

**3D Characterization of Force Transmission Structures and the Effect of
Particle Scale Properties on the Force Chains in Natural Silica Sand**

**A Dissertation Presented for the
Doctor of Philosophy
Degree
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DEDICATION

To Shahin, my mother, for her endless love, support and encouragement even from Heaven

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ABSTRACT

The mechanics of granular materials at the macro-scale is significantly affected by the particle-scale properties and the complex interaction of particles. Current continuum models do not address the discrete nature of the particles and their interactions and consider granular materials as homogenous materials without any inherent structures. Models that can account for all of particle-scale parameters will be very complex to implement successfully in general multi-scale finite element codes. The limited state of knowledge about the influences of particle-scale properties and their interactions influences the efficiency of such models. A comprehensive study of these properties, interactions, and their effects on the mechanics of granular materials can potentially improve the efficiency of micro-scale numerical models. Force transmission systems in granular media are affected by the particle-scale properties. This dissertation employed synchrotron micro-computed tomography (SMT) and 3D x-ray diffraction (3DXRD) microscopy to characterize crystal structure of individual particles, and force transmission structures in a specimen of natural Ottawa sand loaded under a 1D confined compression. The evolution of force structures and their properties at different load steps were examined in detail. Statistical methods were employed to investigate the effects of particle properties such as the crystal structure, contact network, and particle morphology on the force transmission structures and their evolution uncovering possible correlations between particle properties, force structures, and particle fragmentation that can be used as predictive measurements to enhance numerical models.

The complex network framework that was developed to characterize the force structures was also used to characterize the evolution of the contact network of sand particles in different specimens subjected to axisymmetric triaxial compression. A complex network property called subgraph centrality (SC) was used to quantify the variation of the contact network around individual particles. The effect of different particle-scale properties such as particle morphology and particle kinematics on the evolution of the SC of the particles during the compression of the specimen was also investigated. The change in the SC of the particles was related to the volumetric strain of the specimens to connect an abstract mathematical notion to a mechanical property that is easier to understand and measure.

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CHAPTER1

INTRODUCTION

Motivation

Current continuum models do not effectively address the discrete nature of granular particles and the complex particle scale interaction between particles such as rotation, translation, interlocking, particle morphology, crystal structure, particle size effects, and contact network. Moreover, the limited state of knowledge and experimental properties measurements of such particle properties affects the accuracy and efficiency of such models. Experimental measurements that can characterize the effect of the particle scale properties on the constitutive behavior of granular materials can significantly enhance the development of micro-mechanics-based continuum models. Force transmission systems in granular materials are significantly affected by the interactions between particles. A comprehensive experimental study on the mechanics of force transmission systems and the effects of particle-scale properties and interactions on such mechanisms can improve the numerical models commonly used to simulate the constitutive behavior of granular materials.

3D Experimental Imaging techniques such as x-ray computed tomography (CT) and synchrotron micro-computed tomography (SMT) have been widely used to quantify the kinematics of the bulk and individual sand particles (Amirrahmat et al. 2018; Amirrahmat et al. 2018; Cil et al. 2014; Desrues and Andò 2015; Druckrey and Alshibli 2017; Druckrey et al. 2016; Druckrey et al. 2018; Fonseca et al. 2012; Hall et al. 2010; Hasan and Alshibli 2012; Jarrar et al. 2018; Lenoir et al. 2007; Viggiani et al. 2014). However, CT or SMT images cannot measure the inter-particle forces, the deformation, and the strains of individual particles. Discrete element method (DEM) has been widely used to characterize

force structures in granular materials (Cates et al. 1998; Cates et al. 1999; Cil and Alshibli 2012; Majmudar and Behringer 2005; Peters et al. 2005; Radjai et al. 1998; Tordesillas et al. 2010; Tordesillas et al. 2009; Zhang et al. 2010). The DEM simulations for modeling particle morphology and quantifying individual particle strains in assemblies of a large number of particles are limited. Moreover, there is still a need for experimental measurements of contact stresses and the force transmission structure in granular materials. A few experimental methods have been used to measure the deformation and the strains of particles in granular materials. Neutron diffraction techniques have been employed by Penumadu et al. (2009), Hall et al. (2011), Wensrich et al. (2012), Kisi et al. (2014), and Kisi et al. (2014) to measure the global and local lattice strains within granular assemblies. The method adopted the concept of a gauge volume and calculated the average lattice strain within the volume gauge. Even though the gauge volume can be selected to have small dimensions to provide local average lattice strains of a small number of particles, this method fails to provide measurements for lattice strains within individual particles.

3D x-ray diffraction microscopy (3DXRD) is a promising non-destructive technique that has been used in recent years to measure the average lattice strains within individual synthetic and natural sand particles. Hall et al. (2011) used 3DXRD and radiograph scans to identify the lattice strain of individual particles in a small assembly of sand particles. Alshibli et al. (2013) and Cil et al. (2014) used 3DXRD and SMT imaging to quantify the lattice strain within particles of a specimen composed of three ASTM 20-30 Ottawa sand particles. Hall and Wright (2015) employed x-ray CT images and 3DXRD methods to quantify the lattice strains within particles in an assembly of 96 sub-spherical single crystal

synthetic quartz particles. The SMT images and the lattice strains of individual particles were combined to investigate the kinematics and grain-scale structural evolution of the assembly during the 1D loading of the particles. Cil et al. (2017) used SMT images and 3DXRD lattice strain measurements to quantify the evolution of the lattice strain of individual particles within an assembly of 35 natural Ottawa sand particles. Hurley et al. (2018) utilized 3DXRD and CT images to quantify the lattice strains and mechanics of synthetic angular particles fabricated from a single crystal synthetic α -quartz material. The primary purpose of the Hurley et al. (2018) study was to investigate the mechanics of 1D compression of angular particle packings and identify the crystal structure and lattice strains of individual particles during the loading. The specimen was composed of 313 particles.

Even though 3DXRD and SMT or CT images have been used to investigate the force structures and mechanics of granular materials, limited studies were successful in investigating the mechanics and force structures in an assembly of a large number of natural sand particles with various crystal structures and morphology.

Objectives and Original Contributions of the Dissertation

This dissertation expands on the works aforementioned studies to develop an experimental framework to inspect the force transmission mechanism in assemblies of particles independent of the crystal structure of the material, dimension of the specimen, and the number of particles in the specimen. This study utilizes SMT imaging techniques and 3DXRD lattice strain measurements to characterize the force transmission structures in

specimens of natural Ottawa sand comprised of a large number of particles subjected to 1D compression loading.

