Advanced Powder Technology xxx (2017) xxx–xxx

Contents lists available at ScienceDirect

Advanced Powder Technology

journal homepage: www.elsevier.com/locate/apt

Original Research Paper

Structural analysis and tracking of micron-sized glass particles during shear deformation: A study based on time-resolved tomographic data

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ARTICLE INFO

Article history:
Received 3 December 2016
Received in revised form 7 April 2017
Accepted 4 May 2017
Available online xxxx

Keywords:
Shear flow
Glass beads
Micro shear-tester
Particle tracking
X-ray microtomography

ABSTRACT

The interplay between structure and mechanical properties of fine and cohesive granular matter is of wide interest and far from being well understood. In order to study this relationship experimentally, it is desirable to record as much information on the particles and their motion behavior as possible during a shear experiment – ideally, the trajectory of every single particle. Observing the particle movements offers deep insights into changes in the mechanical behavior of the bulk (e.g., densification, loosening or formation of failure areas) and into the behavior of single particles. However, obtaining particle-level information on the dynamics of an entire shear-tester experiment remains a great challenge. In this paper we present an experiment and analysis methods which allow the extraction of the trajectories of almost all particles within a shear-tester. A fully functional micro shear-tester was developed and implemented into an X-ray microtomography device. With this combination we can visualize all particles within small bulk volumes of the order of a few µl under well-defined mechanical manipulation. The processing of time-resolved tomographic data makes it possible to localize and track particles despite large angle increments of up to 5° between tomographic measurements. We apply our methods to a torsional shear experiment with spherical micron-sized particles (~30 µm) and analyze the structural evolution of the sample. In addition, particle tracks provide detailed insights into the formation and evolution of the shear band.

1. Introduction

Shear flow of granular media is ubiquitous in nature and of industrial importance when it comes to the handling and processing of bulk solids (e.g., flow through hoppers [1,2], bunkers and silos [3,4]). In the physics of granular matter [5], among many other interesting phenomena, understanding the flow properties, i.e., the stress response to an applied strain rate, has been in the focus of research [6]. At slow, quasi-static deformation, there arises a strain rate independent creep regime [7,8]. The localization of strain within the bulk, often referred to as failure zone or shear band, represents a unique feature of this quasi-static regime, which was addressed by many researchers in the past [9–12] and can be observed, e.g., in glassy systems [13] and solidifying metals [14] as well. However, the interplay of structure and mechanical properties of the bulk solid is still not deciphered sufficiently, especially when it comes to cohesive granular matter.

Experimental investigations of this interplay are challenging because they require detailed information on particle properties, packing structure and dynamics over the course of a shear experiment – ideally, down to single-particle trajectories. Sophisticated experimental setups are capable of determining the properties of individual micron-sized particles [15] and of imaging the inner structure of a bulk solid nondestructively, e.g., via computer tomography [16,17]. However, detailed experimental information on the dynamics of small-size particles and on their trajectories in 3D is still particularly rare.

An alternative route of investigation is offered by numerical simulations with the discrete element method (DEM), which provide valuable insight into the mechanical behavior of granular matter [18–20]. Although DEM simulations output fine-grained information on particle trajectories and even forces acting between them, the accuracy of the physical behavior in the models is limited by computational constraints and the high complexity in contact mechanics of micron-sized particles [21]. Consequently, even when following the DEM approach, experimental data on

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http://dx.doi.org/10.1016/j.apt.2017.05.002
0921-8831/

the trajectories of micron-sized particles under shear is highly needed for model validation.

The main challenge of tracking all particles within a bulk volume is that it requires 3D image data in a sufficiently high spatial and temporal resolution. Standard methods, which rely on one or more 2D cameras to analyze the dynamics in a flow, provide only aggregated information or are restricted to transparent liquids containing a relatively small number of tracer particles (e.g., particle image velocimetry [22–24] or particle tracking velocimetry [25,26]). Moreover, to our knowledge, these techniques have not yet been applied to micron-sized particles. An alternative approach is presented in [27], where confocal laser microscopy is used to obtain 3D images of particles under lateral shear. While this imaging technique allows for a high temporal resolution, it limits the size of the sample in z-direction and requires particles to be surrounded by a fluid with matching refractive index. This is not possible for all particle materials or when investigating dry powders. Interestingly, partial photobleaching of individual particles enables the detection of particle rotations.

In this paper we use a fully functional micro-sized shear-tester [16], which can be fitted into an X-ray microtomography (XMT) device. In contrast to [16], we conduct and analyze a torsional shear experiment with constant normal load and analyze the motion of almost all micron-sized spherical particles on particle level. Using the XMT, a series of 3D images is recorded in the course of the experiment. While the XMT provides high-quality 3D images at very high spatial resolution, each measurement is time consuming. Therefore, only a limited temporal resolution can be realized. To overcome the problem of tracking particles despite the low temporal resolution, we propose a method to estimate the average particle movement at any location in the shear cell directly from the image data, building on ideas from [28]. Our approach is data-driven and explores the rotational symmetry of the shear cell. Based on this first estimate, we are able to extract the trajectories of almost all individual particles in the shear cell. We utilize this data to examine the initial shear band formation and its evolution over time in full detail. In addition, we analyze structural inhomogeneities in the sample over time.

