MECHANICAL STRENGTH AND MICROSTRUCTURE OF GREEN CERAMIC BODIES

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ABSTRACT

The green mechanical strength of ceramic wall and floor tile bodies is an important property, since it conditions the mechanical behaviour of these materials during the pre-firing operations. In this study, the variation that tile dry mechanical strength and porosity undergo with increased pressing pressure has been determined, for different agglomerate-size fractions of an industrial spray-dried powder with various moisture contents.

The fracture mechanism of this type of product has been determined. It has been shown that although the porosity of the pressed compact largely determines its mechanical strength, at equal porosity, the compacts formed from spray-dried powders with a larger moisture content exhibit higher dry mechanical strength values. These results have been interpreted on the basis of the influence that the spray-dried powder moisture content has on the elastic-plastic deformation of the agglomerate during pressing, on the microstructural uniformity of the resulting material and on the elastic expansion of the newly pressed compact.

Finally, a semi-empirical equation is proposed, which relates dry mechanical strength of the material to its porosity and to the deformability of the spray-dried powder used to form the compact.

1. INTRODUCTION

One of the most important tile properties in ceramic tile manufacture is the body's green mechanical strength. Its importance lies in the fact that the materials must withstand the stresses that they undergo during pre-firing operations without deteriorating. In drying and glazing, as well as in successive conveying, storage, and heat-treatment operations, tile is subjected to mechanical stresses (blows as a result

of transport), and thermal stresses (expansion and contraction), which may give rise to deterioration in the materials.

These reasons clearly indicate the considerable importance of green mechanical strength for single-fired ceramic wall and floor tile manufacture. However, although studies abound in the literature on the mechanical properties of fired ceramic materials, hardly any work has been reported on the fracture of the green ware [1][2][3][4][5]. The present study therefore addresses the fracture mechanism exhibited by green ceramic bodies, as well as the effect of pressing powder characteristics and porosity of the compact on its dry mechanical strength.

1.1. FRACTURE IN CERAMIC MATERIALS

Generally, green or fired ceramic materials that undergo tensile stress exhibit brittle fracture on being subjected to deformations that rarely exceed 1%. It has experimentally been shown that fracture starts at cracks existing within the material, which might have existed prior to testing or during testing itself.

According to the type of fracture they exhibit, materials may be classified as [6][7]:

A) *Pure brittle*. Fracture arises at pre-existing cracks in the material, critical deformation, i.e. maximum deformation prior to rupture, being round 0.1%.

B) *Semi-brittle*. Fracture arises at cracks produced or extended during testing, with critical deformation lying within the range 0.1 to 1%.

C) *Ductile*. In this case, critical deformation lies above 1%, with plastic flow arising, which may be considerable, often followed by a brittle type of fracture.

The origin of rupture in ceramic materials may therefore be due to the presence of inherent cracks, microplasticity, or both effects jointly. In these last two cases, subcritical cracks grow to a critical size by microplasticity, subsequently producing brittle fracture.

1.2. RELATIONSHIP BETWEEN MECHANICAL STRENGTH AND CHARACTERISTICS OF THE COMPACT

Numerous theoretical models have been proposed to allow interpreting and predicting the mechanical strength of materials. In these models, mechanical strength has been related to some characteristics of the particles or agglomerates that make up the pressing powder (particle or agglomerate size, modulus of elasticity, etc.), or to certain properties of the compact (porosity, modulus of elasticity, etc.)

These theoretical models can be divided into two large groups: classical models and crack propagation models.

i) Classical models

In a dry or almost dry tile, the clay particles lie in contact with each other, making it quite likely for their mechanical strength to arise as a result of short-range forces of an ionic or Van der Waals type, which operate at the contact points, or as a result of solid bonds produced by the crystallization of dissolved materials [8][9].

Thus, the simplest method of determining tensile strength, s, is based upon calculating the number N of interparticle bonds that break per unit fracture surface area, and assessing the force, f_0, of a single bond. In this case:

$$\sigma = N \cdot f_0 \quad (1)$$

In order to calculate N, ideal particle packings are used. Assuming there is an isotropic, random packing of monosized spheres (D) having porosity ε, the value of N is given by:

$$N = \frac{(1-\varepsilon)\, n}{\pi\, D^2} \quad (2)$$

Where n is the mean value of the bonds per particle. The relationship between the number of bonds per particle and the porosity of the particle packing is:

$$N = \frac{\pi}{\varepsilon} \quad (3)$$

Substituting (2) and (3) into (1) yields:

$$\sigma = \frac{1-\varepsilon}{\varepsilon} \cdot \frac{f_0}{D^2} \quad (4)$$

On the basis of thermodynamic models, Rumpf [5] derived quite a similar formula to this. The determination of f_0 is difficult, since several kinds of interaction forces may arise (Van der Waals or capillary forces, hydrogen bonds, electrostatic forces, etc.). Therefore, Rumpf proposed different equations for calculating the respective force, as a function of the prevailing adhesion mechanism [5][9].

ii) Models based on crack propagation

In these models, the material is assumed to break as a result of crack propagation, in accordance with Griffith's criterion [10]. Consequently, interparticle forces may not be summed to obtain tensile strength.

Griffith proposed the following equation:

$$\sigma = \frac{1}{Y} \cdot \left(\frac{2 \cdot E \cdot \gamma_i}{C} \right)^{1/2} \quad (5)$$

where Y is a constant that depends on the geometry of the test specimen and test conditions, E the modulus of elasticity of the material, γ_i surface energy for fracture start and C crack size.

