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The Effect of Temperature on Flow Properties of Fine Powders

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Changes of cohesive flow properties of powders at high temperature are observed in many industrial applications, such as fluidized bed reactors, in the filtering of hot gases, granulators and dryers. In this work a modified annular Schulze shear cell was used to measure flow properties of a FCC powder, a corundum powder and fly-ashes between room temperature and 500°C.

1. Introduction

Many works in literature investigated the effect of the increasing temperature on powders in fluidization conditions (Formisani *et al.*, 2002) but very few studies concern with the analysis of the powder flow properties at high temperature by means of conventional testers and procedures commonly employed at ambient conditions, i.e. uniaxial tester and shear cell. However, the effect of the temperature on powder flow properties has been often interpreted by assuming the presence of softening and sintering phenomena of the particles affecting the interparticle forces (Kamiya *et al.*, 2002).

2. Experimental

2.1 High Temperature Annular Shear Cell

In this work, a standard annular Schulze shear cell was modified in order to measure powder flow properties at high temperature. Figure 1 shows a schematic of the new

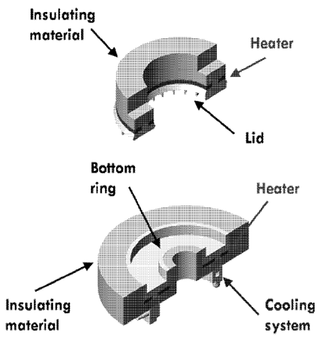

Figure 1: Schematic of the High Temperature Annular Shear Cell

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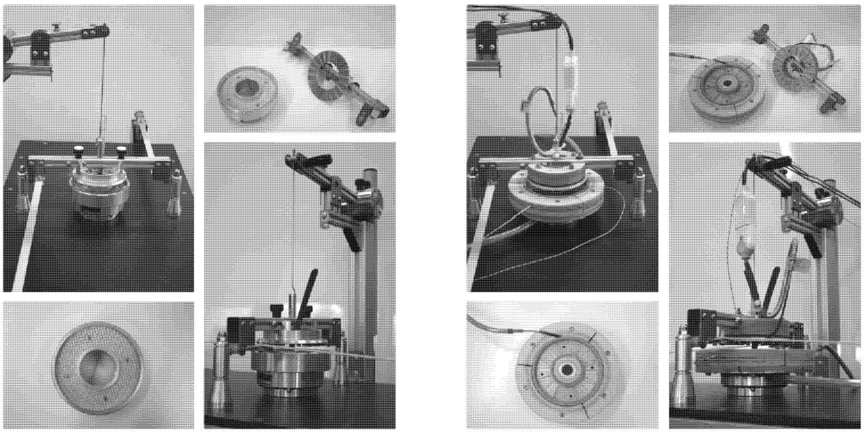

Figure 2: The commercial SV10 Schulze shear cell (on the left) and the modified High Temperature Annular Shear Cell (on the right)

High Temperature Annular Shear Cell. It consists of a bottom annular trough containing the powder sample and an annular lid placed on the top of the sample. The lid is fixed at a crossbeam connected by two tie-rods to two load beams. These allow to measure the shear force acting on the shear plane inside the powder sample by the rotation of the bottom ring relative to the lid for a specified normal load, exerted by weight pieces placed on a hanger connected to the crossbeam.
To heat up the cell and the powder sample contained in it, electric heaters were introduced below the cell bottom and on the lid. In order to minimize the temperature gradient within the sample and for a safe operation of the cell, a covering insulating material was placed around the cell. A cooling system was designed to cool the cell base where it is in contact with the gears of the rotation mechanism. Water is used as cooling medium, flowing inside a cavity in the base of the cell. Figures 2 shows some photos of the modified High Temperature Annular Shear Cell and of the commercial one. The main dimensions of the bottom ring and of the lid are reported in Table 1 together with

Table 1: Data of the High Temperature Annular Shear Cell

Bottom Ring:	
inner diameter	60 mm
outer diameter	120 mm
nominal height	10 mm
internal volume	95.08 mm
Lid:	
inner diameter	62 mm
outer diameter	118 mm
Power supplier:	
lower heater	600 W
upper heater	260 W

Table 2: Size distribution parameters of experimental materials

	FCC powder	Corundum	Fly-ashes
d_{10} (μm)	37.1	54.8	3.5
d_{50} (μm)	72.8	83.6	27.2
d_{90} (μm)	134.1	127.0	123.1
d_{SV} (μm)	41.4	79.3	7.5

the heaters power. Finally, a temperature control system was developed to achieve a constant temperature in the powder sample.

2.2 Materials

Measurements of the flow properties were carried out with a FCC powder, fly-ashes and a corundum powder. For this latter Formisani *et al.* (2002) found change of the porosity with the temperature both in fixed and fluidized beds. According to these findings these changes are not predictable taking into account only the hydrodynamic properties. The parameters of the particle size distribution of the experimental materials are listed in Table 2.