The complex network framework that was developed to characterize the force transmission mechanism is used to characterize the evolution of the contact network of sand specimens subjected to axisymmetric triaxial loading. A complex network analysis property called subgraph centrality (SC) is used as a metric to characterize the evolution of the contact network of particles both in particle-scale and mesoscale. This metric will improve current metrics that are used to characterize the fabric of granular material in particle-scale (contact number and clustering coefficient) as such metrics are limited to a specific particle and particles that are directly in contact with that particle and provide little information about the evolution of contacts in particles within the vicinity of a specific particle (meso-scale). The following is a summary of the main objectives of the dissertation:

- 1- An algorithm was developed to match the 3DXRD measurements to their corresponding particles in a porous media using 3D SMT images and 3DXRD measurements. The algorithm is independent of the dimension, shape, and the material of the specimen, and can be used in any porous media. It was employed to investigate the crystal structure of individual particles.
- 2- An algorithm was developed to characterize the force structures in the specimens based on the particle stresses and contact network. The definition of the force structures was adopted from Peters et al. (2005). However, the algorithm is enhanced, and a depth-first search algorithm was used to incorporate branching of force structures.

- 3- The evolution of the properties of the force structures such as spatial shape (i.e., force chains versus particle clusters), size, and length of the structures during different load steps were investigated.
- 4- The effects of particle-scale properties such as kinematics, crystal structure, contact number, and morphology on the fracture of individual particles, and formation and onset of force structures were examined using statistical methods to uncover possible correlations between particle-scale properties and force transmission structures in granular materials.
- 5- The evolution of the fabric of the sand specimens subjected to triaxial loading is examined using SC to expand the definition of contact number to include the evolution of the contact network of the particles in mesoscale. The effect of specimens' initial density, confining pressure, particle morphology, and particle kinematics on the variation of the subgraph centrality (SC) of the particles was investigated. Sc is connected to the variation of the volumetric strain of the specimens to provide a link from an abstract mathematical notion to a mechanical property.

Table 1.1 illustrates the roadmap for the different tasks in this dissertation.

Table 1.1. The timeline of different tasks in the dissertation

Task/timeline	2016	2017	2017	2017	2018	2018	2018	2019	2019	2019	2020
	Fa.	Sp.	Su.	Fa.	Sp.	Su.	Fa.	Sp.	Su.	Fa.	Sp.
Litreture review Research Plan											
SMT and 3DXRD scans											
Matching framework development											
Image processing											
Characterizing internal structure/ force structures											
Image processing characterizing particle-properties											
First paper											
Statistical analysis											
Characterizing the complex network properties in triaxial specimens											
Second paper											
Third Paper											
Dissertation											

Literature review

A summary of the literature is provided in this section to summarize the state of knowledge, the gaps in the literature, and how this study will fill this gap and contribute to the understanding of the force transmission structures and effects of particle-scale properties

on such structures. There are many studies reported in the literature that investigated the micro-mechanics of granular materials. The review reported in this proposal is limited to the literature that investigated the micro-mechanics of granular materials using experimental methods or characterized force transmission systems in granular materials to avoid prolonging the discussion. The experimental studies that investigated different aspects of the micro-mechanics of granular materials such as kinematics, morphology, and contact networks are reviewed first. The experimental and numerical studies that characterized the force transmission systems in granular media are discussed as well in more detail.

Experimental studies

The micromechanical behavior of granular materials has been extensively evaluated experimentally. Different methods such as photoelasticity, 2D and 3D image correlation methods and x-ray CT have been employed to study different properties of granular materials such as particle size, morphology, kinematics, and contact networks. Digital imaging techniques and computed tomography have been used to characterize particle morphology in granular materials. 2D and 3D images and image processing techniques have been used to study the particle shape and size (Al-Rousan et al. 2007; Alshibli et al. 2015; Bessa et al. 2012; Fonseca et al. 2012; Garboczi and Bullard 2013; Garbout et al. 2013; Kwan et al. 1999; Wang et al. 2005). The surface texture of particles has been measured using image processing methods, fractal geometry, fuzzy uncertainty texture spectrum, and optical interferometry (Alshibli et al. 2015; Lee et al. 1998; Wang et al. 2005).

The kinematics of granular materials has been studied using experimental methods as well. Digital image correlation methods have been used to explore the kinematics of particles and to study the mechanism of strain localization in granular materials (Hall et al. 2010; Rechenmacher and Finno 2004; Rechenmacher 2006). CT images and volumetric digital image correlation methods were used to investigate the localization of deformation in granular materials at different compressive strains (Andò et al. 2012; Hall et al. 2010; Hasan and Alshibli 2012; Sjö Dahl et al. 2012). Particle tracking algorithms have been developed to quantify the kinematics of individual particles during subsequent images, and 3D CT and SMT images (Amirrahmat et al. 2018; Andò et al. 2012; Desrues and Andò 2015; Druckrey and Alshibli 2017; Hall et al. 2010).

Even though SMT images have been proven to be very successful in quantifying the kinematics of granular materials, they cannot measure the inter-particle forces, the deformation, and strains within individual particles. Photo-elastic material has been utilized to characterize the distribution forces in 2D assemblies of disks and rods. Wakabayashi (1950) used small ring-shaped pieces of Phenolite in the sand specimen to quantify the stress in different points inside the specimen. Drescher and De Jong (1972) employed photo-elastic discs with various sizes to measure the variation of internal stresses and strain rate tensor in a 2-dimensional (2D) granular assembly. Majmudar and Behringer (2005) adopted 2D photo-elastic discs to investigate the variation in the distribution of the inter-particle contact forces during pure shear and isotropic compression. A few methods have been used to investigate the deformation and strain within individual particles as well. Neutron diffraction techniques have been adopted by Penumadu et al. (2009), Hall et al.

(2011), Wensrich et al. (2012), Kisi et al. (2014), and Kisi et al. (2014) to measure the global and local lattice strains within granular assemblies. The method utilized the concept of a volume gauge and calculated the average lattice strain inside the volume gauge. Even though the volume gauge can be selected to have small dimensions to provide local average lattice strains of a small number of particles, this method fails to provide measurements for lattice strains within individual particles. As mentioned earlier, 3DXRD is a promising non-destructive technique that has been used in recent years to measure the average lattice strains within individual particles as well.

