Powder Technology 270 (2015) 121–127

Contents lists available at ScienceDirect

Powder Technology

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

An apparatus for confined triaxial testing of single particles

Henrik Jonsson [a], Johan Gråsjö [a], Josefina Nordström [a], Niklas Johansson [b], Göran Frenning [a],*

[a] *Department of Pharmacy, Uppsala University, Box 580, SE-751 23 Uppsala, Sweden*
[b] *Department of Physics and Astronomy, Uppsala University, Box 516, SE-751 20 Uppsala, Sweden*

ARTICLE INFO

Article history:
Received 15 August 2014
Received in revised form 8 October 2014
Accepted 10 October 2014
Available online 20 October 2014

Keywords:
Compression
Triaxial
Single particles
Confined conditions
Apparatus design
Contact mechanics

ABSTRACT

A novel triaxial apparatus employing overlapping rigid boundaries has been designed and constructed for experimental measurement of contact forces under confined compression of single granules in the mm-scale. The performance of the apparatus was evaluated by performing uniaxial and triaxial compression experiments on ideal elastic–plastic materials. Compression curves were compared with the fully plastic Abbott–Firestone contact model and with results from FEM simulations. The increase in contact force associated with confined conditions was observed in the compression curves from triaxial compression experiments, as supported by predictions from simulations using single particle contact models. Hence, a new method for the assessment of mechanical behaviour of single particles under confined compression can be considered as established.

1. Introduction

Compression of granular materials is a central process within several industries, including metallurgy, pharmaceutics and ceramics [1,2]. To fully understand the factors influencing the performance of this process, it is necessary to assess the process characteristics on all levels, including the particle, powder and compact scales and the principles that connect them. Our main focus here is to study the mechanical behaviour at the particle level, by sorting out and experimentally study the behaviour of single particles under confined conditions by triaxial compression using a novel device designed for this purpose.

There is a multitude of devices available for the experimental characterisation of the mechanical response of single particles. An example is the classical indentation equipment where characterisation of the material and its behaviour under mechanical loading is performed by pressing a small indenter into the particle surface [3]. On a larger scale, the mechanical response of single particles is commonly assessed by using a texture analyser to uniaxially compress the particle of interest [4]. On a small scale, Atomic Force Microscopy (AFM) is a common method for measuring mechanical characteristics, either employing a cantilever with a microfabricated tip running over the sample or by using the colloidal probe technique [5].

Several micromechanical contact force relationships have been developed using the single particle approach. The classical Hertz solution [6] as well as extensions to this (see for instance Thornton et al. [7] and references therein) concerning the elastic contact between spheres is one example. Another example is the similarity solution suggested by Storåkers et al. [8] for plastic deformation from particle–particle contacts.

However, it has been known for long (see for example the work by Fischmeister and Arzt [9]) that the forces present in the powder bed in a conventional compaction process cannot be described simply as a sum of contact forces arising from an assembly of independently deforming elastic–plastic particles. The reasons for this are that at high degrees of deformation, the contact surfaces on the particle start to impinge and hence cease to be independent of each other. Mesarovic and Fleck [10] stated that this phenomenon causes the conventional models to become invalid at as low relative densities of the powder bed as about 0.8, whereas powder compaction processes need to proceed up to relative densities as high as above 0.95 in order to form an end product of the desired quality.

For the mapping of confined single particle compression behaviour, the research has thus far been restrained to modelling by employing finite and discrete element methods (FEM/DEM) and/or by the formulation of analytical solutions. For example, Frenning [11] developed an analytical model for the micromechanical forces upon plastic deformation of particles that was validated using the FEM. Gonzalez and Cuitiño [12] attempted to overcome the restriction of independent contacts by the formulation of an analytical contact model for elastically deforming particles using an extension of the Hertz theory. Harthong et al. [13–15] have developed a contact model using a combined FEM/DEM analysis including Voronoi tessellations (as introduced to the area by Arzt [16]) specifically for studying high levels of particle densification under confined conditions. However, despite progress in contact model

---

* Corresponding author. Tel.: +46 18 471 43 75; fax: +46 18 471 42 23.
E-mail address: Goran.Frenning@farmaci.uu.se (G. Frenning).

http://dx.doi.org/10.1016/j.powtec.2014.10.016
0032-5910/

development, the dominating opinion within the research field still seems to be that new contact models for high degrees of densification are needed.

The challenge is therefore, as described above, to mimic the spatially confined situation to which an individual particle is exposed during a bulk powder compaction process, and thus enable the approximation of forces acting on the particle in such situations, something that is unachievable by compression experiments using conventional material testing equipment. The aim of this work is to set up and validate a novel apparatus that is designed for triaxial compression of single granular particles in the mm-scale. In the longer term, results extracted from the experiments performed with the apparatus will be used for the evaluation of micromechanical contact models as well as the development of new ones. The first step is, however, to investigate the ability of the apparatus to perform the type of compression experiments it is intended for. The paper is organised so that the general principle of compression is explained first, after which the design of the apparatus is presented. Finally, data from simple experiments performed by the apparatus are reported and discussed.