Our dataset is extraordinary in that it provides experimental particle-level information on the dynamics within the entire shear cell. Thus, our methods allow to fully analyze and compare experiments both on particle level and macroscopically. Similar information is usually only obtained by DEM simulations. Therefore, our methods offer an increased validation depth for DEM simulations and a reliable basis for model calibration, although a direct comparison of our experiment to DEM simulations exceeds the scope of this paper. In contrast to [27], our methods are applicable to dry powders and to almost all particle materials. Finally, the micro shear-tester is also well suited for determining shear flow properties of powders which are only available in small quantities, e.g. for screening processes.

The paper is structured as follows. Sections 2.1 and 2.2 are devoted to the model material and experimental setup. The methodology of image segmentation, image-based measurement of local shear deformation and particle tracking is described in Section 2.3. Results of a detailed analysis of structural inhomogeneity as well as shear band formation and evolution are presented in Section 3 and discussed in Section 4.

2. Material and methods

2.1. Material

In this study we use a fine and slightly cohesive powder which consists of solid borosilicate glass microspheres (BSGMS 27–32 μm, CoSpheric LLC, USA; BSGMS in the following). An image of several microsphere particles taken on a scanning electron microscope (SEM) is presented in Fig. 1 (inset). The figure emphasizes the almost uniform spherical shape and similar size of the particles, but also shows a non-negligible surface roughness. We measured the particle size distribution using laser diffraction (Helos, Sympatec GmbH, Germany) after dispersing the particles with ultrasound for 30 s in an aqueous environment. The results, which are shown in Fig. 1, indicate a narrow mono-disperse size distribution of the glass particles with median value $x_{50,3} \approx 30$ μm.

Particle stiffness and adhesion forces were determined in [28] using nanoindentation and atomic force microscopy (AFM), respectively. Nanoindentation was carried out with the Triboindenter (Hysitron, Inc, USA), placing single particles between a glass object slide and a flat punch. They were deformed using a force-controlled approach with a maximum loading force of 2 mN. Then, Young's modulus has been calculated from the resulting stress-strain curve taking Hertz theory as a basis. The AFM measurements have been realized by the XE 100 (Park Systems, Korea) using the colloidal probe method for pairs of particles. One of the two particles was attached to the apex of a tip-less cantilever with UV-hardened glue. The other one was attached to the object slide with nail varnish. Then, both particles were brought into contact and the force needed to break this contact was determined. Since both measurement techniques are subject to strong fluctuations, nanoindentation has been repeated for 58 particles and AFM has been applied to 100 pairs of particles. The mean values and standard deviations reported in [28] are $E = 15 \pm 7$ GPa for the elastic modulus and $F_c = 82\ \text{nN} \pm 60\ \text{nN}$ for adhesion forces. From a methodological point of view we do not expect a major influence of the variability on our results.

2.2. Experiment

2.2.1. X-ray microtomography (XMT)

The fundamental component for a detailed microstructural investigation is the nondestructive examination with the XMT, which enables an image-based analysis. We use a high-resolution tomography device (MicroXCT-400, Zeiss (Xradia), Germany). For this study an acceleration voltage of 50 kV and a current intensity of 200 μA were applied at the X-ray source. These parameters result in the best outcome for high-contrast images. According to the sample diameter of 2 mm, a ten-fold optical magnification is used to ensure a reproduction of the entire sample diameter with a resolution of 2.2 μm (1.1 μm before binning). A single detector

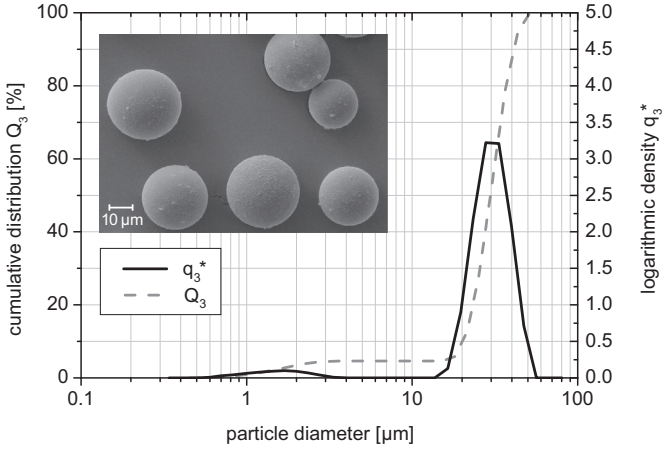

Fig. 1. Particle size distribution (mass distribution, logarithmic density and cumulative distribution function) and SEM picture (inset) of BSGMS glass particles.

Please cite this article in press as: L. Handl et al., Structural analysis and tracking of micron-sized glass particles during shear deformation: A study based on time-resolved tomographic data, Advanced Powder Technology (2017), http://dx.doi.org/10.1016/j.apt.2017.05.002