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APPARATUS FOR MEASURING FRICTION INSIDE GRANULAR MATERIALS – GRANULAR FRICTION ANALYZER

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Abstract

Flow of granular materials is a complex process but it is important to measure, because the flow of granular material during processing, handling and transportation strongly influences the quality of the final product and its cost. Flowability of granular materials depends on the characteristics of the material and on the conditions at which flow is occurring. Existing methods of measuring flowability of powders are described in this paper, and a new methodology is introduced to measure friction between granular materials under pressure induced with uniaxial compression. Apparatus also allows analysis of conditions at which granular material starts to flow when exposed to uniaxial compressive load, i.e., zero-rate flowability. We call the apparatus the Granular Friction Analyzer (GFA).

The concept of the GFA was tested by measuring four different materials with different average particle sizes. It was observed that as the particle size decreases so does its zero-rate flowability. This is in agreement with powder literature. Therefore, it can be concluded that in general the GFA method can be a very useful tool to study friction between granular materials and conditions at which the granular material flow initiates, i.e. zero-rate flowability of powders under pressure. However, further improvements are required to increase its sensitivity and accuracy.

Keywords: Granular materials; Inherent friction; Zero-rate flowability; Granular flow; Metal powders; Uniaxial compression

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1 Introduction

Granular materials are present in our everyday life whether as raw materials, intermediate or final products. For instance, one finds them in the food industry from cereal grains to dehydrated milk; in the pharmaceutical industry different powders are blended and compressed to produce tablets; metal, polymeric or ceramic parts can be produced through different powder sintering techniques; moreover, granular materials are present in nature as sand and rocks. In all these examples, the flow of powder during processing, handling and transportation strongly influences the quality of the product and its cost.

A granular material is defined as a collection of discrete solid particles that are filled with an interstitial fluid, usually air. From this definition, one can notice that they cannot be classified either as solids or liquids, leading to a complex behavior difficult to understand and predict [1]. In the literature, several terms are used interchangeably to describe granular materials, such as bulk solids, particulate solids and powder.

Within the world of granular materials, metal powders constitute an important part of the industry. In this context, one important topic of research nowadays is to study the flowability of powders in powder metallurgy because an increase in flowability represents an increase in the productivity of the overall process and a reduction in the cost of transportation of powders. During powder metallurgy granular materials are transported and molded under pressure.

Another example of utilizing granulated materials is in new emerging dissipative granular technology [2,3]. In this technology damping elements are made of granulated polymeric materials and encapsulated in a flexible container. Good performance of such damping elements can be achieved by pressurizing granular material to a pressure level, at which the damping element's frequency for absorbing a maximum energy substantially matches the frequency of mechanical excitation. This is possible because damping properties of polymeric (elastomeric) materials are frequency (time), temperature and pressure dependent [4,5]. In this instance also flowability of polymeric particles is important, since increased flowability ensures the equal distribution of pressure inside damping elements.

Flowability is defined as the ability of a powder to flow and results from the combination of material physical properties, which along with the equipment used for handling or processing governs how easy a granular material can flow. There are several techniques used for measuring flowability some of them require simple apparatuses while others use sophisticated setups. Simple traditional apparatuses as Hall-flowmeters and angle of repose devices provide a comparative analysis of which particular powder flows better with respect to another under certain conditions of measurement [6,7]. On the other hand, more sophisticated setups as shear cells and powder rheometers can provide more quantitative information as cohesion and angle of friction that influence the flowability of powders [8,9]. Nevertheless since flowability changes according to the conditions at which flow is occurring, measuring devices more or less should resemble the handling or processing equipment in which the powder will be used [10]. An overview of the flow properties of the granular systems including voids, granular porosity and random packing characteristics is summarized in the paper of Traina et al. [11]. Since most of the measuring techniques available in the market work under gravitational and shear flows, the present paper aims to introduce a new method of characterization where the driving force for granular flow is high pressure [12,13]. Hence, apparatus allows investigation of the so-called zero-rate flowability induced by pressure. More precisely the apparatus allows investigation of friction within granular materials exposed to

pressure generated by uniaxial compression. As a special case apparatus allows determination of conditions at which granular material will start to move.

2 Existing Flowability Measuring Techniques

As previously mentioned, flowability is defined as the ability of a powder to flow. It results from the combination of material physical properties and the physical characteristics of the equipment used for handling and processing. In this context, flowability is not an inherent material property because it depends on the conditions under which the flow takes place [14]. It is important to distinguish between flow property data and flowability. The former refers to the characteristics of the powder alone which affect its flowability (e.g. cohesion strength, particle shape, surface roughness, angle of friction, etc.), whereas the latter is a description of the ability of material with certain flow properties to flow under certain conditions.

There are different approaches to estimate flowability of powders under different conditions. A general classification of flowability characterization methods is (i) traditional instrumentation, which have gravity as the driving force for flow and then either time, mass or angle of repose of the material is measured [6,7]; and (ii) modern instrumentation where more complex setups composed by many accessories are moved simultaneously to produce and control force or torque that leads to flow of the powder [8,9]. Within the traditional methods one finds the "Hall-flowmeter" and the angle of repose measurement; on the other hand, shear cells, powder rheometers and optical methods constitute the main modern instrumentation available today to measure flowability [15,16]. In the following sections a short overview of such characterization techniques is given, mainly to clearly distinguish the proposed new measuring technique from the currently available ones. The comprehensive overview is given in the book of Schulze [17].

Hall-flowmeter is a widely used standard method of powder characterization where the driving force is simply gravity. It measures the time required to discharge several grams of powder through an orifice of a standard funnel (DIN ISO 4490, ASTM B 213, MPIF No.3). As other flowability characterization methods, this test offers only a means of comparison and evaluation because in many of the cases the powder does not flow through the small orifice of the funnel. This characterization method is suitable for evaluating flow of particles in hoppers, silos and similar containers.

