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Fracture Mechanics of Green Products

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Abstract

The tensile strength of ceramic green samples (binderless zirconia powder) was investigated within the formalism of linear fracture mechanics. Dry pressed samples were found to obey fairly well the classical laws (Weibull's statistics, the relationship between stress intensity factor and fracture energy). The comparison between the mechanical properties of pressed and cast samples showed that the interparticle forces responsible for the cohesion depend strongly on the shape-forming process. The mechanical interlocking of particle asperities is of primary importance in the strength of pressed samples. Moreover, for a given density the critical defect size is larger in cast than in pressed samples, while the pore size distribution is sharper (which is usually considered as a proof of 'better' microstructure). This sheds light on the differences between cracks in granular materials and pore structure.

Die Zugfestigkeit keramischer Grünkörper (Zirkoniumdioxidpulver ohne Binder) wurde im Rahmen der Formalismen der Bruchmechanik untersucht. Trocken gepreßte Proben genügen weitgehend den klassischen Gesetzmäßigkeiten (Weibullstatistik, Zusammenhang zwischen dem Spannungsintensitätsfaktor und der Bruchenergie). Ein Vergleich der mechanischen Eigenschaften gepreßter und durch Schlickerguß hergestellter Proben zeigt, daß die für die Zugfestigkeit verantwortlichen Kräfte zwischen den einzelnen Teilchen im Grünkörper stark vom Formgebungsprozeß abhängen. Bei gepreßten Proben ist das mechanische Verhaken der Teilchen von entscheidender Bedeutung. Des weiteren ist bei gegebener Dichte die kritische Defektgröße in schlickergegossenen Proben höher als in gepreßten Proben, wobei jedoch die Größenverteilung der Poren ausgeprägter ist, was üblicherweise ein Kriterium für ein günstigeres Gefüge darstellt. Dieses Ergebnis verdeutlicht den Unterschied zwischen der Porosität und den Defekten in einem porösen Material.

La cohésion de pièces céramiques crues (poudre de zircone sans liant) a été étudiée dans le cadre du formalisme de la mécanique de la rupture. Les pièces obtenues par compaction obéissent bien aux lois classiques (statistique de Weibull, relation entre le facteur d'intensité de contrainte et l'énergie de rupture). La comparaison entre les propriétés mécaniques de pièces obtenues par coulage et pressage montre que les mécanismes microscopiques de cohésion sont différents dans les deux cas. Notamment, l'accrochage mécanique des particules intervient de façon déterminante dans la cohésion des pièces pressées. Par ailleurs, quoique la répartition de taille des pores, pour une densité donnée, soit plus étroite pour les pièces coulées que pour les pièces pressées (ce qui est généralement admis comme la preuve d'une 'meilleure' microstructure), la taille de défaut critique est plus grande. Ce résultat éclaire la différence entre la porosité et les défauts dans un matériau granulaire.

1 Introduction

Ceramic green products are often handled and machined before sintering; in order to reduce costly scraps, they must be strong enough to sustain these operations. The problem of green strength is also encountered during several shape-forming processes: demoulding, drying, pyrolysis, etc. A comprehensive understanding of granular material cohesion is thus needed for the engineer to be able to predict the probability of fracture during production. Tensile strength of granular materials is also an interesting field from a scientific point of view, since it provides information on the structure of packings[1] and on the particle interaction forces.[2]

The purpose of this article is to compare the current theories of powder tensile strength with experimental data from ceramic green products. It will be shown that the use of fracture mechanics is necessary if the mechanisms of cohesion are to be understood. The use of different shape-forming

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processes will allow a discussion of the interaction laws responsible for the cohesion in a green product.

2 Theoretical Models for Green Tensile Strength

2.1 Classic model

Usually, a ceramic green compact submitted to a tensile loading breaks in a brittle way. As a consequence, the most simple method for calculating its tensile strength σ_t is to count the number N of interparticle bonds cut by the fracture surface, and to multiply it by the strength f_0 of a single bond:

$$\sigma_t = N f_0 \tag{1}$$

The calculation of N depends on the assumptions concerning the packing structure. If it is a random isotropic packing of monosized spheres (radius r) with relative density ρ, simple geometric calculation shows that

$$N = \frac{\rho . n}{4\pi r^2} \tag{2}$$

where n is the mean number of bonds per particle. A classical approximation for n is[3]

$$n = \frac{\pi}{1-\rho} \tag{3}$$

which leads to

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

Rumpf,[3] on the basis of a thermodynamic model, obtained a formula which is very close to this. Other hypotheses concerning the packing may be used,[1,4] but in the present authors' opinion the random isotropic packing model is better. Furthermore, Tiedge[5] applied the model of Hartley *et al.* to latex powders and showed a discrepancy between theory and experiment.

