



# Bulk flow properties of sieved samples of a ceramic powder at ambient and high temperature



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## ARTICLE INFO

### Article history:

Received 2 July 2015

Received in revised form 11 November 2015

Accepted 14 November 2015

Available online 1 December 2015

### Keywords:

Powder flow properties

High temperature

Interparticle forces

Powder cohesion

van der Waals forces

## ABSTRACT

The flow properties of five samples of a ceramic powder, characterized by different particle size distributions were measured at ambient temperature and at 500 °C with the High Temperature Annular Shear Cell. A significant increase of powder cohesion was observed at high temperature. A model combining a continuum approach and a particle–particle interaction description was used to correlate the powder tensile strength with the interparticle forces. The dependence of the tensile strength on powder consolidation and temperature is correctly described by the model.

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## 1. Introduction

Flow properties of powders play a key role in several industrial process units, such as fluidized bed reactors, granulators and dryers. Several of these units require high temperature operations, which, in turn, may change powder cohesion with respect to that at ambient temperature.

The flow behaviour of granular materials is generally described by using a continuum mechanics approach. In particular, the stress distribution inside a bulk solid is described with the Mohr–Coulomb analysis, according to which the local state of stresses is represented by Mohr's circle on normal stress,  $\sigma$ , and shear stress,  $\tau$ , plane, in which the yield condition is represented by a line of slope  $\tan \varphi_i$  and intercept  $C$ , where  $\varphi_i$  is the static angle of internal friction and  $C$  is the powder cohesion. Furthermore, in the Mohr–Coulomb analysis, the granular material is assumed to be a solid in the failure condition, that is a solid in which the Mohr circle is always tangent to the Coulomb yield locus [1,2]. Following the Mohr–Coulomb analysis, the stress distribution within powders [3–5] can be estimated for the design of handling and storage equipment [6,7] provided that powders are appropriately characterized. For design purposes, the most used powder characterization devices are translational and rotational shear cells [8–10]. As it is known, particle properties, such as size and shape, [11,12], and mechanical properties [13–17] affect the flowability of powders. In fact, the powder flowability is related to the type and magnitude of

the interactions between particles acting at the microscopic scale. Many studies have highlighted the significant role of interparticle interactions by directly measuring the forces between particles [18–20]. Furthermore, it is very important to evaluate the flow properties at realistic process conditions, such as high temperature [21–23], high humidity [24–29] and very loosely compacted conditions [30–32], like in fluidised bed, that can affect the flow behaviour. The role of interparticle forces becomes more significant at low consolidation levels, as in aerated and fluidised beds, where body forces due to gravity are counterbalanced by the drag force exerted by the upper fluidising flow. In this case, the powder behaviour is determined by the relative weight of the interparticle forces compared to the mass and hydrodynamic forces [33–36].

Previous studies on the effect of temperature on fluidization, by means of, bed expansion measurements [37,38] and bed collapse tests [39–43], showed a change of fluidization behaviour with temperature which cannot be fully explained by taking into account only fluid dynamic forces. The authors provided an interpretation based on the hypothesis that temperature could increase the interparticle forces and their relative weight with respect to body forces. Direct measurement of interparticle forces as a function of temperature is difficult to perform and it is affected by a significant uncertainty [19]. Alternatively, it is possible to measure bulk properties such as the powder cohesion as a function of temperature and to correlate the cohesion change with interparticle force variations. Moreover, it is also possible to link measured flow properties and fluidization behaviour of powders [44–46]. Powder cohesion, in fact, is related to the intensity of interparticle forces such as van der Waals, capillary and electrostatic forces. These forces, in

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turn, can be affected by temperature as a result of changes of particle hardness, liquid bridge formation or variations of the particle dielectric properties. Few studies available in the literature have addressed the experimental evaluation of powder flow properties at high temperature. The first attempt was carried out by Smith et al. [47], who preheated powder samples of MgSO<sub>4</sub> and CaSO<sub>4</sub> up to 750 °C, moved them into a Jenike shear cell and performed shear tests immediately without any control of the temperature. More recently, Ripp and Ripperger [48] designed a temperature controlled annular shear cell for the Schulze shear tester operating from 80 °C to 220 °C. Besides shear testers, split cells were also used to evaluate the flow properties of granular materials up to 1000 °C [49,50]. Instead, Zimmerlin et al. [51] measured the torque necessary to the rotation of an impeller into a bed of different samples of cohesive powders up to 700 °C. Experimental results showed an increase of the unconfined yield strength with increasing temperature for all the analysed powders. The High Temperature Annular Shear Cell (HTSC) developed at the University of Salerno is suitable to measure powder yield loci up to 500 °C [23,52]. This cell was used to assess the temperature effect on flow properties of samples of fluid catalytic cracking catalyst (FCC powder), fly ashes, natural corundum and synthetic porous  $\alpha$ -alumina and glass beads. However, experimental evidences did not reveal a univocal effect of temperature in the tested range (between ambient temperature and 500 °C). In that case it was shown that in spite of the hardness of the ceramic material tested, plastic behaviour of the material at the contact point had to be introduced in the model evaluation to justify all the experimental observations tested. In that case, however, the wide particle size distribution of the samples and the lack of knowledge of the effect of temperature on the material yield strength did not allow a complete proof of this finding.