Another type of characterization typically used in industry because of its basic use and high reproducibility is the angle of repose (AOR) measurement [7]. In this method powder is spilled into a funnel that is held at a fixed height above a flat base, or the funnel moves up gradually to allow the sample to flow out. One requirement of these funnel-based methods is that the powder must be able to flow through an orifice, which sometimes may not happen. In this case dynamic methods can be used to measure the dynamic angle where either a cylinder or a tilting table turns until slipping of the powder occurs and then this angle is measured [7].

There are several relations in literature of how to relate AOR with flowability of a powder, Raymus [18] suggests that angles of repose below 30° indicate good flowability, 30°- 45° some cohesiveness, 45°- 55° true cohesiveness, and >55° very high cohesiveness, i.e. very limited flowability. Even though there are some standards (ISO 4324, ISO 8398, ASTM C1444, BS 4140-9, IS-6663), due to the lack of agreement in the best design or size of equipment and the way that a test should be done, one can conclude that the

angle of repose measurement is a practical method (quality control method) to verify if the powder being produced today has better or worse flow properties than the one produced yesterday, more than a characterization method [7]. More recent paper by Lumay et al. [19] comprehensively reviews different standard as well as newer methods for measuring flow properties of powders and grains.

Shear cells are powerful characterization tests that allow the calculation of the angle of internal friction and the cohesion strength of a material according to the Mohr Coulomb criterion [8].

Shear cells measure the relationship between the shear stress (which leads to flow of the material) and the normal stress. The cohesion of the material is obtained by extrapolating to zero several measurements of the relationship between shear stress and normal stress in the shear cell. The cohesion of a granular material corresponds to the value of shear stress required for flow in the absence of other normal force [7]. Once cohesion is known, the friction angle can be obtained for different shear stress values. Several modifications of shear cell have been implemented to improve the sensibility of the equipment as in [9,20,21] but all of them work under the same principle described above. Comprehensive comparison of ring shear testers is presented in the paper by Schulze [22] which summarizes the results of the 2008 round robin project on ring shear testers. The round robin showed that despite different ambient conditions the range of obtained results was quite narrow.

Another type of equipment used is the powder rheometer FT4 developed by Freeman technology Ltd, Gloucestershire, UK [9]. The principle of this rheometer is based on recording the forces required to induce deformation and flow of powder inside a vessel container. The force is transmitted through a sophisticated twisted blade that is moved along a predetermined helical path [9].

Flowability of powders is related in the FT4 by two main parameters the "basic flow energy" (BFE) and the "specific energy"(SE). Both BFE and SE are measures of the work done by the system to induce flow of the powder and depend on the mode of the helical blade. Both parameters BFE and SE can provide a good relative mean of comparison of flowability of powders with different characteristics as size distribution or different external conditions as moisture [9].

Moreover the FT4 rheometer can be adapted with a shear cell that allows us to calculate the friction angle and cohesion of the powder under the principle of a standard shear cell, i.e. to measure the relationship between the shear stress (related in this case with the torque of the shear cell adaptor) and the normal stress (function of the vertical load induced by the cell adaptor) [9].

An alternative way to measure flowability is by optical means through the so-called "Particle Image velocimetry" (PIV) and "Particle tracking velocimetry" (PTV) [15,16]. In PIV the whole flow field is visualized at different points by tracer particles that are illuminated with a sheet of laser light that is pulsed to obtain images of the particles on photographic film or a video [15].

In turn, PVT is based in flow-visualization techniques such as streak photography and stroboscopic photography where the path of the particles is traced by means of multiple exposures or a photograph film with fluorescent markers. In this method you can track the particle displacements that are short compared to the mean spacing between individual particles paths, moreover, it is possible to track individual particles because the number of particle displacements per area of the image is relative small [15].

None of the described techniques have pressure as the means of causing flow of powders; therefore a new technique is needed to measure the flow of powders under pressure generated by uniaxial compression.

More precisely, the presented apparatus allows studying the internal friction of granular materials as well as conditions at which flow of powders will be initiated.

3 Granular Friction Analyzer

As it was presented, the different equipment available in the market for measuring flowability of granular material are based on gravitational forces or shearing failure. The Granular Friction Analyzer (GFA) is a novel apparatus for studying inherent friction in granular materials that are exposed to uniaxial compression loading and conditions at which flow of granular material will be initiated – zero-rate flowability. The apparatus was developed at the Center for Experimental Mechanics (CEM), Faculty of Mechanical Engineering, University of Ljubljana.

Fig. 1 shows schematically the main parts of the apparatus. The GFA is composed of a hollow cylinder with one of its ends closed that is loaded with granular material. Once the cylinder is filled with particles, a force is applied by means of a piston (Fig. 1a); such force will induce a pressure inside the cylinder that at the same time induces elastic deformations in the axial and tangential directions of the external wall of the cylinder. The strain in the external face of the cylinder can be measured experimentally, e.g., by means of a strain gages setup (Fig. 1b), or optically. Finally, Fig. 1c shows the dimensions of the cylinder. One can notice that the powder inside the cylinder is subjected to a confined compression; Ma and Ravi-Chandar [23] developed an apparatus that works under the same principle, however such compression was applied to solid materials and their goal was to characterize the stress state of the inserted solid material.

Fig. 1. Schematic of the GFA: a) cylinder filled with the granular material subjected to a compressive load, b) strain sages setup that is used in the apparatus to measure strain evolution along the length of the cylinder and c) dimensions of the cylinder

The goal of the GFA is to obtain the pressure profile along the length of the cylinder and to introduce such profile as a measure of inherent friction between particles of granular materials. The concept of the GFA is valid because forces through the granular material and the pressure distribution along the length of the cylinder is a measure of how force is transmitted from the piston to the powder and from the powder to the cylinder. In this context, one expects that depending on the friction properties of the powder, the pressure will be transmitted in a different way along the cylinder, thus allowing us to obtain a method for determining inherent friction between particles and condition at which the granular material will start to move. By applying force at different rates the GFA may be used for studying inherent friction between granular materials in dynamic-equilibrium as well. Such information is of importance for pharmaceutical industry in production of tablets, for example.