The determination of f_0 is difficult. Several interaction forces might occur (van der Waals, capillary, hydrogen bonds, electrostatic forces, interlocking of the particles, etc.); even in the case of pure van der Waals force, the calculation is not easy, since the usual formula for two spheres is no more valid when the spheres are in contact and their shape modified by mutual collapse.[6] For the sake of simplicity the case of the 'classic' van der Waals force is considered:

$$f_0 = \frac{Ar}{12 z^2} \tag{5}$$

where z is the distance between particles and A the Hamaker constant. When the particles come into contact, z is not equal to 0, since the formula would lead to an infinite force. Instead, one usually considers that the particles cannot come closer than an equilibrium distance z_0 which depends on the characteristics of the material. Thus

$$\sigma_t = \frac{A}{48 z_0^2} \frac{\rho}{1-\rho} \frac{1}{r} \tag{6}$$

The main question arising with this model is the choice of z_0. Most authors suppose that the minimum distance between two particles is about a few angströms (Dahneke[7] uses about 3 Å). With $A = 5 \times 10^{-19}$ J, $r = 0{\cdot}5$ μm and $\rho = 0{\cdot}5$, one has $\sigma_t = 0{\cdot}23$ MPa, which is the good order of magnitude for ceramic powder tensile strength.

However, eqn (6) is not satisfactory for several reasons. On the one hand, the choice of z_0 is rather arbitrary. Indeed, it has been shown[8] that separate measurements of f_0 and σ_t do not fit the formula, by a factor of about ten. On the other hand, it shows that even if f_0 is not a pure van der Waals force but something else (capillary force, etc.), the dependence of σ_t on ρ is the same (i.e. $\rho/(1-\rho)$). But this dependence fits neither experiments on ceramic powders[9] nor on latex powders.[5] The model does not reproduce the strong rise of σ_t with ρ which is observed experimentally.

Kendall *et al.*[2] and Adams *et al.*[10] have investigated these problems, and proposed using the theory of linear fracture mechanics for a better understanding of granular material tensile strength. York *et al.*[11] applied this theory to pharmaceutical excipients.

2.2 Fracture mechanics

Since the fracture is brittle, it is to be expected that the fracture plane does not break as a whole but as the result of crack opening and propagation. As a consequence, the interparticle forces cannot be summed to obtain the tensile strength (on the contrary, the rupture energies of bonds can be summed, which gives the rupture energy of the packing). An extensive description of linear fracture mechanics may be found in Refs 12 or 13.

Assuming a pure van der Waals attraction between particles, Kendall *et al.*[2] calculated the rupture energies of several ordered packings. The rupture energy was found to depend roughly on the fourth power of the packing density. The application of the Griffith criterion led to the following formula for the tensile strength:

$$\sigma_t = \frac{15{\cdot}6 \rho^4 \Gamma_R^{5/6} \Gamma_E^{1/6}}{\sqrt{2rc}} \tag{7}$$

where ρ is the relative density, c is the crack size, r the radius of the particles, and Γ_R and Γ_E are surface energies measured by rupture and modulus experiments respectively. Assuming a constant crack size, the fit with experimental $\sigma_t(\rho)$ results is better than eqn (6) but not yet perfect: the experimental tensile strength rises even faster than ρ^4.[9] The model of Kendall *et al.* is also discussed by Abdel-Ghani *et al.*[14]

As a consequence, the present authors decided to investigate the tensile strength of ceramic green samples in order to determine the mechanisms of cohesion and fracture.

3 Experimental

The ceramic powder is a binderless zirconia powder (YZ3 TECMIC, Rhône-Poulenc, France) whose mean size is 0·65 micron.

The first idea was to measure the tensile strength through both three-point bending or the diametral compression test (the so-called 'Brazilian test'). However, despite several published results,[15] the experiments showed that the tensile strength measured with the Brazilian test was about three times smaller than the strength measured with the three-point bending test. This discrepancy might be ascribed to a bad stress repartition in the disk: no padding was used in the present case, despite the fact that several authors[16,17] stressed its importance on the results. Furthermore, the disks were obtained by uniaxial compaction and ejection: ejection may have caused some microcracking in the samples. As a consequence, the results were all obtained by three-point bending test (distance between grips $L = 24\,\text{mm}$, thickness $w = 4{\cdot}5\,\text{mm}$, width $b = 4{\cdot}5\,\text{mm}$).