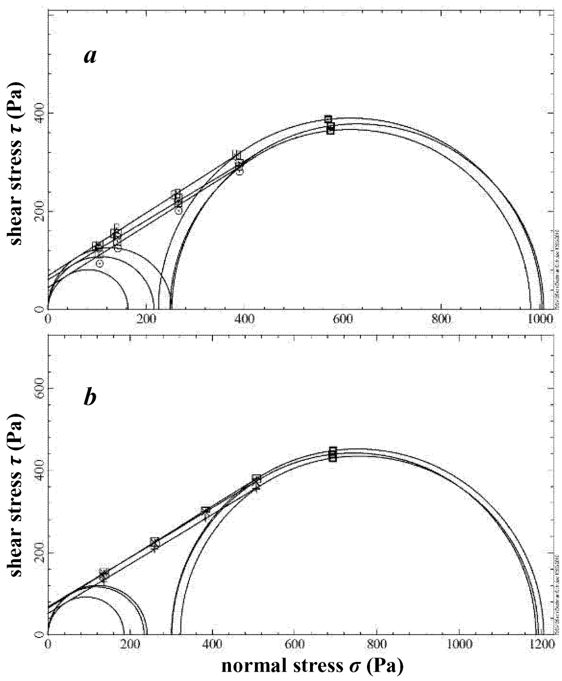

Figure 3: Measured yield loci for FCC powder:

a. test at a normal consolidation stress about 1000 Pa at 20°C (○), 250°C (□) and 500°C (▯)

b. test at a normal consolidation stress about 1200 Pa at 20°C (▭), 250°C (+) and 500°C (×)

2.3 Procedure

The experimental procedure applied mainly followed the standard procedure for shear tests with the annular Schulze shear cell. In this case, samples were pre-treated in an oven at 200°C to remove the volatile components (i.e. moisture content). In order to achieve the desired operating temperature before starting the shear test, some time was waited to let the temperature reach the desired value.

3. Experimental Results

In order to evaluate the equivalence of the measurements performed by the modified cell and conventional testers, a comparison between yield loci of FCC powders measured by the High Temperature Annular Shear Cell and the standard Schulze Ring shear Tester SV10 cell was performed at 20°C for two specified normal load. A good agreement between the experimental results was found, confirming the correspondence of the measurements carried out by the modified High Temperature Annular Shear Cell and the original SV10 Shear Cell (Tomasetta *et al.*, 2010)

To highlight the effect of temperature on the powder flow properties yield loci for certain consolidation loads were evaluated at different temperatures. All the experimental condition tested are listed in Table 3 together with the main parameters describing the flow properties.

Figure 3 reports the yield loci of the FCC powder measured at 20°C, 250°C and 500°C

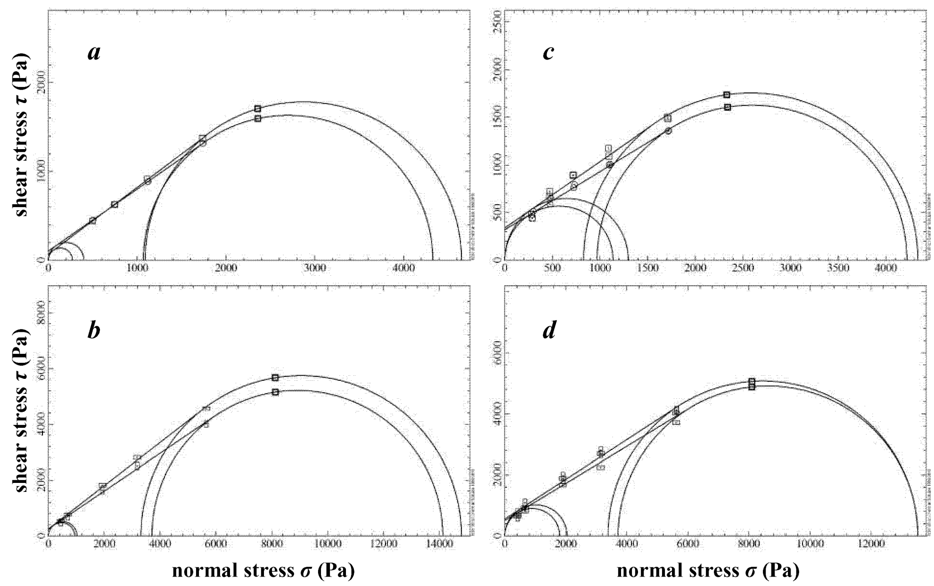

Figure 4: Measured yield loci:

- *for corundum powder at a normal consolidation stress about 4000 Pa (a) at 20°C* (○) *and 500°C* (□); *about 14000 Pa (b) at 20°C* (▯) *and 500°C* (▭)
- *for fly ashes at a normal consolidation stress about 4000 Pa (c) at 20°C* (○) *and 500°C* (□); *about 14000 Pa (d) at 20°C* (▯) *and 500°C* (▭)