3.1 Working principle of GFA

Even though pressure distribution in axial direction is the final result that we seek from the apparatus, what it is being measured experimentally are strains in the tangential (ϵ_θ) and axial (ϵ_z) directions along the length of the cylinder. Fig. 2 show schematically the expected results for the strain evolution in a Newtonian fluid, and in a granular material, respectively. In the case of the fluid (Fig. 2a), it is known that the hydrostatic pressure is constant along the length of the cylinder and it is proportional to the tangential strain, whereas the axial strain occurs just due to Poisson effect. In the case of the granular material (Fig. 2b), it is expected that the pressure is maximal close to the piston where the force is applied, followed by a pressure drop. The pressure drop occurs because the force is distributed through layers of particles and there is formation of force chains that attenuate the pressure along the cylinder and increase the friction force between particles and inner wall of the cylinder [14,24]. In this case, the tangential strain is maximal at the position where the force is applied and drops in the same way as pressure does, whilst the axial strain increases as the tangential decreases because the force is been transmitted to the wall as an axial load due to friction. In powder technology the relation between axially applied pressure and pressure in radial direction is known as ‘Janssen effect’ [25]. For more details we refer the reader to the paper of Ovarlez et al. [26].

Fig. 2. Expected results for tangential and axial strain along the length of the cylinder of the GFA a) for a fluid and b) for a granular material

The pressure along the length of the cylinder can be calculated by assuming that the tangential strain measured, e.g., with strain gages, ($\varepsilon_{\theta(m)}$) comes from two sources: pressure and axial force due to friction, then it can be expressed as:

$$\varepsilon_{\theta(m)} = \varepsilon_{\theta(P)} + \varepsilon_{\theta(axial)} \quad (1)$$

Where ($\varepsilon_{\theta(P)}$) is the tangential strain due to pressure and ($\varepsilon_{\theta(axial)}$) is the tangential strain due to axial due to friction. If we consider that the tangential strain due to axial load $\varepsilon_{\theta(axial)}$ is the Poisson ratio (ν) times the axial strain that is measured in the axial direction ($\varepsilon_{z(m)}$), the following equation is obtained:

$$\varepsilon_{\theta(axial)} = \nu \varepsilon_{z(m)} \quad (2)$$

From Equation (1) and Equation (2), one can calculate the tangential strain due to pressure:

$$\varepsilon_{\theta(P)} = \varepsilon_{\theta(m)} - \nu \varepsilon_{z(m)} \quad (3)$$

The tangential strain due to pressure is used to calculate the internal pressure using the equation for a hydrostatic problem; the equation for hydrostatic problem is given as [27]:

$$\varepsilon_{\theta(P)} = \frac{1}{E} \frac{r_i^2}{r_0^2 - r_i^2} \left(1 + \frac{r_0^2}{r^2} \right) p_i \quad (4)$$

where E is the Young's modulus of the cylinder, r_i and r_0 are the inner and outer radii respectively, $r_i \leq r \leq r_0$, and p_i is the internal pressure. From Equation (4), the pressure can be extracted as a function of the tangential strain due to pressure inside the cylinder:

$$p_i = \frac{\varepsilon_{\theta(P)} E (r_0^2 - r_i^2)}{r_i^2 \left(1 + \frac{r_0^2}{r^2} \right)} \quad (5)$$

By introducing $\varepsilon_{\theta(P)}$ from Equation (3) into Equation (5) and considering that $r_0^2/r^2 = 1$ in our case because we always measure the strain at the outer face of the cylinder, yields the final equation to obtain the pressure along the axial direction of the cylinder as a function of the measured strains in tangential and axial direction:

$$p_i = \frac{(\varepsilon_{\theta(m)} - \nu \varepsilon_{z(m)}) E (r_0^2 - r_i^2)}{2r_i^2} \quad (6)$$

Fig. 3 demonstrates schematically the measuring capabilities of the GFA, i.e. evolution of pressure along the cylinder at a fixed force.

Fig. 3. Measuring capabilities of the GFA apparatus

Fig. 3 illustrates how one can compare the inherent friction properties two granular materials in the GFA, taking as reference the pressure profile of a fluid which is constant along the length of the cylinder, one can determine if certain powder has higher or lower inherent friction by analyzing how steep is the pressure drop along the length of the cylinder, i.e. a granular material with less pressure drop will lower inherent friction (behavior closer to liquid) than another with very rapid pressure drop. Based on the literature, it is expected that powders with a smaller average particle size will exhibit higher inherent friction [14,24]; for this reason Powder 1 in Fig. 3 has a much faster pressure drop than Powder 2.

3.2 GFA engineering solution

The GFA can be subdivided into two systems: (i) Mechanical system, and (ii) strain measuring system. The mechanical system of the apparatus shown on Fig. 4 is composed by the cylinder, piston, measuring plates and a fixation frame that supports and couples the previous components. In this case the strain measuring system is composed of strain gages that are glued on the external face of the cylinder and the completion circuit (1 dummy and 2 resistors placed on a "measuring plate" that is screwed to the fixation frame) that are required to complete the "quarter Wheatstone bridge with temperature compensation" that is used in all circuits. For higher precision, strain gages may be replaced with a more versatile optical strain measuring system.

To complete the strain measuring system, the Wheatstone bridges must be coupled to a signal conditioning system that powers the circuits and allows us to record the measuring signal.

Fig. 4. Exploded view of the GFA mechanical system

Fig. 5 shows the exact location of each strain gage with respect to the top part of the cylinder, for better understanding the cylinder is unfolded in this illustration. The distribution of gages was chosen to be concentrated at the top part of the cylinder where the strain is maximum so the expected drop of strain (tangential strain proportional to pressure) can be recorded at intermediate points before reaching the minimum value.