Green samples were obtained either by compaction or slip-casting. In the former case, the samples were given a rectangular shape by uniaxial compaction (37 MPa) in a rectangular mold ($6 \times 4{\cdot}5 \times 44\,\text{mm}^3$). This mold could be dismantled after compaction, which allowed unmolding of the sample with minimum sliding on the walls. Then isostatic compaction was used to increase the density to the prescribed value (direct uniaxial compaction at high pressure was not possible due to fracture upon unloading). It was observed that isostatic compaction did not alter the rectangular shape.

Slip-cast specimens were cast in a plaster mold, without any binder. The mold was divided with metallic walls so that rectangular samples ($7 \times 5 \times 55\,\text{mm}^3$) were directly obtained. A range of densities was obtained by changing the pH of the slurry with ammonia (pH 8·5 to 10; the solid content was kept at 75%). After one day in the plaster mold at room temperature, the samples were removed and dried for 24 h at 100°C then stored at least for 24 h in a desiccator. They were tested immediately after leaving the desiccator. A few cast specimens were dried at 400°C in order to remove any trace of ammonia, and compared with those dried at 100°C; no difference was observed in the fracture strength.

Fracture strength and Young's modulus were obtained from the following equations:

$$\sigma_t = \frac{3}{2}\frac{FL}{w^2 b} \tag{8}$$

$$E = \frac{1}{4}\frac{FL^3}{y w^3 b} \tag{9}$$

where F is the load, b and w respectively the width and thickness of the specimen, L the distance between grips and y the maximum deflection.

The estimation of the Weibull modulus m was carried out with at least 10 samples. A rigorous determination is quite complicated, since the stress state in the whole sample must be taken into account for the failure prediction,[15] but a good estimation was thought to be obtained from a direct reading of the Weibull plot.

The stress intensity factor was obtained from the equation

$$K_{IC} = Y\sigma\sqrt{a_c} \tag{10}$$

where a_c is the notch depth (the notch width being 0·2 mm) and Y is the well known calibration factor (three-point bending):

$$Y = A_0 + A_1(a_c/w) + A_2(a_c/w)^2 + A_3(a_c/w)^3 + A_4(a_c/w)^4 \tag{11}$$

where

$$A_0 = 1{\cdot}9 + 0{\cdot}0075(L/w)$$
$$A_1 = -3{\cdot}39 + 0{\cdot}08(L/w)$$
$$A_2 = 15{\cdot}4 - 0{\cdot}2175(L/w)$$
$$A_3 = -26{\cdot}24 + 0{\cdot}2815(L/w)$$
$$A_4 = 26{\cdot}38 - 0{\cdot}145(L/w)$$

Fracture energy was obtained from the integration of the stress–strain relationship when the fracture was stable.

Note that it was checked, for both cast and pressed samples, that there was no anisotropy: the strength or stress intensity factor does not change when samples are tested in the direction of pressing (or casting) or in the perpendicular direction.

4 Green Strength of Pressed Products

Before applying linear elastic fracture mechanics to ceramic green products, one must ensure that their behavior is really linear elastic and brittle. Figure 1 shows that the strain–stress relationship is indeed a straight line before failure.

The small irreversible deformation that always occurs during the deformation of a granular materials was found to be negligible. The Young's

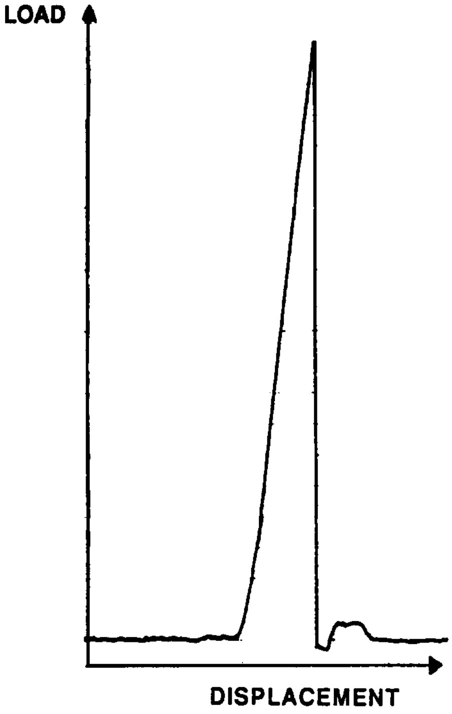

Fig. 1. Load–displacement relationship during the tensile test of a pressed green product. Compaction pressure 50 MPa, density $3{\cdot}32$ g/cm^3.

