Interparticle Friction in Granular Ceramic Materials

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Abstract

The frictional behaviour of dry ceramic materials in the form of spherical beads, sand and spray dried powders has been studied. Measurements were made using standardized flow time, shear cell methods and die pressing. Flow time was found to provide a poor measure of internal friction for coarse material. Internal friction increased with decreasing particle size and deviations from sphericity. Powders with high specific surface area had low apparent and tap density and flowed poorly if at all under gravity loading conditions. However, shear cell and die press experiments showed the coefficient of internal friction measured at load was not sensitive to this parameter. The coefficient of internal friction calculated from shear cell measurements in which the maximum stress was 0.4 MPa was in good agreement with that deduced from die stresses during die pressing to 300 MPa, even when particle fracture occurred at the higher pressures, provided the morphology did not change appreciably.

1. Introduction

The flow characteristics of granular materials depend to a great extent on friction between adjacent particles. This mechanism is of great importance in many problems involving the flow of granular or powdered materials in bulk materials handling and the powder metallurgy and ceramic fabrication industries.

Of engineering interest is the flow behaviour over a wide range of loading conditions ranging from near-gravity loading in hoppers and die filling operations, to high pressure loading of the kind used in crushing or compaction operations. This flow behaviour affects, amongst other things, loads on containment surfaces and uniformity of bulk density.

Interparticle friction is known to depend on particle morphology and size distribution. It is commonly characterised in terms of the average bulk behaviour of representative samples of the granular material. A number of measures are in widespread usage. Some apply to flow and packing under gravity load conditions. Examples include flow-time tests for a known mass, apparent and tap density, and derived quantities such as the Hausner ratio 1). Other measures are based on the response of the granular material to applied load using for example shear cells of the kind developed to study the failure characteristics of soils and similar granular materials 2, 3). The shear cell measurements provide the coefficient of internal friction in the granular body up to applied normal stresses of about 2 MPa. The nature of this internal friction depends on the nature of the load applied to the granular material. At low loads it may be due to just interparticle friction processes as relative movement occurs between adjacent particles. At higher loads after particle interlocking occurs, it may be some complex combination of this and the yield or fracture properties of the particle material. Measurements of axial and radial stresses in rigid die pressing during either quasi-static 4, 5, 6) or dynamic 7) compaction, or triaxial cell stress measurements 8) can also provide measures of internal friction at applied loads at least 2 orders of magnitude higher than those available in shear cells.

Considerable progress has been made in characterising the flow behaviour of specific granular materials 9, 10) but the linkage between micro-mechanical features and macroscopic measures of internal friction is not well understood.

This paper describes a study of the frictional characteristics of several dry ceramic granular materials including coarse beads, sand and spray dried powders, in which the influence of particle morphology, size and size distribution on internal friction was considered. The internal friction was characterised using a number of methods valid individually over
a range of applied stresses. Thus, the generality of the measures of internal friction, and their relation to morphology and size characteristics could be determined.

2. Experiment and analysis

2.1 Granular materials

Particle size, shape factor and specific surface area were chosen to characterise the granular material studied. Many descriptors are used to characterise morphology. Those adopted in this study were chosen as representative of those in widespread use. Shape factor provides a simple measure of geometry effects at the particulate level, whereas specific surface area is related, at least in part, to particle surface texture.

In all, 15 different samples of granular material were used in the study. These are shown together with important particle parameters in Table 1. The diameter and shape factor were calculated from two dimensional images generated on a Quantimet 570 Image Analyser. The mean diameter shown is the spherical equivalent given by \( \left( \frac{4A}{\pi} \right)^{0.5} \) whereas the shape factor is \( \frac{P^2}{4 \pi A} \) in which \( P \) is the perimeter and \( A \) the projected area. So defined, the shape factor for a sphere is 1. For materials 13, 14 and 15, the specific surface area was measured using the BET method. For the coarser powders for which a figure is shown it was estimated for comparative purposes assuming a closely spherical shape. As such it is a lower limit only approached by material with near unity shape factor and no surface roughness or porosity.

