A microscopic investigation into the breakage behavior of calcareous origin grains in 1D compression

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**ABSTRACT**

Breakage in granular material has gained extensive research attention recently. This grain scale behavior would cause significant change to the macroscopic response. The development of x-ray micro-tomography technique makes it possible to non-destructively examine the microstructure of an assemblage at different stages of loading. In this study, micro-focused X-ray CT scanned images are used to visualize the development of grain crushing under one-dimensional compression. Initially uniformly graded carbonate grains are prepared into a tailor-made oedometric cell such that X-ray CT scanned images can be taken at different stages (loading and unloading) of the test. Image processing is carried out to isolate individual grains from the assemblage. The evolution of grain size and shape, and void size is examined based on the processed 3D images. The results show that the crushed grains are more rounded. The void space becomes smaller and varies within a narrower range as the compression continues.

**Keywords:** breakage, microtomography, one dimensional compression, carbonate grain

1 INTRODUCTION

Granular soils yield and exhibit irrecoverable deformation when compressed to high stresses. Excessive deformation may lead to serviceability and overall stability problems. This macroscopic “yielding” has been shown to be originated from grain crushing (Coop and Lee 1993; Yamamura et al. 1996; Nakata et al. 2001b; Cheng et al. 2004). Investigating grain breakage and its evolution therefore help develop a more complete constitutive framework of granular soil.

Extensive laboratory investigations have been carried out to correlate breakage behavior with initial density, stress level, loading manner etc (Hardin 1985; Lade et al. 1996; Coop et al. 2004). More recently, techniques like SEM and QicPic analysis shed further light on the microscopic characteristics of the grains after the testing (Cavarretta et al. 2010; Miao and Airey 2013). However, the analyses can only be made at the end of a test on a dismantled assemblage. The grain interaction and its evolution during a test, however, cannot be examined.

By taking advantage of the micro-focused X-ray CT scanning, this paper describes at the microscopic level the breakage of calcareous origin grains under 1D compression. With an appropriate image analysis, the test offers us the opportunity to observe the assemblage at the particulate-level at different stages of a loading-unloading cycle. In this paper, the evolution of grain characteristics and void space during the compression test is presented. The results are derived from the 3D analysis of the scanned images.

2 MODIFIED OEDOMETER TEST WITH MICRO-X RAY CT SCAN

2.1 X-ray CT system and loading equipment

The X-ray CT system used in this study is TOSCANNER-32300 FPD which is housed in the X-Earth Center of Kumamoto University (Fig. 1). The soil sample is scanned at a spatial resolution of 36 um at different stages of the loading-unloading cycle. The CT images in the present study have 16-bit gray values.

The tailor-made 1D compression equipment as shown in Fig. 2 comprises a loading chamber at the bottom, the miniature load cell in the middle, and the loading system on the top. In this device, the load is applied manually via a gear system. The cylindrical soil sample has a height of 35 mm and a diameter of 35 mm. It is contained in aluminum holder aiming at the best scanning resolution as compared to a steel one. The
2.2 Testing material and preparation of specimen

The calcareous sand used in this study has been described in Yan and Shi (2014). In this investigation, the grains were first sieved to retain only grains in the range of 1.18-2 mm. To achieve a low density sample, the grains were air pluviated into the container to form a dry and loose assemblage. 1D staged loading was then applied to reach as high as 16.9 MPa, which was then followed by a two-staged unloading. The volumetric compression curve is shown in Fig. 3. Micro-focused X-ray CT scanning was carried out at some selected stages during the loading and unloading (as shown also in Fig. 3). Table 1 summarizes the information.

![Fig. 1. The designed one-dimensional compression apparatus](image1)

![Fig. 2. The designed one-dimensional compression apparatus](image2)

### 3 IMAGE PROCESSING

The three-dimensional scanning gives a stacked of two-dimensional 16 bit grey-scale images. The thickness of each slice of the image is equal to the unit length of a voxel which corresponds to a 36 μm cube in this study. Intensity value of a voxel, which has \(2^{16}\) levels for this 16 bit system, is a function of the degree of attenuation of the X-ray after passing through the materials subject to scanning. Image processing techniques, including filtering, binarization, erosion-filling-dilation, and watershed segmentation, are then required to transform the raw images into useful data revealing microscopic characteristics of the assemblage. In this study, the analysis is conducted with the help of the open source program Fiji and the image processing toolbox in MATLAB.

![Fig. 3. The volumetric compression curve. Numbers in the figure indicate the states where CT scanning was carried out.](image3)

| Description | Loading stage | Overburden stress (MPa) | Void ratio |
|-------------|---------------|-------------------------|------------|
| Loading     | 1             | 0                       | 1.359      |
|             | 2             | 1.2                     | 1.227      |
|             | 3             | 2.9                     | 1.103      |
|             | 4             | 5.8                     | 0.937      |
|             | 5             | 12.6                    | 0.687      |
|             | 6             | 16.9                    | 0.609      |
| Unloading   | 7             | 7.7                     | 0.609      |
|             | 8             | 0                       | 0.625      |

Table 1. Details of the compression test.
Watershed segmentation separates individual grains. For irregular grains having extensive connections, the morphological watershed algorithm has been proved to give promising results (Al-Raoush and Alshibli 2006). Firstly, a distance transformation is applied to the binary image to find the local maxima in each region. Then, the watershed algorithm works in a way to flood from each local maximum until ‘water’ comes from different directions meet. ‘Dams’ will then be constructed at the meeting locations, where they are taken as the contacts among the grains.