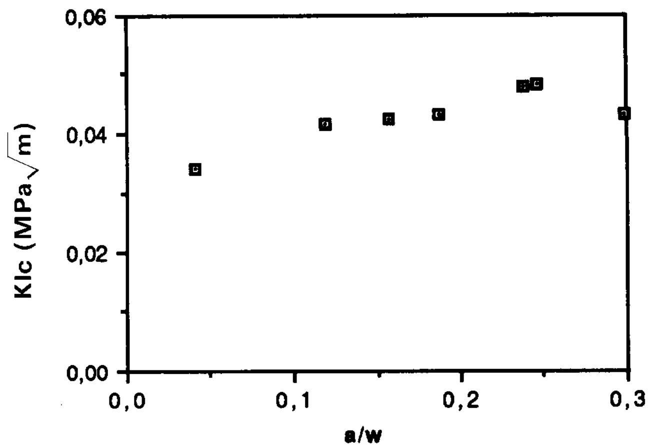

Fig. 3. K_{IC} as a function of a/w. Compaction pressure 50 MPa, density $3{\cdot}32$ g/cm^3; error bars are indicated.

modulus was thus calculated from the slope of this curve.

The next step was to verify whether the results obeyed uniaxial Weibull statistics. This is not at all obvious, since other statistical laws are possible.[16] Figure 2, however, shows that the results obtained for 18 experiments lie on a straight line in the Weibull plot, with $m = 20$.

Figure 3 shows the measured K_{IC} as a function of the ratio a/w; a constant value is obtained. It was not necessary to introduce an 'effective crack length' $a + \Delta a$, as in Ref. 10, to take into account a plastic zone at the crack tip. The large plastic zone observed by Adams *et al.* probably arises because the powder (sand) used contains a binder (polyvinyl alcohol). However, it is remarkable that the K_{IC} obtained with ceramic powder is roughly the same order of magnitude as the value obtained by Adams *et al.* ($K_{IC} = 0{\cdot}1$ MPa$\sqrt{\text{m}}$ for the same porosity of $0{\cdot}44$).

It is now necessary to verify that the sample crack length and dimensions obey the criteria for the validity of stress intensity factor measurements in linear elastic fracture mechanics. These criteria are:

$$a \geq 2{\cdot}5(K_{IC}/\sigma_e)^2 \quad (12)$$

(i.e. the notch length is large compared with the plastic zone)

$$w \geq 5(K_{IC}/\sigma_e)^2 \quad (13)$$

(i.e. the sample thickness is large compared with the plastic zone)

$$b \geq 2{\cdot}5(K_{IC}/\sigma_e)^2 \quad (14)$$

(i.e. the sample width is large compared with the plastic zone)

where σ_e is the elastic limit ($\sigma_e = \sigma_t$ in this case). Thus one should have $a \geq 1{\cdot}25$ mm, $w \geq 2{\cdot}5$ mm and $b \geq 1{\cdot}25$ mm. The experimental notch depth a is slightly too small. However, the good results shown prove that the criteria were satisfied within the required limits.

It is known that the measured critical stress

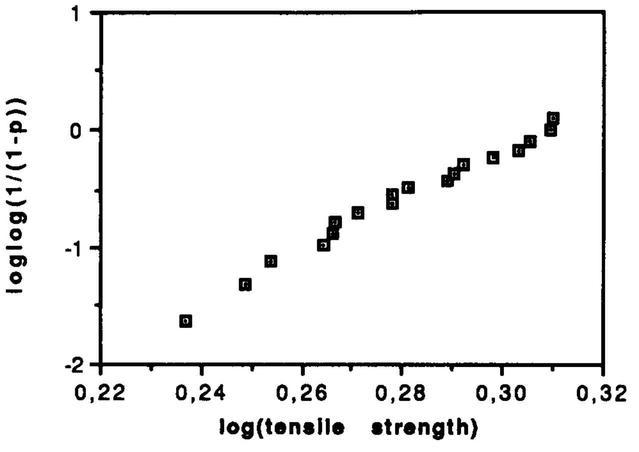

Fig. 2. Weibull plot for a pressed product (threshold stress $\sigma_u = 0$). Compaction pressure 50 MPa, density $3{\cdot}32$ g/cm^3.

