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Mechanical behaviour of brittle cement grains

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Before attempting a modelling approach to the crushing mechanisms of "cement clinker" it was necessary to characterize the mechanical behaviour of this material up to breakage. The diametrical compressive test was chosen, as it is the closest to the typical loading within a packing of grains and a very convenient experimental approach for the analysis of mechanical behaviour law up to fragmentation of brittle granular material. The modellization of the grain as a hard brittle core surrounded by a thin crumbly skin leads us to associate the contact setting with the crumbly skin behaviour and the breakage with the brittle core behaviour. This analysis allowed us to define scaling laws for the skin thickness and breakage load. The breakage force increases as a power law of the grain diameter with an exponent of 1.5 and it is shown that the mean prefactor K_0 is an intrinsic characteristic parameter proportional to the tenacity of the material.

1. Introduction

Breaking grains in order to obtain a finely divided powder is an important process in many industrial applications. However in most cases it is energetically very inefficient.

This is especially true for civil engineering materials such as cement where 40% of the energy consumed is devoted to the crushing process. The material we tested "cement clinker", consists of granules of cement. It is the physical state of cement powder at furnace output just before crushing.

The mechanical properties of the material to be broken up are obviously a key element in understanding the fragmentation process. The literature is poor on this subject and the most important characterization of clinker mechanical behaviour was made by Forgeron [1]. Cement clinker itself was not studied, but instead samples of heat sintered cement powder, a reconstituted material whose mechanical properties differ widely from cement clinker itself. In the present study we chose to use real "cement clinker" in order to be closer to the industrial application.

2. Materials and methods

In industrial breakers the grains of material to be crushed are submitted to compressive load up to breakage. In the laboratory instead we used the diametrical compressive load test, which is very convenient for the analysis of the mechanical behaviour of a brittle granular material. All the loading tests are conducted on a universal testing machine (Instron 4505), with imposed crosshead speed, under quasi-static conditions. Crosshead displacement and load are recorded as a function of time. The diameter of the granules of clinker used in the tests ranges from some tenths of a mm to several cm. In order to limit dispersion we retained only spheroid grains of uniform aspect. This criterion leads us to exclude grains with too heterogeneous a structure (this structure results from the manufacturing process). Practically, due to the available testing machine configuration, the diameter of the smallest grains studied was of millimeter scale.

For a structurally heterogeneous material such as cement clinker we had to consider two very distinct stages as shown in Fig. 1: the initial setting of contacts (1 in the figure) – a local crushing process controlled by the macroscopic roughness of the particle – followed by the classical linear behaviour of brittle material up to breakage (2 in the figure). The main differences with glass balls [2] (used as the brittle materials reference) appeared in the first part of the compressive load test. Creating an area of contact is different in the two cases. The modellization of the grain as a hard brittle heart of diameter $\approx \phi$ surrounded by a thin crumbly skin lead us to associate the contact setting with the crumbly skin behaviour and the breakage with the brittle heart behaviour (numbered respectively 1 and 2, in Fig. 1).

The fracture load (i.e. the load needed to create a cross crack) follows, as for a glassy material, a power law as a function of the particle diameter with an exponent close to 1.5. The dispersion we observed on experimental results can be analysed statistically using Weibull laws, or better with a specific failure criterion which allows us to define a material-dependent tenacity-like parameter characterizing the hard brittle core of the grain.

