



# Analysis of industrial reactive powders flow properties at high temperature



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## ABSTRACT

Changes of bulk flow properties of two different types of titanium dioxide powders were measured at room temperature and 500 °C using the High Temperature Annular Shear Cell. A significant increase of the macroscopic bulk flow properties was observed with increasing temperature, in particular with regard to the unconfined yield strength. A theoretical modelling procedure was proposed with the aim to relate the measured properties to the microscopic interactions between particles. The results indicated that the model might provide a good match with the experimental data if proper values for the model's parameters are taken into account.

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## 1. Introduction

Changes with temperature of flow properties of particulate solids are usually observed in many industrial process units, such as fluidized beds. However, a satisfactory understanding of the phenomena that are responsible for such modifications has not been achieved yet. Although a number of research works have been carried out on the influence of process conditions on fluidization, findings are still controversial, as reported by Yates [1] and Lettieri and Macri [2] in their reviews on the subject. Much of the controversy still remains because the relative importance of the interparticle forces (IPFs) and the hydrodynamic forces (HDFs) on the flow behaviour of the particles.

Most of the disagreement on the relative role of HDFs and IPFs on the fluidizability of powders lies in the uncertain nature on the IPFs involved and in the difficulty of measuring them directly. Several authors investigated powders flow behaviour at different temperature through fluidization tests [3–7] and measurements of interactive forces between particles [8–12]. However, a full understanding of the effect of temperature on interparticle interactions and flow properties of bulk solid is still challenging.

Within this framework, powder rheology represents an appealing tool to evaluate indirectly the effects of the IPFs on fluidization. A great deal of research has been carried out over the last sixty years in order to define and measure, by means of simple tests, parameters apt

to characterize and predict the flow properties of solid materials [13–15].

Powder flow behaviours are commonly described in engineering science by using a continuum mechanics methodology, which allows to directly characterize powder rheological properties and flowability by estimating the stress distribution within powders. Many testers are available to this aim, but shear cells are currently the most used devices [16].

Some works attempted to find a link between fluidization and rheological behaviour combining cohesion and flowability changes measured with shear tests to the fluidization behaviour of powders [17–21]. Powder cohesion can be related to the intensity of IPFs such as van der Waals, electrostatic and capillary forces, which are affected by temperature as a result of changes of particle hardness, liquid bridge formation or variations of the dielectric characteristics. However, a more systematic assessment of the independent and the combined effects of process conditions on fluid bed rheology and fluidization still needs to be accomplished.

Other works focused on the evaluation of the effect of temperature on the powder flow properties [22–24]. Tomasetta and co-workers [25–27] recently modified at the University of Salerno a Schulze shear cell (High Temperature Annular Shear Cell, HTASC) to perform measurements up to 500 °C.

Even in condition at which electrostatic effects can be neglected, the effect of temperature on the powder flow properties is clearly a function of the material, in fact, temperature may produce phase changes which may determine or alter the amount of liquid available on the particle surface and turn on and off capillary forces. With the HTASC, Tomasetta

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and co-workers [25–27] evaluated rheological properties at ambient and high temperature for different powder samples: glass beads, FCC powder, fly ashes, alumina and natural corundum. The results showed that there was no significant effect of the temperature in the range 20–500 °C on the shear flow of the FCC powder, alumina, fly ashes and corundum powder. Instead, an increase of cohesion and, therefore, of the unconfined yield strength was observed with temperature for glass beads. More recently, Chirone et al. [28] used the high-temperature annular shear cell developed by Tomasetta and co-workers to characterize the bulk flow properties of five ceramic powder samples with different particle size distributions between ambient temperature and 500 °C. They observed a significant increase of powder cohesion at 500 °C for different cuts of the same powder with a particle size larger than 20 µm, resulting in a lower flowability of the samples. In both cases above, direct observation excluded the significance of electrostatic forces. Furthermore, within the range of temperature explored, the presence of capillary forces could also be excluded and, therefore, any observed variation of powder flow properties had to be explained considering only the action of van der Waals forces. The analysis tool used in both cases above was the model based on the multiscale approach proposed by Rumpf and Molerus [29–31] to predict the effect of temperature on the tensile strength of the powder samples. The main conclusion was that the temperature effects for materials in these conditions could only be explained by assuming a plastic deformation of the particle at the contact points.

Within this framework, this work presents further experimental observations reporting the effect of temperature on the flow behaviour of various titanium ore powders. To this end, rheological measurements were performed using the HTASC to assess the particle-particle interactions at both ambient and high temperature. Furthermore, in this paper, in place of the purely plastic deformation model suggested by Molerus [29], a novel procedure to correlate microscopic interparticle forces to the measured macroscopic bulk flow properties is proposed. This is based on the theoretical modelling approach proposed by Tomas [32,33] that was developed to account for elastic-plastic particle deformation at the contact points. The data will be discussed in order to assess on the best physical parameter to be considered to describe the material yield at the contact point.