This model makes the assumption that the cracks are already formed and that the value of σ is twice or three times smaller than the material's yield stress. Fracture energy is calculated by adding up the bond energies at each interparticle contact point, following an analogous procedure to that of Rumpf.

Fig. 1 schematically summarizes both of the above theories. Many workers feel that the classical models are inadequate, since they do not explain the rupture mechanism observed in materials, nor the weakening effect that cracks have, or the wide scatter in dry mechanical strength values found for apparently identical materials. However, the models based on Griffith's theory are currently widely accepted. Thus, tensile strength of pure brittle materials (type A), can be suitably interpreted using Griffith's theory. Unfortunately, in many cases, the actual mechanical strength values found are different from the ones predicted by theory. This suggests that during testing cracks form, which are larger than the pre-existing ones, by plastic deformation.

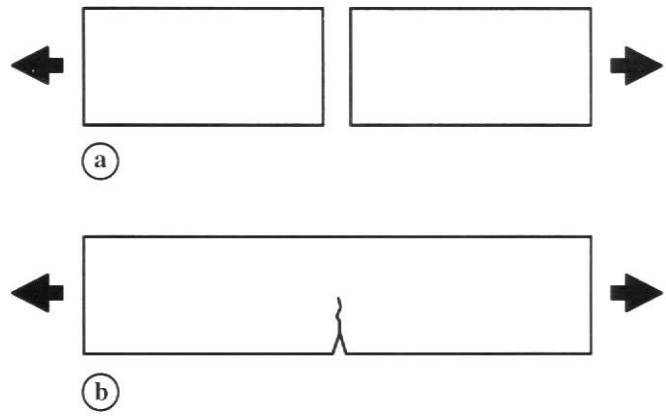

Figure 1. *a) Rumpf's theory assumes that fracture arises simultaneously throughout the material; b) Griffith's theory assumes that rupture occurs as a result of crack propagation.*

2. EXPERIMENTAL

2.1. MATERIALS

An industrial spray-dried agglomerate pressing powder was used, prepared from kaolinitic-illitic natural clays, potassium-sodium feldspar and feldspathic sand.

Agglomerate-size distribution was determined by dry screening and the data have been listed in Table I. True density of the material, measured in a helium pycnometer, was 2.65 g/cm^3.

Table I. Agglomerate-size distribution of the industrial pressing powder (wt%).

Mesh aperture (μm)	Differential percentage (%)
>750	0.57
750-500	10.43
500-400	25.46
400-300	26.54
300-200	19.37
200-125	14.77
<125	2.86

2.2 EXPERIMENTAL PROCEDURE

2.2.1. Pressing powder preparation

An industrial spray-dried powder (PI) was used from which three agglomerate-size fractions were taken referenced PA, PB and PC. The first corresponded to fraction 750-500 μm, the second to fraction 400-300 μm and the third to fraction 200-125 μm.

2.2.2. Pressing powder characterization

The yield pressure was established of powder agglomerates PA, PB and PC. This was done by monitoring the increase in bulk density that the powder bed underwent as pressing pressure was raised (compaction curve), defining the yield pressure locus as the point in the curve at which the first slope change occurs [11][12].

2.2.3. Forming test specimens

Using the four pressing powders, prism-shaped test specimens were formed sized 1.5x8x0.7 cm by unidirectional pressing, in a laboratory hydraulic press. These test specimens were formed at varying pressures (250, 400 and 550 kg/cm^2) and different pressing moisture contents (0.040, 0.055 and 0.070 kg water/kg dry solid), with a view to obtaining different microstructures in the green compact.

2.2.4. Test specimen characterization

Test specimen bulk density, ρ, was determined by the method involving immersion in mercury [13]. This value was then used to calculate porosity ε, by means of the following equation:

$$\varepsilon = 1 - \frac{\rho}{\rho_r} \quad (6)$$

where ρ_r is true density.

The determination of the surface roughness parameters of the test specimens was carried out with a roughnessmeter. Twenty-five, 10 mm-long roughness profiles were determined at each of the surfaces to be studied, spaced at 0.1 mm.

Mechanical strength was determined by three-point cross-bending testing in a universal testing machine, at a constant deformation rate of 1 mm/min. The mechanical strength values, σ, were calculated from the following equation:

$$\sigma = \frac{3\ F_{max}\ L}{2\ a\ b^2} \quad (7)$$

where F_{max} is maximum force prior to rupture, a and b test specimen width and thickness respectively, and L the span between supports.

The modulus of elasticity, E, was determined from the following equation:

$$E = \frac{1}{4} \cdot \frac{L^3}{a\,b^3} \cdot P \quad (8)$$

where P is the slope of the stretch of straight line of the force-deformation curves obtained in the rupture tests.

The mechanical strength data were analyzed by Weibull statistics. Such analysis was carried out by arranging the experimental mechanical strength data in order from small to large, assigning a rupture probability to them F (σ), in accordance with equation:

$$F(\sigma) = \frac{i}{N+1} \quad (9)$$

where σ is the mechanical strength of the i-th test specimen and N is the total number of specimens tested per series. Finally, the Weibull modulus was determined from the slope found on plotting $\ln\ln(1/(1-F(\sigma))$ as a function of $\ln(\sigma)$.

