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Measuring shear properties and normal stresses generated within a rotational shear cell for consolidated and non-consolidated powders

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ABSTRACT

Powder behaviour and particularly the measurement and prediction of flow properties remains of great importance to industry. Analytical studies over many decades have increased understanding, but it is modern instrumentation, sometimes using empirical methodologies that is providing new means of characterising powders and better understanding of the elements of powder processing. Progress is rapid and every study reveals the potential for further research.

This work uses a rotational shear cell to evaluate five very different powders from a large particle ceramic (bi-modal size distribution: 60 and 170 μm) to 4 μm limestone. The focus of the study was firstly to determine the yield loci of all materials after 9 kPa pre-consolidation and then to use a different shear cell control methodology to investigate the stresses automatically generated normal to the shear plane, as shearing proceeds.

Non-consolidated or unstressed powders are of special interest and shear tests were done at close to zero consolidation stress, including the measurement of generated normal stress. The ratio of this generated normal stress to applied shear stress was derived for a wide range of pre-shear conditions and the results suggest that this parameter is differentiating and useful. Values for the coarse and the fine powders varied by a factor of about 2 and up to 3 when the powders were non-consolidated.

1. Introduction

The characterisation of the flowability of powders is important for process control and the design of powder processing/handling devices. Powder characteristics are determined by their physical properties such as particle size, distribution and shape and also by many environmental factors such as temperature, humidity and the stresses applied during storage and processing, such as gravity, vibrations, air pressure, and external loading from filling devices.

Powders are complex, not least because a given mass of powder can contain variable amounts of air which can radically change its rheology or flow properties, perhaps by a factor of 100 or even 1000. The initial state of the powder prior to testing is therefore particularly important and so 'conditioning' to ensure a reproducible packing condition is essential if the data is to be both reproducible and repeatable [1]. Traditional test methods [2–5] often lack an initial conditioning stage which removes the powder's history and operator variability.

This study focuses primarily, on shear testing and the behaviour of consolidated and non-consolidated powders. The study of very lightly stressed powders with shear cells is relatively new and the work follows on from an earlier study [1] where measurements close to zero normal consolidation stress were made.

The objective of this work was to present shear test results focusing on the generated normal stresses measured during shearing at both high and very low consolidation stresses. For the evaluation of a range of different materials a powder rheometer [1] was used for pre-conditioning, rotational shear testing and the measurement of compression and dynamic flow properties.

2. Materials

The materials characterised in this study have very different physical properties as shown in Table 1.

3. Method

For the evaluation of a range of different materials a powder rheometer (FT4, from Freeman Technology) was used for (1) pre-conditioning, (2) rotational shear testing (3) dynamic testing and (4) compressibility testing. Dynamic testing and conditioning used a 48 mm diameter blade and a powder sample contained in a 50 mm bore, borosilicate test vessel. An automated, 18 segment, 48 mm diameter rotational shear cell accessory was used for all shear testing using a 85 ml sample. The most important test stages are described below; for more details please refer to [1].

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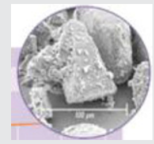
Table 1
Material properties

Measurements	Ceramic	Spray dried lactose	Coarsely milled lactose	Finely milled lactose	Limestone CRM116
D_{50} particle size (μm)	150*	130	100	20	4
Particle/granule shape	Spherical	Spherical	Angular	Angular	Angular
Conditioned Bulk Density (g/ml)	1.20	0.64	0.75	0.46	0.74
Materials morphology					

*A bi-modal distribution with one peak at 60 μm and the second at 170 μm.

3.1. Conditioning — how to prepare a powder for evaluation?

It is essential to produce a standardised packing condition as a preliminary to each test cycle. Fig. 1 shows this conditioning process which comprises of a traverse of a blade downward and then a traverse upward. During this traverse the blade gently lifts the powder and drops it behind the blade, each particle coming to rest behind it. Conditioning involves gentle displacement of the whole powder sample in order to loosen and slightly aerate the powder. This process removes any packing history such as pre-consolidation or excess air.

3.2. Shearing

After pre-conditioning, the material is tested without any further handling, with the rotational shear cell operating in the force control mode, see Fig. 2. As the shear cell is rotated the torque (shear stress) is measured whilst the force (normal stress) is kept constant. Therefore, during shearing the shear head can displace vertically to keep the normal stress constant if the sample dilates or compresses. This is the normal measuring mode as used to obtain the Section 4.1 data. The shear cell can also be operated in height control mode in which the vertical position of the shear head is fixed. This mode was used to obtain the generated normal stress data presented in Sections 4.2 and 4.3.

4. Results and discussions

4.1. Measuring yield loci relative to 9 kPa pre-consolidation normal stress

Fig. 3 shows the yield loci for all five powders after pre-shearing at an arbitrary 9 kPa pre-consolidation (PC) normal stress. The FT4 instrument was operated in automated force control mode to allow individual shear tests to be done with applied normal stresses over the range from 80% down to 5% of the PC stress. The 9 kPa pre-consolidation and pre-shear was applied before each test.

For coarse, non-cohesive powders the yield loci are near straight lines passing close to zero, indicating minimal cohesion and unconfined yield strength (UYS) values. However, the fine powders exhibit much greater shear strength and significant cohesion values (*Y* axis intercept) even though the yield locus typically becomes more convex below about 30% resulting in reduced values of cohesion and UYS. This convexity possibly reflects the much greater compressibility, by a factor of 10, of the fine powders in relation to the coarse, see Fig. 4. It may also be explained by the recovery from pre-consolidation at the lower levels of applied normal stress. Another possible influence is the effect of generated pressure (see Section 4.2) that is significant at these lower normal stresses, though it is not clear how it might reduce the shear stress values. What can be said with confidence is that the cohesive powders become different powders when their air content is changed by consolidation so it is unsurprising that the yield loci is not a straight line.

Table 2 shows the various measured parameters including the UYS derived from a Mohr Circle analysis. Apart from the internal angle of friction, all values are lower than in an earlier study [1] on account of the data points extending down to 0.5 kPa normal stress. The current cohesion value for limestone of 0.66 kPa is considered more accurate than the 1.05 figure from the earlier study where the data was extrapolated from 3 kPa normal stress.

Fig. 1. Photo of the end of a conditioning traverse showing the blade moving upwards out of the powder.

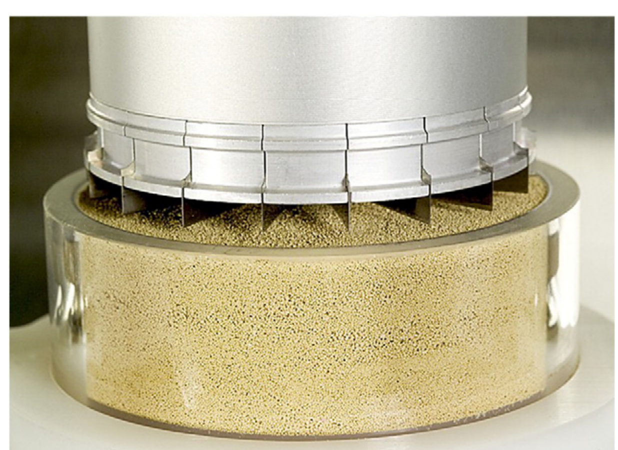


Fig. 2. Photo showing glass vessel and shear head with thin blades before moving into the powder.