2. General considerations

The mechanism of compression employed by the apparatus involves overlapping rigid boundaries, following the principle shown in Fig. 1. Upon operation, the boundaries are allowed to simultaneously, yet independently, slide over one another, gradually shrinking the rectangular box that they constitute. This enforces the sample under testing to undergo confined compression, since there is no possibility for the particle to compensate for the imposed contact forces by expanding spatially in any direction. An apparatus employing this mechanism of operation was first developed by Hambly [17] for triaxial compression of soil specimens. The principle has later been adopted for the same purpose by Airey and Wood under the name *The Cambridge True Triaxial Apparatus* [18]. Ibsen and Praastrup further developed the apparatus, constructing a device referred to as *The Danish Rigid Boundary True Triaxial Apparatus* [19]. As far as we know, this mechanism has not been employed before for the compression of single granular particles in the mm-scale. For this purpose, somewhat different issues must be considered. On one hand, no drainage is needed, since the samples intended for testing are virtually dry. On the other hand, the samples of interest (and thus the boundaries) are significantly smaller, posing higher demands on precision of the apparatus.

3. Apparatus design and coordination of movements

3.1. Apparatus design overview

The movements needed for the compression procedure are effectuated by three orthogonally positioned linear actuators with a loading capacity of 400 N each (*M238.5PL*, *Physik Instrumente GmbH & Co*, *Karlsruhe*/*Palmbach*, *Germany*, described further in Section 3.2). Three of the boundaries (hereafter referred to as *punches*, denominated X_1, Y_1 and Z_1) are attached to the actuators which themselves are mounted to an outer aluminium frame, as seen in Fig. 2. The three opposing punches (X_2, Y_2, Z_2) are attached to rigid aluminium cylinders, which themselves also are mounted to the outer metal frame. Upon operation, the linear actuators are set to move towards a defined position at a desired rate (see Section 3.3). The distances the punches have moved are detected by position sensors integrated in the actuators and accordingly registered by in-house computer software designed in *LabVIEW 2013* (*National Instruments*, *Austin*, *TX*, *USA*). The computer software simultaneously stores compression force data as registered by load cells mounted to all of the punches (see Section 3.2).

In order to obtain the compression procedure of the particle according to Fig. 1, the axial movement of a punch must impose lateral movements of two of the neighbouring punches (directions are defined by the coordinate system in Fig. 2). This is accomplished by an inner coordination device (further described in Section 3.3) that acts as a movement conveyor for the punches.

To enable the required lateral punch movements and to reduce frictional resistance, the actuators and aluminium cylinders connected to the Y_1, Z_1, X_2 and Y_2 punches are mounted to the outer metal frame via ball bearing rails (*LLTHC 15 U-T1 P3*, *SKF Sverige AB*, *Gothenburg*, *Sweden*).

3.2. Punch assembly

Each of the linear actuators (X_1, Y_1, Z_1) is assembled with components as shown in Fig. 3. The original actuator component consists of the parts designated *1–3* in the figure. The actuator (1) is mounted to the metal frame of the apparatus via a threaded part (2), which is screwed into place. The piston (3) is the axially movable component of the system, controlled from the computer software. To the piston, a bronze piece (4) for connection to the inner coordination device (Fig. 4, see Section 3.3) is attached. The distance between the flanges on the bronze piece is 6 mm, in order to perfectly fit in the milled rectangular openings in the coordination device. At the end of the assembly, a steel punch is attached. The rod shaped tip of the punch is

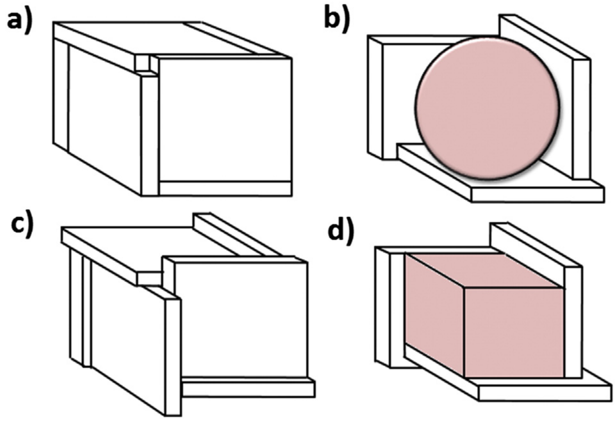

Fig. 1. The mechanism of operation of the rigid boundary triaxial apparatus. Six partially overlapping rigid boundaries form an initially cubic cavity (a) into which a particle is placed (b). During operation, the boundaries are allowed to slide past each other (c), eventually compressing the particle into a rectangular shape (d).

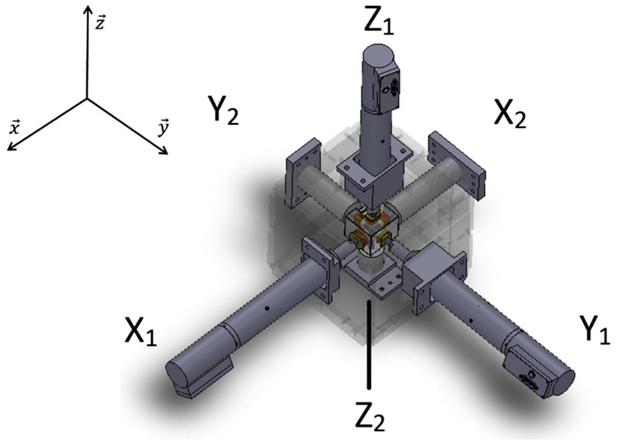

Fig. 2. Drawing of the triaxial apparatus seen obliquely from above, involving denomination of the punches. For clarity, the metal frame is shown as shaded. Also shown is the coordinate system defining the directions of punch movements.