Numerical studies modeling contact forces

The force transmission structures in granular materials have been studied using numerical models. DEM models are widely used to characterize the force structures in granular materials (Bouchaud et al. 2001; Cates et al. 1998; Cates et al. 1999; Cil and Alshibli 2012; Majmudar and Behringer 2005; Makse et al. 2000; Peters et al. 2005; Radjai et al. 1998; Tordesillas et al. 2010; Tordesillas et al. 2009; Zhang et al. 2010). The DEM models are used to quantify the distribution of inter-particle forces. Cates et al. (1998) studied the evolution of force transmission systems in a 2D assembly of fragile materials due to a jamming process. Radjai et al. (1998) investigated force distribution and the effect of the contact network on these distributions in a packing of rigid particles. Makse et al. (2000) inspected the effect of the compressibility of spherical particles on the force structures with 3D assemblies. The results were compared with the rigid spheres to investigate the effect of compressibility on the behavior of the assemblies. Peters et al. (2005) developed an algorithm that characterized the force structures with assemblies of 2D disks based on the

contact networks of the disks and principal stresses within individual disks. The algorithm was used to characterize the force structures during the advancement of punch into a half-space assembly of the disks. Tordesillas et al. (2009) examined the force structures in sphere assemblies and their failure due to buckling under strain-controlled compression. Tordesillas et al. (2010) studied the distribution of contact forces and the evolution of force structures in assemblies of disks subjected to biaxial loading. Finite element method combined with DEM has also employed by Imseeh and Alshibli (2018) to investigate the evolution force structures within an assembly of 35 natural Ottawa sand particles subject to a 1D confined compression.

Characterization of the evolution of the contact network in granular materials

There is an extensive number of studies in the literature that have investigated the evolution of the fabric of granular materials due to external stress or strain. One of the standard metrics that has been used to quantify the fabric of granular materials is the contact number of the particles in an assembly of particles (Al-Raoush 2007; Amirrahmat et al. 2018; Jang and Frost 2000; Kahn 1956; Kuhn 1999; Makse et al. 2000; Rothenburg et al. 2004). The contact number of particles provides localized information about the evolution of the structure of the granular materials, and it is relatively easy to quantify. However, it is limited as it fails to capture the effect of particles that are surrounding a specific particle but are not in direct contact with it. It is limited to particle-scale and cannot capture the effect of surrounding particles at mesoscale. Extending the definition of contact number to incorporate the behavior of particles in mesoscale can significantly enhance the effectiveness of such a metric in characterizing the mechanics of granular materials.

Network theory is the mathematical analysis of the structure of systems that are represented by networks that are defined as different subjects (nodes) and links that connect the nodes to each other. These links can represent different types of relationships between the nodes. Complex network analysis and its properties have been recently employed to characterize the evolution of the fabric in granular materials.

Smart and Ottino (2008) studied the evolution of fabric in 2D granular assemblies at the particle's mesoscale and global scale. They studied three different phenomena; namely pressure variance in disperse packings, the effect of contact loops on the stability of granular packings, and heat transfer in granular packings. Arevalo et al. (2009), Arevalo et al. (2010), and Arevalo et al. (2010) used molecular dynamics simulations and complex network properties such as contact number, clustering coefficient, and node distance to investigate the evolution of packing in a 2D assembly of disks subjected to an isotropic compression. Tordesillas et al. (2010) employed complex network properties to study the evolution of force chains and force cycles in an assembly of 2D spheres constrained to move along a plane subjected to a variety of compression and penetration external loads. Walker and Tordesillas (2010) studied the topological evolution in dense 2D granular packings from a complex network perspective. They studied the properties of the contact network of the particles such as degree, clustering coefficient, and centrality (i.e., importance) measurements. The relationships between the buckling of force structures, shear bands, and nonaffine deformation of the assembly with the evolution of the aforementioned network properties were investigated as well. Tordesillas et al. (2012) examined the variation of cluster conformations in an assembly of 1568 bidisperse sphere disks

subjected to cyclic shear under constant volume. Walker and Tordesillas (2012) characterized the rearrangement of particles in a granular assembly subjected to biaxial compression. They summarized the rearrangement of the particles in the evolution of the properties of the contact network of the particles as well as the evolution of the building structures of the assembly. Arevalo et al. (2013), and Arevalo et al. (2013) used complex network properties to investigate the difference in the evolution of the fabric of 2D granular packing subjected to tapping and regular compression loads. Shi et al. (2014) used statistical complex network properties to assess the percolation of nano-rods and their effect on the macroscopic conducting behavior of synthetic materials. Walker et al. (2014) studied the macroscopic response of photo-elastic disk assemblies subjected to prolonged cyclic pure shear under uniform compression. They used the clustering coefficient of the particles to expose the variation of the cyclic patterns that provide the support and stability for the force structures in the specimens. Walker et al. (2015) explored the structural transition of fabric in an assembly of near-frictionless granular material by tracking the transition of the 3D conformations formed by the contact network of the particles subjected to uniaxial compression. They showed how different properties such as clustering coefficient of the contact network evolved with loading. They also tracked the evolution of the prevalent conformations with loading to show how these small fundamental structures change with loading and unloading. Walker et al. (2015) quantified the variation in the number and shape of the motifs and super-families (Milo et al. 2002; Xu et al. 2008) to study the topological change in the contact network of 3D triaxial experiments. The evolution of the super-families and motifs (i.e., small structural patterns formed frequently)

of the contact networks was investigated to show how these fundamental structures evolve during the shearing and how they contribute to the change in stability of the force transmission structures and micro shear bands.

Dissertation outline

This dissertation consists of four Chapters focusing on quantifying the lattice strain, and stress within the particles in a specimen subjected to 1D compression. The lattice strains and stresses and contact network of the particles were used to characterize the force structures within the specimen. The effect of particle-scale properties on the variation of internal stresses within the particles and fracture of the particles was investigated to expose possible correlations. The complex networks framework developed to characterize force structures was used to characterize the evolution of the contact network of the particles within sand specimens, subject to axisymmetric triaxial loading. Versions of each Chapter either has been or will be published in peer-reviewed technical journals. Chapters 2 and 3 involve the concurrent employment of 3DXRD and SMT images to characterize the force transmission structures as well as the effect of the particle-scale properties on the stresses within the particles.

Chapter 2 presents a detailed description of a framework that utilizes the 3D SMT images and 3DXRD scans to quantify internal stresses within the particles in a specimen of sand particles subject to a confined compression loading. The quantified stresses and the contact network of the particles, obtained from the SMT images, were used to characterize the force structures within the specimen as well. A detailed analysis of the different mechanisms of force transmission (force chains and particle clusters) is provided in chapter

2. The variation of the length-scale properties of the force structure is also investigated in chapter 2.

Chapter 3 utilizes the particle stresses, quantified in chapter 2, as well as particle-scale properties characterized from SMT images. Statistical analysis is used to expose possible correlations between the particle-scale properties such as particle shape, contact network, particle kinematics, crystal structure, and position within the specimen with particle stresses. The effect of the contact network, particle internal stress, kinematics, position, and internal particles' stress on the fracture of the particles is also investigated.