In contrast to load, displacement exhibits non-classical behaviour. Analysis of cycling loading tests allows us to define the displacement at the end of the crushing stage of contact setting and gives us a simple

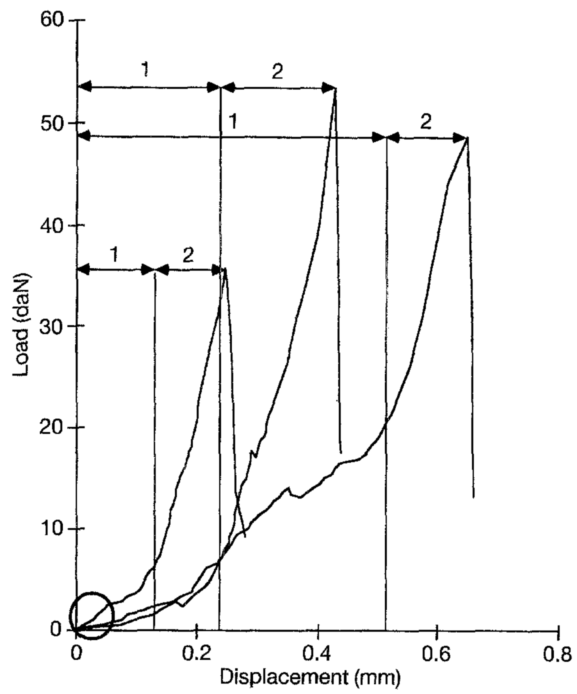

Figure 1 Typical load-displacement curves obtained in similar conditions for grains of equal size (mean diameter 6 mm). In spite of their disparity the general shape of the the curves suggests a separation into two parts: 1. surface crushing: flattening of contact areas by local crumbling; 2. Classical linear behaviour up to breakage of the grain.

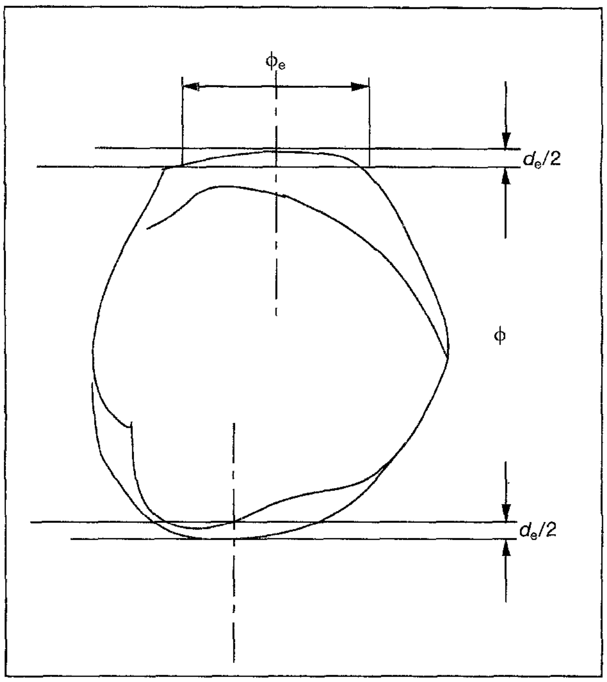

Figure 2 Geometrical model of the grain shape at the end of the crushing stage. The crumbling process of the skin is assumed to be local and confined to the vicinity of the contact areas.

rule to determine its value directly on the classical load–displacement curve. This value corresponds to half the crumbly skin thickness in our model (Fig. 2). Its linear dependence on grain diameter is confirmed by surface contact measurements as will be seen later.

Let us first discuss the second stage because of its greater importance for the breaking process.

3. Behaviour law before failure

The later part of the loading curve shows the typical behaviour of a brittle material up to failure. In order to scale breakage load and displacement with grain size, we analysed the loading curves of about 400 grains (diameter range 1.1–15 mm) tested up to breakage in similar conditions.

3.1. Breakage load

We attempted to fit the variation of the breakage load F_r with diameter ϕ to a power law

$$F_r = K\phi^{\alpha} \quad (1)$$

The analysis (Fig. 3) leads to a prefactor mean value K_0 of 3.36 and an exponent of 1.5 ± 0.1, which is consistent with the empirical Bond's law [3] (fracture energy scaling $\phi^{2.5}$). The transition size (or "critical size" in Kendall's sense [4]) below which energy increases inversely with grain diameter could not be observed although we tested 0.5 mm diameter grain beds.