Materials 1, 2 and 3 were solid beads made from yttria stabilized tetragonal zirconia polycrystal (Y-TZP) supplied by ICI Advanced Ceramics, Melbourne. These were chosen for their near spherical geometry and smooth surface as shown in Fig. 1 for material 3, that of nominal diameter 0.1 mm. The surface details shown were typical of that observed for all the bead material (1, 2 and 3). Materials 4, 5 and 6 were precisely measured blends of the bead material, blend 6 corresponding to the maximum packing density possible from spheres of these sizes 11). These spherical particle systems were included in the study to unambiguously decouple size and shape effects on frictional behaviour.

Commercially available sands from two local sources were also studied. One, sand (a), was from Stradbroke Island. A micrograph of this material is shown in Fig. 2. This in its "as supplied" form was material 7 in the study. The sieve analysis for this sand was done according to BS 410-1986 and the results are shown in Fig. 3. Two of the sieve fractions of this

| No. | Material | Mean Dia. (µm) | Mean Shape Factor | Specific Surface Area (m²/g) | Apparent Density (kg/m³) | Tap Density (kg/m³) |
|-----|----------|---------------|-----------------|------------------------------|----------------------------|---------------------|
| 1   | Y-TZP monosized beads | 946 | 1.05 | 0.0010 | 3655 | 3656 |
| 2   | Y-TZP monosized beads | 294 | 1.06 | 0.0034 | 3581 | 3698 |
| 3   | Y-TZP monosized beads | 94  | 1.04 | 0.0106 | 3272 | 3752 |
| 4   | 60/40 wt% of 2/3 | 103 | 1.0 | 0.0096 | 3789 | 4128 |
| 5   | 70/30 wt% of 2/3 | 108 | 1.0 | 0.0092 | 3848 | 4096 |
| 6   | 66.4/24.5/9.1 wt% of 1/2/3 | 116 | 1.0 | 0.0085 | 4044 | 4136 |
| 7   | SiO₂ sand (a) (as supplied) | 290 | 1.48 | 0.0038 | 1412 | 1550 |
| 8   | 212-300 µm fraction of 7 | 250 | 1.19 | 0.0045 | 1432 | 1552 |
| 9   | 150-212 µm fraction of 7 | 190 | 1.16 | 0.0051 | 1382 | 1531 |
| 10  | SiO₂ sand (b) (as supplied) | 310 | 1.28 | 0.0036 | 1486 | 1613 |
| 11  | 212-300 µm fraction of 10 | 220 | 1.22 | 0.0051 | 1537 | 1651 |
| 12  | 150-212 µm fraction of 10 | 180 | 1.52 | 0.0063 | 1416 | 1568 |
| 13  | Y-TZP (SY-ULTRA) | 0.92 | 1.17 | 15.4 | 1132 | 1294 |
| 14  | Y-TZP (SYP-ULTRA) | 31.34 | 1.14 | 7.1 | 995 | 1137 |
| 15  | Y-TZP (TIOXIDE) | 28.38 | 1.09 | 19.9 | 885 | 1043 |
material were chosen as materials 8 and 9 for the study of particle size effects. It can be seen from Table 1 that these sub-samples were made up of particles of nearly spherical shape.

Fig. 1 SEM of Y-TZP Beads of Nominal Diameter 0.1 mm. The other bead materials had very similar morphology.

Fig. 2 SEM of Material 7 - Stradbroke Island Sand (As received).

Fig. 3 Sieve Analysis of Material 7 - both in the "as received" state and after pressing to 300 MPa.

The other sand (b) was from Moreton Bay which in its "as supplied" state is shown in Fig. 4. This was also subjected to a sieve analysis and the results are shown in Fig. 5. In its "as supplied" state and in sieved sub-samples, it formed materials 10, 11 and 12 in the study.

Commercial spray dried zirconia powders were also studied (materials 13, 14 and 15). These were all of yttria stabilized tetragonal zirconia polycrystal (Y-TZP). Like the bead material described above, they were also supplied by ICI Advanced Ceramics, Melbourne. Two of these (13 and 15) were without binders. The third (14) was manufactured with a proprietary inbuilt binder system. Optically, all had similar morphology consisting of agglomerates made up of sub-micron fundamental particles (Figs. 6a and b). However, agglomerate size and size distribution varied significantly between the spray dried powder samples, as shown by the size distributions given in Figs. 7a, 7b and 7c.