Fig. 5. Strain gages distribution along the cylinder length (cylinder is unfolded); all dimensions are in mm.

Fig. 6 shows schematically the complete setup of the GFA measuring process in a flow chart form. First a force is transmitted to the granular material by means of the piston, and this force induces an internal pressure that leads to strain in the outer face of the cylinder. When the strain gages in the cylinder are connected to a Wheatstone bridge a signal of voltage that is related to a strain in the cylinder is recorded. The force sensor and the Wheatstone bridges in the strain measuring system are connected to the amplifier/signal conditioning that powers the circuits and increases the output signal to a recordable level and minimizes the noise in the signal; finally the analog signal from the amplifier is converted into a digital format through a data acquisition (DAQ) card and then is stored in a PC through a LabVIEW (National Instruments, Austin, TX, USA) interface.

Fig. 6. Mechanical and strain measuring system of the GFA, flowchart of the measuring process

3.3. GFA Calibration

Granular materials present a complex behavior with solid and liquid-like behavior, therefore we cannot use any granular material to perform calibration of the apparatus. The calibration of the GFA must be done by performing a test in which the strain conditions of the problem are known: either by pressurizing a fluid inside the cylinder or by applying pure axial force. Since the sealing between the pressurizing piston and the cylinder is not sufficient for pressurizing liquids, the calibration was performed by exposing the cylinder to pure axial loading.

The axial load experiment is depicted in Fig. 7. In this test the empty steel cylinder is subjected to a known force in the axial direction. The strain in the axial direction $\varepsilon_{z(m)}$ is proportional to the applied force F over the contact area A , see Fig. 7, and the tangential strain $\varepsilon_{\theta(m)}$ is accounted through Poisson's ratio ν . The governing equations of this elastic problem are:

$$\varepsilon_{z(m)} = \frac{1}{E} \frac{F}{A} = \frac{F}{E\pi(r_0^2 - r_i^2)} \quad (7)$$

$$\varepsilon_{\theta(m)} = \nu \varepsilon_{z(m)} = \frac{F\nu}{E\pi(r_0^2 - r_i^2)} \quad (8)$$

Since the loading force, F , and the inner, r_i , and the outer, r_0 , radii of the cylinder are measured independently, and both strains are measured with strain gauges using 1/4 Wheatstone bridge with temperature compensation, one can use the two equations to determine Young's modulus, E , and Poisson's ratio, ν , of the material from which the measuring cylinder is manufactured.

$$E = \frac{F}{\varepsilon_{z(m)}\pi(r_0^2 - r_i^2)}, \quad (9)$$

$$\nu = \left| \frac{\varepsilon_{\theta(m)}}{\varepsilon_{z(m)}} \right| \quad (10)$$

Fig. 7 Axial Load Experiment

Taking into account the relation between the measured, V_{out} , and energizing, V_{in} , voltage and measured strain, $\varepsilon_{measured}$, for a quarter Wheatstone bridge [28],

$$\frac{V_{out}}{V_{in}} = \frac{S_g}{4} \varepsilon_{measured}, \quad (11)$$

one may express both measured strains as,

$$\varepsilon_{z(m)} = \frac{4V_{z,out}}{V_{in}S_{g,z}}, \quad (12)$$

$$\varepsilon_{\theta(m)} = \frac{4V_{\theta,out}}{V_{in}S_{g,\theta}}. \quad (13)$$

In view of this we may rewrite the Equations (9) and (10) as,

$$E = \frac{V_{in}S_{g,z}}{4V_{z,out}} \cdot \frac{F}{\pi(r_0^2 - r_i^2)} \quad (14)$$

$$\nu = \frac{\varepsilon_{\theta(m)}}{\varepsilon_{z(m)}} = \frac{V_{\theta,out}}{V_{z,out}} \cdot \frac{S_{g,z}}{S_{g,\theta}} \quad (15)$$

The accuracy of the Young's modulus, and Poisson's coefficient, ν , may be evaluated with parametric error analysis of the Equations (14) and (15),

$$\Delta E \cong \left| \frac{\partial E}{\partial V_{in}} \Delta V_{in} \right| + \left| \frac{\partial E}{\partial V_{z,out}} \Delta V_{z,out} \right| + \left| \frac{\partial E}{\partial S_{g,z}} \Delta S_{g,z} \right| + \left| \frac{\partial E}{\partial F} \Delta F \right| + \left| \frac{\partial E}{\partial r_0} \Delta r_0 \right| + \left| \frac{\partial E}{\partial r_i} \Delta r_i \right|, \quad (16)$$

and

$$\Delta \nu \cong \left| \frac{\partial \nu}{\partial V_{\theta,out}} \Delta V_{\theta,out} \right| + \left| \frac{\partial \nu}{\partial V_{z,out}} \Delta V_{z,out} \right| + \left| \frac{\partial \nu}{\partial S_{g,z}} \Delta S_{g,z} \right| + \left| \frac{\partial \nu}{\partial S_{g,\theta}} \Delta S_{g,\theta} \right|, \quad (17)$$

which leads to

$$\frac{\Delta E}{E} \cong \left| \frac{\Delta V_{in}}{V_{in}} \right| + \left| \frac{\Delta S_{g,z}}{S_{g,z}} \right| + \left| \frac{\Delta F}{F} \right| + \left| -\frac{\Delta V_{z,out}}{V_{z,out}} \right| + \left| -\frac{2r_0}{r_0^2 - r_i^2} \Delta r_0 \right| + \left| \frac{2r_i}{r_0^2 - r_i^2} \Delta r_i \right|, \quad (18)$$

and

$$\frac{\Delta \nu}{\nu} \cong \left| \frac{\Delta V_{\theta,out}}{V_{\theta,out}} \right| + \left| \frac{\Delta V_{z,out}}{V_{z,out}} \right| + \left| -\frac{\Delta S_{g,z}}{S_{g,z}} \right| + \left| -\frac{\Delta S_{g,\theta}}{S_{g,\theta}} \right|. \quad (19)$$