The dispersion on F_r values is important, but all the experimental points are included between two limiting curves following a power law with the same exponent α and prefactors equal to 1.6 (lower curve) and 6.0 (upper curve) respectively. This suggests a dispersion law for the prefactor K independent of the diameter ϕ.

This plot suggests that it is possible to correct for the diameter effect by introducing a reference diameter ϕ_0, the equivalent breaking force F_0 that would be needed to break a grain of diameter ϕ_0. This equivalent force F_0 is related to F through

$$F_0 = F\left(\frac{\phi_0}{\phi}\right)\alpha \quad (2)$$

Choosing a unit reference diameter ϕ_0 is equivalent to studying the prefactor K of Equation (1).

Assuming $\alpha \approx 3/2$ this prefactor has the dimension of tenacity ($\mathrm{Nm}^{-1/2}$). Applying the concepts of brittle fracture mechanics [5] to the hard core of the grain and using a scaling argument we derive the relation between K and the tenacity K_c as follows.

Let σ denote the average stress applied to a structure made from a material of tenacity K_c. The ratio K_c/σ is dimensionally equivalent to the square root of a length L called the "Griffith length" [6]; that means the length of the largest macroscopic fault allowable under these conditions. Let ϕ be a characteristic size of the structure. At breakage L has the value ϕ and the rupture load F_r is of the order of $\sigma\phi^2$. According to the definition of L we have:

$$F_r \approx K_c\phi^2/\sqrt{\phi} \quad (3)$$

From a comparison of Equations (1) and (3) we may conclude that the prefactor K is proportional to the tenacity K_c and can be studied as an intrinsic feature of the material.

The statistical distribution of K can be fitted to various trial functions. We made various attempts along these lines: first a Weibull-like law:

$$F(K) = 1 - \exp(-(K/K_0)^{\alpha}) \quad (4)$$

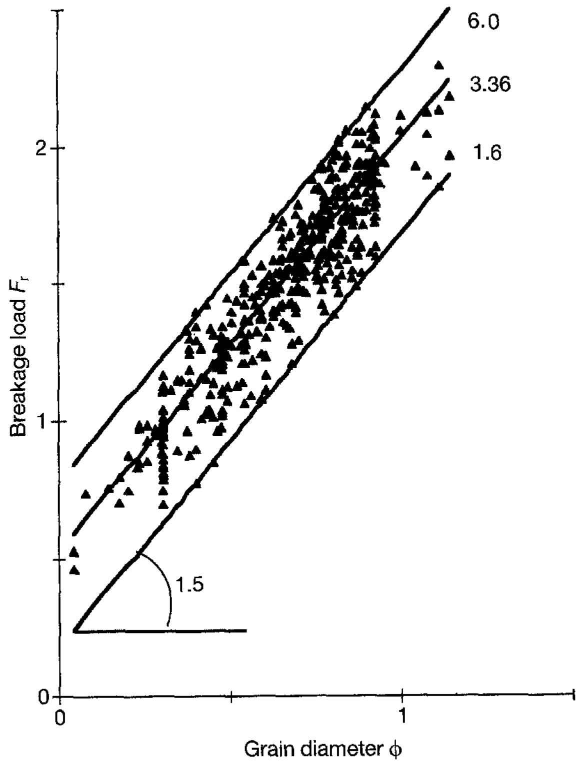

Figure 3 Breakage load F_r versus grain diameter ϕ: log–log plot. The breakage force increases as a power law of the grain diameter with an exponent of 1.5 and a mean prefactor of 3.36 (middle curve). The experimental points are included between two limiting curves following the same power law with prefactors respectively equal to 1.6 (lower curve) and 6.0 (upper curve). This suggests a dispersion law for the prefactor K independent of the diameter ϕ.

This law with a high exponent ($\alpha = 4.18$ and $K_0 = 3.83$) fits correctly the first part of the K distribution (values less than the average), but a lower exponent ($\alpha = 2.26$ and $K_0 = 3.53$) gives a better fit above the average value and takes into account the large force tail of the K distribution.

