Research project proposal for the IFPRI theme:

MECHANISM OF FORMATION AND GROWTH OF POWDER LAYERS ON PROCESS EQUIPMENT SURFACES AT LOW STRESSES

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Outline of the problem

Several papers and books in the literature deal with the adhesion of single particles on walls. Mostly, the effect of particle and surface roughness is highlighted and the action of the different kinds of interparticles forces is described. Instead, the build-up (make up) of powder layers on the equipment surface during operation at ambient temperature is not described nor quantified in the literature with the exception of dedicated patents [1] and of the vast literature on slag accumulation phenomena in combustors and gasifiers (e.g. [2])

Considering flow within containers, in granular mechanics it is generally assumed that the wall friction, at equal system conditions, is lower than the internal friction. This is reasonable considering the microscopic interactions that can be assumed at the wall and within the material. In fact, in process and storage equipment, it is reasonable to assume that the wall roughness is generally lower than the roughness of particle-particle contacts in an internal shear plane and that the number of particle to wall contacts per unit surface is generally lower than the number of particle-particle contacts in an internal shear plane. The first effect is dominating in case of prevailing frictional interactions between particles and between particles and wall. The second effect is dominating in the case of cohesive forces between particles and adhesive forces between particles and wall. Therefore, the occurrence of the formation of a stationary layer during powder shear, whatever its thickness, can only be associated to increased particle-particle cohesive and particle-wall adhesive forces in the layers close to the surface.

More complex phenomena take place in particle layer build up in dilute systems, in which the interaction between particles and wall occur with particles hitting the wall. The nucleation of a static particle layer on the wall depends on the possibility that the elastic energy accumulated by the particles hitting the wall is not able to produce the work necessary to win the adhesion forces between the particles and the wall at the rebound. The buildup of layers is related to the rate of accumulation of new adhering particles (on the wall or on the particle layer already formed) and the tendency of the hitting particles to remove the adhering particles.

From the description above, it is clear that the experimental set up, useful to measure the initiation and the accumulation of a particle layer on the walls of a container with a dense particulate system, cannot be the same of the experimental setup to be used to measure the nucleation of a layer of particles and its growth in a dilute system, in which many other phenomena than powder shear occur. Nevertheless, experiments in powder shear may provide useful information on the interparticle forces acting in particulate system that can be used to understand the behavior of dilute systems. For this reason, first we will focus our attention on the layer formation in dense powder shearing during this proposed project. The study of the formation of a static layer in pieces of equipment with particles travelling in dilute systems could be addressed later in a possible extension of the present project.

Several system conditions can determine a local increase of interparticle cohesive forces, which can justify the formation of a static layer of particles on the wall of a container hosting a powder. These may be local changes of air relative humidity, for example due to lower temperatures at the wall, which can determine local capillary condensation of water. With hydrophilic materials, local changes of air humidity can induce the soaking and consequent softening of some of the particles phases that, by deformation, may increase the contact areas between particles and between particle and wall and, consequently, also the interparticle forces. Even worse effects can be observed with temperature fluctuations at the wall that can determine caking phenomena due to the precipitation of solids in the capillary bridges of changing volume or the hardening of material at contacts. Temperature increases at the wall can also determine a local variation of van der Waals interactions. In this case, the principal effect of temperature is related to the change of particle hardness. In fact, at higher temperatures hardness decreases and determines larger contact areas between particles and, consequently, larger contact forces. More significant can be the effect of temperature increase at the wall when it determines the partial melting of particulates or the fusion of low melting phases in the particulate systems. In this case, capillary forces set up determining a significant increase of interparticle cohesion.

Scope of the project

Scope of the project is the understanding of 1) the changes of interparticle forces at the wall that can determine the formation of a static powder layer in shear flow occurring at low but quantified compressive stresses and 2) of the conditions leading to the growth of a static layer of particles during powder shear.

In this project, the temperature will be used to change the intensity of interparticle forces and to determine the formation of a static particle layer on a wall. The study will include the use polymeric particulates and the investigated temperatures will be in the range ranges between 100 and 300 °C. Experiments with sub millimeter particulates made of polymer, carried out below the polymer melting temperatures, will describe the temperature effects on van der Waals interactions due to changes in the material hardness and thus in the particle contact area. The effect of temperature on the flow properties of these systems will be studied with the help of a High Temperature Annular Shear Cell (HT-ASC) [3] developed at the University of Salerno (UNISA). To characterize the effect of temperature on the powder properties, the cell will be used according to the established procedure in which the sample is uniformly heated up to the desired temperature of the shear test. Furthermore, the apparatus, will be modified in order to dynamically study the temperature profile condition that can lead to the formation of a static layer on a wall sample on which model shear conditions are realized.

