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# INVESTIGATION OF POWDER PROPERTIES USING ALTERNATING STRAIN PATHS

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**Abstract:** Steady-state flow (ssf) of powders has been investigated using alternating strain paths, with precompacted powder samples sheared in alternating directions. The dependency of ssf on the level of precompaction is shown.

Keywords: powder, biaxial shear tester, steady flow, precompaction strain path

### 1. Introduction

In powder mechanics, engineers need material models of the bulk solids they want to handle, in order to design silos and discharge units. The constitutive, macroscopic models often used today make the analytic design of simple geometries or design by Finite Element Methods (FEM) possible for many bulk solids. Still, calculation based on the Janssen equation [1] are carried out nowadays in order to predict wall pressures in silos. This equation is based on simple, macroscopicmechanical assumptions. Jenike has introduced the flow function for bulk solids [2]. The Jenike method is state of the art for silo design in process engineering. With his method, Jenike has developed a shear tester the usage of which is an established and internationally defined standard [3]. For each bulk solid, shear tests are carried out to determine the required bulk solid parameters.

Especially, for the handling of very cohesive bulk solids, predictions of their behaviour are needed which include time dependency and cohesion. Since the "oldfashioned" engineering models in process engineering yield exaggerated dimensions, better predictions are now expected from "modern" Discrete Element Methods (DEM). These methods vary considerably. Some prescribe the microstructure of the bulk solid before the calculation (*e.g.* Cellular Automata [4]). However, the popular molecular dynamics method [5] computes the bulk solid's structure considering the deformations of each particle-particle contact as the source of interparticular forces. Conversely, the contact dynamics method [6] determines these forces on the basis of their mechanical effects (to prevent penetration to keep stability of the ensemble). All of them share the restriction to a number of particles (currently about one million). For a cohesive micron-sized bulk solid with a void ratio of about 70%, one needs about  $10^{14}$  particles to simulate a representative volume used in shear tests. Therefore, the DEM will not replace continuum mechanical models in our days. But we can expect them to contribute to our understanding of – for example – compaction, creeping, relaxation and steady-state flow.

Whichever DEM one chooses, its predictions can only be as good as the calibrating experiments carried out on an actual bulk solid. The true biaxial shear tester of the Institute of Mechanical Process Engineering of the Technical University of Braunschweig is a device which has already been successfully used for the further development and validation of an existing continuum mechanical model for bulk solids [7]. It also allows us to investigate of steady-state flow of bulk solids [8] and time dependent effects such as relaxation and creeping [9]. For all these phenomena, macroscopic observations and descriptions exist but an understanding of the microscopic process is still missing. Therefore, our true biaxial shear tester has been redesigned and extended. Equipped with more precise technology, it now also allows us to calibrate microscopic models. Thus, our true biaxial shear tester may serve as a link between the microscopic (DEM) and the macroscopic (continuum mechanical) description of bulk solid behaviour.

# 2. Description of the test device

2.1. The true biaxial shear tester



Figure 1. The TUB true biaxial shear tester

The true biaxial shear tester allows the deformation of a brick-shaped bulk solid specimen. The deformation mechanism is composed of a bottom, a top plate and four side plates, which can be moved independently but always stay perpendicular to the adjacent ones. The bottom and top plates are fixed at a distance of 36mm from each other. The lateral load plates can be moved towards each other from a maximum distance of 130mm to a minimum distance of 70mm (Figure 1). The movements of the lateral load plates are produced by four synchronized motors. Their rotational speed of maximum 4000rpm is reduced by a factor of 100 with a cycloid gearing.