As pointed out by Neville [7] a distribution law of the form:

$$F(K) = (K/K_0)^{\alpha}/1 + (K/K_0)^{\alpha} \quad (5)$$

is a good representation of the statistical criterion for failure in linearly elastic materials containing distributions of microcracks such as cement clinker. In Fig. 4 such a distribution with an exponent $\alpha = 5.45$ and $K_0 = 3.36$ gives a better fit for the overall experiment. It is noteworthy that K_0 corresponds to the averaged value (over all experimental points) of the prefactor K of the breakage power law (F_r versus ϕ). For the large values of K belonging to the tail of the distribution the experiment deviates from the fit, but it is readily seen in Fig. 3 that the difference is independent of the grain diameter. At the other end of the distribution, when K tends towards zero, the two functions $F(K)$ of Equations (4) and (5) tend towards a power law $(K/K_0)^{\alpha}$ with a high exponent as a consequence of the existence of a cut-off at low breakage load.

These results allow us to define, for each grain, a mean breakage load F_c related to the diameter ϕ by:

$$F_c = K_0 \phi^{\alpha} \quad (6)$$

with an exponent $\alpha = 3/2$ and a prefactor $K_0 = 3.36$.

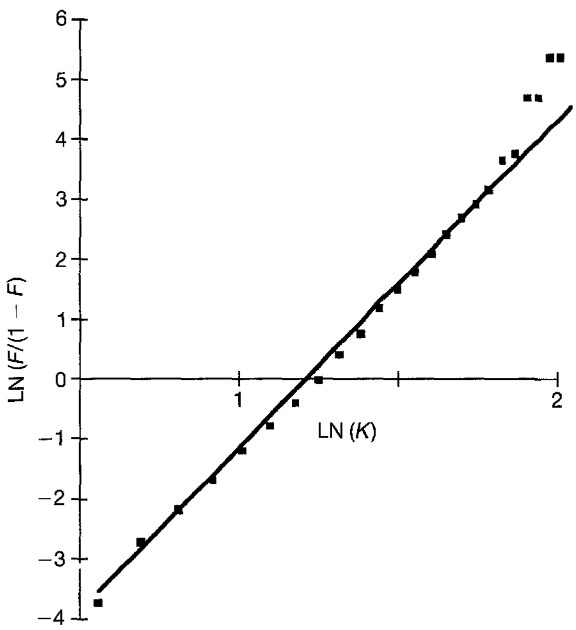

Figure 4 Statistical study of the cumultive probability of the prefactor K of the power law of Equation 1 (breakage load F_r versus grain diameter ϕ). For the large values of K belonging to the tail of the distribution the experiment deviates from the fit but it is readily seen in Fig. 3 that the difference is independent of ϕ.

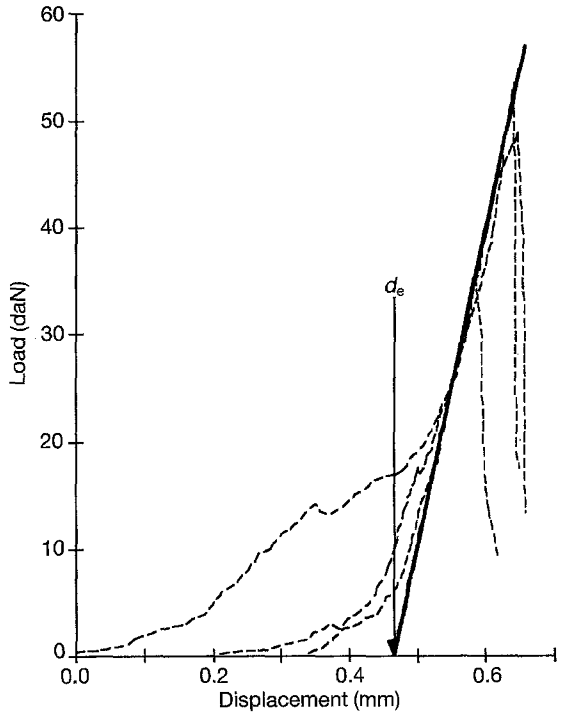

Figure 5 The three loading curves of Fig. 1 superposed by shifting along the displacement axis display the same linear end behaviour. The values of displacement (d_e included) on the x axis are those of the reference curve (not shifted).

