Flow properties of ductile ceramic granules – Challenges in characterization

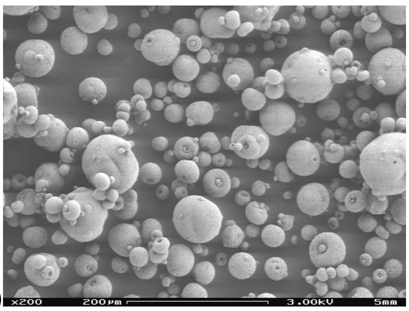
B. Lang[1], M. Schäfer[2], D. Höhne[2], M. Nebelung[1]
[1]Fraunhofer-Institute for Ceramic Technologies and Systems Dresden, [2]TU Bergakademie Freiberg

1. Introduction

Flow behavior is a very important property of granules during different processing steps, e.g. handling during storage and transport as well as the homogeneity of the die filling in compaction process. In the latter case, inadequate material properties of the final compartment (e.g. compact) can result, if the flow behavior does not meet the requirements. Thus, characterization of the flow behaviour is important. Although, a lot of research has been done in the field of flow behaviour of primary particle systems and the basic principles [2-6], there are only a few papers focusing on the field of ceramic granule systems. The aim of this paper is to investigate the flow behaviour of such granules, selectivity for ductile granule systems with good flowability. In this context one important premise is to analyse the applicability of different commercial measurement methods (shear tester). Moreover, challenges in characterization of the granules will be shown and discussed as well.

2. Test method and material

Flowability experiments were carried out with different shear cell devices: Schulze ring shear tester RST-01.01, modified Peschl RO-200 with ring shear cell and FT4 Powder Rheometer torsional shear cell with different diameters (25mm and 50mm). The mentioned devices and the measurement procedures are well described in literature and standards [1;4]. Measurements under five different consolidation stresses with values of 1.4 kPa, 5.4 kPa, 10.2 kPa and 20.4 kPa had been carried out. For two of the consolidation stresses ten measurements were done each testing the reproducibility and for the other stresses three measurements.

(1) 

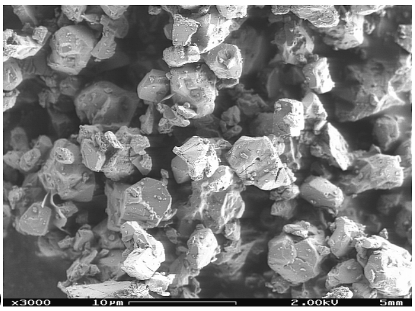
(2) 

Fig. 1: (1) Al_2O_3-Granules and (2) Limestone CRM-116 (FESEM)

For applicability tests of the different shear cell devices a commercial, free flowing Al_2O_3 granule system was used as test material (Fig. 1 and Table 1). Reference material was Limestone (CRM-116).

For the comparison of the flow behaviour of primary particle systems with granular system two further systems (TiO_2 – granules and Al_2O_3 primary particle) are analyzed as well (Table 1).

Table 1: Properties of the analyzed test materials

	Al_2O_3 Granules	Limestone (CRM-116)	TiO_2 - Granules	Al_2O_3 Primary Particles
granule or particle size $x_{10}/x_{50}/x_{90}$ [µm]	31/66/120	0.7/3.7/6.9	63/125/500	487/707/989

mechanical strength σ_B [MPa]	1.97 MPa	- [1)]	0.04 MPa	- [1)]

[1)] primary material with high mechanical strength

3. Results

Comparative measurements

A comparison of the yield loci (σ-τ-points) for measurements with the different shear cell devices is shown in Fig. 3.