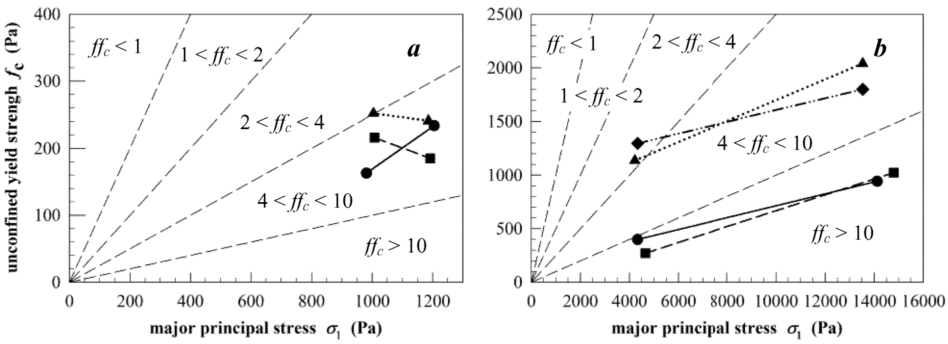

Figure 5: Measured flow functions:

a. for FCC powder at 20°C (●, solid line), 250°C (■, dashed line) and 500°C (▲, dotted line)

b. for corundum powder at 20°C (●, solid line), 500°C (■, dashed line) and fly ashes at 20°C (▲, dotted line) and 500°C (◆, dash-dotted line)

for major principal stresses of about 1000 Pa and 1200 Pa 3. Measurements performed at the lower consolidation levels do not appear fully reliable. This is probably due to the contribution of the forces exerted on the cell lid by the electric wiring of the lid heater. These contributions are difficult to be completely neutralized or accurately accounted for and have a non negligible effect on the points of the yield loci at low σ values. For this reason, subsequent tests were performed at higher consolidation levels. Furthermore, inspection of Figure 3, does not show a significant change of the flow properties of FCC powder with the increasing temperature, as confirmed by the estimated flow functions showed in Figure 5a.

Figure 4 reported the measurements carried out for corundum and fly-ashes at higher consolidation loads about 4000 Pa and 14000 Pa. Also in this cases, the flow properties of powder samples do not show significant change with the temperature.

Measured flow functions for the whole set of materials are shown in Figure 5b.

4. Conclusions

The High Temperature Annular Shear Cell proved to be able to measure the flow properties of powders at temperatures up to 500°C. For the tested materials, experimental results do not reveal significant effect of temperature on the flow properties. This result was somehow expected for FCC powders which are commonly fluidized in catalytic cracking units and, therefore, it is desirable that the flow properties of this powder do not change up to the operating temperature of 650÷750°C. On the other hand, the results for corundum are not in agreement with the data reported by Formisani *et al.* (2002). In fact, in the absence of electrostatic and capillary forces, they attributed the increase of the voidage in fixed and fluidized state at high temperature as an increase of the van der Waals forces and not only to the effect of the increase of viscosity of the fluidizing gas. Further work will investigate powders in which cohesive interparticle interactions are not exclusively related to van der Waals kind of forces.

Table 3: Effect of temperature on powder flow properties – experimental results

Shear cell	*Material*	T (°C)	σ_1 (Pa)	φ (°)	C (Pa)	φ_e (°)	f_c (Pa)
SV10	FCC	20	997	32.1	60	37.8	216
SV10	FCC	20	1211	31.4	61	36.1	217
HT-ASC	FCC	20	981	32.3	45	36.6	163
HT-ASC	FCC	20	1205	31.8	65	36.9	234
HT-ASC	FCC	250	1008	31.1	61	36.9	216
HT-ASC	FCC	250	1192	31.0	52	35.0	185
HT-ASC	FCC	500	1004	32.7	69	39.4	252
HT-ASC	FCC	500	1186	31.1	68	36.5	241
HT-ASC	Corundum	20	4329	34.9	104	37.1	399
HT-ASC	Corundum	20	14126	34.1	250	35.7	943
HT-ASC	Corundum	500	4652	37.0	68	38.3	271
HT-ASC	Corundum	500	14795	37.7	251	39.3	1023
HT-ASC	Fly ashes	20	4132	31.8	274	38.1	983
HT-ASC	Fly ashes	20	13197	32.8	418	35.8	1536
HT-ASC	Fly ashes	500	4662	36.8	367	44.9	1465
HT-ASC	Fly ashes	500	13297	33.0	566	37.0	2086

List of symbols

- C cohesion (Pa)
- d_{10} diameter for which 10% of the volume of the particles has a smaller diameter (µm)
- d_{50} volume median diameter (µm)
- d_{90} diameter for which 90% of the volume of the particles has a smaller diameter (µm)
- d_{SV} Sauter mean diameter (µm)
- f_c unconfined yield strength (Pa)
- ff_c flow function (-)
- T temperature (°C)
- φ angle of internal friction (°)
- φ_e effective angle of internal friction (°)
- σ normal stress (Pa)
- σ_1 major principal stress (Pa)
- τ shear stress (Pa).

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