From Equations (12) and (13) we can similarly evaluate accuracy of measured strains,

$$\frac{\Delta \varepsilon_{z(m)}}{\varepsilon_{z(m)}} \cong \left| \frac{\Delta V_{z,out}}{V_{z,out}} \right| + \left| -\frac{\Delta V_{in}}{V_{in}} \right| + \left| -\frac{\Delta S_{g,z}}{S_{g,z}} \right| \quad (20)$$

and

$$\frac{\Delta \varepsilon_{\theta(m)}}{\varepsilon_{\theta(m)}} \cong \left| \frac{\Delta V_{\theta,out}}{V_{\theta,out}} \right| + \left| -\frac{\Delta V_{in}}{V_{in}} \right| + \left| -\frac{\Delta S_{g,\theta}}{S_{g,\theta}} \right| \quad (21)$$

Calibration was performed by loading cylinder 30 times using different forces. Strain in the axial and tangential directions was measured at three points of the cylinder and averaged. Accuracies of the measuring sensors that constitute the equipment, i.e. load cell, powering system and strain gages; were taken from the corresponding data sheets provided by their manufacturer. Deviation of the inner and outer radii of the cylinder were measured 30 times at different positions. Table 1 lists the accuracies of all measured quantities.

Table 1. Accuracy of measured quantities

Geometry and force					
F [N]	4424.93	ΔF [N]	16.99	$\Delta F/F$	0.0038
r_0 [mm]	10	Δr_0 [mm]	0.01	$\Delta r_0/r_0$	0.0010
r_i [mm]	8	Δr_i [mm]	0.01	$\Delta r_i/r_i$	0.0013
Axial direction (z direction)					
V_{in} [V]	4.9	ΔV_{in} [V]	0.01	$\Delta V_{in}/V_{in}$	0.002
$V_{z,out}$ [V]	0.000474	$\Delta V_{z,out}$ [V]	0.000015	$\Delta V_{z,out}/V_{z,out}$	0.032
$S_{g,z}$	2	$\Delta S_{g,z}$	0.02	$\Delta S_{g,z}/S_{g,z}$	0.01
Tangential direction (θ direction)					
$V_{\theta,out}$ [V]	0.000157	$\Delta V_{\theta,out}$ [V]	0.000006	$\Delta V_{\theta,out}/V_{\theta,out}$	0.041
$S_{g,\theta}$	1.99	$\Delta S_{g,\theta}$	0.019	$\Delta S_{g,\theta}/S_{g,\theta}$	0.01
Calculated quantities					

E [MPa]	202209	ΔE [MPa]	11645	$\Delta E/E$	0.0576
ν	0.332	$\Delta \nu$	0.03065	$\Delta \nu/\nu$	0.0923
$\varepsilon_{z(m)}$	-0.0001935	$\Delta \varepsilon_{z(m)}$	-0.00000846	$\Delta \varepsilon_{z(m)}/\varepsilon_{z(m)}$	0.0437
$\varepsilon_{\theta(m)}$	0.0000642	$\Delta \varepsilon_{\theta(m)}$	0.00000338	$\Delta \varepsilon_{\theta(m)}/\varepsilon_{\theta(m)}$	0.0526

The final step of the calibration is to determine the accuracy of the computed pressure that is calculated from tangential and axial strains, thus both strains contribute to the accuracy. Such accuracy can be evaluated from the parametric error analysis of the Equation (6),

$$\Delta p_i \cong \left| \frac{\partial p_i}{\partial \varepsilon_{\theta}} \Delta \varepsilon_{\theta(m)} \right| + \left| \frac{\partial p_i}{\partial \varepsilon_z} \Delta \varepsilon_{z(m)} \right| + \left| \frac{\partial p_i}{\partial \nu} \Delta \nu \right| + \left| \frac{\partial p_i}{\partial E} \Delta E \right| + \left| \frac{\partial p_i}{\partial r_0} \Delta r_0 \right| + \left| \frac{\partial p_i}{\partial r_i} \Delta r_i \right|, \quad (22)$$

which leads to

$$\frac{\Delta p_i}{p_i} \cong \left| \frac{\Delta \varepsilon_{\theta(m)}}{\varepsilon_{\theta(m)} - \nu \varepsilon_{z(m)}} \right| + \left| -\frac{\nu \cdot \Delta \varepsilon_{z(m)}}{\varepsilon_{\theta(m)} - \nu \varepsilon_{z(m)}} \right| + \left| \frac{\varepsilon_{z(m)} \cdot \Delta \nu}{\varepsilon_{\theta(m)} - \nu \varepsilon_{z(m)}} \right| + \left| \frac{\Delta E}{E} \right| + \left| \frac{2r_0 \cdot \Delta r_0}{r_0^2 - r_i^2} \right| + \left| -\frac{2r_0^2 \cdot \Delta r_i}{r_i (r_0^2 - r_i^2)} \right| \quad (23)$$

Using measured and calculated values from Table 1 accuracy of pressure was calculated and results are shown in Table 2, where also contribution of each parts of Equation (23) to the total error is shown.

Table 2. Accuracy of calculated quantities for pressure

	$\Delta p_i/p_i$	$\Delta p_i/p_i$ in %	Contribution of error to total error in %
$\left \frac{\Delta \varepsilon_{\theta(m)}}{\varepsilon_{\theta(m)} - \nu \varepsilon_{z(m)}} \right $	0.0263	2.63	16.70
$\left -\frac{\nu \cdot \Delta \varepsilon_{z(m)}}{\varepsilon_{\theta(m)} - \nu \varepsilon_{z(m)}} \right $	0.0219	2.19	13.87
$\left \frac{\varepsilon_{z(m)} \cdot \Delta \nu}{\varepsilon_{\theta(m)} - \nu \varepsilon_{z(m)}} \right $	0.0462	4.62	29.30

$\left \frac{\Delta E}{E} \right $	0.0576	5.76	36.54
$\left \frac{2r_0 \cdot \Delta r_0}{r_0^2 - r_i^2} \right $	0.0056	0.56	3.52
$\left -\frac{2r_0^2 \cdot \Delta r_i}{r_i(r_0^2 - r_i^2)} \right $	0.0001	0.01	0.07
Total	0.1576	15.76 %	100

Results from Table 2 show that in this case the error in measured pressure is 16%, where the highest contribution to total error represents error from E modulus with 36%, followed by Poisson's ratio (ν) with 30%, the contribution of tangential strain (ε_θ) and axial strain (ε_z) to total error is with 17% and 14%. Error coming from geometry (r_0 and r_i) is responsible for minor part of error with approximately 4%.

