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# Evolution of particle breakage studied using x-ray tomography and the discrete element method

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**Abstract.** Particle breakage can significantly change the fabric (size and shape of particles and contact network) of a granular material, affecting highly the material's macroscopic response. In this paper, oedometric compression tests are performed on zeolite specimens and x-ray computed micro-tomography is employed, to acquire high resolution 3D images of the specimens throughout the test. The images are processed, to describe breakage spatially and quantify it throughout the test and gain information about the mechanisms leading to particle breakage. In addition to the image processing, the discrete element method (DEM) is used to study the initiation and likelihood of particle breakage, by simulating the experimental test during the early stages of loading and using quantitative results from the images to inform and validate the DEM model. A discrete digital image correlation is used, in order to incrementally identify intact grains and simultaneously get results about the strain field within the specimen, as well as the kinematics of individual grains and fragments. In the initial stages of breakage, there is a clear boundary effect on the spatial distribution of breakage, as it is concentrated at the moving boundary (more than 90% of total breakage) and circumferentially (more than 70% of total breakage) close to the apparatus cell. The DEM model can reproduce the bulk response of the material until the point where substantial breakage governs the macroscopic response and it starts to soften. Additionally, there is an initial indication that the spatial distribution of the force network matches the localisation of breakage radially, but it does not seem to localise close to the loading platen. This analysis will enrich our understanding of the mechanisms and evolution of particle breakage.

## 1 Introduction

Particle breakage can significantly change the fabric (size and shape of particles and contact network) of a granular material, affecting highly the material's constitutive behaviour. The discrete element method (DEM) has been used to numerically model breakage and investigate the underlying micro-mechanics that govern the fracturing behaviour (for example, [1-3]). In many cases, the lack of experimental 3D micro-scale information regarding particle deformation, significantly limits the validation of numerical models. Additionally, the models might lack the necessary input to accurately reproduce breakage behaviour.

The main objective of this work is to gather quantitative information regarding breakage of a granular material (zeolite) subjected to strain-controlled oedometric compression, using x-ray computed micro-tomography (XCT). The use of 3D imaging with XCT to study particle scale mechanics has been increasing recently (for example, [4-6]), enabling a direct comparison of various micro-mechanisms with DEM models (for example, [7-9]). The high resolution of the

images in this work allows the extraction of quantitative data about the fabric of the material and the visualisation of the spatial distribution of deformation.

## 2 Testing material and experimental campaign

A total of 6 tests were performed on zeolite specimens, all with similar initial grading (very uniform) and relative density (medium density), with a  $D_{50}$  of 1.36mm. The specimens were loaded until the majority of the particles break. Experiments were carried out with 3 different loading rates: 25 $\mu\text{m}/\text{min}$ , 50 $\mu\text{m}/\text{min}$  and 100 $\mu\text{m}/\text{min}$ , before the XCT was performed, and it was determined that the strain rate did not affect the response of the zeolite. The macroscopic response of the tests (Figure 1) shows a good match of the initial stiffness, which suggests the tests are reproducible, and a well-defined yielding. From the response it is expected that significant breakage would start to occur at an axial stress of 2MPa (Point LS3 in Figure 1). The different stages of loading (LS#), corresponding to the necessary pauses to perform

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the XCT, are indicated on Figure 1 for test OSC1 (tested at an axial loading rate of  $50\mu\text{m}/\text{min}$ ).

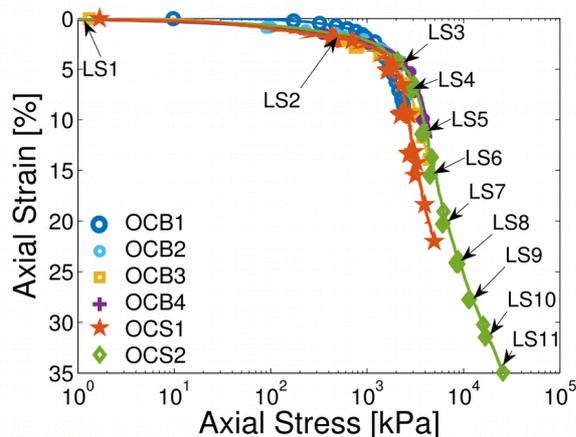


Figure 1. Macroscopic response of oedometric tests.