Chapter 4 incorporates the complex network analysis to characterize the evolution of the contact network of the particles in sheared sand specimens. SC is introduced as a new metric that can characterize the evolution of the contact network of the particles better than current standard metrics such as contact number and clustering coefficient. The evolution of 4 node structures (i.e., basic contact structure with 4 particles) is investigated in more detail to see how the change in these structures affects the change in the contact network of the particles. The relationship between particle-scale properties and variation of the Sc of the particles is investigated in Chapter 4 as well.

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CHAPTER 2
3D EXPERIMENTAL MEASUREMENT OF EVOLUTION OF FORCE CHAINS
IN NATRUAL SILICA SAND

A version of this chapter is published by Siavash Amirrahmat, Wadi Imseeh, Khalid alshibli, Peter Kenesei, Zaher Jarrar, and Hemant Sharma:

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My contribution to this paper is:

- 1- The analysis of the data and creating the framework for data analysis (matching algorithm, force chain algorithm, analyzing the 3D images, creating force chains using complex network methods, implementing the codes, and validating the results)
- 2- Most of the writing and the Figures

Abstract

The mechanisms of force transmission in granular materials is a classic physics problem that has been addressed since the 19th century when Heinrich Rudolf Hertz investigated the interaction between two similar objects that were in contact under compression. However, the study of force transmission mechanisms in assemblies of more particles has proven to be a formidable problem due to the complex nature of granular materials. In recent years, synchrotron micro-computed tomography (SMT) and three-dimensional x-ray diffraction microscopy (3DXRD) have been employed to study the mechanics of granular materials experimentally. Combining SMT and 3DXRD offers unique 3D experimental measurements of the internal structure, kinematics of particles such as rotation and translation, and lattice strains of individual sand particles. In this paper, in-situ SMT and 3DXRD scans were acquired at multiple load steps for a specimen composed of 2705 natural Ottawa sand particles that were subjected to one-dimensional (1D) confined compression. An algorithm was developed to combine SMT images and 3DXRD lattice

strain measurements and was used to characterize the constitutive behavior of sand particles. The results were used to identify the crystal structure and the evolution of the stresses and lattice strains of individual sand particles. Another algorithm was developed to characterize the force structures within the specimen. Force structures were identified, and their properties such as length and their evolution through the experiment were examined. The contact number of the particles is a particle-scale property that affects the mechanics of granular materials. The effect of the contact number of sand particles on the onset and evolution of the force structures was also investigated and discussed.

Introduction

Granular materials are composed of discrete particles that have complex particle-scale properties and particle-to-particle interaction. They resist applied stress via a network of complex force structures that develop within the mass of the material. Different experimental methods have been reported in the literature to characterize the force structures/chains within granular materials. Photo-elastic materials have been widely used to visualize force transmission structures/chains. For example, Drescher and De Jong (1972) used photo-elastic discs with various sizes to measure the variation of internal stresses and strain rate tensor in a 2-dimensional (2D) granular assembly. Majmudar and Behringer (2005) used 2D photo-elastic discs to investigate the variation in the distribution of the inter-particle contact forces during pure shear and isotropic compression. Zhang et al. (2010) used a 2D assembly of photo-elastic particles to monitor the evolution and onset of force structures under a cyclic, pure shear loading. The results of these studies provided clear evidence of force structures that carry most of the applied stresses at material

boundaries. Most of the high inter-particle forces are distributed among the particles within these force chains rather than within all particles within the bulk mass of granular materials. There are a few continuum-based numerical models such as the works of Manzari and Dafalias (1997) and Andrade and Borja (2006) that used a state parameter to model the variation in the deformation behavior of different elements due to the variation in the void ratio to address the shortcoming of traditional numerical models that are based on Cam-clay or modified Cam-clay models. However, even these advanced models fail to account for the effects of the discrete nature of sand and most of the particle-scale properties such as contact network of the particles, kinematics of the particles and particle morphology on the constitutive behavior of sand. This is due to the fact that a model that can address all these parameters would be too complex to formulate and implement. Moreover, the limited number of 3D experimental measurements of the particle scale properties such as contact network, and its effects on the constitutive behavior of granular materials at a larger scale hinders modelers desire to test new theories and constitutive models. There is a pressing need to measure particle-scale properties and their effects on the force structures and mechanics of force transmission in granular materials experimentally, which will significantly encourage new developments and validation of micromechanics-based constitutive models.

X-ray computed tomography (CT) is a 3D x-ray imaging technique that has been used to measure the kinematics, particle contact network, deformation and strain field at representative elementary volume in granular materials in situ (e.g., Hall et al. (2010) Desrues and Andò (2015) Druckrey et al. (2018) Amirrahmat et al. (2018). Even though

the CT and SMT imaging techniques have proven to be excellent in quantifying the kinematics of individual particles, they cannot measure inter-particle forces, the deformation, and strains within individual particles. Discrete element method (DEM) has been widely used to characterize force chains in granular materials (Cates et al. 1998; Cates et al. 1999; Delaney et al. 2010; Majmudar and Behringer 2005; Makse et al. 2000; Peters et al. 2005; Radjai et al. 1998; Tordesillas et al. 2010; Tordesillas et al. 2009; Zhang et al. 2010). Visualizing force chains is a common output of most DEM codes. However, there are still limitations on running DEM simulations for particles shapes that closely match the physical shapes of natural sand particles (Kawamoto et al. 2018), especially if one wants to simulate a large number of particles with complex morphology. Also, there is a need for experimental measurements of contact stresses and the force network structure in order to validate the aforementioned models and provide predictive parameters that can enhance the accuracy of such models. A few experimental methods have been used to measure the deformation, and the strain of particles in granular materials. Neutron diffraction techniques have been employed by Penumadu et al. (2009), Hall et al. (2011), Wensrich et al. (2012) , and Kisi et al. (2014) to measure the global and local lattice strains within granular assemblies, where they introduced the concept of a volume gauge and calculated the average lattice strain inside the volume gauge. Even though the volume gauge can be selected to have small dimensions to provide local average lattice strains of a small number of particles, this method fails to provide measurements for lattice strains within individual particles.