4 GFA apparatus performance demonstration

For the purpose of GFA apparatus performance demonstration five different materials provided by BASF (Germany) were analyzed: 4 stainless steel powders and 1 type of polyoxymethylene (POM) pellets. In Table 3, their average particle size is shown. In this demonstration we limit ourselves to study the effect of the average particle size only. Behavior of granular material is of course affected by grains size distribution, grains shape, grains surface roughness and cohesive forces which however are not the subject of the current paper.

Table 3. Average particle size of materials tested

Material Identification	Type of material	Average particle size [μm]
Fraction I	Stainless steel 316LW spherical powder	41,7
Fraction II		23,7
Fraction III		20
Fraction IV		6,8

POM	Polyoxymethylene copolymer spherical pellets	4000
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4.1 Methodology of experiments

The measuring procedure is divided in 4 stages. First, the powder sample should be prepared and consolidated by tapping the cylinder. It is important to point out the importance of sample conditioning as part of the sample preparation process in order to prevent excess of air in between the particles. To release the excess of air from the sample and yield the same arrangement of particles inside the cylinder, tapping of the cylinder is necessary during and after filling the cylinder with powder: this operation must be done as repeatable as possible. The second stage consists of placing the filled cylinder into the frame of the apparatus, and balancing of the circuits. During the third stage, the test is performed by applying the load. The final stage is the cleaning of the cylinder after the test.

Before performing any test, one has to determine the right amount of powder sample to be used during the experiments, this is done by performing preliminary experiments that follow the same steps described, but with the only difference that the amount of powder is changed until satisfying the condition that the position of the piston after applying the desired load is about 17 mm above the first measuring point (in our case 103.5 mm from the top of the cylinder). In order to determine the height to which the powder should be loaded, Finite Element Analysis (FEA) was performed. The analysis was performed by applying 30 MPa of hydrostatic pressure along the distance of 225 mm, measured from the bottom of a 300 mm stainless steel cylinder, as shown in Fig. 8. From FEA, it was observed that due to the fixed end at the bottom of the cylinder, there is an area of tangential strain instability at about 20 mm from the bottom of the cylinder and at about 15 mm below the point where the hydrostatic pressure is being applied. Therefore, the distance of 17 mm from the first measuring point was chosen in order to avoid the instability zone.

Fig. 8. Tangential strain at external surface versus length for measuring cylinder

All materials were tested 10 times following the described protocol. In order to artificially increase the number of measuring points along the length of the cylinder, the amount of powder sample was deliberately changed and in this particular case two different distances were used as schematically shown on Fig. 9. When determining the measuring position the minimal distance of 15 mm from the first measuring point has to be taken into account in order to avoid the instability zone. Using this procedure pressure profile curves composed of 6 points instead of just 3 were obtained. The force that was applied during the tests was $F = 4428 \pm 25 \text{ N}$ corresponding to a maximum pressure $p = 22.025 \pm 0.125 \text{ MPa}$.

Fig. 9. Position of piston for determination of pressure profiles

4.3 The GFA index

The inherent friction of the granular material is represented by axial distribution of pressure within the measuring cylinder. If the cylinder is filled with a Newtonian fluid the pressure would be uniform throughout the volume of the measuring cylinder, i.e., constant along the axial direction, as schematically shown in Fig. 3. On the other hand, when the cylinder is filled with a granular material pressure distribution is not constant, it is changing along the axial direction of the cylinder, as shown in Fig. 3. For easy comparison pressure distribution along the axial direction of the cylinder may be normalized by the pressure, which would be generated within the cylinder if it would be filled with a Newtonian fluid, hence $p_{NF} = F / A$. Such two situations are shown more vividly in Fig. 10. As a measure of the inherent friction we may define a parameter, which we will call “GFA” index, and it is defined as

$$GFA_{index} = \frac{S_{granular}}{S_{fluid}} = \frac{\int_0^L p(z) dz}{p_{NF} \cdot L} = \frac{A}{FL} \cdot \int_0^L p(z) dz, \quad (24)$$

where $S_{granular}$ is the area of the pressure profile of a granular material, and S_{fluid} is the area of the pressure profile corresponding to a Newtonian fluid, F is the applied force, A is the inner cross section area of the cylinder, $p(z)$ is the pressure distribution along the cylinder and L is an agreed upon “standardized” length of the cylinder. In our case we have used $L = 188 \text{ mm}$.

The maximum value of the "GFA index" is 1 and indicates that tested granular material does not exhibit any inherent friction as a Newtonian fluid. As the "GFA index" decreases it indicates amplification of inherent friction in a granular material. In this case the area may be calculated by using trapezoidal rule, as it is shown in Fig. 10 where the total area of the curve corresponds to the sum of 6 trapezoids that are formed from the number of measuring points used to calculate the pressure profile. By increasing the number of measured points determination of the GFA index will be of course improved.

Fig. 10. Schematic illustration and definition of the "GFA index"

5 Results and discussion

Following the methodology described in section 4, the pressure profiles of all metallic powders and POM material were measured. In line with the definition of GFA index measured pressures were normalized by the maximal pressure, which corresponds to the applied force divided by the cross section area of the cylinder. The maximum pressure varied slightly from test to test within the range from 21.9 to 22.2 MPa.

