JOURNAL OF MATERIALS SCIENCE 27 (1992) 3305–3308

Tensile strength of ceramic powders

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The tensile strength of granular materials has been extensively studied and is of great technological importance. Several theoretical or experimental relationships between density and strength have been proposed. This paper compares these models with experimental data from zirconia powders. It is shown that the most important parameter determining the strength of a green compact is not density but compaction pressure. A micromechanical model, which gives a qualitative understanding of ceramic powder behaviour under tensile stress, is also presented.

1. Introduction

Fracture of pieces is a common problem in ceramic technology. It is encountered in every shape-forming process: compaction (during ejection); slip casting (during drying); and injection (during pyrolysis). Thus a fundamental understanding is needed of both the driving forces for fracture (residual stresses after compaction, for example) and the mechanisms of cohesion in a green compact. The former will be achieved through the study of powder rheology, and finite element calculation [1–3]. The latter is discussed in this paper.

The most simple way for determining the tensile strength of a green compact is to count the number of particle/particle bonds in the failure plane [4]. This calculation is tedious but quite simple. In this case we suppose that all bonds display the same cohesion, f_0, and are broken at the same time. The result for a random packing of monosized particles is [3]

$$\sigma t = \frac{f_0 \rho}{4(1-\rho)r^2} \tag{1}$$

where ρ is the relative density, and r the particle radius. For a powder without binder, the attraction force between two particles is the Van der Waals force [5–7]

$$f_0 = \frac{Ar}{12z_0^2} \tag{2}$$

where A is the Hamaker constant and z_0 the smallest possible distance between two particles. Of course, other factors may also contribute to the cohesion (hydrogen bonds or capillarity, for example) but we will suppose them negligible. Combining Equations 1 and 2

$$\sigma t = \frac{A\rho}{48z_0^2(1-\rho)r} \tag{3}$$

For oxides, A is $\approx 10^{-19}$ J [5]; the estimated value for z_0 is 0.4 nm; using $r = 0.5$ μm and $\rho = 0.5$, we obtain $\sigma t = 0.03$ MPa which is a rather good order of magnitude, as we will see.

However, this calculation is not very satisfactory as the particle/particle bonds crossing the fracture plane are not broken at the same time but (as might be expected in a brittle material) one after one, as the result of a crack propagation. This point has been widely discussed by Kendall [8] and Adams *et al.* [9]; in fact, only the rupture energies of these contacts might be summed. Kendall calculated the tensile strength of several regular packings of monosized particles, and extrapolated the behaviour of a random one from these data. His formula is

$$\sigma t = \frac{15.6\rho^4 \Gamma_R^{5/6} \Gamma_E^{1/6}}{(2rc)^{1/2}} \tag{4}$$

where c is the crack length, and Γ_R and Γ_E are surface energies measured by rupture and modulus experiments, respectively. This method is indeed better; however, the notion of 'crack' is not well defined in a granular material: when does a pore become a crack?

With $\sigma t = 0.1$ MPa (Fig. 1), $r = 0.5$ μm and $\rho = 0.5$, we obtain

$$\Gamma = 100c^{1/2}$$

where $\Gamma = \Gamma_R = \Gamma_E$ is the classical surface energy. In

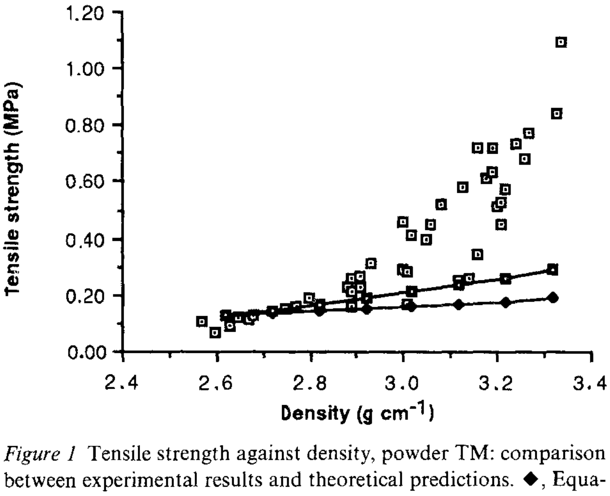

Figure 1 Tensile strength against density, powder TM: comparison between experimental results and theoretical predictions. ◆, Equation 3; ■, Equation 4; □, experiment.