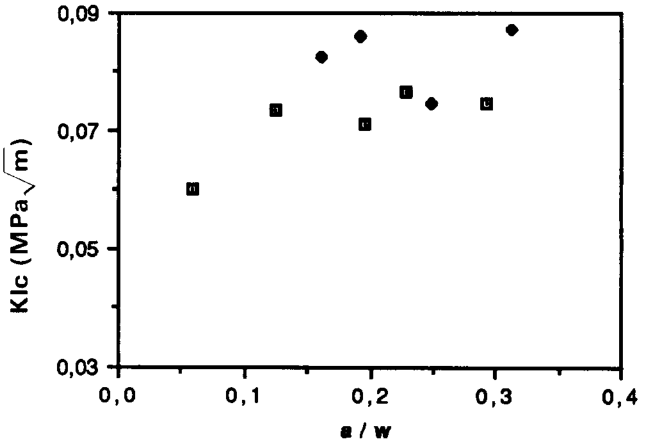

Fig. 4. K_{IC} as a function of a/w for different notch width (□, radius = $0{\cdot}1$ mm; ◆, radius = $0{\cdot}2$ mm). Compaction pressure 150 MPa, density $3{\cdot}48$ g/cm^3.

intensity factor depends on the notch radius if this radius is too large. Figure 4 seems to show a slight increase of the measured K_{IC} when the notch radius increases from 0·1 to 0·2 mm. However, notches 0·1 mm wide are difficult to machine. So, even if 0·2 mm is too high a value for a perfect determination of K_{IC}, it is thought to give a good basis for a comparison between different specimens.

In order to check the predictions of the theory, the experimental value of the fracture energy will now be compared with the value obtained from the theoretical formula:

$$G = \frac{K_{\mathrm{IC}}^2(1-\nu^2)}{E} \tag{15}$$

where ν is Poisson's ratio. For a green density 3·32 g/cm^3 (compaction pressure 50 MPa) one has

$$G_{\mathrm{measured}} = 1{\cdot}25\ \mathrm{J/m^2} \quad \text{(from load/deflection curve)}$$
$$G_{\mathrm{calculated}} = 1{\cdot}28\ \mathrm{J/m^2} \quad \text{(from eqn (15))}$$

showing excellent agreement between theory and experiment. This result proves that linear fracture mechanics can be applied successfully to ceramic green products.

The last item of information that can be obtained from these results is the critical defect size:

$$a_{\mathrm{c}} = (K_{\mathrm{IC}}/Y_0\sigma_{\mathrm{t}})^2 \tag{16}$$

where $Y_0 = 1{\cdot}12\sqrt{\pi}$. One obtains $a_{\mathrm{c}} = 170\ \mu\mathrm{m}$. This may be rather surprising, since mercury porosimetry does not show such large pores (see Fig. 9). But the equivalent crack a_{c} is not necessarily a true pore. Even if there were such pores, they may be closed by small openings (so that they are not detected by mercury porosimetry), or their total volume may be too small to be measured. Attempts were made to observe these defects by SEM micrographs, but polished surfaces were too difficult to realize on green products. The comparison between slip-casting and dry-pressing will shed light on the question of critical defects.

As explained before, Kendall *et al.*[2] tried to predict the rupture energy of granular materials on the basis of a microscopic model. The following expression was obtained:

$$G_{\mathrm{Kendall}} = 56\rho^4(\Gamma^5/E^2d^2)^{1/3} \tag{17}$$

where Γ is the interfacial energy, E is the Young's modulus, d is the average diameter of the particles, and ρ is the solid volume fraction.

With $\Gamma = 80\ \mathrm{J/m^2}$, $E = 200\ \mathrm{GPa}$, $\rho = 0{\cdot}55$ and $d = 0{\cdot}5\ \mu\mathrm{m}$, the predicted fracture energy is $3{\cdot}53\ \mathrm{J/m^2}$, which is not so far from the experimental results. However, it will be shown in the following that the main hypothesis from Kendall *et al.* (the interaction force between particles is only the van der Waals force) is not true. Abdel-Ghani *et al.*[14] provided an interesting review of the work of Kendall *et al.*

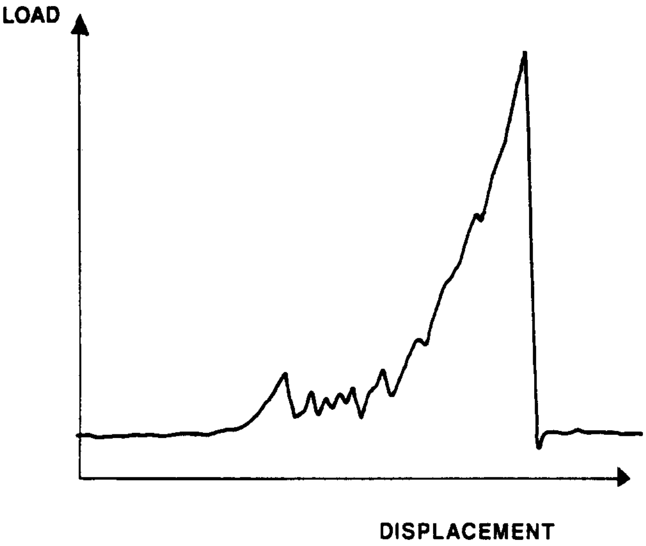

Fig. 5. Load–displacement relationship during the tensile test of a slip-cast green product. Density 3·59 g/cm^3.