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Figure 2. Process control strategy of biaxial shear tests

Then a high-precision thread converts the rotational movement into translatoric one with a maximum speed of 66 microns per second. Due to this design, maximum shear rates of 0.1 per cent per second can be reached. Via four servo amplifiers, the velocity can be set in steps of 0.03 microns per second. This corresponds to an increment of the strain rate by about  $3 \cdot 10^{-5}$  per cent per second. The movements of each load plate are measured with a photo-optical gauge which can measure relative displacements of less than 0.2 microns. On the specimen's border, the complete stress state is measured with three five-component load cells (LC 1-3), which are installed in the bottom and in the two perpendicular load plates. An extension to six load cells is planned. The servo amplifiers are controlled by a process computer, which is also used for data acquisition, conversion and recording. The LabView<sup>TM</sup>-based control program is drawn in Figure 2. During tests, silicon grease is spread on the bottom, lateral and top plates of the tester. They are then covered with highly flexible rubber membranes in such a way that the membranes are deformed evenly with the borders of the powder specimen. The friction between the specimen and the load plates is thus minimised and no significant shear stresses comparable to the normal stresses can be observed. Therefore, the normal stresses on the specimen borders are principal stresses. The principal stress axes coincide with the deformation axes.

#### 2.2. The model bulk solid

The mechanical properties of several powders have been investigated using the true biaxial shear tester, such as limestone [8]. In this paper, we will also show experimental data obtained with carbonyl iron powder (CIP). The prefix "carbonyl" indicates the production of this powder by thermal decomposition of iron pentacarbonyl to pure iron. During the decomposition process, spherical particles develop (see Figure 3) forming shell-like layers around nuclei. We use a batch of about-2micron-sized primary particles. We have chosen this powder, with its spherical and stiff primary particles, in order to simplify DEM simulations of the experiments.



Figure 3. SEM photographs of Carbonyl Iron Powder (CIP), photograph width: left  $-30\,\mu m$ , right  $-9\,\mu m$ 

## 3. Basis of the test procedure

As described above, the measured results obtained with our true biaxial shear tester shall also form the basis for other research groups in the field of powder mechanics. Therefore our definitions have to be very clear and intelligible in all aspects of the test process.

## 3.1. Test process control

The process data are given by a process file which has to be assembled before the beginning of the test. The file is structured in three sections: "test info", "process section" and "legend". For each section, test type and the relevant measurement and regulation parameters are defined. The LabView<sup>TM</sup>-based measure and control program then reads the process file and performs the test (Figure 2). For each test type, its own process file has to be programmed, reusing the existing program modules. Thus, the program is kept very flexible and can be extended for future tasks.

#### 3.2. Strain control

All test procedures presented in this paper are strain controlled, *i.e.* strains or strain rates are set for the test procedure. The tests are stopped once a given strain level is reached. The distances between every two opposite load plates at the beginning of the test,  $L_{x_0}$ ,  $L_{y_0}$  and  $L_{z_0}$ , are known, just as the actual distances  $L_x(t)$ ,  $L_y(t)$  and  $L_z(t)$  at an instant t,  $L_z(t)$  being equal to  $L_{z_0}$  for any t. Since, in bulk solid mechanics, investigations are carried out for finite strains, we do not use the conventional strain:

$$\varepsilon_{\rm conv} = \frac{L - L_0}{L_0},$$

which is less convenient than the natural strain, first introduced by Ludwik [10] with the natural-strain increment  $d\varepsilon = \frac{dL}{L}$  for the following reason. The increment  $d\varepsilon$  – also called "effective specific strain increment" – describes the change in length related to the actual length. Integrating this term we obtain:

$$\varepsilon = \int_{L_0}^{L} \frac{dL}{L} = \ln \frac{L}{L_0} = \ln(1 + \varepsilon_{\rm conv}).$$

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We define respectively the volume strain increment:

$$d\varepsilon_V = \frac{dV}{V},$$

and obtain:

$$\varepsilon_V = \ln \frac{V}{V_0} = \ln \frac{(L_x L_y L_z)}{L_{x_0} L_{y_0} L_{z_0}} = \varepsilon_x + \varepsilon_y + \varepsilon_z$$

for the volume strain.

Since we do not neglect – as is done when using conventional strain – higher order terms of the strains, the definition of natural strain allows for easier handling in the framework of strain-controlled tests at constant volumes. Our maximum deformations usually do not exceed 30%, corresponding to a difference  $\varepsilon - \varepsilon_{\rm conv}$  of about 4%.