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our case, Γ is about 80 J m^{-2} (from tensile strength on sintered parts) which gives an unrealistic value for c. From his experiments on titanium dioxide, Kendall [10] found $\Gamma = 14$ J m^{-2}, and $c = 60$ µm. This corresponds to higher values of σt.

2. Experimental procedure

The tensile strength of dry-pressed samples of zirconia powder was measured using the so-called diametral compression test (Fig. 2). Despite some limitations concerning the exact stress state in the disk [11], it is usually recognized that this test gives a good approximation of the true material tensile strength [12–14]. Another example of a tensile test, where the green is tested in the compaction mould, is given in [15].

The specimens were 3 cm in diameter, their height between 0.5 and 0.8 cm. Pieces were heated 24 h at 100 °C before the test. All were broken along the diametral plane, which is the usual criterion for the test validity.

To assess the influence of particle size, we used a commercial zirconia powder named TM (YZ_3 from Rhône-Poulenc) in the following, which was classified with a sedimentation process to obtain three powders of different particle size (Table I). This powder was also granulated (without binder) by spray drying to investigate the influence of granulation (powder Tg). Another very different zirconia powder (D; YZ_3 from Daïchi Corporation) was also used.

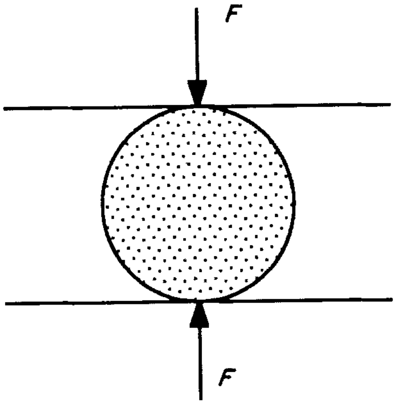

Figure 2 Diametral compression test: the stress state is tensile along the diameter joining the loading points.

TABLE I Powder characteristics (granulometer: Cilas)

Powder	Mean diameter d_{50} (µm)	Standard deviation $\frac{d_{90}-d_{10}}{d_{50}}$	Specific surface (m^2 g^{-1})
TM	0.59	2.30	7.00
TMf	0.33	1.76	9.47
TMG	0.92	2.07	5.97
TMm	0.41	1.92	7.5
Tg	80 (*granules*)	2.50	7.00
D	0.76	3.5	

3. Results and discussion

Although our powders are not monosized, we may expect Equation 3 or 4 to predict at least approximately their behaviour (the values of A/z_0^2 in Equation 3, and $\Gamma/c^{1/2}$ in Equation 4, were chosen to fit the experimental value of σt for $\rho = 0.5$). By contrast, Fig. 1 shows clearly that it is not the case. Had we taken into account other interparticle forces, neither equation would apply as its density dependence would not change.

Tensile strength against density curves for our different powders (Fig. 3) allowed us to determine the tensile strength as a function of mean particle size, for a given density. This curve is compared with the theoretical predictions (Fig. 4). Once again, the theoretical relationships are not verified. Thus another model is needed.

Fig. 5 shows the tensile strength as a function of compaction pressure: for a given pressure, the tensile strength is the same for all the powders, although the density might be very different. Even the Tg and D powders, whose tensile stresses are rather different for a given density, lie on the same curve.