Fig. 4 SEM of Material 10 - Moreton Island Sand (As received).

Fig. 5 Sieve Analysis of Material 10 - both in the "as received" state and after pressing to 300 MPa.

2.2 Flow behaviour

The flow behaviour of the materials were experimentally measured for three different conditions.

(1) Free flow

(2) Shear under limited stress (interparticle friction)

(3) Die compaction (internal friction)
2.2.1 Free flow
Free flow behaviour and apparent density were measured using the Hall flowmeter according to standards ASTM B212 and B213. Tap density was found by repeated tapping of a known sample in a measuring cylinder. Prior to these tests, powders were oven dried and cooled in a desiccator. The tests were then conducted at prevailing atmospheric conditions within 15 minutes removal of the granular material from the desiccator. In Table 2 the Hausner ratio is tap/apparent density and the apparent and tap densities are shown in the form of relative densities. These were calculated using solid phase densities of 6046, 2682, and 2671 kg m\(^{-3}\) for the Y-TZP, sand (a) and sand (b) respectively.

2.2.2 Shear measurement
An annular shear cell was used to measure the flowability characteristics (angle of interparticle friction) of the bead and sand materials (materials 1-12 in Table 1). The annular cell could not be used with the finer spray dried powders because these materials worked their way up the clearance in between the annular ring and its housing leading to errors in the friction measurement. Instead, a direct (Jenike) cell was used with these powders.

The angle of internal friction was calculated using the Mohr-Coulomb yield criterion which can be written 12),

\[
\tau = \sigma \tan \theta + c
\]  

(1)
**TABLE 2** - FRICTIONAL CHARACTERISTICS

| No. | Material                  | Flow Time (sec) | μ Shear Cell | μ Die Press | Hausner ratio | Relative Density (%) |
|-----|---------------------------|-----------------|--------------|-------------|---------------|----------------------|
| 1   | Y-TZP monosized beads     | 30.2            | 0.24         | 0.47        | 1.00          | 60.5                 |
| 2   | Y-TZP monosized beads     | 18.9            | 0.31         | 0.58        | 1.01          | 59.2                 |
| 3   | Y-TZP monosized beads     | 16.4            | 0.65         | 0.70        | 1.15          | 54.1                 |
| 4   | 60/40 wt % of 2/3         | 14.7            | 0.40         | 0.55        | 1.09          | 62.7                 |
| 5   | 70/30 wt % of 2/3         | 15.3            | 0.30         | 0.64        | 1.06          | 63.6                 |
| 6   | 66.4/24.5/9.1 wt % of 1/2/3| 21.9            | 0.29         | 0.47        | 1.02          | 66.9                 |
| 7   | SiO₂ sand (a) (as rec’d)  | 59.6            | 0.58         | 0.60        | 1.10          | 52.6                 |
| 8   | 212-300 μm fraction of 7  | 60.0            | 0.58         | 0.55        | 1.08          | 53.4                 |
| 9   | 150-212 μm fraction of 7  | 57.1            | 0.58         | 0.60        | 1.11          | 51.5                 |
| 10  | SiO₂ sand (b) (as supplied) | 58.5            | 0.58         | 0.63        | 1.09          | 55.6                 |
| 11  | 212-300 μm fraction of 10 | 57.7            | 0.55         | 0.56        | 1.07          | 57.5                 |
| 12  | 150-212 μm fraction of 10 | 56.7            | 0.58         | 0.55        | 1.11          | 53.0                 |
| 13  | Y-TZP (SY-ULTRA)          | 0.54            | 0.67         | 1.14        | 18.7          | 21.4                 |
| 14  | Y-TZP (SYP-ULTRA)         | 0.72            | 0.66         | 1.14        | 16.5          | 18.8                 |
| 15  | Y-TZP (TIOXIDE)           | 0.41            | 0.64         | 1.18        | 14.6          | 17.3                 |

where $\tau$ is the shear stress, $\sigma$, the normal stress, $\theta$, the angle of interparticle friction and $c$, the cohesion. The coefficient of internal friction is given by $\tan \theta = \mu$. The granular materials considered in this work were cohesionless, and so the value of $c$ was therefore zero.