3DXRD is a promising non-destructive technique that has been used in recent years to measure the average lattice strains within individual particles. Hall et al. (2011) used 3DXRD and radiograph scans to identify the lattice strain of individual particles in a small assembly of Ottawa sand particles. Alshibli et al. (2013) and Cil et al. (2014) used 3DXRD and SMT imaging to quantify the lattice strain within particles of a specimen composed of three ASTM 20-30 Ottawa sand particles. Hall and Wright (2015) employed x-ray CT images and 3DXRD methods to quantify the lattice strains within particles in an assembly of 96 sub-spherical single crystal synthetic quartz particles. The SMT images and the lattice strains of individual particles were combined to investigate the kinematics and grain-scale structural evolution of the assembly during 1D compression of the particles. Cil et al. (2017) used SMT images and 3DXRD lattice strain measurements to measure the evolution of lattice strain of individual particles within an assembly of 35 natural Ottawa sand particles. Hurley et al. (2016) used CT, 3DXRD, and optimization methods to quantify the inter-particle forces in an assembly of 77 spherical quartz particles and characterize force chains within the assembly. Hurley et al. (2018) utilized 3DXRD and CT images to quantify the lattice strains and mechanics of synthetic angular particles fabricated from a single crystal synthetic α -quartz material. A single crystal block was chiseled to produce fragments, and these fragments were subjected to ball milling and vibratory sieving. A specimen composed of 313 particles with a grain size passing US sieve #60 (250 μm) and retained on US sieve #80 (170 μm) were used in their study. The main purpose of the study was to investigate the mechanics of 1D compression of angular particle packings and to identify the crystal structure and lattice strains of individual particles during the loading.

Even though 3DXRD and SMT or CT images have been used to investigate the force structures and mechanics of granular materials, limited studies were successful in investigating the mechanics and force structures in an assembly of a large number of natural sand particles with various crystal structures and morphology. This paper builds on the works of the aforementioned studies and combines 3DXRD measurements and SMT images to study the lattice strain of individual particles within a specimen of natural Ottawa sand composed of 2705 particles that was subjected to 1D confined compression. Lattice strains within the particles were measured using 3DXRD and were used to calculate the stresses within the particles by adopting the appropriate anisotropic constitutive model of α -quartz crystal. The force structures within the specimen were characterized at different load steps to investigate the evolution of the force structures during the experiment. The effects of contact number of particles on the onset and formation of the force structures were examined as well.

Experiment

Particle Size fraction between the US sieve #100 (0.149 mm) and sieve #140 (0.105 mm) of natural silica sand known as F75 Ottawa sand with a specific gravity of 2.65 (mined from Ottawa Illinois and marketed by US Silica company) was used in this study. This sand is composed of 99% α -quartz mineral and has been used widely by the geotechnical community. The confined 1D compression of the specimen was conducted using a special apparatus described in Cil et al. (2017). Referring to Figure 2.1a, the 1D compression cell consists of an acrylic specimen mold, a plastic tube to encase the sand grains, a loading system with a stepper motor applying axial compression, two load cells and an acrylic

jacket that provided support for the top and bottom cell endplates. The acrylic mold surrounding the specimen has an outside diameter of 15 mm and an inner diameter of 2 mm. It is difficult to drill a smooth small-diameter hole inside acrylic as hard silica sand particles would lodge and indent the rough wall of the mold during compression. Moreover, the lateral forces between the rough surface of the acrylic mold and particles increases and create substantial friction between the wall and particles. In order to offer a smoother surface to interact with the sand particles, a plastic tube with an inner diameter of 1.650 mm was inserted inside the acrylic mold. The acrylic mold was used to provide the necessary lateral support for a k_0 stress path during the experiment. Sand particles were deposited inside the plastic tube using dry pluviation with the help of a very small aluminum funnel that was specially fabricated for this purpose. The specimen had a height of 3.720 mm prior to loading and initial density of 1.41 g/cm^3 (initial void ratio is 0.879). Two load cells with resolutions of 0.35 N and 0.56 N were used together at the top and the bottom of the specimen to measure the applied axial load and account for friction between sand grains and plastic tube. A stepper motor was used to apply axial compression at the top of the specimen at a constant displacement rate of 0.2 mm/min. The specimen was compressed to four pre-defined global axial strains of 4.35%, 6.71%, 8.35%, and 11.47% and the experiment was paused at each strain to acquire SMT and 3DXRD scans.

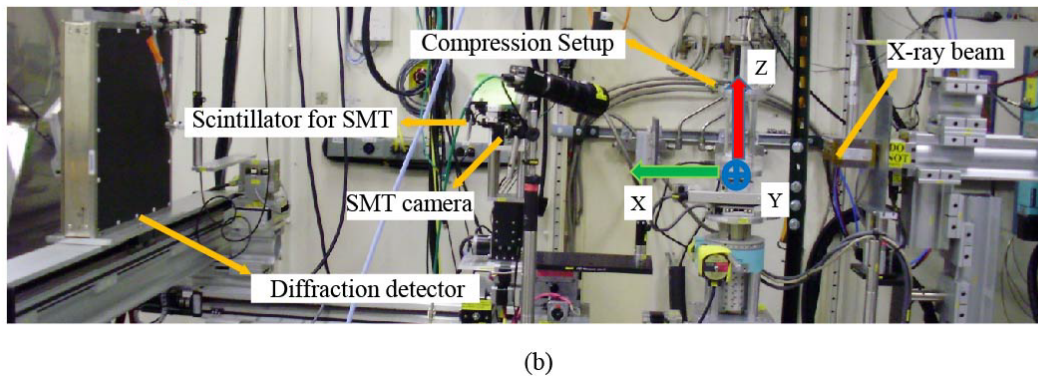
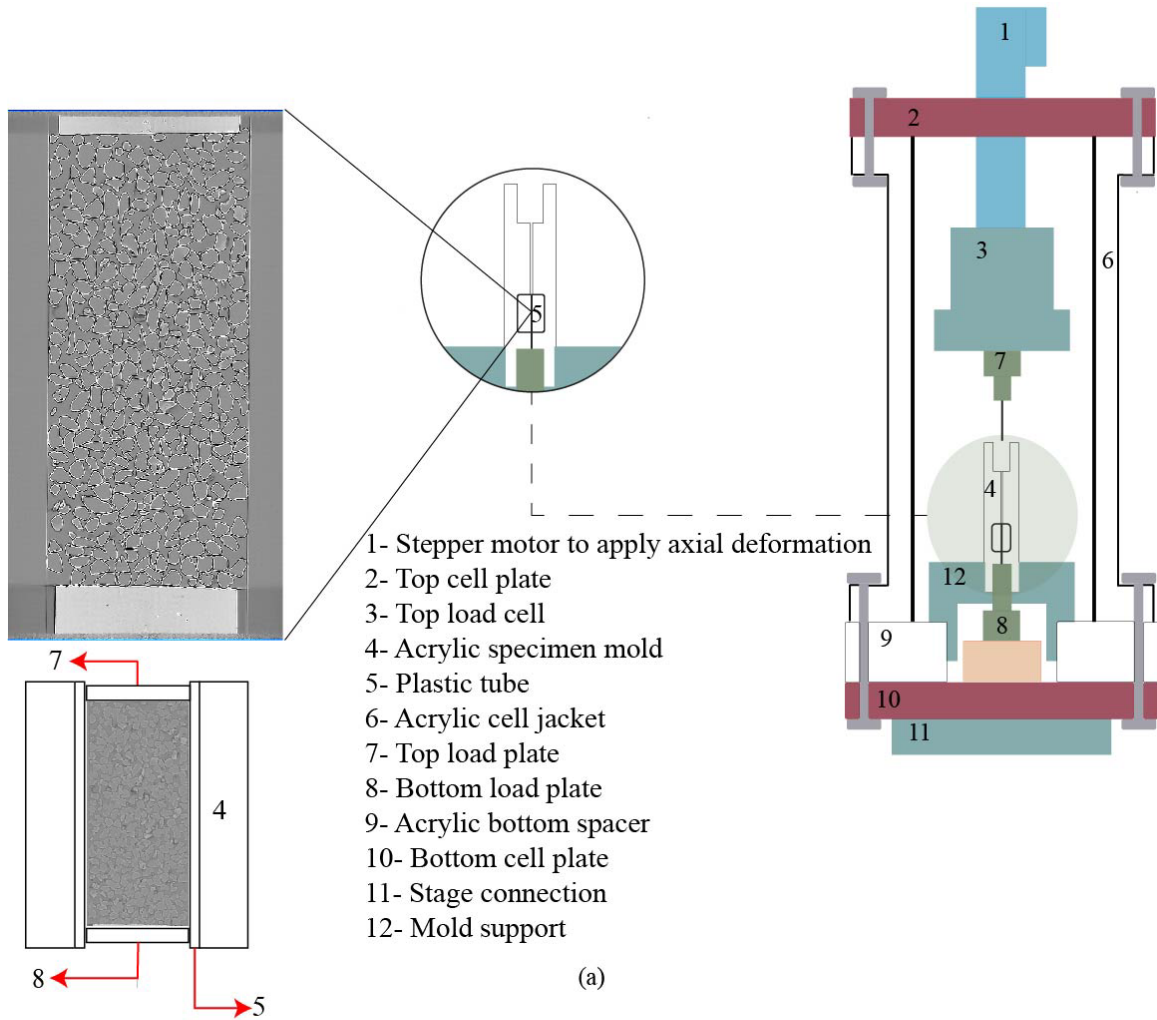