Experimental set up

The High Temperature - Annular Shear Cell (HT-ASC)[3] developed at UNISA operates on the same workbench of the original manual tester developed by Schulze for shear test at room temperature. Figure1a shows a schematic of the HT-ASC. It consists of a bottom aluminum annular trough containing the powder sample and an aluminum and steel annular lid placed on the top of the sample like the original cell in the Schulze tester. As for the original shear cell, the lid is fixed at a crossbeam connected by two tie-rods to two load beams. These allow measuring the shear force acting on the shear plane developed inside the powder sample

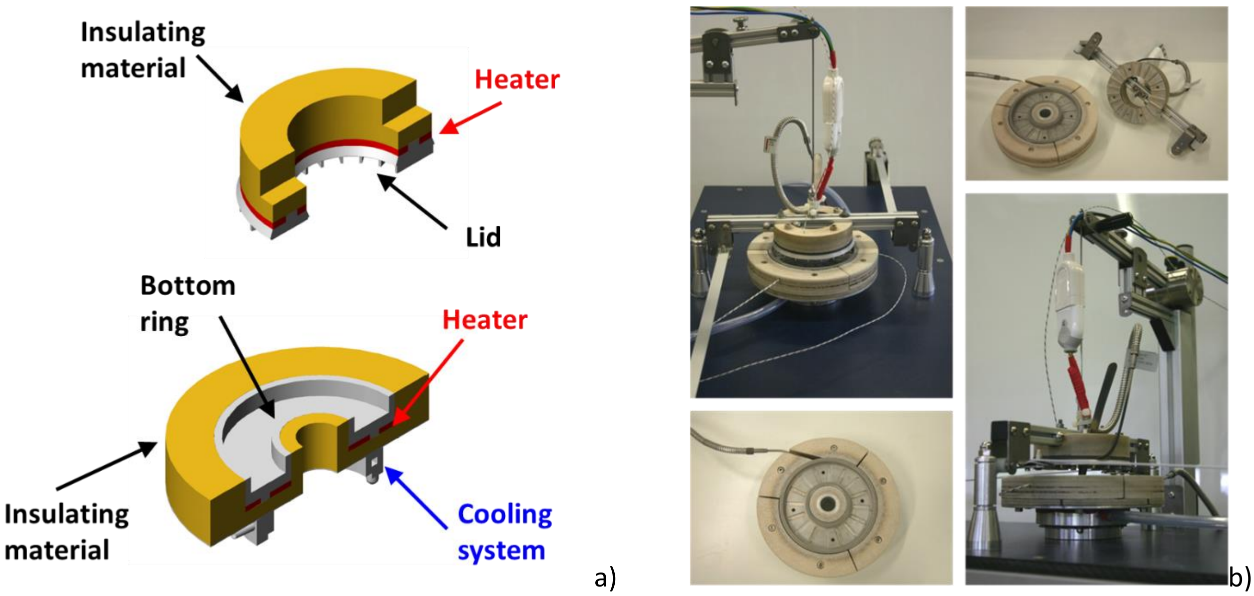

Figure 1 Schematic of the High Temperature - Annular Shear Cell (HT-ASC) (a) and its operation on the Schulze RST (b)

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.

Electric heaters were introduced below the cell bottom and on the lid to heat the cell and the powder sample contained in it. In order to reduce the heat flux from the external surface of the cell and then to minimize the temperature gradient within the sample and, for safe operation of the cell, a covering insulating material was placed around the trough of the cell and above the lid. A cooling system was designed and built to cool the cell base where it is in contact with the gears of the rotation mechanism of the tester. Some pictures of the HT-ASC operating on the Schulze workbench are reported in Figure1b. The material used for the construction of the HT-ASC allows a safe operation of the system up to 500°C.

A temperature control system was developed to achieve the set temperature in the powder sample. It consists of three J thermocouples measuring the temperature at three different depths of the sample connected to a data acquisition board. A dedicated software was developed to read the three temperatures and regulate the heating power in order to keep the temperature as uniform as possible. In particular, the procedure acquires and digitizes the three temperature values. The top and bottom temperature, T_{UP} and T_{DOWN}, are compared with the set-point temperature, which can be the same or different for the two separate PID software loops that are used to control separately the heating power on the lid and on the trough.