3. RESULTS AND DISCUSSION

3.1. FRACTURE MECHANISM IN GREEN CERAMIC BODIES

In order to determine how green ceramic bodies behave during fracture, test specimens with different porosities were subjected to three-point cross-bending tests. Testing was done at a constant rate of 0.05 mm/min, continuously monitoring the relation applied load/resulting deformation. Fig. 2 shows a typical curve, in which the following three stretches can be distinguished.

a) An initial non-linear area, probably caused by the settling of the bending system and plastic deformation at the contact points.

b) A straight stretch, in which deformation is always a linear function of the applied load.

c) A slightly curved stretch, in which deformation is not a linear function of load and which ends with failure of the test specimen.

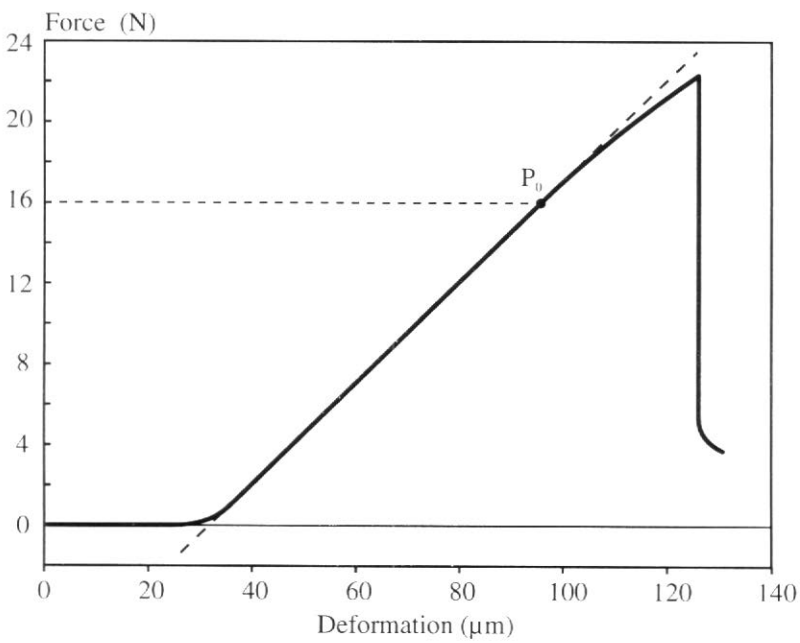

Figure 2. *Load/deformation curve obtained in a cross-bending test with rupture of the sample.*

It has been shown that on lowering the porosity of the tested specimen, both the breaking load and the slope of the straight stretch increase in the corresponding plots. The values of the modulus of elasticity can be calculated from this slope, by using Equation (8).

The point at which the values deviate from linearity, P_0, defines the elastic limit of the material. Thus, when a load is applied below this value, the arising permanent deformation is virtually negligible. However, on going beyond this bound, appreciable plastic deformation arises, since when the load is reduced the load/deformation curve does not return along the same path (Fig. 3).

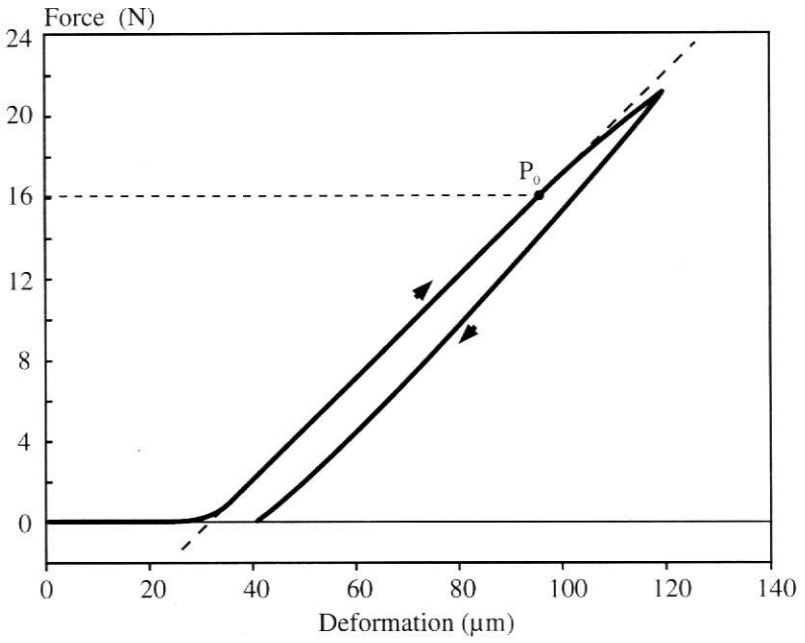

Figure 3. *Load/deformation curve obtained in a cross-bending test without rupture of the sample.*

The elastic limit would therefore indicate the point at which crack development and extension start as a result of plastic deformation, arising at already existing defects [7][14][15]. In flexural testing, rupture occurs at or near the surface of the test specimen that is subjected to tensile stress (Fig. 4). Thus, surface texture of the test specimens could noticeably affect test outcomes.

Figure 4. *Development of a crack at the surface of a test specimen subject to tensile stress.*

In Fig. 5, a schematic illustration depicts what has been set out above, by schematizing the most likely fracture mechanism for green ceramic tile. On exceeding the elastic limit, it becomes quite likely that a crack or existing defect at the surface

will grow. This crack could then branch out between the agglomerates, giving rise to multiple cracks. Finally, when the system of cracks reaches a critical size, C, failure occurs.