Figure 2.1. (a) Schematic of the 1D compression apparatus;(b) Experimental setup at the APS beamline 1-ID

SMT imaging and 3DXRD scans

The SMT and 3DXRD scans were acquired at beamline 1-ID of the Advanced Photon Source (APS), Argonne National Laboratory (ANL), Illinois, USA. The high precision setup (Benda et al. 2016) provided by the beamline allowed the specimen to translate along any direction (Figure 2.1b), and rotate about the vertical axis, and permitted tip-tilt alignments. The x-ray beam defined the horizontal x-axis; the rotation axis defined the z-axis and was carefully aligned vertically and perpendicular to the x-axis. The third orthogonal direction is denoted as y-axis using a right-hand coordinate system. The axis of the cylindrical specimen and thus the loading direction was aligned with the z-axis. The SMT detector was mounted on a horizontal translation stage that allowed the reproducible motion of the detector between the specimen and the diffraction detector. The x-ray beam passed through a single crystal scintillator and the radiographic images were captured using 1960 x 1080 pixel resolution PointGrey Grasshopper3 CMOS camera. The scans were acquired using a 2 mm wide and 1.1 mm high x-ray box beam that had an energy of 61.32 keV. X-ray scans were acquired using incremental rotations of 0.2 for the total rotation of 360° and an exposure time of 0.25 seconds resulting in a total of 1800 frames. The SMT scans were reconstructed to obtain the 3D structures with a spatial resolution of 1.17 $\mu\text{m}/\text{pixel}$ which are referred to as SMT images for simplicity.

The 3DXRD scans were also acquired at the same load steps using the same setup and x-ray beam. For each load step, four scans each with a height of 0.95 mm were collected and saved to cover the entire height of the specimen. The beam size was 1.3 mm $\times$ 0.7 mm to ensure a consistent beam intensity through the height of the scan for accurate measurements

of lattice strains. The 3DXRD scans were acquired at an angular rotation interval ($\Delta\omega$) of 1° for a total rotation of 360° . An amorphous silicon flat-panel detector (GE Revolution 41RT, (Lee et al. 2008) with the dimensions of $410 \text{ mm} \times 410 \text{ mm}$ was positioned at a distance of about 1392 mm from the specimen. It has a resolution of 2048×2048 pixels with a pixel size of $200 \times 200 \text{ }\mu\text{m}$. Prior to loading, SMT images and 3DXRD scans were acquired for the initial state of the specimen. Then, the specimen was compressed until the global target strain was reached (i.e., 4.35%, 6.71%, 8.35%, or 11.47%.) and loading was paused to acquire the next scan.

Data analysis and results

Figure 2.2a shows the global axial displacement versus the axial force relationship where the axial force generally increased except during the relaxation periods when the axial force dropped since loading was paused to acquire the SMT and the 3DXRD scans. The friction between the particles and the plastic tube remained relatively constant throughout the experiment (3.0 N during the first load step to 3.7 N at the fourth load step). The slight increase in the friction was due to the change in the packing of particles and some sand particles pushed against the plastic tube, which increased the friction between the particles and the tube. Figure 2.2b depicts the variation of the void ratio versus the global axial stress. The stresses are calculated based on the axial loads during the scans after the relaxation phase was finished. The specimen was subjected to vertical stresses as high as 9 MPa at fourth load step which are higher than typical vertical stresses common in many geotechnical projects. However, there are field conditions where sand can experience very high stresses such as sand around the tip of a driven pile, deep drilling, high strain loading,

and mining activities. The objective of this paper is to investigate the evolution of force structures and force transmission mechanisms within sand using innovative nondestructive 3DXRD and SMT techniques. Therefore, high stresses need to be applied to cause particle fracture and also cause the force structures to develop and evolve. The specimen exhibited a large initial change in void ratio during the first load step as it was in a loose state. However, after the first load step, the variation in the void ratio was much smaller when compared with the first load step. The void ratio of the specimen increased during the third load even as the specimen was compressed, which indicates an increase in the diameter of the specimen (i.e., bulging) during this load step. In order to characterize bulging in the specimen, the profile of the middle section plastic tube wall at different loads was superimposed on each other. The left boundary of the plastic tube (from the middle axial section) at different loads were superimposed on top of each other, and the right boundaries were plotted in Figure 2.3g to show bulging of the specimen. The SMT images offered an excellent visual aid to assess the kinematics and rearrangement of the particles while the 3DXRD measurements provided measurements of the deformation and lattice strains of individual particles.