Materials

As above mentioned, two different cuts of model particulate solids and a further real powder will be used in the experiments.

The first particulate material made of sub-millimetric polymeric particles (100-600µm) will be used below the melting temperature for experiments in which the presence of van der Waals forces only will be assumed so that the effect of temperature on the interparticle forces could be governed by changes of the material hardness [4,5].

The second material will be a mixture of a ceramic non-porous particulate (e.g. sand) in the sub-millimetric range (300-1000 µm) and of a micrometric polymeric powder (1-30 µm). It will be used in temperature ranges including the melting temperature of the polymer. The presence of a molten phase will introduce strong changes in the interparticle forces [6].

The real industrial powder will be chosen with the help of the IFPRI members and possibly provided by them on the basis of their experience of materials showing the formation of static layers in conditions accessible with the proposed experimental set up

Procedures and required changes to the set up

Shear experiments will be carried out in two different modes: with uniform temperature and with a temperature gradient.

The uniform temperature mode will be used for the measurement of internal powder properties and wall friction with temperature as a system parameter. In this mode the set point values of the temperature control loops on the lid and at the bottom heater will be the same. The internal flow properties and the wall friction will be measured with the usual procedures applied to the ring shear testers [7] after the uniform temperature in the system is reached and verified by reading same temperature at the three thermocouples. The trough of the cell will be adapted in order to carry out wall shear experiments at high temperature.

The temperature gradient mode of use of the shear cell will be adopted in a separate set of measurements focused on the dynamics of growth of the static particle layer. In these experiments, the cell will be used with the heated lid hosting a wall coupon. The temperature at the lid will be increased gradually up to a level at which the shear strength at the lid wall is higher than the internal shear strength at the vanes tip. The schematic situation is reported in Figure2 showing that, at the beginning of the heating process, when the temperature is uniform, the internal shear strength at the vanes s_i is larger than the shear strength at the wall s_w (Figure2a and b). Therefore, the shear plane is at the wall. Instead, when the temperature profile is such that the internal strength at the vanes s_i is smaller than the strength at the wall s_w (Figure2c), the shear occurs at the vane tip.