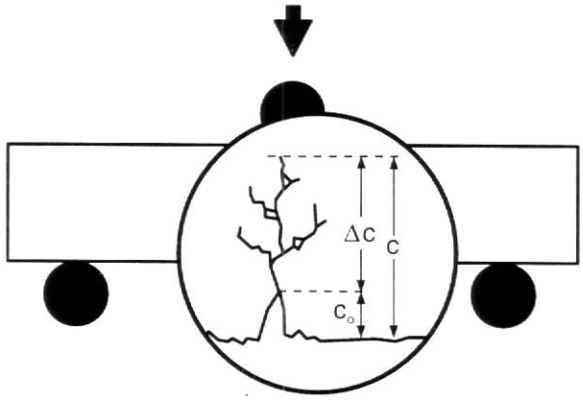

Figure 5. *Fracture mechanism proposed for green ceramic materials.*

The material considered in the study does not exhibit purely brittle but rather semi-brittle behaviour, since the propagation of numerous cracks is required for fracture to occur. These conclusions are consistent with the values of the Weibull modulus obtained [6][16], and with the high critical deformations observed. The values of the Weibull modulus range from 20 to 55 (depending on the forming and pressing powder variables used), while critical deformation is found to lie at 0.4%

In order to be able to apply Griffith's theory to materials with semi-brittle behaviour, a series of further considerations are required [14][15][16], however, of a qualitative nature. In accordance with the theory, mechanical strength, σ, can be expressed by the following quotient:

$$\sigma = \frac{\textbf{function (number of contacts)}}{\textbf{function (defect size)}} \qquad \textbf{(10)}$$

The number of interparticle contacts and size of the fracture-starting crack or defect depend, in turn, upon a series of factors that will be dealt with below.

3.2. FACTORS INFLUENCING DRY MECHANICAL STRENGTH

3.2.1. Influence of pressing powder moisture content and pressing pressure

Three series of test specimens were formed from pressing powder PB. Within each series, pressing moisture content was kept steady and the applied pressure was modified. Fifteen test specimens were prepared for each pair of values (pressure, moisture content), in order to average the results. The moisture contents involved were 0.040, 0.055 and 0.070 kg water/kg dry solid, while the pressures used were 250, 400 and 550 kg/cm^2.

With a view to determining the effect that the pressing variables have on the specimen's mechanical strength, σ, and on its relationship to porosity, ε, the values of

σ have been plotted versus ε in Fig. 6. The figure shows that dry mechanical strength rises considerably on lowering test specimen porosity. This fact is due to the increased number of contacts that arise among particles and/or agglomerates on decreasing porosity [12][17] [18].

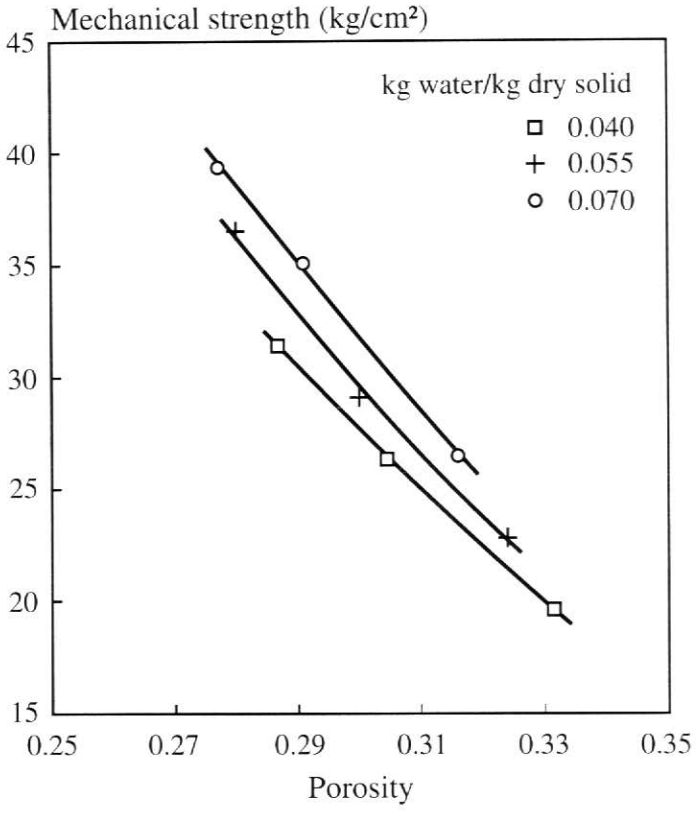

Figure 6. *Variation of dry mechanical strength with porosity for different pressing moisture contents.*

However, at the same porosity, mechanical strength rises slightly with increased pressing powder moisture content, thus showing that the relationship between dry mechanical strength and porosity of the material is not independent of the forming variables.

One of the causes possibly contributing to the fact that the specimens pressed at higher moisture contents exhibit greater dry mechanical strength may lie in the plasticizing effect of the water. Thus, as agglomerate moisture content is raised, they become more deformable, facilitating sliding and enhancing particle packing [19]. However, when the granules are dry, particle packing is less stable, which yields greater after-pressing expansion, and therefore a reduction of the contact surface produced during compaction.

Another possible cause that may serve to explain the outcomes, is the fact that as granule moisture content is reduced, the microstructure of the compact becomes less uniform. Thus, agglomerates and/or parts of these, which were not wholly deformed during pressing as a result of their low plasticity, may act as fracture-starting defects in the material.