3.2. Breakage behaviour

In order to avoid fluctuations introduced by the crumbling process we shifted the load curves obtained for each of the 400 tested grains along the axis of displacement in order to superpose their end parts. A linear function, with a slope β, fits the different curves correctly, as shown in Fig. 5. It was found that the slope of linear behaviour is independent of grain size and a mean stiffness β equal to $300 \pm 50\ \mathrm{kg\,m^{-1}}$ was determined.

4. Surface crushing

Using this linear relation between load F and displacement d and the corresponding measured breakage values F_r and d_r, we compute, for each tested grain, what we call the "crushing end displacement" d_e as

$$d_e = d_r - F_r/\beta \qquad (7)$$

d_e is easily determined as the extrapolated displacement (at zero load) of the linear breakage behaviour of the static loading tests.

4.1. Cyclic loading and incremental displacement

To show that d_e is a good measure of the irreversible part of the grain strain variation, the compressive load test is performed as a series of successive load–unload cycles. Each cycle consists of a loading (up to load F) followed by a complete unloading (down to zero) of the grain under test. The maximal load reached at the end of the i-th cycle is given by $i^*\Delta F$. The increase of load ΔF between two successive cycles represents a given percentage (5% in our tests) of the expected average breakage load F_c, which is a function of the grain diameter (Equation 6). We define the irreversible incremental displacement Δd, at load F, as the difference in measured crosshead displacement (at zero load – end of cycle) between two successive cycles (Fig. 6). The evolution of Δd as a function of F follows the grain irreversible deformation and then characterizes the crushing stage. Therefore we observe (Fig. 6 – insert), as expected, large values for Δd during the crushing phase, then low variations beyond. The graph $\Delta d(F)$ – at given ΔF, F is proportional to the cycle number – shows the same behaviour, independent of grain size. This curve (main graph) clearly displays the crumbling phase and the low value of the reduced displacement during the final cycles. This allows us to neglect the small variation of the irreversible part of the displacement during this last stage – Δd would be strictly zero valued for a purely elastic material – and to identify d_e with the irreversible component of the displacement (mainly generated by the crushing process).

In order to confirm this analysis we tested grains whose contact faces were previously planed with abrasive paper. Loading curves looked as expected, the central part of the graph, which corresponds to the crumbling phase, disappeared, and loading curves exhibited only linear behaviour up to fracture.

In our experiments we observed that the crushing stage (first part of the compressive test curve) is always associated with the local crumbling of the areas of contact between grain and crosshead. More and more asperities get in contact with the testing machine crosshead surfaces, offering increasing force to the flattening of the grain surface, but the compressive load increases moderately compared with the displacement. We can now identify the thickness of the crumbly skin of our model of grain to half of the crushing end displacement d_e.