5 Comparison between Slip-cast and Dry-pressed Specimens

Figure 5 shows that the strain–stress relationship during the tensile test of a cast sample is characteristic of a brittle failure. However, it is seen that several small instabilities occur during the loading sequence. They may be ascribed to the crushing of small surface asperities.

The differences between the slip-cast and dry-pressed products will now be described, by plotting their mechanical characteristics versus density.

Figure 6 shows that for a given density cast samples are weaker than pressed samples. From eqn (10) this may come either from a smaller critical stress intensity factor K_{IC} or from a larger critical defect a_{c}.

The critical stress intensity factor of cast samples is indeed smaller than for pressed samples (Fig. 7).

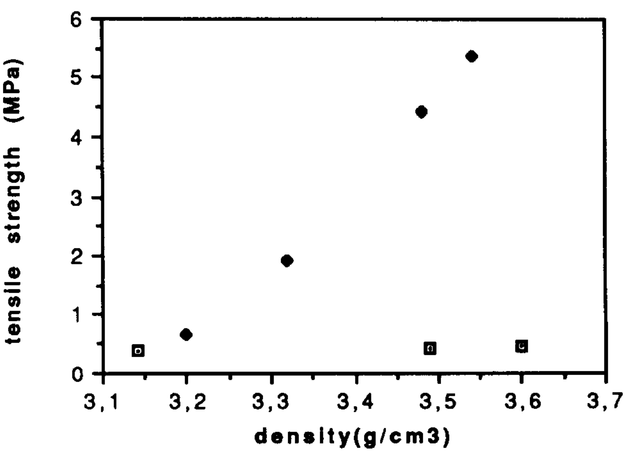

Fig. 6. Mean fracture strength versus density, slip-cast (⊡) and dry-pressed (◆) samples.

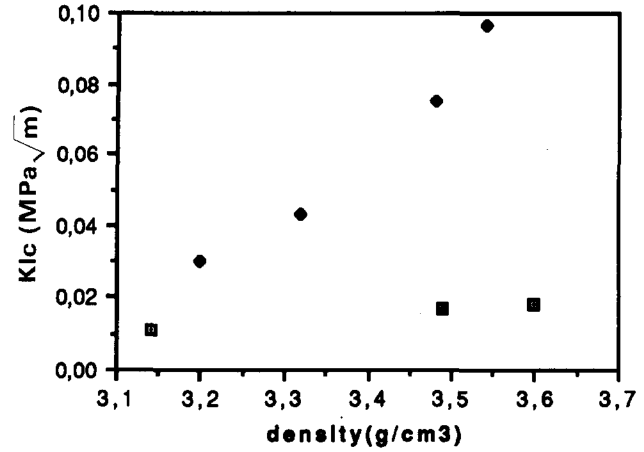

Fig. 7. Critical stress intensity factor versus density, slip-cast (□) and dry-pressed (◆) samples.

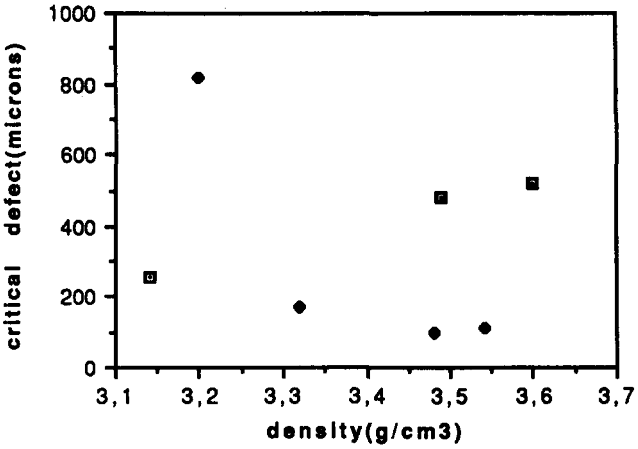

Fig. 8. Critical defect size versus density, slip-cast (□) and dry-pressed (◆) samples.

The critical stress intensity factor is an intrinsic property of the material, which does not depend on the microstructure of the samples (cracks, etc.). Thus the microscopic mechanism of interparticle cohesion is different in pressed and cast samples. This can be explained easily: during casting, the particles fall slowly one over the other. After drying, the interaction force between them is only the van der Waals force or hydrogen bonding. By contrast, during dry pressing the interparticle forces are high enough to cause a real interlocking of the surface asperities. As a consequence, for a given density the green strength is far higher. The occurrence of mechanical interlocking is also discussed in Refs 18–20.