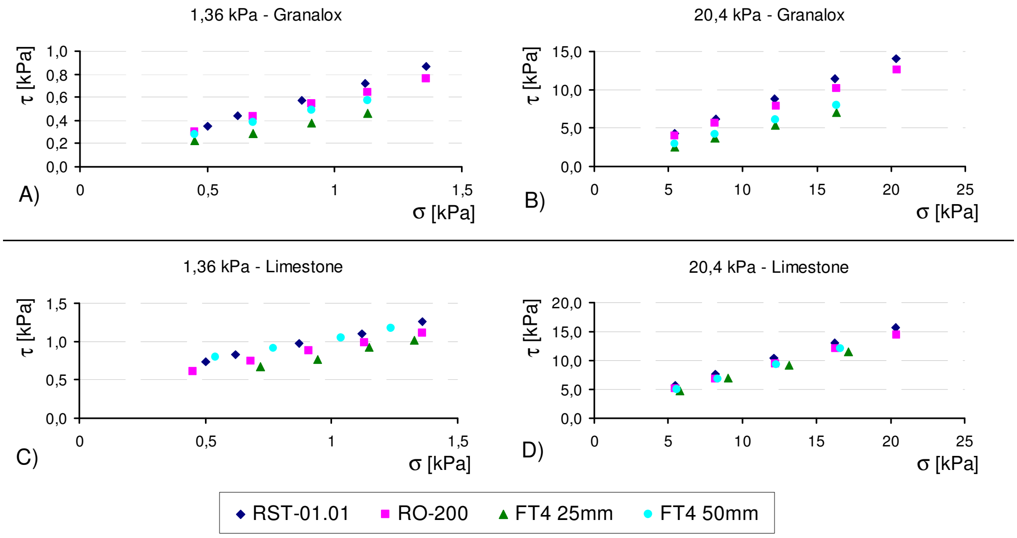


Fig. 3: Comparison of yield loci for different shear cell devices, consolidation stresses and material systems

The results of measuring Limestone with the different shear devices under high load are similar (D). More differences for low loads could be detected (C). At both levels the lowest values are measured with the FT4 25 mm shear cell. In the case of the Al_2O_3 granules (A and B) there much bigger differences had been obtained, with the lowest values for both FT4 shear cell devices. This could be due to the difference: ring shear cell – torsional shear cell. The differences between the ring shear devices (RST-01.01 and RO-200) could be caused by dissimilarity in lid geometry.

By extrapolating the measured σ-τ-points (yield loci) to the x-axis the cohesion τ_c as a flowability value is received. One basis of the variation of this value the reproducibility of the measurements was analyzed. The results for the lowest and highest consolidation stress for all shear devices are shown in Fig. 2. For the free flowing granule system the reproducibility is worse than for the Limestone powder. This is true for all shear devices and consolidation stresses. Especially in the case of low stress strong fluctuations of the flowability value could be detected. At low stresses the random variations are big relative to the measured value. This and the necessary extrapolation especially in the case of good flowing materials could lead to a high fluctuation of the cohesion and other flowability values.

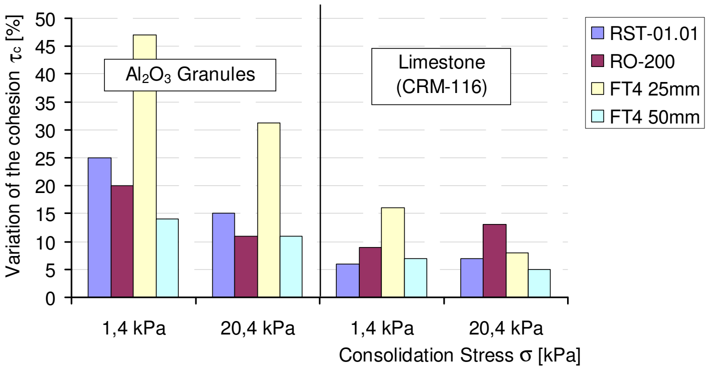


Fig. 2: Variation of the cohesion τ_c for the measurements with different shear cell devices (1.4 kPa & 20.4 kPa consolidation stress; 10 measurements each)

Mechanical properties: Influence on flowability

In Fig. 4 the flow function measured for the analysed systems are plotted. In the case of granules there is a progressive σ_1/σ_c-trend visible, i.e. a decline of flowability with increasing load. In comparison, the most primary particle systems show an improve of flowability [4]. This behaviour is also visible for the analysed Limestone (CRM-116) and the Al_2O_3 primary particle system (Fig. 4).