In order to explain this result, let us introduce a very simple model. Let P be the compaction pressure, and s the total surface of the interparticle contacts crossing a plane perpendicular to the compaction axis, per unit

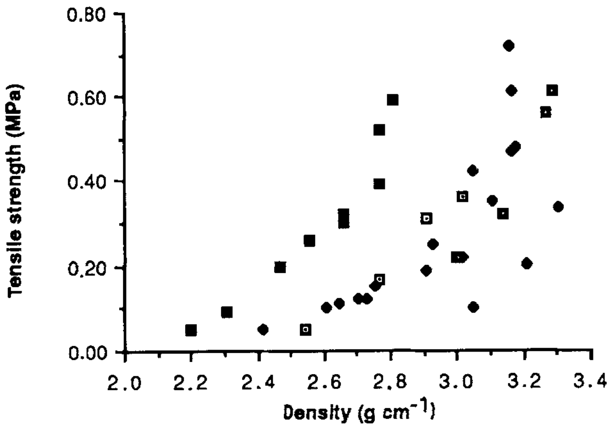

Figure 3 Tensile strength against density for the different powders. □, TMf; ◆, TMm; ■, TMG; ◆, Tg; ■, D.

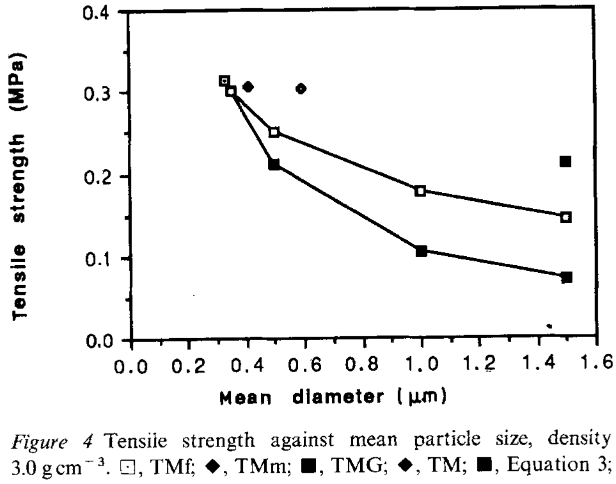

Figure 4 Tensile strength against mean particle size, density 3.0 g cm^{-3}. □, TMf; ◆, TMm; ■, TMG; ◆, TM; ■, Equation 3; □, Equation 4.

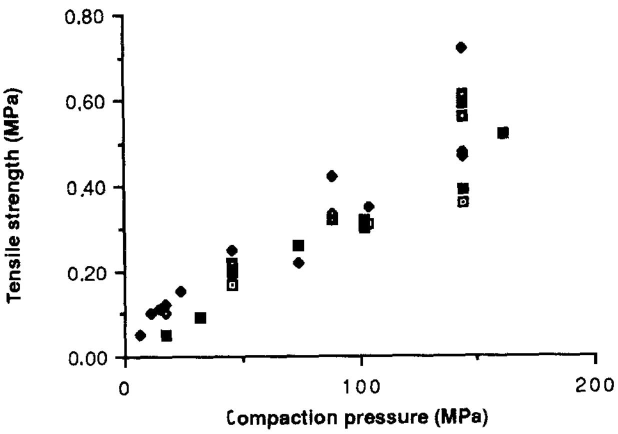

Figure 5 Tensile strength against compaction pressure for different powders. □, TMf; ◆, TMm; ■, TMG; ◆, Tg; ■, D.

surface of this plane (Fig. 6). The mean stress acting on s is

$$\sigma = P/s$$

Considering the small surface area of interparticle contacts, s must be very small. Thus, σ might overcome the yield strength σ_y of the material, causing some crushing of the particle asperities. The equilibrium is obtained, through rearrangement and crushing, when $\sigma = \sigma_y$. σ_y being approximately constant for all our powders, s must be the same for a given compaction pressure. Now suppose that σt depends on s only: then σt is the same function of P for all the powders, which is observed in Fig. 5. An explanation of this kind has also been given by Thompson [16].

This model is of course too simple: interparticle cohesion cannot be described only by a simple 'contact area' concept. It probably also involves some mechanical interlocking of particles, which depends on their roughness. Moreover, the strength of the green compacts is certainly influenced by their flaw size. However the model gives some physical insight into the mechanism of cohesion.