However, Fig. 8 shows that the smaller tensile strength of cast samples arises also from their larger critical defect size (except for low densities). This is rather surprising, since it is usually believed that slip-cast microstructures are 'better' than pressed microstructures. Indeed, mercury porosimetry experiments show that the pore size distribution of cast samples is sharper (for a given density) than the pore size distribution of pressed samples (Fig. 9).

This is another example showing that the defect size is not directly related to the pore size distribution but is concerned with another scale of the structure which is intermediate between the particle size and the sample size.

The Young's modulus is also higher for dry-pressed samples (Fig. 10). This may come from the interlocking of asperities in these samples (stiffer

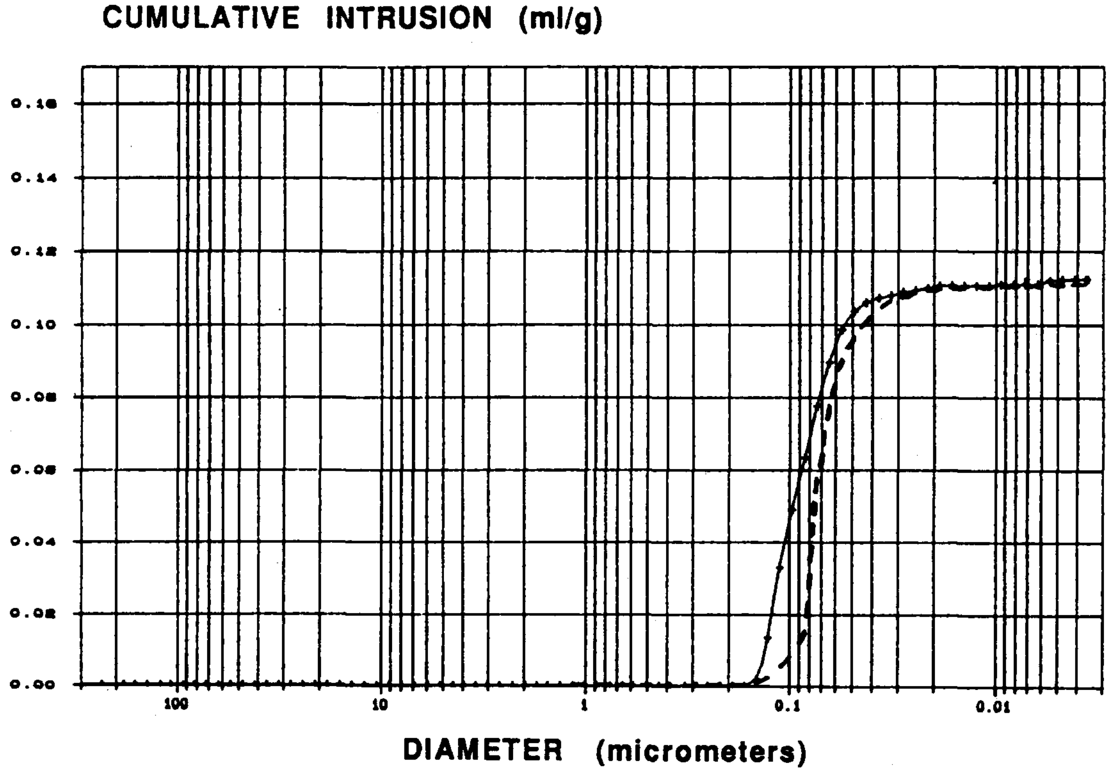

Fig. 9. Pore size distribution for pressed (+++) and slip-cast (– – –) samples. Density 3·45 g/cm^3.

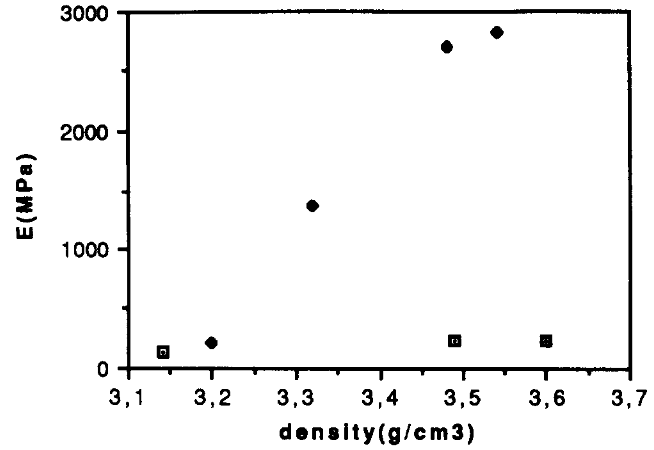

Fig. 10. Young's modulus versus density, slip-cast (⊡) and dry-pressed (◆) samples.

particle contacts) and/or from the occurrence of larger cracks in cast samples.