Fig. 11a shows the "GFA index" for five granular materials as function of the average particle size. In Fig. 11a we have used logarithmic scale for the particle size in order to show all five results on the same diagram. Fig. 11b shows the "GFA index" for four metallic granular materials only. Here the particles size coordinate is shown in linear scale. The results shown represent average of ten measurements, and the corresponding variance was calculated by using Student's t-distribution.

From the results one can observe that Fraction IV presents the lowest "GFA index" which indicates that it has the highest inherent friction. This agrees with [14,17,24] where authors state that as particle size decreases cohesion increases and thus inherent friction increases. Fractions III and II present almost the same GFA index and Fraction I has the highest "GFA index" indicating the lowest inherent friction among the metal powders. Despite the large experimental error observed in measurement on POM, the results evidence that POM have much lower inherent friction with respect to metal powders because of the lower friction between the POM pellets. Large experimental error in POM measurements may be accounted to unfavorable ratio between the size of the POM pellets and the inner diameter of the testing cylinder.

We can observe that the value of the "GFA index" of POM is approximately 3 times higher than the value of the "GFA index" for the metallic powders, however, it is important to notice that the difference in average particle size is more than 100 times, see Table 3!

Fig. 11. a) GFA index for metal powders and POM, b) GFA index for metal powders (close up look)

5.1 Modeling of the GFA index

Inherent friction of granular materials depends on a type of material, topology of particles, and their size. Since particles of all metallic granular materials were spherical we may try to find a model describing the interrelation between their inherent friction, expressed with GFA index, and average particle size. In general the interrelation may be exponential or polynomial. To determine this we need to plot the data in a double logarithmic coordinate system where polynomial interrelations become linear functions, so the function,

$$y = a_n x^n + a_{n-1} x^{n-1} + \dots + a_1 x + a_0 \approx a_n x^n, \quad (14)$$

Then becomes

$$\log y = \log a_n + n \log x \quad (15)$$

For our data this is shown in Fig. 12, where experimental data are shown as squares.

Fig. 12. Log-log plot of average particle size and GFA index

Since the relation between the average particle size and the GFA index in a double logarithmic coordinate system appears to be linear we may conclude that the interrelation between the GFA index and corresponding average particle size may be expressed as

$$GFA = a \cdot \Theta^n, \quad (25)$$

or

$$\log GFA = \log a + n \log \Theta \quad (26)$$

where a and n are constants and Θ denotes the average particle size of analyzed metallic granular materials. By fitting the data, shown in Fig. 12, with a linear function we find that $\log a = -1.00$ and $n = 0.09$, and

$$GFA = 0.1 \cdot \Theta^{0.09} \quad (27)$$

In a first approximation we may consider that $a \cong n \cong 0.1$, which leads to interesting relation between the GFA index and average particle size of granular materials

$$GFA = 0.1 \cdot \Theta^{0.1} \quad (28)$$

Fig. 13. GFA index and particle size for four metal fractions. Solid line represents GFA model, points represent measured values

Comparison of experimental data and the analytical prediction using the two models are presented in Figs 12 and 13 as dashed line for the model shown in Equation (27) and as a solid line for the model given in Equation (28). It is interesting to see that in linear scale, Fig. 13, both models fit the data reasonably well. However in double logarithmic scale, one clearly sees that the model shown in Equation (28) fails completely. This indicates that parameters a and n are not the same.

The new GFA apparatus and the proposed analytical model represent an interesting approach for studying the inherent friction of granular materials. In particular, it would be interesting to understand how a and n depend on material properties, grains size distribution, grains shape, grains surface roughness and cohesive forces.

6 Conclusions

Proposed GFA apparatus proves to be an interesting tool for studying the inherent friction of granular materials exposed to pressure loading and conditions at which granular material will start to flow. It consists of a hollow cylinder where granular materials can be pressurized by means of a piston; the pressure drop in axial direction of the cylinder is estimated by measuring strains at the outer surface of the

cylinder along its length. The GFA apparatus was calibrated by performing experiments where the axial load is known without powder and the maximum error in measured strain was estimated to be 15.76% at the applied force 4450 N. From the parametric error analysis of Young's modulus, E , Poisson's ratio, ν , and internal pressure, p_i , it can be seen that most of the error in the estimated pressure comes from the strain measurements, therefore it is important to measure strains at the outer surface of the cylinder more accurately, say by using one of the optical methods, such as speckle interferometry.

A new parameter, GFA-index, used to assess the inherent friction of different granular materials has also been introduced. The GFA-index is the ratio of the integral of the pressure distribution along the agreed upon "standardized" length of the cylinder, L , divided by the integral over the same distance assuming that pressure is constant, which is the case if cylinder would be filled with a Newtonian fluid.

The GFA prototype was utilized to test five fractions of stainless steel 316LW and granules of polyoxymethylene of different average particle size in order to demonstrate the concept and capabilities of the apparatus. Obtained results proved the simplicity and power of the GFA apparatus. Obtained results were in line with the data from the literature. However, demonstration experiments have shown that measuring strains at only few locations is not sufficient, which suggests that further work is required to improve the accuracy of this first GFA prototype.

Important recommendations towards improving the accuracy of the GFA include: standardization of conditions during the sample preparation (such as powder filling and tapping of the cylinder); the implementation of a sensor to control the displacement of the piston during the test; and measuring strains at the outer surface with an optical method that allows obtaining the complete pressure profile along the cylinder instead at just three positions. The most appealing alternative is to use speckle interferometry.