Fig. 4(a) shows a typical grey-scale raw image obtained from the scan while Fig. 4(b) shows the same image after the mentioned image processing. Note that individual grains can be clearly seen in Fig. 4(b).

Fig. 4. The 2D images before and after image processing: (a) grey-scale raw image; (b) segmented image.

4 RESULTS

4.1 Grain size evolution

Traditionally, the grading curves obtained by mechanical sieving before and after a compression test are used to illustrate grain breakage (Marsal 1967; Turcotte 1986; Fukumoto 1992). With the aid of micro-focused X-ray CT scanning and proper image analysis, the evolution of grain size at different intermediate stages of a compression test can now be tracked without dismantling the specimen. In this study, as a first trial, the ellipsoid fitting algorithm is used to idealize the segmented grains into ellipsoid. The three principal axes are then determined (Ollion et al. 2013). The shortest principal axis length (herein denoted by \( R_x \)) is used to represent the size of a grain as 99\% of \( R_x \) fall below 2 mm, which fits the initial sieving.

Fig. 5 shows the particle size distribution (PSD) obtained by the aforementioned image processing at different stages of a compression test. Curves labeled by 1-8 correspond to PSD at different stages (see Fig. 3 and Table 1) of the test. As shown in the figure, insignificant breakage takes place below 1.2 MPa (curves 1 and 2). When overburden stress increases from 1.2 to 16.9 MPa (curves 3 to 6), grain crushing can be readily seen. During unloading, curves 6 to 8 essentially overlap with each other which indicate that no noticeable breakage has occurred. In the same figure, the initial and final PSD obtained by mechanical sieving (labeled by sieve_initial and sieve_final, respectively) are also shown. It is worth noting that neither curve matches with the one derived from the CT scanned images. In particular, PSD obtained from mechanical sieving shows more smaller-size grains than the one derived from image analysis. It is anticipated that during the 1D confined loading most cracks initiated within the grains are closed cracks with fragments clustering in their original position. It is not easy to distinguish the cracks and separate the grains during the image analysis. It results in fewer smaller-size grains to be identified in the image analysis. Yet, this is not a problem in mechanical sieving.

Fig. 5. PSD curves derived from CT scanned images and mechanical sieving.

4.2 Grain shape evolution

Grain shape also evolves during compression (Miao and Airey 2013; Yan and Shi 2014). Based on the dimensions of ellipsoids, a 3D characterization of the particle geometry is carried out. Shape parameters including sphericity, elongation and flatness are defined in Eq. (1) to (3) as shown below (Lin and Miller 2005; Al-Rousan et al. 2007).

\[
\text{Sphericity} \quad S = \frac{S_{EO}}{S_{OB}} = \frac{1}{36\pi V_{OB}^2} \\
\text{Elongation ratio} \quad ER = \frac{R_x}{R_z} \\
\text{Flatness ratio} \quad FR = \frac{R_y}{R_z}
\]

where \( S_{OB} \) and \( V_{OB} \) are the surface area and volume of the ellipsoid; \( S_{EO} \) the surface area of its equivalent sphere; \( R_x, R_z, \) and \( R_y \) are the length of the ellipsoid along the longest, intermediate, and shortest principal axis respectively. Note that the value of the shape
parameters lies between 0 and 1.

Fig. 6 summarizes the cumulative density function against different shape parameters at different compression stages. A clear increase in sphericity during loading can be seen. Slight increase in elongation and flatness ratio can also be visualized though not as obvious as the sphericity. In short, the particle morphology and surface roughness change during compression loading and the grains become more rounded. In all cases, the change of shape parameters is negligible during unloading.

4.3 Void size evolution

The processing of void from the CT scanned images is similar to that of the grains. Following the same procedure of distance transformation and morphological watershed, the void space is segmented into isolated volume. Here the diameter of an equivalent volume of sphere is used to characterize the void size as shown in Eq. (4).

\[ \text{Void size } D = \sqrt{\frac{6V_{\text{eq}}}{\pi}} \] (4)

Fig. 7 summarizes the void size distribution during loading and unloading. Increasing the overburden pressure decreases the percentage of bigger voids and more smaller-size voids are created. Furthermore, void size tends to varies within a smaller range as the overburden stress increases (note that the void size is plotted in a logarithm scale in the figure). During the unloading, very little change in the void size can be seen.

5 CONCLUSIONS

One dimensional compression test was conducted on crushable grains using a tailor made oedometer cell which allows micro-focused X-ray CT scanning to be conducted at different stages of the loading without sample dismantling. Image processing including filtering, binarization, erosion-filling-dilation, and segmentation is performed on the grey-scale raw images to obtained 3D images of the assemblage. The results clearly show noticeable grain breakage during the compression when the macroscopic stress goes beyond a certain threshold. Furthermore, such a breakage is not observable during unloading. The crushed grains are more rounded as concluded by the grain shape parameters evaluated from the 3D images. Void size becomes smaller as loading continues. Besides, the void size varies within a smaller range as test continues. During unloading, however, void size does not change much.
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