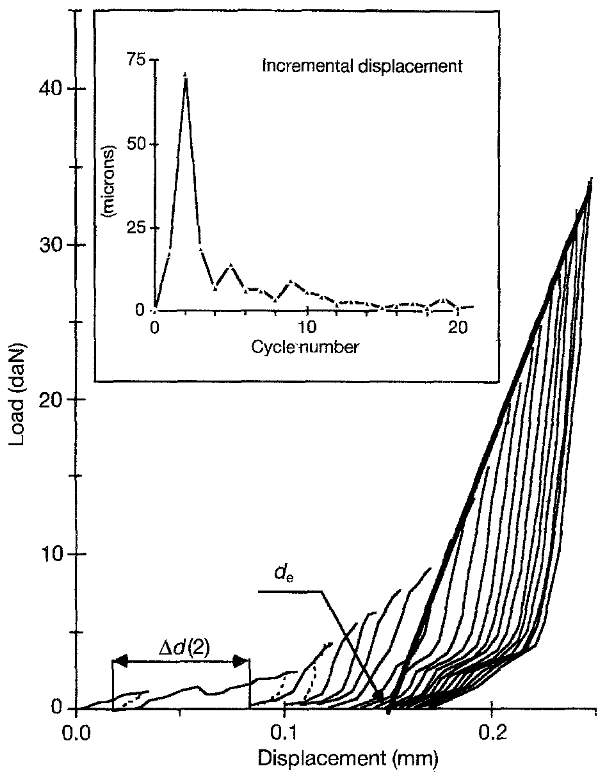

Figure 6 The main graph represents the typical response of a grain to a cyclic compressive loading test (unloading part of cycle not drawn for clarity). The maximal load reached at the end of the i-th cycle is given by $i^*\Delta F$, where ΔF is a given percentage (5% in our tests) of the theoretical breakage load F_c, a known function of the grain diameter. The insert represents the evolution of the incremental displacement as a function of the cycle number.

4.2. Contact setting

If we enlarge the force – displacement curve at a very low level of load (Fig. 7) we observe fully reversible elastic – but non-linear behaviour which ends when the first asperity crumbles. Due to the early occurrence of crumbling this first stage is difficult to analyse. However, if we take into account only the first points of the graph – inside the circle drawn near the origin on Fig. 1 – it is possible to define a power law relation $F \propto d^{\lambda}$ between the load F and the displacement d with an experimental exponent $\lambda \approx 1.5$–2.0. The exponent of the elastic behaviour depends on the shape of the first asperity. The flattening law is consistent with the prediction of Hertz's theory of contact [8, 9] which gives exponents ranging from 1.5 (spherical punch) to 2 (wedge shaped punch).

4.3. Scaling law

The grain compression at the end of the crushing stage "crushing end displacement" d_e varies with diameter ϕ as

$$d_e = \gamma\phi \qquad (8)$$

The mean value $\langle\gamma\rangle$ of the distribution of γ (Fig. 8) is equal to 0.037, and the standard deviation is equal to

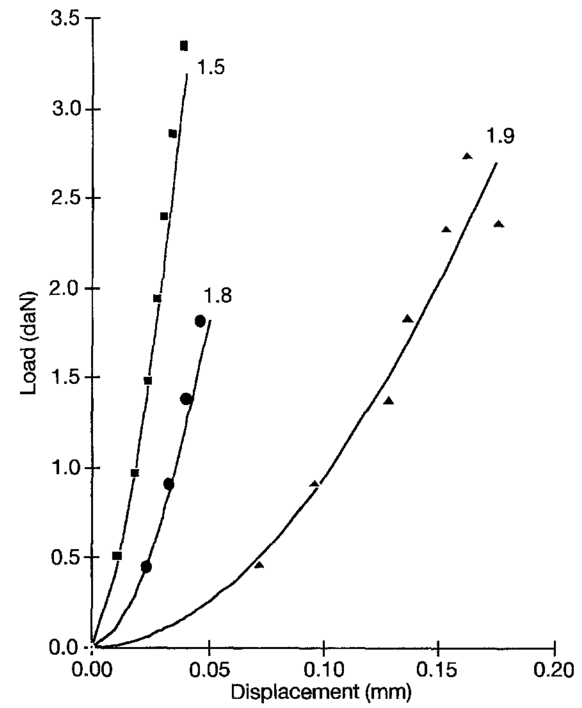

Figure 7 The experimental exponent $\lambda \approx 1.5$–2.0 of the $F \propto d^{\lambda}$ relation describing the elastic behaviour at the beginning (area inside the circle drawn on Fig. 1) of the load–displacement curve depends on the shape of the first asperity. The flattening law is consistent with the prediction of Hertz's theory of contact which gives exponents ranging from 1.5 (spherical punch) to 2 (wedge shaped punch).