With a view to assessing the relative influence that each of the foregoing causes has, the modulus of elasticity has been plotted as a function of porosity in Fig. 7. It can be observed that this parameter, just as mechanical strength, rises as pressing powder moisture content is raised. This increase may be assumed to be almost exclusively due to the increased number of interparticle contacts, since the size of the defects or cracks has a negligible effect on modulus of elasticity values [2].

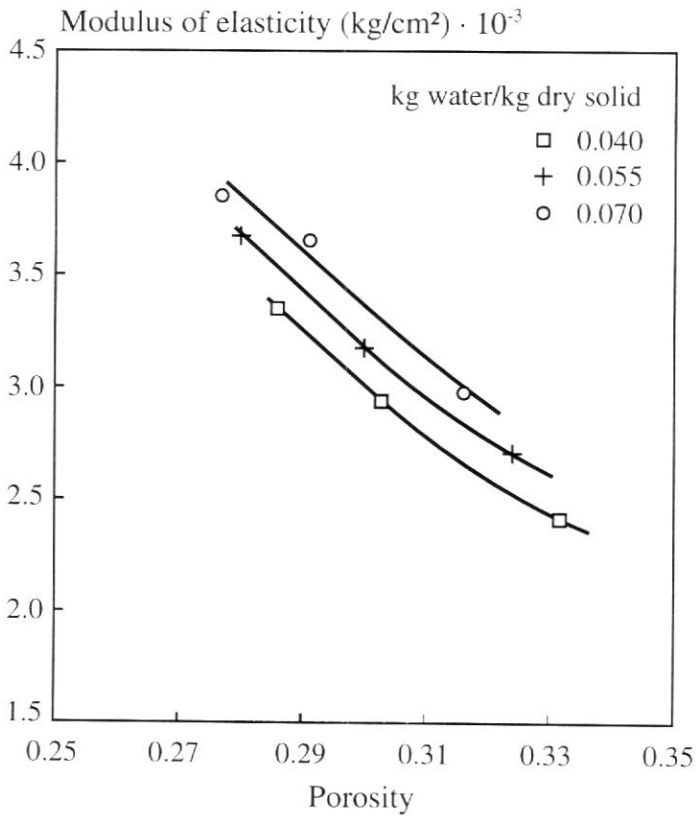

Figure 7. *Variation of the modulus of elasticity with porosity for different pressing moisture contents.*

In Fig. 8 a plot is shown of dry mechanical strength versus the modulus of elasticity. If the drop observed in mechanical strength were due to the weakening effect produced by more severe defects, a group of straight lines would be obtained as a function of moisture content; however, it can be observed that the points fit a single straight line quite satisfactorily.

It may therefore be inferred that at the same porosity, the compacts formed from moister granules exhibited greater dry mechanical strength, because the actual contact surface among particles and/or agglomerates was higher.

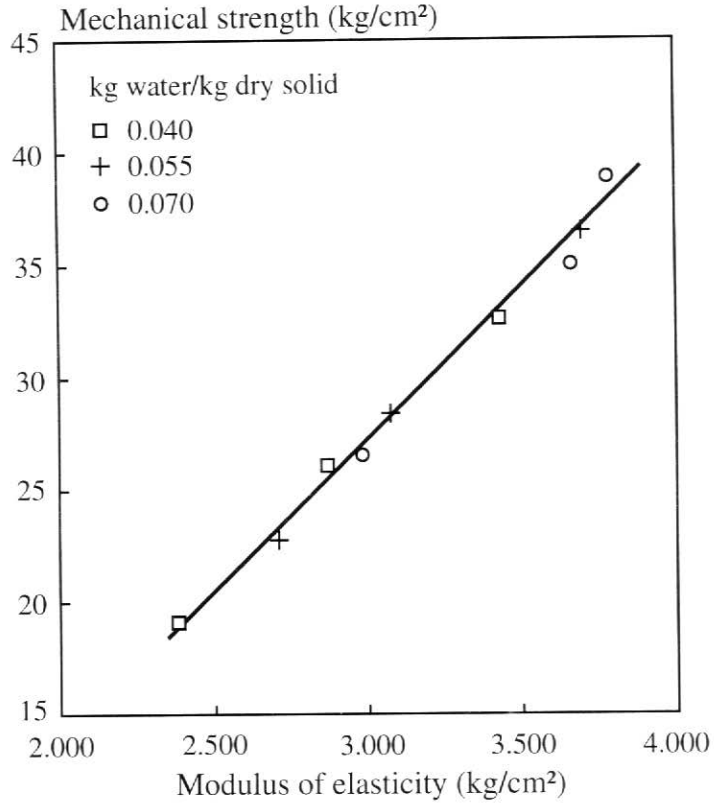

Figure 8. *Relationship between dry mechanical strength and modulus of elasticity for different pressing moisture contents.*

3.2.2. Influence of agglomerate size

Four series of test specimens were formed from pressing powders PI, PA, PB and PC. Within each series, pressing pressure was modified (P = 250, 400 y 500 kg/cm²) and agglomerate moisture constant was kept steady (X = 0.055 kg water/kg dry solid). Fifteen test specimens were prepared for each pressure value, to allow averaging the results.

Figure 9 shows plots of the dry mechanical strength values versus porosity. On examining this figure it can be observed, that at the same porosity, mechanical strength grows as agglomerate size decreases. It can furthermore be observed that the curves corresponding to industrial pressing powder PI, with a mean agglomerate size approaching 400 μm, and those of spray-dried powder PB are superimposed.