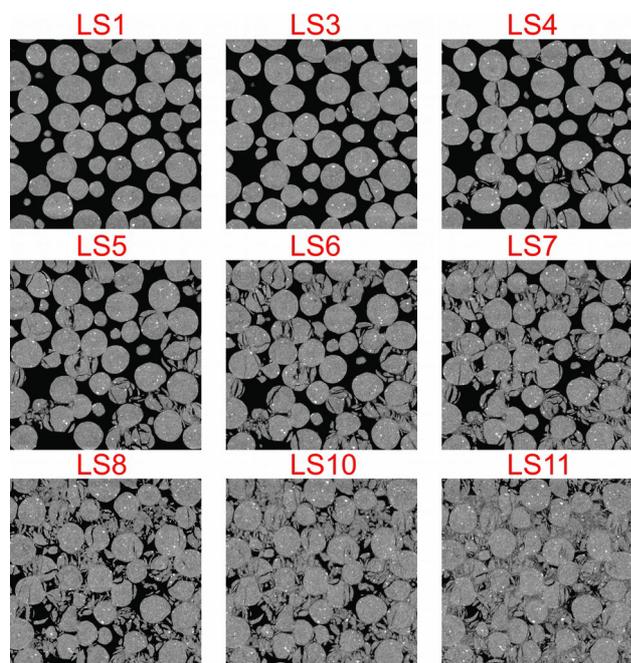


Figure 2. Extent of breakage of test OCS1 (zoomed section from XCT images).

The oedometer cell was custom-made out of PEEK (PolyEtherEtherKetone), specifically to enable XCT imaging. Two cells were built and the respective specimens have a ratio of diameter/height of 4.5/1.5 (OCB#) and 1.5/1.5 (OCS#). For brevity, in this work only results from test OCS1 are presented and compared to the DEM model. The mean diameter of each particle comprises of approximately 120 pixels (voxel size of  $12\mu\text{m}$ ), which provides plenty of detail to identify contacts and individual fragments. Figure 2 shows the evolution of breakage during testing of specimen OCS1. From a visual investigation, it is concluded that breakage develops irregularly in space and time, well after yielding (LS8), similarly to what was observed in [9]. Thereafter breakage seems to develop continuously and uniformly.

### 3 Discrete Digital Image Correlation

A discrete Digital Image Correlation (DIC) is employed, in order to incrementally identify intact and broken grains and simultaneously get results about the strain field within the specimen and the kinematics (translation and rotation) of individual grains. It is based on the code TomoWarp2, described in [10], where in this case the correlation window is adapted to each particle. Figure 3 lists the basic steps of the DIC algorithm and presents an example of a result. The grains that break are analysed regarding their spatial distribution, coordination number right before breakage, the size and shape of the intact grains and the fragments (whenever they could be identified), and the evolution of the grading of the whole specimen.

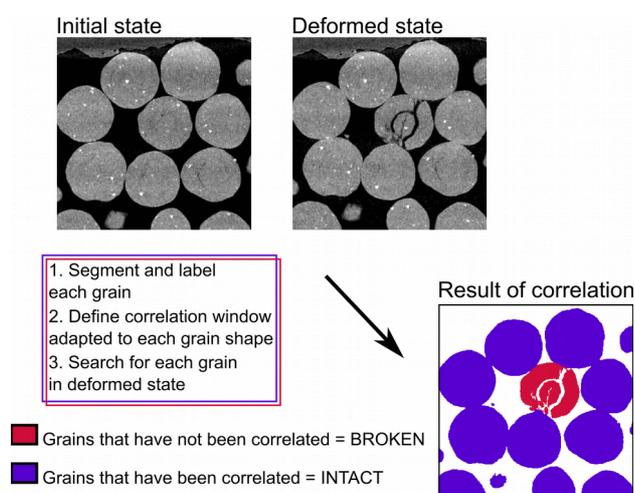


Figure 3. Schematic describing the discrete DIC algorithm.

### 4 Discrete Element Method

For the DEM simulations, a Hertz-Mindlin contact model was used to study the changes in the fabric of the specimen right before the majority of breakage occurs (Point LS4 in Figure 1). Right after this point, there is a substantial amount of softening in the macroscopic response caused by the extensive breakage that cannot be simulated by a simple Hertz-Mindlin contact law. This can also be seen in Figure 4 where after 7% of axial strain (LS4) the numerical and experimental results start to deviate.