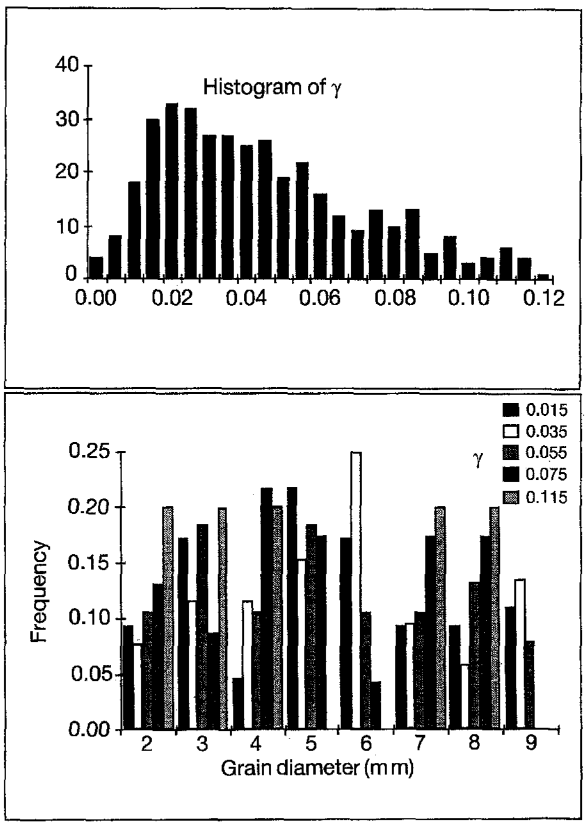

Figure 8 Above: histogram of the ratio γ: "crushing end displacement" d_e to grain diameter ϕ. *Below*: Normalized distributions of grain diameter for five values, 0.015, 0.035, 0.055, 0.075 and 0.115, covering the variation range of γ.

0.024. The dispersion on d_e values is important but, as shown in the lower part of Fig. 8, the normalized distributions of grain diameter – for different values of the factor γ covering its range of variation – spread over the diameter range of the tested grains. This suggests a dispersion law independent of the diameter ϕ for the prefactor γ.

4.4. Contact area measurements

As explained above the compressive load increases moderately compared with the displacement during the crushing stage of the compressive test. All the grain deformation is concentrated in the vicinity of the areas of contact and the measure of the scaling law of the contact area with the grain diameter ϕ is another way to characterize the crushing process.

It is not possible to measure the contact areas at the end of the crumbling stage, since this would imply removing the grain from the testing device. But, as shown in Fig. 6, the grain strain remains very low between crushing and breakage (quasi-zero incremental displacement). As a consequence the increase of the surface of contact remains very low and we can identify, with a good approximation, the contact area at the end of the crumbling process and at breakage. We reconstructed the tested grain from its fragments in order to measure the contact areas. It is rather difficult to measure precisely these areas because we have to restore the grain after breakage; however we observed that they depend on the grain size but they are independent of the extent of the crumbling phase. For each reconstituted grain we measure the values S_1 and S_2 of the two contact areas and compute the "equivalent diameter" ϕ_e of the contact area so that the area of a circular disk of diameter ϕ_e is equal to the average contact area. As shown in Fig. 9 the equivalent