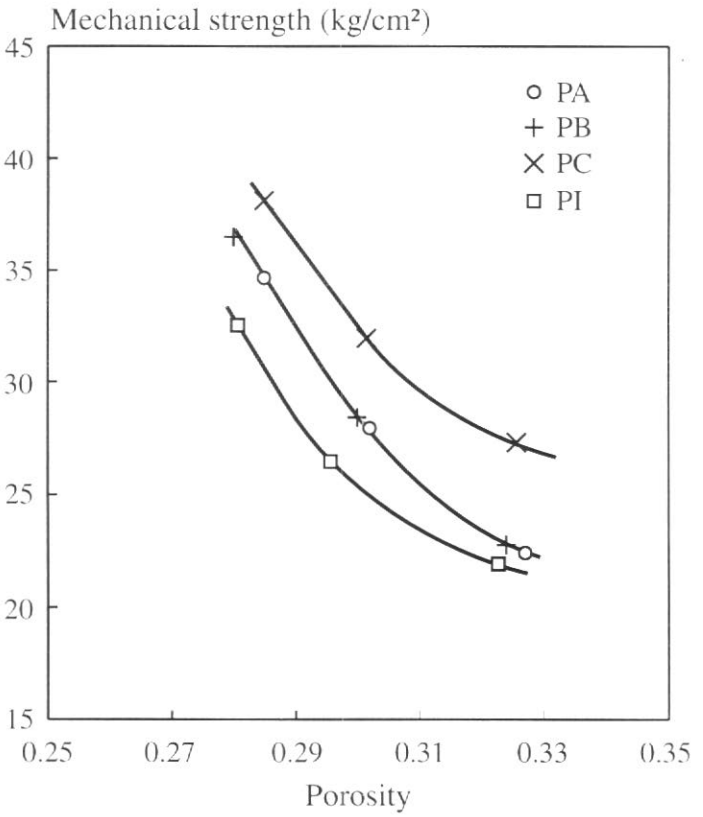

Figure 9. *Variation of dry mechanical strength with porosity for different spray-dried powders.*

The reasons for an increase in dry mechanical strength in a material on lowering agglomerate size can be found in the greater number of contacts among particles and/or agglomerates, and the reduction of the size of the fracture-starting defect.

In order to determine how granule size impacts the number of contacts among particles and/or agglomerates, a plot has been made in Fig. 10 of the modulus of elasticity as a function of test specimen porosity. On examining this figure, it may be observed that despite the slight increase in this parameter, and therefore in the number of interparticle contacts, this increase alone cannot explain the observed rise in dry mechanical strength.

With a view to assessing the influence that the second cause mentioned above has, a plot has been made in Fig. 11 of dry mechanical strength versus modulus of elasticity. On analysing this plot, it can be observed that the data do not fit one single straight line, but rather fit several straight lines quite well as a function of the powder used. This would appear to confirm that agglomerate size affects the dimensions of the fracture-starting defect, contrary to what happened with pressing powder moisture content.

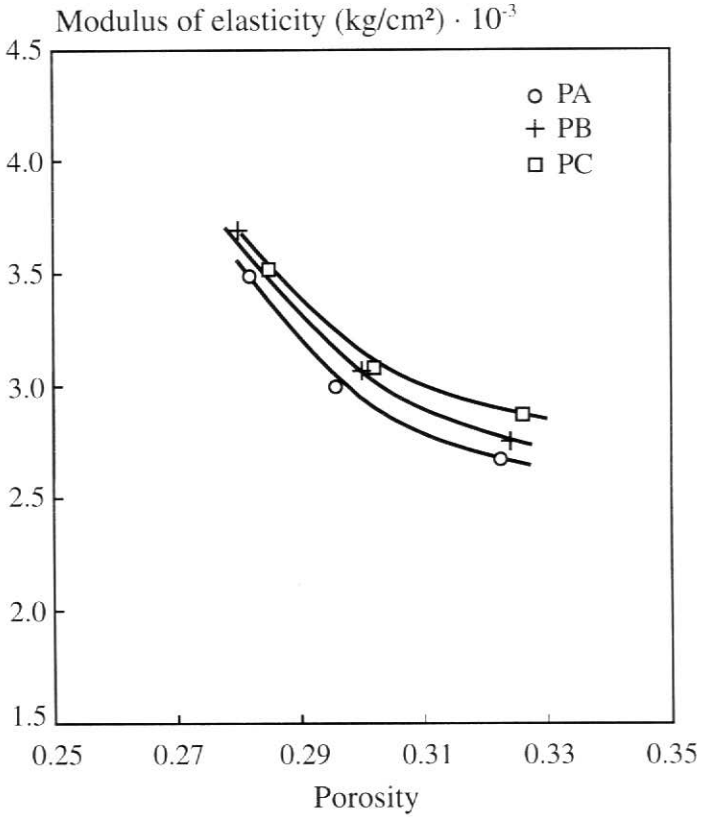

Figure 10. *Variation of the modulus of elasticity with porosity for different spray-dried powders.*