## 3.3. Test evaluation

Stresses and strains are recorded every 0.1 seconds. We will display the data by either plotting stresses and strains versus time or plotting stress and strain paths, *i.e.* plotting  $\sigma_y(t)$  versus  $\sigma_x(t)$  and, respectively,  $\varepsilon_y(t)$  versus  $\varepsilon_x(t)$ . As  $L_z(t) = \text{const.}$ and thus  $\varepsilon_z(t) = 0$ ,  $\varepsilon_z(t)$  is not plotted. Compressive strains, as well as compressive stresses, will be defined positive.

## 4. Steady-state flow investigation

Apart from investigating the process of compaction [11], we want to pay special attention here to the investigation of the process of steady-state flow (ssf). This process has been investigated macroscopically [8, 12, 13], but the microscopic, physical explanation is still missing. Macroscopically, steady-state flow is characterised by constant (steady) stresses, whereas microscopically it ought to show (non-steady) particle-particle contacts moving, breaking up and rebuilding.



Figure 4. Steady-state flow investigation of limestone [8]

Steady-state flow of fine cohesive limestone powder has already been investigated using the true biaxial shear tester in its former configuration [8]. The bulk solid specimen was compressed with the ratio  $\frac{\dot{\varepsilon}_x}{\dot{\varepsilon}_y} = 1$  (or other ratios of 0, 0.5, 2) until a volume strain  $\varepsilon_{Vssf}$  was reached (see Figure 4, point 1). Then, the specimen was deformed while respecting the condition  $\varepsilon_x + \varepsilon_y = \varepsilon_{Vssf}$ , *i.e.* keeping the





specimen's volume constant. After a short period of deformation (point 2), constant principal stresses  $\sigma_{I,II\,ssf} = f(\varepsilon_{V\,ssf})$  appeared (point 3). Their values were independent of the choice of the ratio  $\frac{\dot{\varepsilon}_x}{\dot{\varepsilon}_y}$  and they remained approximately constant (steady-state, point 3).

Whether  $\sigma_x$  or  $\sigma_y$  becomes the first principal stress  $\sigma_I$  depends only on the direction of the shearing ( $\dot{\varepsilon}_x > 0$  or < 0) during the shearing phase at  $\varepsilon_{V ssf}$ . Similar observations could be made with Carbonyl Iron Powder (see Figure 5). After a very short shearing period (between points 1 and 2) steady-state flow can be observed. In

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Figure 8. Strains (left) and stresses (right) versus the time



Figure 9. First steps of alternating shearing with higher precompaction



Figure 10. Alternating shearing with higher precompaction

order to extend our tests, we now do not stop the test, as seen in Figure 4, at  $\varepsilon_x \approx \varepsilon_{V ssf}$ and  $\varepsilon_y \approx 0$ , but change the direction of shearing before, the specimen volume still remaining constant, see Figure 6. In this direction, we shear again until constant stresses  $\sigma_{I,II\,ssf}$  are reached (already before having reached  $\varepsilon_x \approx 0$  and  $\varepsilon_y \approx \varepsilon_{V\,ssf}$ ). Then the direction of shearing is changed again, Figure 7. It can be observed that although reaching steady-state flow in the first shearing period, it becomes more and more difficult to reach in the following periods, at this compaction level. The stress maxima even decrease with each step, as can be seen in Figure 8 (see also [14]). In the test described so far, the sample was compacted up to 20kPa before shearing. In a new test, the compaction level in z-direction is made higher in order to reach a higher stress level in x- and y-direction. The sample is now compacted up to 35kPa before shearing, Figure 9. As can be seen, steady-state flow is reached in the second period. Even in the following periods of alternating shearing, the same stress levels of steady-state flow are reached, Figure 10.

## 5. Conclusion

The true biaxial shear tester is a suitable tool for fundamental research in powder mechanics. The choice of carbonyl iron powder as the test substance allows one to investigate the steady-state flow of powders, already known for other bulk solids such as limestone. Comparison of the experiments presented here with discrete element simulations is facilitated by the choice of this powder [15]. Steady-state flow of carbonyl iron powder can be observed at the beginning of shearing, but if one wants to reproduce steady-state flow also after changing the direction of shear, a certain level of precompaction has to be reached. Then, steady-state flow can be observed during several phases of alternating shearing.

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