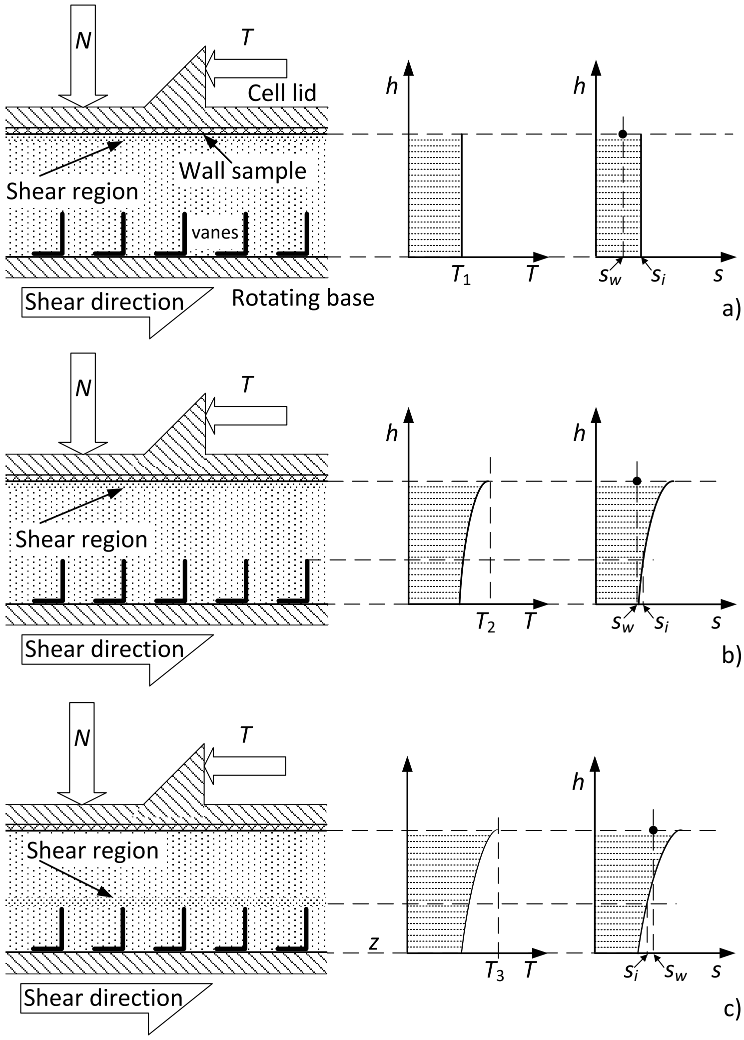

Figure 2 Evolution of the shear cell experiment at variable temperature gradient ($T_1<T_2<T_3$): a) initial condition with uniform temperature; b) Start of the heating process; c) final stage of the heating process. In a) and b) the internal strength at the vanes s_i is larger than the material adhesion s_w and the shear occurs at the wall. In c) the internal strength at the vanes s_i is smaller than the material adhesion s_w and the shear occurs at the vane height.

In order to carry out experiments with a temperature profile in the sample, the existing cell will have to be modified as it is shown in Figure3. The cell lid will have to be adapted to hold an annular wall coupon in contact with the heated surface. The cell base will require a number of more significant changes. First, vanes will be placed at the cell bottom. Then the trough side walls, presently made with the same material of the bottom wall, will be made of fused silica (quartz). The lower thermal conductivity of the wall will avoid thermal bridges between parts of the powder samples at different temperatures. Furthermore, fused silica, that can withstand temperatures up to 1500°C, is transparent in the visible and near infra-red (NIR) range and, therefore, can be used in conjunction with a NIR camera to read the material temperature[8]. The NIR camera fixed on the rotating base will be calibrated to measure the temperature profile. Images sequences taken in the visible range will be used with a Particle Image Velocimetry (PIV) procedure to derive the particle movements behind the wall. Since the camera is moving with the base, particles adhering on the heated wall surface fixed on the lid will be seen as in movement.

The outcome of the experiments with the new cell will be the time evolution of the temperature profile in the powder sample in the shear cell and of the presence of a static shear layer by applying a temperature gradient. The main variables that will be investigated are the extremes of the temperature gradients, the normal force applied in the shear experiments and the surface quality of the wall sample. With the second model system also the mixture composition, i.e. the amount of liquid bridges, formed will be investigated. These results will be compared with the theory hypothesized above and reported in Figure 2. In order to verify the theory, the internal and wall strength as a function of the temperature, previously measured with the conventional HT-ASC, will be needed. The measured powder shear properties at changing temperature will be used to derive the necessary information.

Project time deployment and expected outcome

The time distribution of the activities in the project is shown in the Gantt chart reported in Figure 4.

At the end of year 1, the experimental procedure will be completely defined with the construction of the new apparatus, its functionality testing and some first exploratory measurements of the first particulate to be investigated. In fact, by that time this material will be also completely characterized for internal and wall shear at relevant temperatures. Some preliminary results on the applicability of the theoretical framework will be also available.

At the end of year 2, the complete thermal flow characterization (internal and wall shear) of both model materials will be accomplished and tested for the static layer formation.

At the end of the last year (year 3), the complete thermal flow characterization (internal and wall shear) of the real material chosen for the experiments will be accomplished and tested for the static layer formation. The results will be completely analyzed and the theoretical framework confirmed.

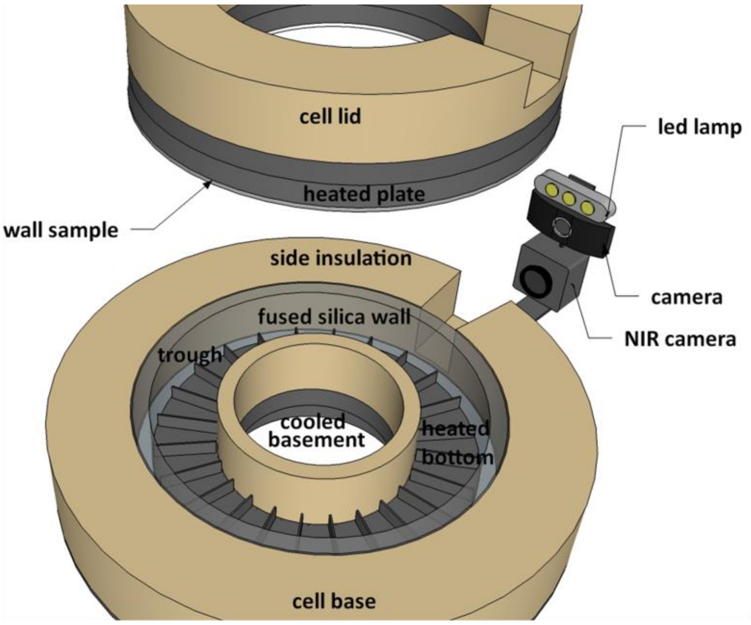

Figure 3 Modified set up of the shear cell base for experiments with a vertical temperature gradient.