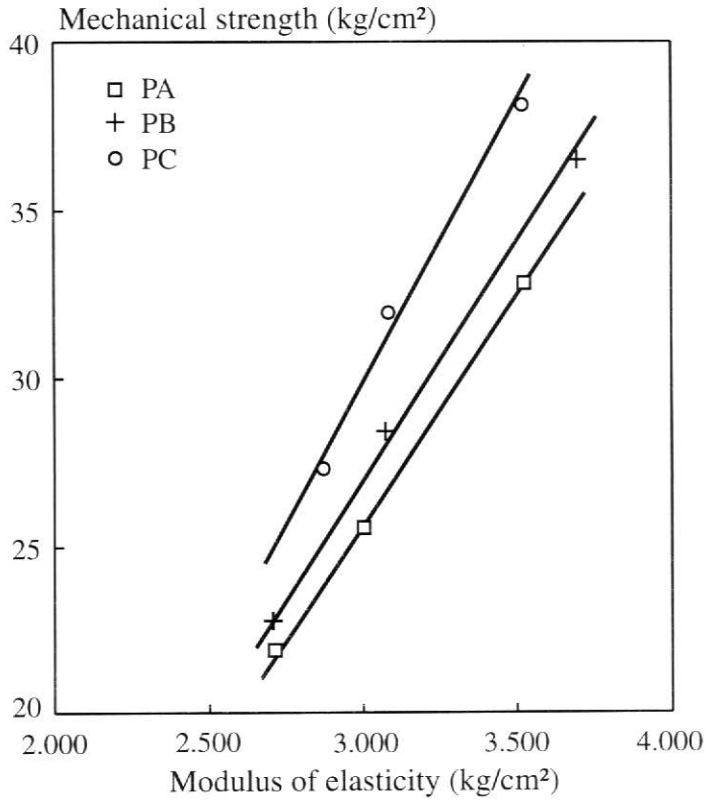

Figure 11. *Relationship between mechanical strength and modulus of elasticity for different spray-dried powders.*

In view of the results obtained, and owing to the fact that in flexure tests the fracture is quite likely to start at the surface that is subjected to tensile stress, the surface roughness was determined of four test specimens formed at the same porosity and obtained from the four studied pressing powders. The selected roughness parameter was the so-called R_{ZISO} parameter [20][21], which is considered the most suitable one for statistically describing the severest surface defects. Table II lists the results obtained. The surface defects are shown to become greater as agglomerate size is raised.

Table II. *Roughness and dry mechanical strength of materials formed from different spray-dried powders (P=400 kg/cm^2 and X=0.055 kg water/kg dry solid).*

Powder	R_{ZISO}		Mechanical strength	
	Mean (μm)	Deviation (μm)	Mean (kg/cm^2)	Weibull modulus
PA	22.2	5.5	26	25
PB	19.9	2.8	29	33
PC	15.7	1.5	32	40
PI	19.7	4.5	29	25

It can thus be observed that the mean values of parameter R_{ZISO} obtained for the test specimens formed from powders PB and PI are quite alike; however, because the agglomerate-size distribution of the latter powder is much wider, the R_{ZISO} values exhibit a greater standard deviation. This means a wider defect-size distribution, and therefore lower Weibull moduli, as the degree of scatter found for the mechanical strength values is greater (Table II).

3.3 RELATIONSHIP BETWEEN DRY MECHANICAL STRENGTH AND POROSITY OF THE COMPACT AND PRESSING POWDER CHARACTERISTICS

The data obtained in the foregoing sections allow formulating the following relationships among the variables:

$$\textbf{Number of contacts} = \textbf{function}\ (e, D, G) \tag{11}$$

$$\textbf{Defect size} = \textbf{function}\ (G) \tag{12}$$

where ε is porosity of the compact, D is agglomerate deformability and G is agglomerate size.

Thus, Eqs. (10), (11) and (12) yield:

$$\sigma = \textbf{function}\ (\varepsilon, D, G) \tag{13}$$

With a view to obtaining a semi-empirical equation that describes the joint effect of porosity of the compact, ε, and the pressing powder characteristics (deformability, D, and mean granule size, G) on mechanical strength, the relationships between

mechanical strength and these variables must be established beforehand. Thus, the effect of porosity on mechanical strength can be adequately described by an equation of the type [17]:

$$\sigma = c_i \cdot \varepsilon^{-2/3} + d_i \quad (14)$$

where c_i and di are two characteristic parameters of the pressing powder used.

On the other hand, the relationship that exists between mechanical strength and agglomerate size can be expressed by the following equation [22]:

$$\sigma = K \cdot G^{-\alpha} \quad (15)$$

where K is a characteristic material constant.

With regard to agglomerate deformability, it has been shown that this parameter is closely related to agglomerate yield pressure [12]. Fig. 12 reports the values corresponding to spray-dried powders PA, PB and PC, which shows that yield pressure drops with agglomerate moisture content, and at a given moisture content, with an increase in agglomerate size.