Figure 11 compares the fracture energies of cast and pressed samples. At low densities they are nearly equal, but the increase with density is steeper for pressed than cast samples.

Let G_1 be the fracture energy of cast samples; it comes from the van der Waals interaction between particles, and depends on the number of contacts which are broken by the fracture surface. In Rumpf's model (eqn (2)) one should have $G_1 = \alpha[\rho/(1-\rho)]$, while in the model of Kendall *et al.*[2] one should have $G_1 = \alpha\rho^4$. Figure 12 shows that Kendall's model is more efficient (note that for both models the coefficient α has been calculated so that $G_{1(\text{calculated at } 3{\cdot}1\ \text{g/cm}^3)} = 0{\cdot}75\ \text{J/m}^2$).

Assuming that the fracture energy G of pressed samples can be divided into two parts corresponding respectively to the van der Waals interaction and to the mechanical interlocking of asperities, then $G = G_1 + G_2$. Figure 11 shows that the influence of G_2 becomes important for compaction pressures above 40 MPa. From previous experiments it is the authors' opinion that G_2 depends mainly on the compaction pressure and not on density.[9]

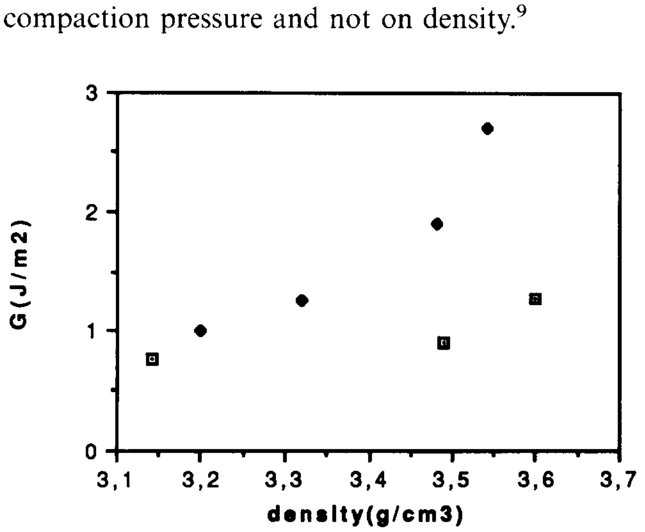

Fig. 11. Fracture energy versus density, slip-cast (⊡) and dry-pressed (◆) samples.

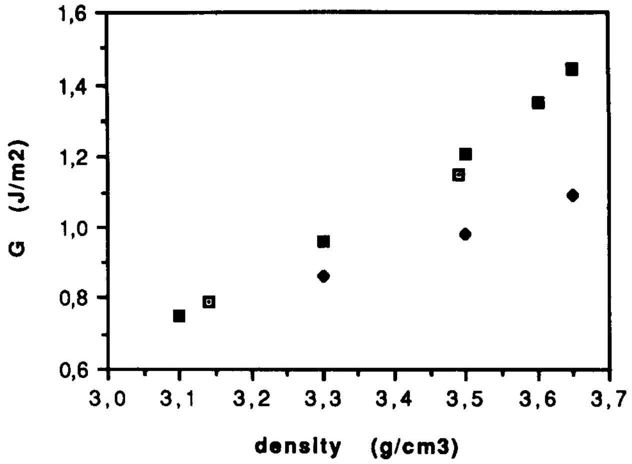

Fig. 12. Fracture energy G_1 of cast samples versus density; experimental (⊡) and theoretical predictions: Rumpf (◆), Kendall *et al.* (■).

6 Conclusion

Linear fracture mechanics have been applied successfully to ceramic green products. The behavior has been shown to be elastic–brittle, the K_{IC} was seen to be constant versus a/w, and the fracture energy predicted with K_{IC} and E was very close to the experimental value.

This method was further applied to slip-cast and dry-pressed samples of different densities. As a general rule, slip-cast samples were found to be weaker (for a given density) than dry-pressed samples. This difference comes from a smaller critical stress intensity factor and a larger critical defect size. The high K_{IC} or fracture energy of pressed samples was ascribed to the mechanical interlocking of the particles caused by the high pressure.

The order of magnitude of the critical defect size is 100 μm; this is very large compared with the pore size as determined from mercury porosimetry. At this scale the microstructure of cast samples was shown to be 'worse' than the structure of pressed samples while it is 'better' (smaller pores, sharper pore size distribution) at the particle scale.

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