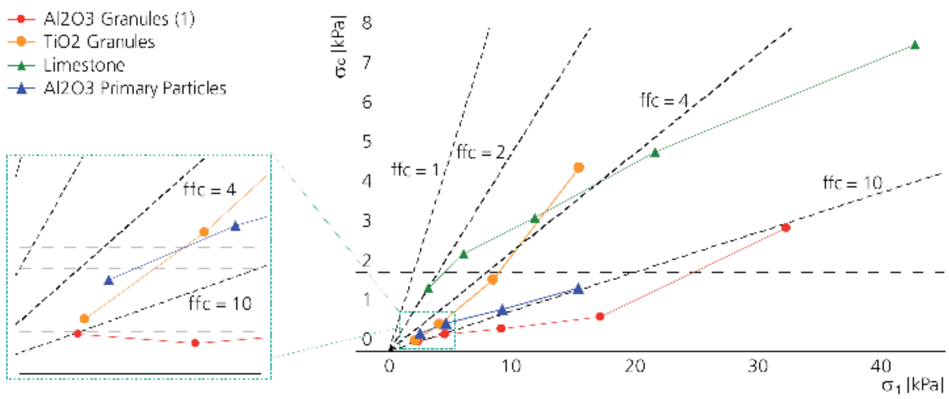


Fig. 4: Flow function for Al_2O_3 granules, TiO_2 granules, Limestone and Al_2O_3 primary particles (RST-01.01)

The reason for the progressive σ_1/σ_c-trend is originated in the mechanical properties of the granules. Because of the structure of the single granule, which consists of small primary particles, pores and organic components, they show a ductile mechanical behavior. This can cause granule deformation, destruction and attrition in dependence of the kind and the value of the load. Thereby, the mechanical strength σ_B of the single granules plays a decisive role - the weaker the granules the greater the extent. On the basis of TiO_2 (σ_B = 0.04 MPa) and Al_2O_3 Granules (σ_B = 1.97 MPa) this is illustrated in Fig. 4. While both show similar flow behavior at low stress, they differ significantly at higher stress. For the soft TiO_2 granules the flowability decreases even at middle stress and the destruction of the granules in the shear zone was optical visible after measurement. In the case of the Al_2O_3 granules an improve of the flowability in the first part of the flow function is visible, which is similar to primary particle systems. At the highest stress a decline occurs - this could be an indication for a possible deformation or destruction of the granules.

4. Discussion

80% of spray dried, ceramic granules can be classified as good or free flowing. Since it is also necessary to distinguish between such good flowing granules a good reproducibility of the flow measurements is important. As it was shown, there is a large variation of the results for low stresses for systems with good flowability, getting better the higher the load.
Due to the mechanical properties of ceramic granules high loads can result in a deformation or destruction of the single granules. In that way the flowability will be influenced negatively. Because the granules are mechanically stressed during many processes (storage and transportation) the handling of granules is carried out under low loads. Analysing the flowability under high loads (better reproducibility) would therefore mean measuring under not process relevant conditions. Besides this could result in measurement values which account a too worse flowability for the system (depending on the mechanical strength of the granules).

5. Conclusion

Extensive experiments with different shear cell devices and test material delivered following findings:

- The cohesive Limestone gives reproducible results for all configurations with only a few restrictions.
- Testing granule systems with good flow properties show high variations of the flowability values especially in the case of low stress.
- Differences in the results of the shear cell devices are shown, with a tendency to lower shear stresses for the FT4 torsion shear cell.
- With increasing load the ductile, ceramic granules could be deformed or destroyed and the flowability decreases significantly, showing a progressive σ_1/σ_c-trend of the flow function.

As discussed, it is not reasonable measuring the flow behavior under high loads because of no process relevance and possible deformation/destruction of the single granules. Therefore the focus for further investigation is on the characterization under small loads. In this context high sensible methods for the characterization are necessary.

7. Reference List

[1] ASTM D 6773, Standard Shear Test Method for Bulk Solids Using the Schulze Ring Shear Tester. 2000

[2] R. Freeman, Measuring the flow properties of consolidated, conditioned and aerated powders -- A comparative study using a powder rheometer and a rotational shear cell. Powder Technology 174 (2007) 25-33.

[3] O. Molerus, Schüttgutmechanik, Springer-Verlag, 1985

[4] D. Schulze, Pulver und Schüttgüter: Fließeigenschaften und Handhabung, Springer-Verlag, Berlin; Heidelberg, 2006

[5] D. Schulze, A. Wittmaier, Messung der Fließeigenschaften hochdisperser Schüttgüter bei sehr kleinen Verfestigungsspannungen. Chemie Ingenieur Technik 74 (2002) 1144-1148

[6] J. Tomas, The mechanics of dry, cohesive powders. powder handling&processing 15 (2003) 296-314