Project leverage into other research activities at UNISA

The project will rely on the continuing research activities on the effect of temperature on powder flow properties and the project could not take the present configuration without the vast research activity carried out on the topic. In the next years the study of the thermal effect on the powder flow properties will focus mainly on topics related to the distribution of powders in Selective Laser Sintering (SLS), an additive manufacturing techniques which alternates the addition of powder layer and selective sintering of the particle in the layer, in order to obtain three dimensional objects. One of the main issues in this field is related to the characterization of powders to understand if these are suitable for the SLS process with respect to the possibility to obtain with powders a layer of good quality layer. At UNISA a PhD student is presently working on this topic and some of his materials are polymeric powders. The experience we are gaining with this project will help us in the right choice of the model material.

In 2019 other two PhD projects on topics related to SLS will start at UNISA. Both these PhD projects are part of Mathegram an Innovative Training Network financed by the EU Commission within the MSCA-ITN program with highly competitive calls. Mathegram is lead by prof Charley Wu at University of Surrey and involves 5 universities, 4 public research centers and 6 major research centers of major companies in Europe. One of the 2 PhDs project at UNISA is related to the flowability of SLS powders, among the other things, will study if

Month	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36
Material definition and purchase	■	■																																		
Design of the modified cell for temperature gradient experiments	■	■																																		
Construction of the modified shear cell			■	■																																
Functional test of the new cell and managing software set-up					■	■																														
Polymer powder characterization with the standard cell			■	■			■	■	■																											
Experiments with the polimer powder with the modified shear cell										■	■	■	■	■																						
Model development calculations and reporting												■	■	■	■	■						■	■	■	■	■						■	■	■	■	■
Sand-polymer mixture characterization with the standard cell															■	■	■	■	■																	
Experiments with the sand-polymer mixture with the modified shear cell																				■	■	■	■	■												
Real powder characterizatio with the standard cell																									■	■	■	■	■							
Experiments with the real powder mixture with the modified shear cell																														■	■	■	■	■		

Figure 4 Gantt of the project

it is possible to use a light compaction of the powders in the layer preparation for the SLS. One of the instrument that can be applied is the forward rolling of a cylinder. The effective possibility to use it is related to the circumstance that the forward rolling cylinder does not detach in its trailing section material from the layer spread, due to the adhesion of part of the layer to the cylinder. This phenomenon occurs when the adhesion force of the compressed powder on the cylinder surface is larger than the internal resistance of the compressed layer, a situation very similar to that to be studied in the project related to the present proposal to IFPRI. Therefore, the understanding on the phenomenon occurring at the SLS roller will help in the right interpretation of the IFPRI project. Also, the experimental set up developed for the PhD project, a roller operating at controlled temperature, may constitute a valid alternative to the experimental set up proposed here, in case something is not working properly.

The second PhD project at UNISA within Mathegram will be on the heat transfer during the laser sintering action in the SLS project. The plan is to use a dedicated set up in which a layer of particles is placed on a fused silica glass and the heat transfer is measured by looking at the evolution of the temperature distribution of the powder above the glass. The similarity with the experimental set up considered for this project is clearly evident as well as the benefit that will derive from similar experimental experience in both projects.

Critical unknowns and get around solutions

The most critical issues of the proposed project are related to the effective functionality of the new cell that will be designed for in which a temperature profile is applied. Problems may arise for the following reason:

1) The apparatus is not sufficiently transparent to visualize the flow in presence of liquid bridges due to sticking of liquid on the cell wall. This problem can be overcome either changing the materials to be investigated or with the design of a different cell, not transparent but with an appropriately designed sensor highlighting the presence of shearing material at different heights. A temperature probe will be required to measure the temperature profile
2) The new apparatus is not able to visualize the position of the shear plane due to important sidewall interactions. In this case different apparatus derived from the one used to study SLS layer deposition will be used to apply compression on the powder over a heated layer and to study its resistance to scratching actions.

Support from IFPRI members

As above mentioned, the real industrial powder will be chosen with the help of the of the IFPRI members.

It would be advisable that they could also provided a material that, on the basis of their experience, has showing the formation of static layers in conditions that can be explored with the proposed experimental set up.

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