## 2. Theoretical framework

In order to relate the microscopic interparticle interactions to bulk flow properties, the model proposed by Tomas [32,33] for steady-state flow criterion of particles can be followed. Such theory is based on the possibility to relate consolidation and non-rapid flow of particulate solids with adhesion forces in particles contacts. The following main assumptions are considered:

1. Particles are organised in a randomly packed assembly and the packing structure is assumed to be isotropic with uniform porosity ( $\varepsilon$ ) in any cross-sectional areas as well as in the powder bulk;
2. Particles are spherical and monodisperse and thus the contact points are uniformly distributed over the particle surface with equal probability;
3. The contact areas are small in comparison to the characteristic particle size, therefore these may be considered as contact points;
4. The transmission of an isostatic state of compressive stress with three equal principal stresses is assumed.

These hypotheses are the basis of the equation derived by Molerus [29] for the relationship between stresses and contact forces for monodisperse particles with characteristic particle size  $d$  and assembly porosity  $\varepsilon$ . In particular it is used to relate the interparticle forces acting in a real bulk solid,  $F_H$ , to the isostatic tensile strength,  $\sigma_t$ , extrapolated from the powder experimental yield locus, i.e. the tensile stress at

which the force necessary for the contact separation equals the adhesive interparticle force  $F_H$  at the contact point:

$$F_H = \sigma_t d_{sv}^2 \frac{\varepsilon}{1-\varepsilon} \quad (1)$$

In Eq. (1)  $d_{sv}$  is the particle Sauter mean diameter, that provides the best estimate of the ratio between particle surface and volume and, therefore it gives the most accurate volumetric concentration of the interparticle contact points, which is the key variable to relate stresses to average interparticle forces values [27].

Tomas [32,33] considered an elastic-plastic particle contact model in order to describe the failure/flowability conditions of a bulk powder. He proposed the following equation for the quantification of the IPFs:

$$F_H = (1 + \kappa) F_{H0} + \kappa F_N. \quad (2)$$

In which  $F_N$  is the compressive normal force transmitted at the contact during the consolidation stage;  $F_{H0}$  is the adhesion force without any consolidation ( $F_N = 0$ ) and any contact deformation intrinsically present in fine powders and related to their cohesive nature;  $\kappa$  is the so-called elastic-plastic consolidation coefficient.

According to Tomas and Molerus, the value of  $F_N$  can be calculated from the normal stress at consolidation  $\sigma_N$  applying the Rumpf equation:

$$F_N = \sigma_N d_{sv}^2 \frac{\varepsilon}{1-\varepsilon}. \quad (3)$$

The coefficient  $\kappa$  describes the influence of plastic contact deformation and so it is a measure of the irreversible particle contact stiffness or softness. It is given by the slope of the linear relationship between the interparticle adhesive force  $F_H$  and the normal force  $F_N$ : a small slope ( $F_H \approx F_{H0}$ ) stands for low adhesion level because of stiff particle contacts, while a large inclination means soft contacts and a resulting cohesive flow behaviour. However, from a physical point of view, it is defined as:

$$\kappa = \frac{\kappa_p}{\kappa_A - \kappa_p} \quad (4)$$

where  $\kappa_p$  is the plastic repulsion coefficient and  $\kappa_A$  is the elastic-plastic contact area coefficient representing the ratio of plastic particle deformation area,  $A_{pl}$ , to the total contact deformation area, which includes the contact area affected by elastic displacement,  $A_c = A_{pl} + A_{el}$ . According to Tomas [32,33], they can be estimated as:

$$\kappa_p = \frac{C_{H,fs}}{6\pi p_f z_0^3} \quad (5)$$

$$\kappa_A = \frac{2}{3} + \frac{1}{3} \frac{A_{pl}}{A_c} = 1 - \frac{1}{3} \sqrt{\frac{h_{c,f}}{h_c}} \quad (6)$$

where  $C_{H,fs}$  is the Hamaker solid-fluid-solid constant,  $z_0$  is the characteristic molecular separation distance, that amounts to about 0.3–0.4 nm [34],  $p_f$  represents the repulsive particle micro-hardness, i.e. the resistance against plastic deformation expressed as the plastic compressive yield strength of the particle material. In addition,  $h_c$  represents the particle height of flattening and  $h_{c,f}$  is the height of flattening for incipient yielding at  $p = p_f$  that can be defined as function of the particle size, the material Poisson ratio  $\nu$  and the Young's modulus  $E$ . The parameter  $h_{c,f}$  is a function of the contact properties and can be estimated by means of the following equation:

$$h_{c,f} = d_{sv} \left( \frac{\pi p_f (1-\nu^2)}{2 E} \right)^2. \quad (7)$$

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