

QUANTITATIVE IN-SITU STUDIES OF GRANULAR COMPACTION

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Summary: Granular compaction is an important field of study, with numerous applications around us. The mechanisms of granular compaction take place at several different length scales, which makes it complex to predict and model. Here quantitative in-situ studies of granular compaction processes have been carried out at multiple spatial scales using x-ray microtomography, image analysis and digital volume correlation.

1. INTRODUCTION

The compaction of powders or granular beds is an important process in a number of industrial applications, including sintering of metals or ceramics, production of pharmaceutical tablets and manufacture of explosive. The process of powder compression is complex and is generally explained by a four-stage model comprising initial particle rearrangement, particle fragmentation, particle plastic deformation and finally elastic deformation of the compact [1]. In order to obtain non-destructive, full-field density information, a number of researchers have applied x-ray microtomography (XMT) [2]. However, we can better understand the physics of the process by tracking deformations of different regions of the bed in-situ. Full three-dimensional measurements of the material deformation are possible using a technique called digital volume correlation (DVC) [3], which has been used for measurements of various materials and applications, including compaction behaviour in granular materials [4].

2. EXPERIMENTAL METHOD

The measurements were carried out at the microCT lab at Luleå University of Technology (LTU), using a ZEISS Xradia 510 Versa system, see Fig. 1(a). The in-situ loading was carried out using a 5 kN compression/tensile stage (Deben CT5000TEC), also visible in Fig.1(a). The microtomography system include a 30-160 kV micro-focused source, a 2Kx2K CCD camera, a tuneable detector system consisting of multiple resolution- and field-of-view pairings, analogous to a light microscope, which enables studies at a number of different spatial scales. The system due to its design also allows maintaining high resolution at large working distances, making it optimal for in-situ studies. The spatial resolution is 0.7 μm , and the minimum achievable voxel size is <70 nm.

The granular material (powder) is placed in one of the sample holders that have been specially produced for this purpose. These come in a few different sizes, optimized for different particle sizes and Field of Views (FOV), but are all having the same principal design with a base made of aluminium, a tube made of PMMA and a punch made of Brass. The diameter of the punch is slightly smaller than the inner diameter of the PMMA tubes, in order to ensure a smooth and frictionless force transmission.

Here a bed of granular sugar with the diameter 10 mm, and initial height 4.5 mm, was studied during compaction. The study was carried out using a 0.4x macro objective, resulting in a spatial resolution of 12.5 μm . Quantitative characterisation of the reconstructed 3D microstructure is carried out using 3D image analysis, which allows us to identify features of special interest, such as grains, pores and cracks, and form various statistical measures of the microstructure, including grain size distribution and porosity. Moreover, the three-dimensional deformation and strain in the material is measured using DVC. Here, the analysis is carried out based on two sets of data from XMT, captured from two different load states (before and after deformation). By repeating this procedure for the entire load cycle all intermediate states can be followed.

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3. RESULTS AND DISCUSSION

Fig.1(b) and (c) show examples of the reconstructed sugar microstructure, obtained with XMT, in unloaded state and when loaded at 500 N, respectively. Here one can see a certain compaction of the material, with more densely packed crystals in (c). Moreover one can also see examples of initial crushing of the crystals (marked with circles) – both in regions near the wall as well as in the bulk. Fig.1(d) shows an example of macroscopic analysis of the 3D displacement field in the z-direction, obtained with DVC, for the load sequence 0N-500N-1.0kN-2-kN-3.5 kN. From the results one can see clear wall friction effects, i.e. differences in the material behaviour between the regions close to the PMMA tube and the bulk region. This effect becomes more severe as the load increases.

It is strength to be able to study the material behaviour at multiple spatial scales. For example the interaction mechanisms near the wall at particle level and how they relate to the wall friction effect that is seen in the macroscopic analysis.