The standard annular cell (Wykeham Furrance Engineering Ltd, type WF-25850) used for the bead and sand measurements has a normal stress capacity of 1.4 MPa. To accommodate testing to higher normal stresses, the load capacity of the cell was increased to 4.7 MPa by reducing the width of annular cross-section, from 15 to 5 mm. This modification also gave the added advantage that smaller samples were needed for a test. It did however lead to an increase in the ratio of wall to powder body shear forces, but for the materials tested, the grain-wall friction was small and, with the exception of the largest beads, the particle sizes were small compared to the annular width. It has been shown that negligible error was introduced by this modification of the annular shear cell.

In the interest of repeatability, it was important to standardize initial density of the test specimens charged in the cell. With like materials, this was achieved in practice with a charging accuracy of 3%. During experiments, all the specimens were subjected to a constant strain rate of 5 mm per minute. For each material, the limiting shear stresses at progressively higher applied normal stresses were plotted in the form of Mohr’s stress diagrams. The coefficient of internal friction ($\mu$) and cohesion ($c$) were obtained from equation 1 applied to the line drawn tangential to the series of Mohr’s stress diagrams.

### 2.2.3 Die compaction

The quasi-static compaction experiments were done in the 25 mm diameter instrumented punch and cup die arrangement shown schematically in Fig. 8.
Following Kuhn 13), the die ring was designed to remain elastic for radial stresses up to 300 MPa. It was made from EN 26 steel hardened to 62 Rc and ground in the longitudinal direction in order to minimise die-wall friction. A strain gauge was mounted circumferentially on the die-ring at an appropriate location to measure strain during die calibration and granular material testing. Further details are given in 14).

Two sets of punches were used, one for calibration and the other for powder compaction experiments. For given depths of material fill in the die, the output from the strain—gauge attached to the die ring was calibrated using oil in the die loaded to known pressures by means of the top punch. Die wall friction was minimized in the loading experiments with granular materials by only using depths of loaded material smaller than that necessary to produce measurable die wall friction. Loading of the top punch was affected using an ‘Instron’ hydraulic testing machine. The accuracy of Instron was 0.2% of full range, corresponding to a die pressure of 1000 MPa.

For testing, a 20 g sample of the granular material was charged in the die and the fill height measured. It was then compacted with a punch displacement rate of 50 mm per minute on an Instron testing machine. The experimental measurements were recorded on an x-y recorder, in the form of a load-strain plot that were subsequently converted to axial-radial stress plots using the die ring strain-gauge calibrations.

The experimental measurements were recorded on an x-y recorder, in the form of a load-strain plot that were subsequently converted to axial-radial stress plots using the die ring strain-gauge calibrations. The frictional characteristics of all materials studied are summarized in Table 2. The major consolidation stress for the shear cell experiments quoted was 0.4 MPa and for the die press experiments, 300 MPa. Flow time measurements were not possible with the spray dried powders. Even after careful drying, these powders were so highly frictional under gravity loading conditions that they would not flow reliably through the standard funnel used for this test.

### 3.1 Particle size effects

The results for the monosized beads provided frictional data in which only particle size was the variable. Comparing the frictional characteristics of the monosized beads (Table 3), two important observations can be made—a) there is in fact a negative correlation between flow time and shear cell measures of internal friction and b) there is a marked increase in internal friction with decreasing particle size. The first observation is important—flow time is commonly used in powder metallurgy and other industries to measures frictional behaviour. With the relatively coarse bead particles used in this part of the study, this measure clearly leads to serious errors in the quantification of internal friction. The second observation, although not new, quantifies the effect very clearly for materials where the only difference between them is particle size—material and geometry are exactly the same. The higher friction with smaller particle size was manifested by higher coefficients of internal friction as measured in both the shear cell and die press (equation 2) experiments, although the absolute values obtained from the die press experiments were higher. Higher friction was also indicated by lower apparent density.