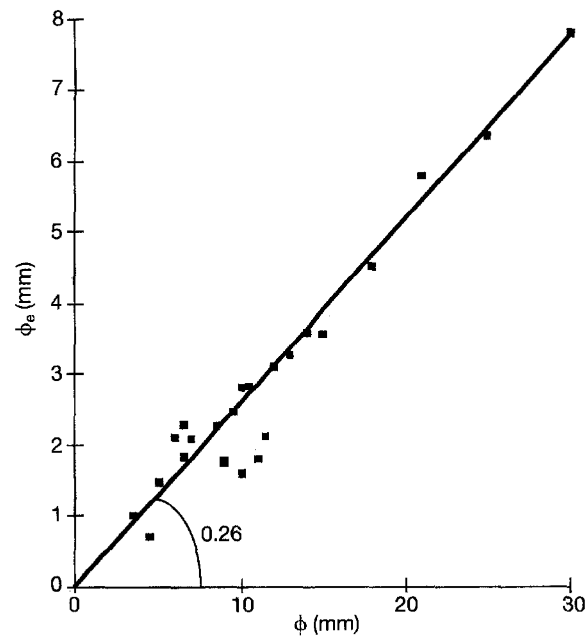

Figure 9 Contact area "equivalent diameter" ϕ_e as a function of grain diameter ϕ. This linear relation is in good agreement with the previously reported linear variation of d_e with ϕ. For a spherical grain (see geometry in Fig. 2) the slope k and the mean value $\langle\gamma\rangle$ of the ratio d_e to ϕ are geometrically related by: $2\langle\gamma\rangle = k^2$. The value $\langle\gamma\rangle = 0.034$ deduced from $k = 0.26$ and the previous mentioned value $\langle\gamma\rangle = 0.037$ are in good agreement.

diameter ϕ_e of the contact area is approximately linearly related to the grain diameter ϕ

$$\phi_e = k\phi \quad (9)$$

with $k \approx 0.26$. This linear relation is in agreement with the previously reported linear variation of d_e with ϕ. For a spherical grain (see geometry on Fig. 2), the three parameters d_e, ϕ_e and ϕ are related in first approximation by: $\phi d_e \approx \frac{1}{2}\phi_e^2$, or $\langle\gamma\rangle = \frac{1}{2}k^2$. The previously mentioned value $\langle\gamma\rangle = 0.037$, and $k = 0.26$; thus $\frac{1}{2}k^2 = 0.034$ is in good agreement.

5. Conclusion

Before attempting a modelling approach of crushing mechanisms of cement clinker it was necessary to characterize the mechanical behaviour of this material up to breakage. The diametrical compressive test was chosen, as it is the closest to the typical loading within a packing of grains and a very convenient experimental approach for the analysis of mechanical behaviour law up to fragmentation of brittle granular material.

Analysis of the loading curve displays two stages, each corresponding to mechanical behaviour we can identify:

- *crushing*: this phase corresponds to the creation of contact areas, a local process, by crumbling asperities of the grain in contact with the crosshead. The average diameter of the contact area varies linearly with the grain size. The grain deformation at the end of this phase also increases linearly as a function of the grain size. This leads to a simple geometrical model. We identify also the process of setting the contact points: the appearance of the loading curve at low load level (first few points) is the same for all the grains. The load–displacement relation follows a power law with an exponent ranging from 1.5 to 2. The progressive flattening of asperities causes a correlative increase of a number of contact points.
- *up to the fragmentation of the grain* (*in fact creation of a cross crack*): introducing normalized displacement to take into account the crumbling effect, the grain exhibits a linear load displacement law up to breakage. As for other brittle materials such as glass, the breakage force increases as a power law of the grain diameter with an exponent of 1.5. The dispersion of prefactors that we observed in the experiments results is analysed according to

$$F(K) = (K/K_0)^\alpha/1 + (K/K_0)^\alpha$$

Modelling of the grain as a hard brittle core of diameter ϕ surrounded by a thin crumbly skin of thickness $d_e/2$ (Fig. 2) leads us to associate the contact setting with the crumbly skin behaviour and the breakage with the brittle core behaviour. This analysis allows us to define K_0 as an intrinsic characteristic parameter proportional to the tenacity of the material.

This work allowed us to characterize mechanical behaviour of cement clinker. This is a study which is necessary to analyse the crushing mechanisms in packed beds. Experimental results on the cohesion of packed beds will be reported in a forthcoming paper.

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