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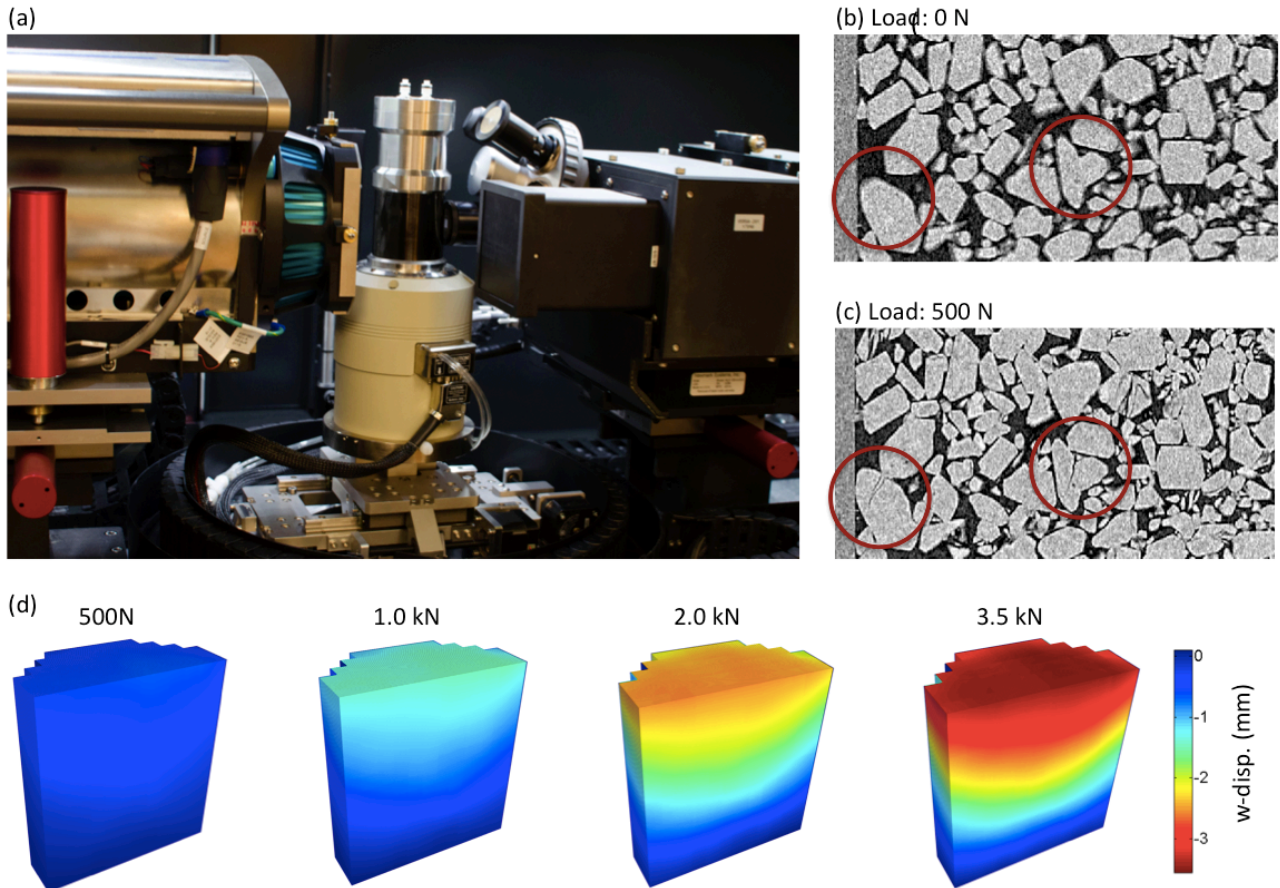


Figure 1. Experimental setup for in-situ measurements of granular compaction, showing the interior of the microtomography system and the 5kN in-situ load stage (a). Mid cross-sections through the granular bed in unloaded state (b) and when loaded at 500N (b), showing examples of initial crushing of crystals (red circles). Macroscopic analysis of compaction obtained with DVC, for the load sequence 0N-500N-1.0kN-2-kN-3.5 kN (d).