For these materials, the tap density changed little with particle size, but because of the lower values for apparent density for the more frictional small

| No. | Material          | Mean Dia. (μm) | Flow Time (sec) | µ (Shear Cell) | µ (Die Press) | Hausner ratio | Relative Density (%) |
|-----|-------------------|----------------|-----------------|---------------|--------------|---------------|---------------------|
| 1   | Y-TZP monosized   | 946            | 30.2            | 0.24          | 0.47         | 1.00          | 60.5                | Tap                |
| 2   | Y-TZP monosized   | 294            | 18.9            | 0.31          | 0.58         | 1.01          | 59.2                | Tap                |
| 3   | Y-TZP monosized   | 94             | 16.4            | 0.65          | 0.70         | 1.15          | 54.1                | Tap                |
sizes; the Hausner ratio was higher for these materials too.

The blends of the monosized beads (Table 4) had friction coefficients somewhere between the extremes associated with the monosized components. Of particular note is the result for material 6, the blend of these 3 component sizes producing the highest possible bulk density. The friction coefficient for this material is only marginally higher than that for material 1, the largest sized component which also had the lowest friction coefficient. This suggests that in appropriately blended size ranges, the frictional behaviour is dominated by the largest particle size present.

### 3.2 Particle geometry effects

The sand based materials (7-12 in Tables 1 and 2) were more irregular in shape than the beads as can be seen from Fig. 1, 2 and 4. As naturally occurring materials they also comprised a blend of sizes. The shape factors listed in Table 1 and the size distributions shown in Fig. 3 and 5 quantify these effects.

The friction coefficients determined in the shear cell for this range of materials are, with one exception, all higher than the monosized or blended beads. Since the size range is similar with the beads, and since there was little difference between any of the sieved sub-samples (materials 8, 9, 11 and 12) and as received materials (7 and 10), it would be reasonable to conclude that the higher friction observed in the sand based materials was largely a consequence of their more irregular shape. Comparing materials 2, 8 and 11 (Table 5), they are all of similar size, but of different shape factor. Flow time, shear cell internal friction and Hausner ratio all indicate higher friction in the more irregularly shaped material.

Specific surface area (ssa) is a mass-based measure. However, the difference in density between the materials studied only varied by a factor of about 2. Even allowing for this density difference, the specific surface area of the spray dried powders (materials 13, 14 and 15) was orders of magnitude higher than that for any other material tested. The friction results for the spray dried powders are compared with other typical values in Table 6. Material 9 was chosen as a comparison material on the basis that its shape factor was similar (Table 1). Unfortunately, it was a coarser powder than the spray dried materials. Nevertheless, allowing for the larger size and the different density material, the coefficients of internal

| TABLE 4 | SIZE EFFECTS - BLENDS OF MATERIALS 1, 2, 3 |
| --- | --- | --- | --- | --- | --- | --- | --- |
| No. | Material | Mean Dia. (μm) | Flow Time (sec) | μ Shear Cell | μ Die Press | Hausner ratio | Relative Density (%) |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 4 | 60/40 wt % of 2/3 | 103 | 14.7 | 0.40 | 0.55 | 1.09 | 62.7 | 68.3 |
| 5 | 70/30 wt % of 2/3 | 108 | 15.3 | 0.30 | 0.64 | 1.06 | 63.6 | 67.7 |
| 6 | 66.4/24.5/9.1 wt % of 1/2/3 | 116 | 21.9 | 0.29 | 0.47 | 1.02 | 66.9 | 68.4 |

| TABLE 5 | SHAPE FACTOR EFFECTS : 212-300μm PARTICLES |
| --- | --- | --- | --- | --- | --- | --- | --- |
| No. | Material | Mean Shape Factor | Flow Time (sec) | μ Shear Cell | μ Die Press | Hausner ratio | Relative Density (%) |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 2 | Y-TZP monosized beads | 1.06 | 18.9 | 0.31 | 0.58 | 1.01 | 59.2 | 59.7 |
| 8 | 212-300μm fraction of 7 | 1.19 | 60.0 | 0.58 | 0.55 | 1.08 | 53.4 | 57.9 |
| 11 | 212-300μm fraction of 10 | 1.22 | 57.7 | 0.55 | 0.56 | 1.07 | 57.5 | 61.8 |

