Microstructural changes, particle tracking and shear localization of fine glass powders

Lisa Handl, Ulm University; Dr. **Lutz Torbahn**, TU Braunschweig; **Aaron Spettl**, Ulm University; Dr. **Harald Zetzener**, TU Braunschweig; Prof. **Volker Schmidt**, Ulm University; Prof. **Arno Kwade**, TU Braunschweig;

1. Introduction

Changes in the microstructure of bulk solids affected by external stresses are relevant for any type of processing, storing and transportation of powders in industry and science. Here, the shear deformation strongly affects the microstructure of powders, and hence, the arrangement of particles and contacts between them. This information is important regarding the movement and mobility of single particles and particle assemblies, and contributes to increase the understanding of particle motion within zones of shear deformation and the formation of shear bands. In this study a micro-shear tester is considered which is implemented in an X-ray computed tomography device to obtain high-resolution 3D images of a fine glass powder under shearing. Particles are segmented by means of image analysis and tracked over a large period of time, which provides detailed information on the behavior of single particles and the formation of a shear band based on experimental data.

2. Material and Methods

Experiment

The particles used in this study are slightly adhesive solid microspheres made of borosilicate glass (BSGMS, CoSpheric LLC, USA) and have a diameter range of 27-32 µm according to manufacturer. For our investigation we use the micro-shear tester developed in [1], which has a cylindrical sample chamber with a diameter of 2 mm and can handle very small sample volumes (6 µL). Its outer wall is formed by a borosilicate glass capillary with a thickness of 50 µm and it is confined by two structural plastic pistons at both the top and bottom. In addition, the entire shear cell is implemented in an X-ray microtomograph (XMT), which allows for detailed 3D imaging of shearing experiments. For the experiment investigated in this study, the particles are sieved into the sample chamber and the normal load is increased up to 0.5 kPa. Then, while keeping the normal load constant, the upper piston and wall are rotated iteratively in steps of 0.5° up to 10° and further, up to an overall shearing angle of 40°, in steps of 5°. After each step the shear cell is imaged with tenfold magnification using the XMT (acceleration voltage: 50 keV, current intensity: 200 µA), resulting in a series of 3D images of the entire shear cell with a resolution of 2.2 µm per voxel side length.

Analysis

Particle positions are extracted from the 3D image data using a watershed segmentation algorithm and represented in cylinder coordinates, (r, φ, z). We use a marker-based watershed transform, where markers are determined by the local maxima of the grayscale images after convolution with an idealized particle mask [2]. A 2D cutout of the original 3D image data and the segmentation result are shown in Figure 1. The segmentation enables us to estimate the particle size distribution and intensity profiles in the rz-plane of the cylindrical sample, allowing us to follow the local compaction and dilation of the powder over time.

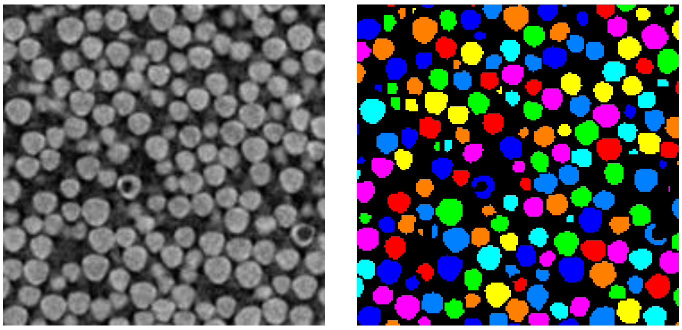

Figure 1: Cutout of one slice of an XMT measurement (left: original, right: segmented).

Based on the centers of mass of the segmented particles, a particle tracking is computed. For time steps with an angle increment of 0.5°, we employ a fast tracking algorithm which aims to (approximately) minimize the sum of squared displacements [3]. In time steps with an angle increment of 5°, minimizing displacements is no longer feasible and we need prior information about particle movements. We can estimate a local angle of rotation and a local translation in z-direction directly from the image data as a function of the radius and height in the sample. The basic idea for this approach, which has been proposed in [1], is to rotate each slice of an image stack by a number of different angles and to determine for which angle it agrees best with the corresponding slice of the subsequent image stack. In the present study, this approach is refined by subdividing each image slice into disjoint rings of equal area which are rotated independently and by including a deformation in z-direction. The quality criterion for comparing two image slices is their correlation. Using this image-based local shear deformation, we can track a large number of particles even in steps of 5°.

In order to validate the method, we applied it to the time step from 5° to 10° of shearing, where the detailed information in steps of 0.5° is available as well, and compared the tracking based on the local shear deformation (ignoring intermediate measurements) to the tracking based on all available data. We found that 98.9 % of the tracks identified with our method were identical to the tracks identified using measurements in steps of 0.5°.