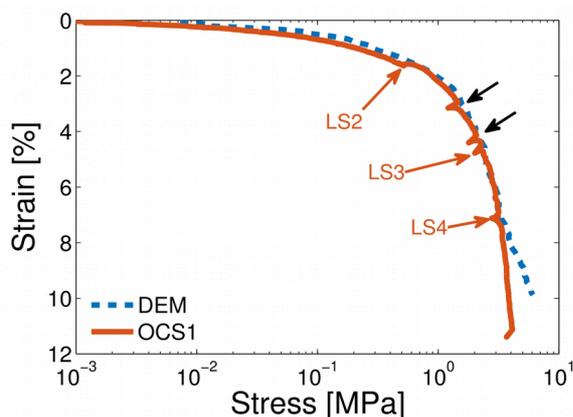
Table 1. DEM particle parameters for test OCS1.

Parameter	Value	Units
Particle density, $\rho$	2180	$\text{kg}/\text{m}^3$
Poisson's ratio, $\nu$	0.25	-
Young's modulus, $E$	4.5	GPa
Coef. of restitution, $e$	0.01	-
Coef. of static friction, $\mu_s$	0.2	-
Coef. of rolling friction, $\mu_v$	0.05	-
Mean diameter, $D_{50}$	1.36	mm
St. Dev. of $D_{50}$	0.0322	-

In this work each particle is represented by a single rigid sphere in the DEM model. An assembly of 1056 grains is generated in a virtual space and left to randomly fill a cylinder that matches the dimensions of the oedometer cell, to simulate the actual specimen and experimental test. For the simulation some friction has been assigned to the boundary (PEEK was also chosen because of its low friction). Table 1 summarises the DEM parameters chosen for this simulation. The material properties ( $\rho$ ,  $\nu$  and  $E$ ) are chosen by performing single-particle compression tests on real zeolite particles and comparing them with DEM simulations of the same test. In this first implementation the contact parameters ( $e$ ,  $\mu_s$  and  $\mu_r$ ), are assigned to match the packing density of the DEM to that of test OCS1, since the filling density will be governed by the frictional behaviour (sliding and rolling) of the particles. Finally, the physical parameters of the DEM model ( $D_{50}$  and St. Dev of  $D_{50}$ ) are an exact match to what has been measured from the images for test OCS1. In future work, in order to simulate breakage a bonded contact model will be used, as described in [11], based on the Timoshenko beam theory which considers axial, shear and bending behaviour of the bond, to create multi-sphere particles to simulate each zeolite grain.

## 5 Results

The main focus of this ongoing work is to quantify the amount of breakage from the images and study the likelihood of breakage with DEM. Figure 4 shows the results from the DEM simulation in comparison to the stress strain response of test OCS1.

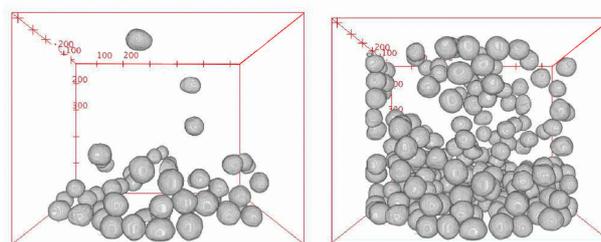


**Figure 4.** Comparison of early loading stages of DEM and test OCS1 macroscopic response.

There are various slight stress relaxations present in the experimental curve (the largest ones marked with the two black arrows in Figure 4), that are attributed to fragmentation of the micro-asperities of the particles and potentially some crack initiation. These asperities, create multiple contacts between two particles, whereas in DEM a contact occurs over an infinitesimal area and there can only be one contact point between two spherical particles; therefore, the overall DEM response is expected to be slightly stiffer than the experimental. This has been

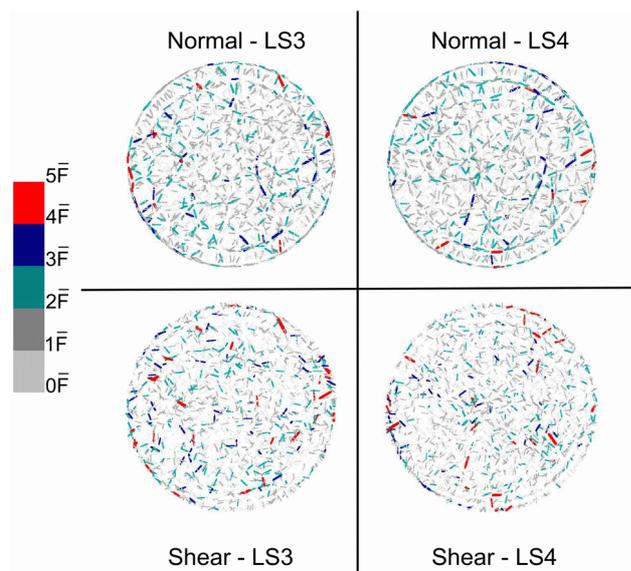
accounted for by assigning a rolling friction to the particles.