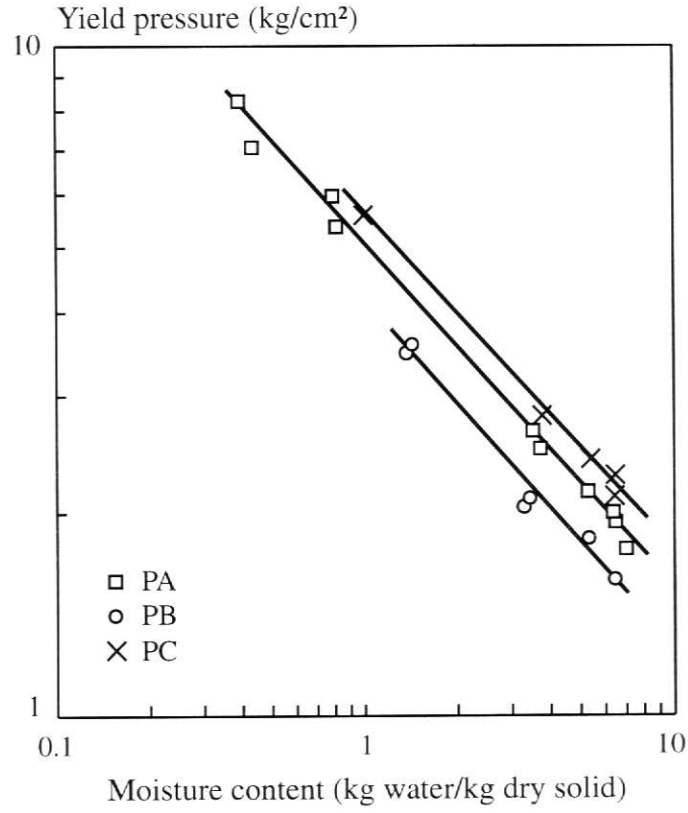

Figure 12. *Variation of yield pressure with pressing moisture content for powders PA, PB and PC.*

A literature survey showed no equation which, with other factors being equal, could relate agglomerate yield pressure, P_f, to the mechanical strength of the compact; however, it is quite likely that this relationship will be of a potential type, so that:

$$\sigma = P_f^{-\beta} \quad (16)$$

Bearing in mind the above, it has been attempted to fit the experimental data by non-linear correlation to the following equation:

$$\sigma = \Delta (\varepsilon^{-2/3} - \varepsilon_o^{-2/3}) G^{-\alpha} P_f^{-\beta} \quad (17)$$

The value of G considered for pressing powders PA, PB and PC was 0.0625, 0.0350 and 0.0162 cm , which yielded the following values for the parameters of this equation:

$\varepsilon_0 = 0{,}443$ $\Delta = 33$ $\alpha = 0{,}273$ $\beta = 0{,}501$

A plot is shown in Fig. 13 of the theoretical mechanical strength values, calculated from the proposed equation versus the experimental data. It can be observed that the results fit a straight line of unity slope quite well, thus substantiating the validity of Equation (17).

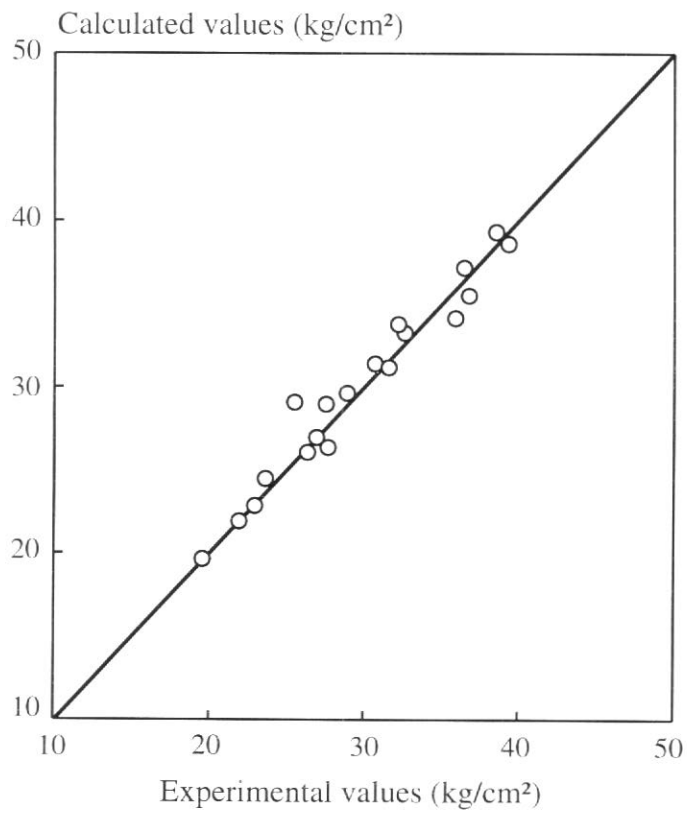

Figure 13. *Verification of the validity of Equation (17).*

As had been foreseen, the moisture content of the pressing powder, which considerably affects porosity of the compact and granule deformability, is shown to have a marked effect on dry mechanical strength. On the other hand, although agglomerate size also influences this property, its effect is less significant than that observed for pressing moisture content.

4. CONCLUSIONS

The results obtained in the present study allow drawing the following conclusions:

- Green ceramic compacts exhibit semi-brittle behaviour, with fracture starting at pre-existing cracks or defects, which grow by microplasticity during testing.

- Dry mechanical strength considerably rises on reducing the porosity of the compact, owing to the increased number of contacts among particles and/or agglomerates.

- At equal porosity, mechanical strength of the materials formed from moister granules, was higher than that obtained on using drier granules and greater pressing pressures. This is because of the influence that moisture content has on elastic-plastic deformation of the agglomerate during pressing and therefore on the number of contacts among particles and/or agglomerates.

- Although agglomerate size hardly influences the compactness of the resulting material, it has been shown that the test specimens obtained from smaller-sized agglomerates are noticeably more resistant than the ones formed from larger agglomerates. This is because the size of the fracture-starting defect or crack increases as the agglomerates become larger.

- Finally, a semi-empirical equation has been proposed, relating dry mechanical strength to the porosity of the materials and to pressing powder characteristics (agglomerate mean size and hardness), and its validity has been substantiated.

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