| TABLE 6 | SPECIFIC SURFACE AREA (SSA) EFFECTS |
| --- | --- | --- | --- | --- | --- | --- | --- |
| No. | Material | Mean Dia. (μm) | SSA (m²/g) | μ Shear Cell | μ Die Press | Hausner ratio | Relative Density (%) |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 9 | 150-212μm fraction of 7 | 190 | 0.0051 | 0.58 | 0.60 | 1.11 | 51.5 | 57.1 |
| 14 | Y-TZP (SYP-ULTRA) | 31.3 | 7.1 | 0.72 | 0.66 | 1.14 | 16.5 | 18.8 |
| 15 | Y-TZP (TIOXIDE) | 28.3 | 19.9 | 0.41 | 0.64 | 1.18 | 14.6 | 17.3 |
friction were comparable for the materials shown. On the basis of both size and higher ssa, materials 14 and 15 would have been expected to have higher internal friction, but this was not so. The Hausner ratio figures also do not discriminate between these materials. However, the raw figures for both apparent and tap density are much lower for the spray dried material. This suggests that these measures are poor indicators of the frictional behaviour of these materials when under higher than gravity loads.

3.3 Loading effects
The results for the coefficient of internal friction for selected samples are shown in Table 7 at two values of the major consolidation stress. The first (395 kPa) was achieved in the shear cell experiments whereas the second (300 MPa) was achieved in the die pressing experiments. The results for the monosized beads showed effects that were typical of the range of materials tested. A significant fraction of the large beads (material 1) fractured at the higher load. This meant that there was partial loss of the spherical shape throughout the powder body which lead to a significantly higher friction coefficient at the higher loads. In contrast, the small beads (material 3) were stronger and did not fracture at applied pressures up to 300 MPa. It can be seen that the friction coefficient did not change much between the two load conditions.

With the sand materials, in most cases there was a slight increase in friction with increasing load, but the striking feature is that there is really very little change. The results for material 7 shown in Table 6 were typical of this type of material. This result was obtained despite measurable particle fracture in the diepressing experiments, as can be seen from the "before and after" sieve analyses shown in figures 3 and 5. However for this class of material, the fracture debris was similar in shape to the original material.

In contrast, there was much greater fracture in the spray dried powders at the high loads experienced in the die pressing experiments. The results for these materials (13, 14 and 15) in Table 7 show a marked change in the friction between the shear cell and die pressing experiments. There was significantly different friction between the powder samples in the shear cell results, but very little in die pressing results. Bearing in mind the large difference in major consolidation stresses between these measurement techniques, this difference in frictional behaviour is consistent with substantial particle fracture. The large agglomerates would have dominated frictional behaviour at the lower stresses present in the shear cell, in contrast to the die experiments at high loads where the frictional behaviour would have been dominated by the debris from the broken agglomerates. This debris would be typified by the basic crystallite size (~ 0.6 μm), virtually the same for all of 13, 14 and 15 materials. This mechanism is supported by the constancy of friction coefficient across all spray dried materials, despite the large difference in this property between these materials at the lower testing load.

4. Conclusions
At least for coarse granular materials, flow time measures of frictional characteristics give a poor indication of the coefficient of internal friction.

In mono-sized granular material, the internal friction increases with decreasing particle size. In granular materials blended with respect to component particle sizes, the internal friction lies within the range set by the lowest and highest for the component sizes used. With appropriate size blending, a high bulk density can be achieved with little increase in the internal friction over that due to the size component with the lowest friction used. Since in the present work these were the largest particles, the results suggest that, in such blends, the frictional behaviour is controlled by the largest particles present.

Other things being comparable, granular bodies with particles having higher shape factors also have higher internal friction. Powders with high specific surface area were found to have low apparent and tap densities, but more direct measures of frictional behaviour indicated that the specific surface area had negligible effect on the coefficient of internal friction.
At least for hard ceramic granular materials, measurements of internal friction in a shear cell at consolidation stresses of 0.4 MPa give a good indication internal friction during rigid die pressing to consolidation stresses at least up to 300 MPa. This is so even when particle fracture occurs during pressing, provided particle morphology does not change significantly as a result of the fracture.

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