From the DIC results, it is found that no particles break until LS2, 4% of particles (42 grains) break between LS2 and LS3 and 14.5% (153 grains) between LS3 and LS4. At LS4, 18.5% of grains break since the beginning of the test. Figure 5 shows 3D images of all the intact particles that broke; it is clear that the breakage is concentrated close to the moving (bottom) boundary and the cell. Specifically, breakage initiates between LS2 and LS3 and in LS3 more than 90% of all breakage is concentrated in an area with height  $3.8D_{50}$  (measured from the moving boundary) and radially more than 70% of the breakage is concentrated in a maximum distance of  $3D_{50}$  from the cylindrical boundary.



**Figure 5.** 3D images of the intact particles that have broken in LS3 (left) and LS4 (right), from the DIC analysis.

An initial approach is to investigate the distribution of forces, both normal and shear and how they compare to the spatial distribution of breakage. Figure 6 shows 3D images from the DEM results of the normal and tangential force network at LS3 and LS4, with respect to the average normal and tangential force at each loading stage (LS3:  $F_{N,avg} = 3.64N$  and  $F_{T,avg} = 0.35N$ ; LS4:  $F_{N,avg} = 4.56N$  and  $F_{T,avg} = 0.43N$ ). As it can be seen in Figure 6, the normal forces higher than  $4F_{N,avg}$  develop radially, close to the cylindrical boundary and more than 85% of the forces higher than  $3F_{N,avg}$  are lying within a  $3D_{50}$  annular section from the cylindrical boundary. This correlates well with where the majority of breakage is concentrated. Regarding the higher shear forces they do seem to localise, however the pattern is not as clear as for the normal force network. The next step will be to study the kinematics and compare the shear and volumetric strains of the DIC analysis and the DEM results. We expect to see a localised compaction band at the initial stages of breakage close to the moving boundary as indicated by the localisation of breakage and the porosity maps created from the images. The coarse graining of the DEM results could be challenging due to the small number of grains (10 grains per oedometer diameter).



**Figure 6.** 3D images of the force network at LS3 (left) and LS4 (right), from the DEM analysis. [Top: normal and bottom: shear].

## 6 Conclusions

In summary, the recent use of XCT in geomechanics has allowed the extraction of valuable quantitative 3D information, locally or not, for entire specimens during loading. The increasing use of the DEM to study kinematics and deformation of granular materials, has led to a rising need for particle-scale quantitative experimental results (especially regarding the meso-scale) for validation. In this paper, an effort was made to interdependently validate the measurements of both the XCT and the DEM, as well as to quantitatively describe particle breakage. The main findings of this study are:

- The macroscopic response of the tests is reproducible and shows a well-defined yielding.
- Breakage initiates after 3% of axial strain (1MPa of axial stress) and it becomes substantial (18.5% total breakage) after 7% axial strain (8MPa axial stress).
- A discrete DIC is employed that facilitates the incremental study of grain breakage, in terms of fabric and spatial distribution. So far, the initial stages of loading are evaluated and it is found that there is a clear boundary effect on the spatial distribution of breakage, as it is concentrated at the moving boundary (more than 90% of total breakage) and circumferentially (more than 70% of total breakage).
- For the DEM model, initially a Hertz-Mindlin contact law is used to simulate the early stages of loading and the material particles are represented by spheres of the same diameter and overall grading.
- The DEM model can reproduce the bulk response of the material until the point where substantial breakage governs the macroscopic response and it starts to soften.

- From the results of the DEM model the force network shows some localisation of the large forces, more so of the normal forces than the shear.

In the future, breakage will be simulated with the DEM by using a bonded model to create clumps that represent each particle. The fines produced by the DEM simulations will be compared to the XCT fines, that will be measured with the technique described in [5], complemented by the discrete DIC presented in this paper. Also, the 3D images will be further investigated in order to gain more information on the contact network and how that affects the particles that break. Finally, the kinematics (stress and strain field) will be investigated and compared for both the DEM and XCT results.

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