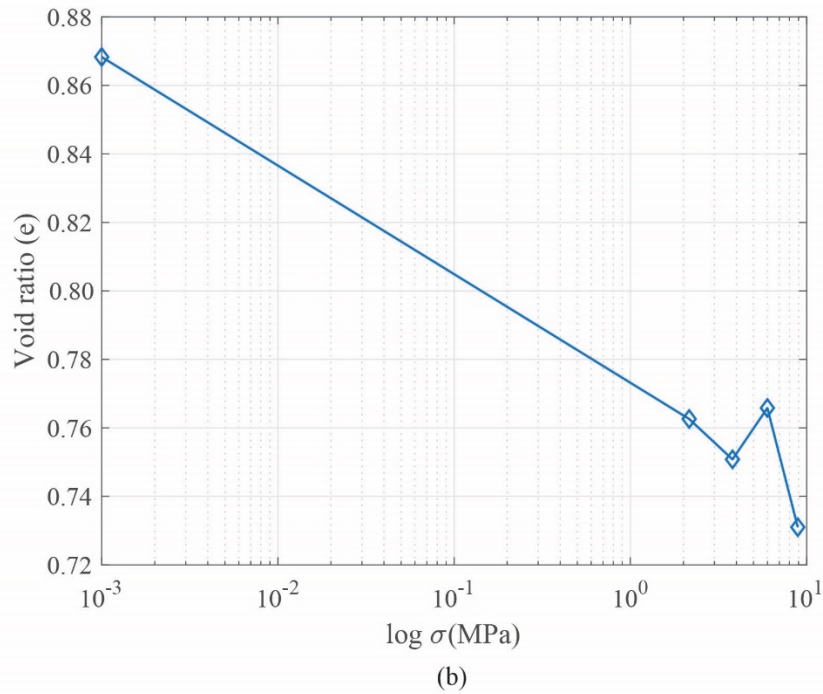
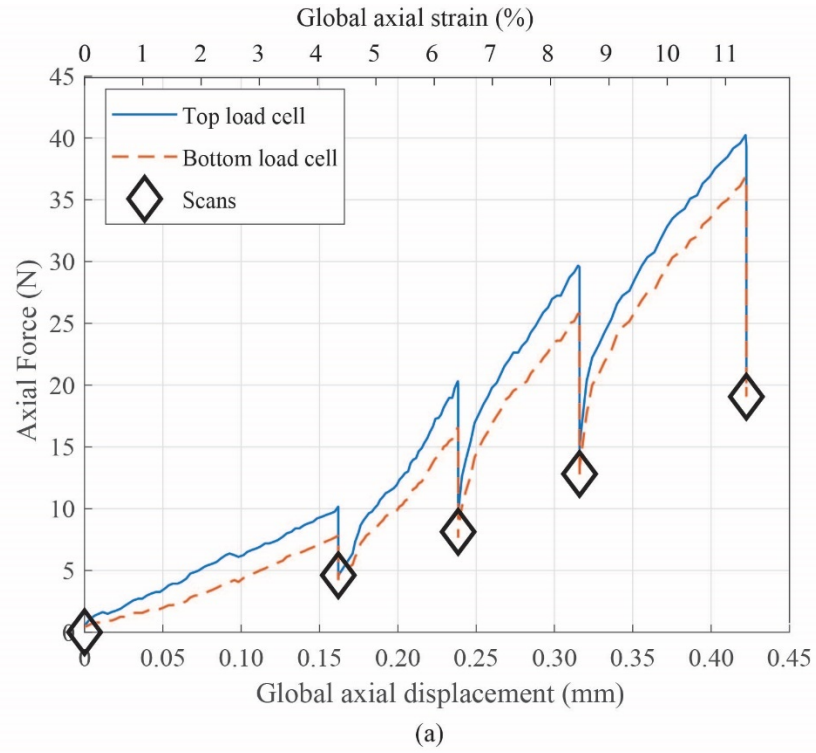


Figure 2.2. (a) Global axial displacement versus axial force relationship during the initial and the following four consecutive load steps as recorded by the data acquisition system (b) The variation of void ratio during different load steps

Figure 2.3 displays central axial SMT slices of the specimen at the aforementioned load steps. During early load steps, particles rearranged throughout the specimen creating a denser packing (Figure 2.3b&c). The plastic tube began to bulge during the third and fourth load steps due to the high lateral pressure in the sand specimen against the tube walls (Figure 2.3d&e) while 19 particles fractured into multiple fragments during the third load followed by the fracture of more particles (187 particles) during the fourth load step. The majority of fractured particles were close to the top and bottom of the specimen adjacent to the loading plates. The fractured particles were identified using the fifth SMT scan at an axial load of 37.5 N and a global axial strain of 14.03%. The fracture of particles affected the analysis of the 3DXRD data as each sand particle disintegrated into multiple fragments which made it difficult to track diffraction spots of a large number of fragments; therefore, this study is limited to the behavior of the particles during the first four load steps, and the fifth step was omitted from the analysis as the ratio of fragmented particles to the total number of particles increased rapidly.