4. Numerical simulation

In order to understand the behaviour of the powder on a microscopic scale, we realised a micromechanical model which simulates on a computer the behaviour of an assembly of rigid discs. In this calculation, the interparticle forces are known (Van der Waals, elastic repulsion, sliding friction) and the computer determines the behaviour of the whole packing in a quasistatic deformation. This program has been used to simulate a pure tensile test on packings of several hundreds of discs [3]; here, the results for small packings will be described in order to emphasise the influence of the packing structure.

For a regular packing, the fracture is brittle and the tensile strength is the sum of the contact forces of the bonds broken by the fracture plane (Fig. 7; the tensile strength in this case is 6 MPa due to the high interparticle cohesion chosen).

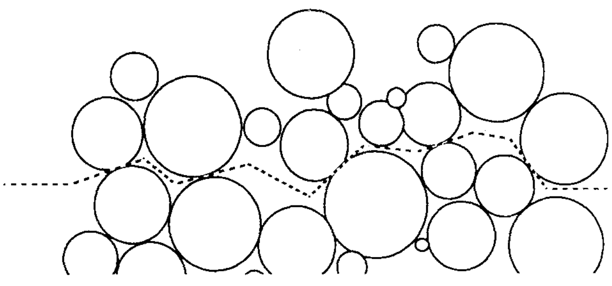

Figure 6 Contacts crossing a plane perpendicular to the compaction axis in 2D.

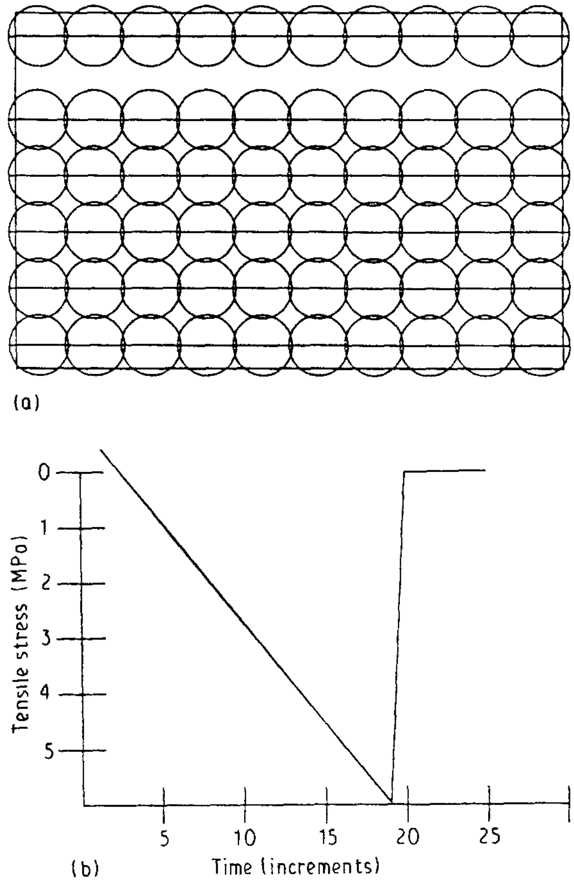

Figure 7 Tensile test on a regular packing: (a) packing and (b) stress/strain relationship.

When the radii of half of the discs are randomly modified by a factor of 5%, the packing is still regular at first sight (the number of neighbours is always four). However, its behaviour during the tensile test is very different (Fig. 8). The tensile strength is now only 4 MPa, the reason for which is clearly seen in Fig. 8: the packing is broken by the propagation of a crack. As a consequence, the fracture becomes more stable. This behaviour is a direct consequence of the strong modification of interparticle forces that arises from the modification of the radii (Fig. 9). This means that the behaviour of a random packing cannot be extrapolated directly from the behaviour of a regular one (this has also been demonstrated by Stauffer *et al.* [17]). This might be the reason for the relative failure of Kendall's model to predict our experimental data.