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7 References

- [1] H.M. JAEGER, S.R. NAGEL, Physics of the Granular State, *Science* (80-.). 255 (1992) 1523–1531. doi:10.1126/science.255.5051.1523.
- [2] I. Emri, B.S. von Bernstorff, F. Brehmer, A. Kalamar, M. Bek, P. Oblak, Sleeper with damping element based on dissipative bulk or granular technology, EP2700838, 2012.
- [3] I. Emri, B.S. von Bernstorff, Dissipative bulk and granular systems technology, EP2700839, 2012.
- [4] N. Tschoegl, W. Knauss, I. Emri, The effect of temperature and pressure on the mechanical properties of thermo-and/or piezorheologically simple polymeric materials in thermodynamic equilibrium - A critical review, *Mech. Time-Dependent Mater.* 6 (2002) 53–99. doi:10.1023/A:1014421519100.
- [5] I. Emri, J. Gonzalez-Gutierrez, M. Gergesova, B. Zupančič, I. Saprunov, Experimental Determination of Material Time-Dependent Properties, in: R. Hetnarski (Ed.), *Encycl. Therm. Stress.*, Springer Netherlands, 2014: pp. 1494–1510. doi:10.1007/978-94-007-2739-7_907.
- [6] N. Vlachos, I.T.H. Chang, Investigation of flow properties of metal powders from narrow particle size distribution to polydisperse mixtures through an improved Hall-flowmeter, *Powder Technol.* 205 (2011) 71–80. doi:10.1016/j.powtec.2010.08.067.
- [7] D. Geldart, E.C. Abdullah, a. Hassanpour, L.C. Nwoke, I. Wouters, Characterization of powder flowability using measurement of angle of repose, *China Particuology.* 4 (2006) 104–107. doi:10.1016/S1672-2515(07)60247-4.
- [8] K. Shinohara, B. Golman, Dynamic shear properties of particle mixture by rotational shear test, *Powder Technol.* 122 (2002) 255–258. doi:10.1016/S0032-5910(01)00422-3.
- [9] 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 Technol.* 174 (2007) 25–33. doi:10.1016/j.powtec.2006.10.016.
- [10] J. Prescott, R. Barnum, On powder flowability, *Pharm. Technol.* 24 (2000) 60–84. citeulike-article-id:11996698\nhttp://cat.inist.fr/?aModele=afficheN&cpsidt=795318.
- [11] K. Traina, R. Cloots, S. Bontempi, G. Lumay, N. Vandewalle, F. Boschini, Flow abilities of powders and granular materials evidenced from dynamical tap density measurement, *Powder Technol.* 235 (2013) 842–852. doi:10.1016/j.powtec.2012.11.039.
- [12] J.A. Moreno López, I. Emri, Effect of Particle Size on the Flow Ability of Micro-sized Stainless Steel 316LW Powder Analyzed with a New Granular Flow Apparatus, J. A. Moreno Lopez, 2012.
- [13] D. Bregant, I. Emri, Development and construction of the measuring device for analysing micro powders flowability, University of Ljubljana, Faculty of Mechanical Engineering, 2013.

- [14] C.S. Campbell, Granular material flows - An overview, *Powder Technol.* 162 (2006) 208–229. doi:10.1016/j.powtec.2005.12.008.
- [15] N. Jesuthasan, B.R. Baliga, S.B. Savage, Use of Particle Tracking Velocimetry for Measurements of Granular Flows: Review and Application, *KONA Powder Part. J.* 24 (2006) 15–26. doi:10.14356/kona.2006006.
- [16] T. Börzsönyi, R.E. Ecke, J.N. McElwaine, Patterns in flowing sand: Understanding the physics of granular flow, *Phys. Rev. Lett.* 103 (2009) 1–4. doi:10.1103/PhysRevLett.103.178302.
- [17] D. Schulze, *Powders and bulk solids: behavior, characterization, storage and flow*, Springer Science & Business Media, 2007.
- [18] G. Raymus, Handling of Bulk Solids and Packaging of Solids and Liquids, in: Green D.W. ; Maloney J.O. (Ed.), *Perry's Chem. Eng. Handb.*, 7th ed., McGraw-Hill, New York, 1997.
- [19] G. Lumay, F. Boschini, K. Traina, S. Bontempi, J.-C. Remy, R. Cloots, et al., Measuring the flowing properties of powders and grains, *Powder Technol.* 224 (2012) 19–27. doi:10.1016/j.powtec.2012.02.015.
- [20] W.P. Schellart, Shear test results for cohesion and friction coefficients for different granular materials: Scaling implications for their usage in analogue modelling, *Tectonophysics.* 324 (2000) 1–16. doi:10.1016/S0040-1951(00)00111-6.
- [21] A.D. Orlando, H.H. Shen, Effect of particle size and boundary conditions on the shear stress in an annular shear cell, *Granul. Matter.* 14 (2012) 423–431. doi:10.1007/s10035-012-0313-8.
- [22] D. Schulze, Round robin test on ring shear testers, *Adv. Powder Technol.* 22 (2011) 197–202. doi:10.1016/j.appt.2010.10.015.
- [23] Z. Ma, K. Ravi-Chandar, Confined compression: A stable homogeneous deformation for constitutive characterization, *Exp. Mech.* 40 (2000) 38–45. doi:10.1007/BF02327546.
- [24] K.K. Rao, P.R. Nott, *An Introduction to Granular Flow*, Cambridge University Press, New York, 2008. doi:http://dx.doi.org/10.1017/CBO9780511611513.
- [25] H.A. Janssen, Versuche über getreidedruck in silozellen, *Zeitschr. D. Vereines Dtsch. Ingenieure.* 39 (1895) 1045–1049.
- [26] G. Ovarlez, C. Fond, E. Clément, Overshoot effect in the Janssen granular column: a crucial test for granular mechanics, *Phys. Rev. E.* 67 (2003) 60302. doi:http://dx.doi.org/10.1103/PhysRevE.67.060302.
- [27] R.G. Budynas, J.E. Shigley, *Shigley's Mechanical Engineering Design*, 8th ed., McGraw-Hill Education, 2008.
- [28] R. Watson, Bonded Electrical Resistance Strain Gages, in: W.N. Sharpe Jr. (Ed.), *Springer Handb. Exp. Solid Mech.*, Springer US, 2008: pp. 283–334. doi:10.1007/978-0-387-30877-7_12.