In order to better assess on this point, in this work the High Temperature Annular Shear Cell (HTSC) was used to study the flow properties of five powder samples of the same ceramic material with different narrow sized particle size distributions. Powder flow properties were measured between ambient temperature and 500 °C in the range in which the effect of temperature on the material yield strength was known from the literature. A model based on the multiscale approach proposed by Rumpf [53] and Molerus [13] was used to predict the effect of temperature on the tensile strength of powder samples.

**Table 1**  
Characteristic sizes of the five samples tested.

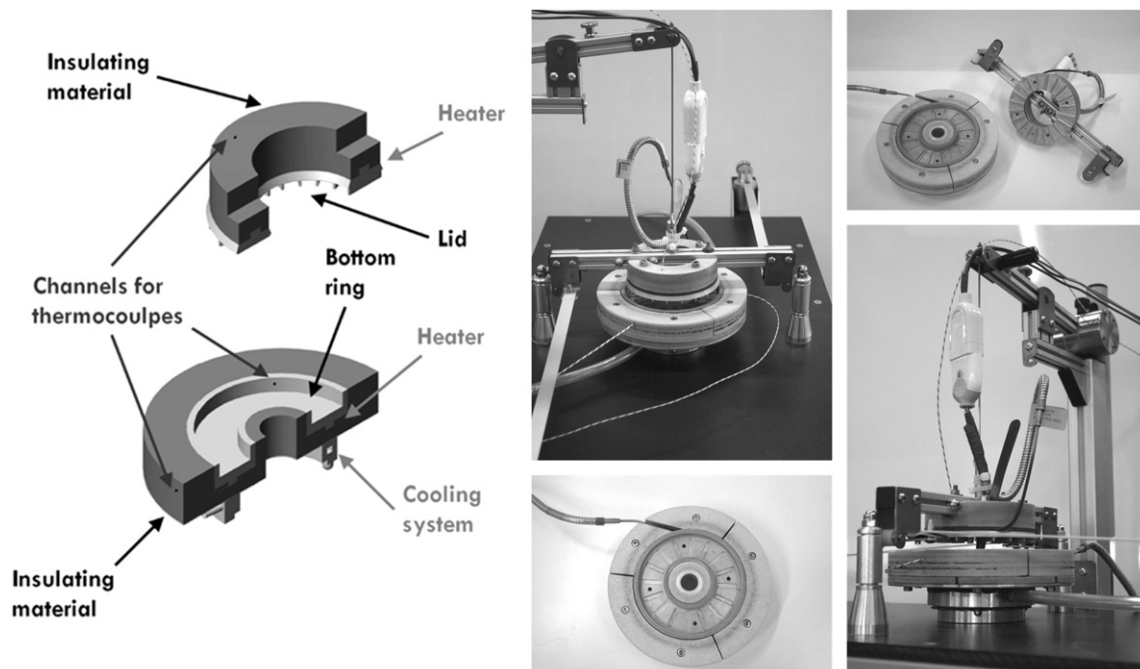
Sieving range, $\mu\text{m}$	<20	20–38	38–63	63–88	>88
$d_{32}$ , $\mu\text{m}$	7	22	41	51	104
$d_{43}$ , $\mu\text{m}$	14	37	63	89	227
$d_{10}$ , $\mu\text{m}$	3	18	38	55	90
$d_{50}$ , $\mu\text{m}$	12	35	61	87	184
$d_{90}$ , $\mu\text{m}$	28	61	95	130	423

## 2. Theoretical framework

Powder flow properties can be correlated to interparticle interactions at ambient and high temperature by means of the microscale approach provided by Rumpf [53] and by Molerus [13]. For van der Waals kind of interparticle forces, model equations can account alternatively for elastic or plastic deformation of the contact points. In this work, the hypothesis of plastic deformation was assumed according to the results obtained by Tomasetta et al. [54]. That study, in fact, indicated that the assumption of plastic deformation provides the correct order of magnitude values of tensile strength and its dependence on consolidation.

The main underlying assumptions of the Rumpf [53] and by Molerus [13] approach are:

1. Particles are organized in a randomly packed assembly.
2. Particles are spherical and monodisperse.
3. The contact areas between particles are small enough in comparison with the particle surface and therefore contact areas can be assumed as contact points.
4. The contact points are distributed over the particle spherical surface with equal probability.
5. The packing structure is isotropic.
6. The transmission of an isostatic state of compressive stress with three equal principal stresses is assumed.
7. The coordination number (i.e., the mean number of contacts of a particle with the adjacent neighbours),  $k$ , and assembly porosity,  $\varepsilon$ , follow the correlation  $k\varepsilon \approx 3.1 \approx \pi$  [56,57].
8. The particle Sauter mean diameter,  $d_{sv}$ , is used as the representative particle size.



**Fig. 1.** Schematic view of the High Temperature Annular Shear Cell (HT-ASC).

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