5. Conclusions

Experimental tensile tests on ceramic green compacts have been performed. For a given compaction pres-

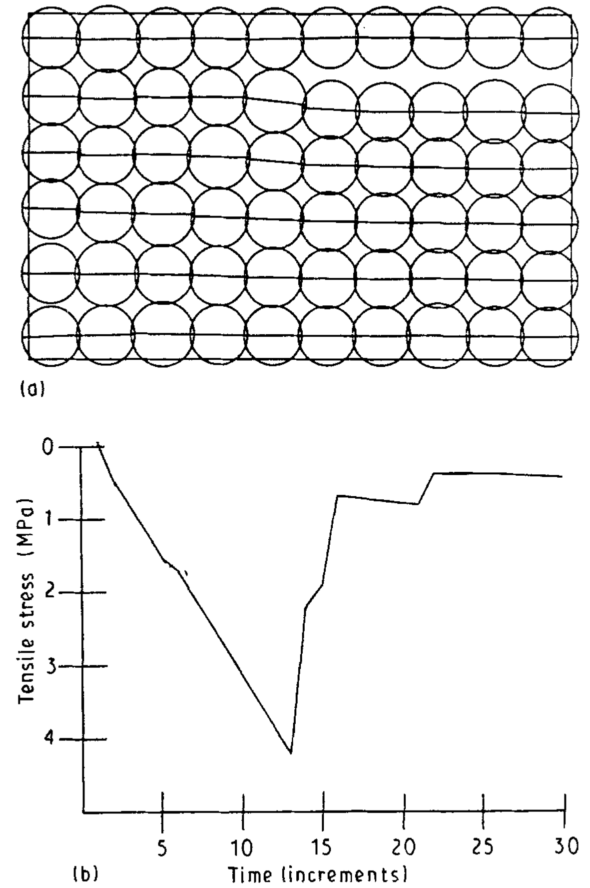

Figure 8 Tensile test on a regular packing with random variation of the radii: (a) packing and (b) stress/strain relationship.

sure, the tensile strength is equal for several zirconia powders, whatever the mean size of the particles or the sample density. This result is in contradiction with most theoretical models; it suggests that the interlocking of particles caused by the compaction loading might be the most important contribution to the overall strength. A micromechanical model has been used to determine the microscopic behaviour of packing during the tensile test. It shows that the randomness of a packing dramatically affects its behaviour. In particular, the tensile strength is seriously decreased compared with a regular packing, because fracture happens through the propagation of a crack.

Acknowledgements

This work is a part of a PhD thesis. This thesis has been directed by M. Abouaf, who is gratefully acknowledged for many helpful discussions. Rhône-Poulenc is acknowledged for giving support.

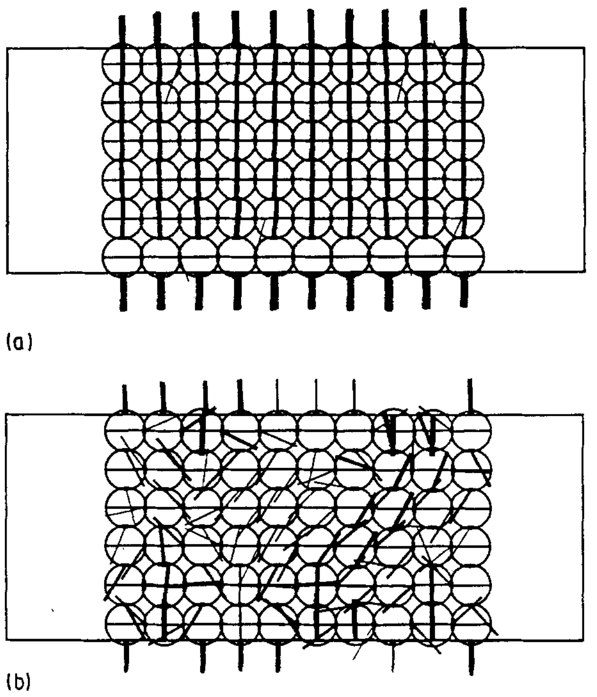

Figure 9 Interparticle forces before the tensile test, in (a) regular packing and (b) "nearly regular packing". The forces are represented by a bolt line, whose thickness is proportional to the force value.

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Received 4 March
and accepted 1 September 1991