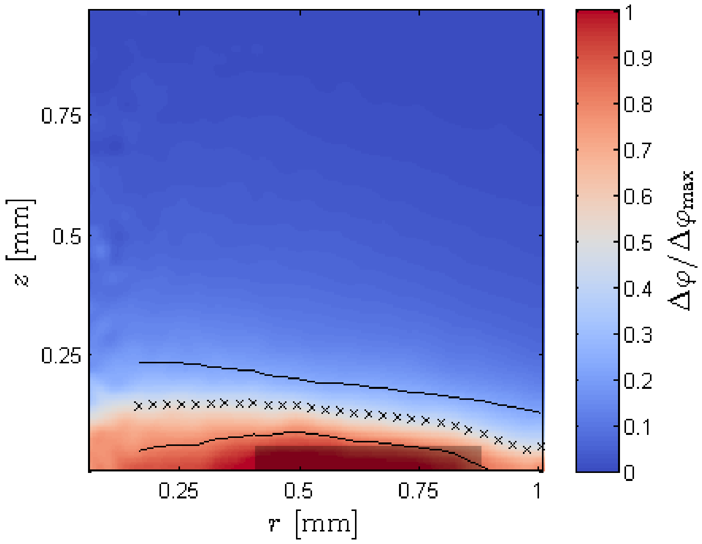

Figure 2: Profile of rotational velocities between 10° and 15° of shearing normalized by the shearing angle and relative to the upper piston and wall. The region shaded in gray is the upper edge of the lower piston. The black points and lines indicate the fitted location, z_{sb}, and width, w_{sb}, of the (radius-dependent) shear band.

One feature of the data, which becomes obvious when looking at the absolute distance particles travel in each time step, is that the axis of rotation is not fixed at the central axis of the cylinder but varies over time. By calculating angles of rotation with respect to the wrong axis, we would overestimate them on one side and underestimate them on the other side of the cylinder. Therefore, the axis of rotation is estimated from the data by fitting circles to particles with the same height and speed and averaging the circle centers at the given height. This way we obtain a (possibly curved) axis of rotation, which adapts to the data in each time step. Then, the angle by which each particle is rotated is calculated relative to the estimated axis of rotation and we can assume that the dynamics in the system do not depend on the angle φ of rotation. Using kernel estimation and averaging in φ-direction, we obtain 2D profiles of rotational velocities and of displacements in r- and z-direction. In order to obtain objective measures of the location and width of the shear band, we fit a model function to the z-coordinates and normalized rotational velocities of particles at the same radius in the sample. The fitting function used to describe the transition from zero to maximum rotational velocity is $v(z) = \frac{1}{2} + \frac{1}{2}\mathrm{Erf}\left(\frac{z - z_{sb}}{w_{sb}}\right)$. A profile of the displacements in φ-direction and the shear band fitted to it can be found in Figure 2.

3. Results

Visually, the segmentation results are very good, see Figure 1. The mean particle diameter estimated from the segmented particles is 30.04 µm. While the size distribution is slightly broader than specified by the manufacturer, it is almost identical at all points of measurement, indicating consistent segmentation results. The distance between the upper and lower piston is 2.2 mm initially and decreases to 2.0 mm during the first 4° of shearing. At the same time, the packing density increases and settles at 0.54 with about 235,000 particles. With our analysis we focus on the measurements starting at 4° of shearing, where the packing density and height of the sample remain approximately constant. The tracking efficiency is larger than 99.5 % for all steps of 0.5° after this point and larger than 97 % for all steps of 5°. In most time steps, the shear band is close to the lower piston and slightly curved at the outer wall of the sample chamber, though it seems to jump up to about 0.4 for several time steps during the first 10° of shearing. The average width of the shear band after stationarity is reached is 229.3 µm, which corresponds to 7.63 particle diameters.

4. Conclusion

Using the example of slightly adhesive spherical glass particles, we present methods for segmenting, tracking and analyzing particles and their dynamics in a shearing experiment based on image data. We explained how particles can be tracked even if a large angle increment is used between the measurements and how errors due to a curved and shifting axis of rotation can be avoided. Finally, we assessed rotational velocities in the rz-plane of the sample and proposed a way to automatically determine the location and width of the shear band as a function of the radius in the sample.

[1] Strege, S.: Röntgenmikrotomographische Analyse der Verdichtung und Scherung feiner und kohäsiver Pulver. TU Braunschweig Diss. 2014

[2] Wenzl, J., Seto, R., Roth, M., Butt, H.-J. and Auernhammer, G. K.: Measurement of rotation of individual spherical particles in cohesive granulates. Granular Matter 15 (2013) pp. 391-400

[3] Crocker, J. C. and Grier, D. G.: Methods of digital video microscopy for colloidal studies. Journal of Colloid and Interface Science 179 (1996) pp. 298-310