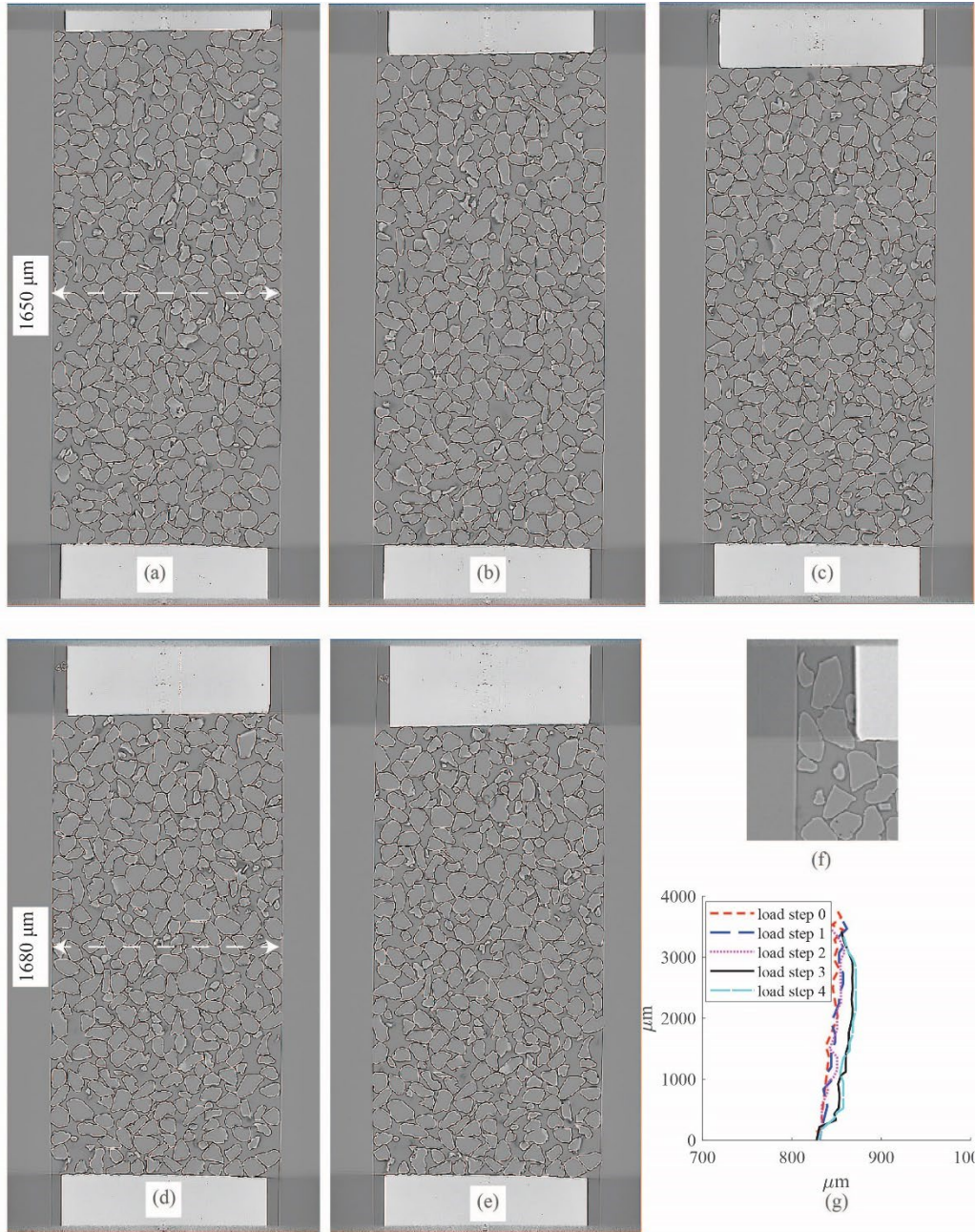


Figure 2.3. SMT images of central axial cross-sections in the specimen at global axial strains of (a) 0%; (b) 4.35%; (c) 6.71%; (d) 8.35%; and (e) 11.47%; (f) Examples of particles that moved into the gap between the load plate and tube inner surface during the third load step; (g) The profile of the right side of the inner surface of the plastic tube in the middle cross-section

Analysis of 3DXRD measurements

3DXRD scans were used to calculate lattice strains within individual sand particles. The incident monochromatic x-ray beam diffracted on the crystals and the diffracted intensity was measured on the detector for those lattice planes which satisfied the geometrical condition formulated in Bragg's law:

$$2d_{hkl}\sin\theta_{hkl} = n\lambda, \quad (2.1)$$

where λ is the wavelength of the incident beam, n is a positive integer (in this case it is 1), and θ_{hkl} is the angle of the incident beam with respect to the lattice planes denoted by h, k , and l Miller indices with distance d_{hkl} (Figure 2.4a). As the specimen rotated during the 3DXRD scan, the lattice planes within the scanned volume came into Bragg condition and could be observed on the Debye–Scherrer rings on the diffraction detector (Figure 2.4b). The precise position (Figure 2.4c) of the diffraction spots were used to quantify the lattice strains within individual particles by evaluating the change in d_{hkl} in Bragg's law. The diffraction analysis used the MIDAS software package (Sharma et al. 2012). In this analysis, after the calibration procedure, first all diffraction spots and their corresponding crystal grains were determined, this procedure is called indexing. Then the diffraction spot positions measured from the same crystal grain were used for fitting its orientation (3 parameters), center of mass position (3 parameters), and lattice strain tensor (6 parameters) simultaneously in a least mean square sense through a 12 dimensional search for the solution of a highly over-determined equation system resulting from the precise measurement of the diffraction geometry and application of Bragg's law. The method is based on the principle that the diffraction spots measured in different directions and at different specimen angle are a direct measure of a certain projection of the strain. Knowing the precise position of the specimen and the diffraction spots provides many samplings of the strain in various projections both in the laboratory

and the crystal frame, which makes this procedure successful for strain tensor evaluation. Hence measuring the ε_{hkl} strain perpendicular to a specific (hkl) lattice plane can be obtained using the following formula

$$\varepsilon_{hkl} = \frac{d_{hkl}^0 - d_{hkl}}{d_{hkl}^0} \quad (2.2)$$

where the parameter d_{hkl} is calculated from Bragg's law, d_{hkl}^0 is corresponding value to the unstrained lattice prior to the loading. In this notation, compressive strains were considered to be positive. The procedure to calculate lattice strains is discussed only briefly here to avoid lengthening the paper. For further details about the setup calibration aspects, calculation and accuracy of the lattice strains the reader is referred to the literature (Alshibli et al. 2013; Borbely et al. 2014; Cil et al. 2017; Moscicki et al. 2009).

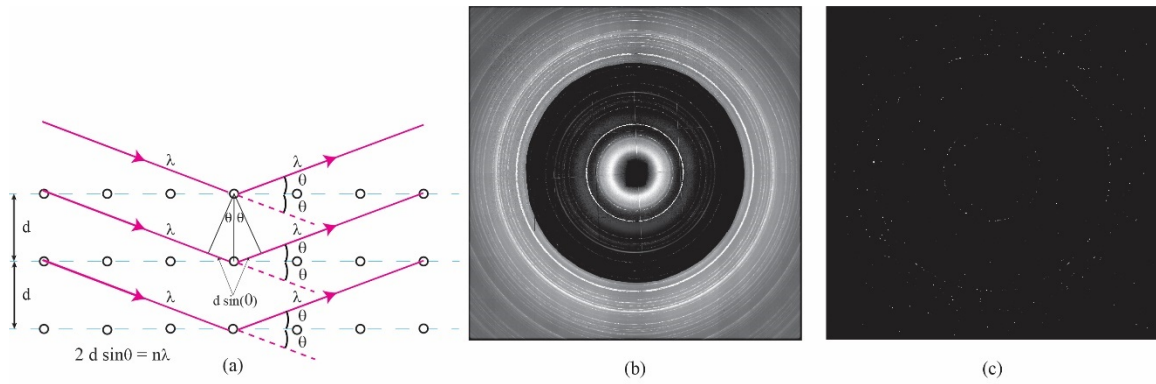


Figure 2.4. (a) Schematic illustration of Bragg's diffraction; (b) Debye Scherrer rings for all projections during the initial step prior to the loading (c) Diffraction peaks for an individual projection during the initial step prior to the loading

Matching algorithm

After processing the raw 3DXRD measurement data with MIDAS, the output file saves for each sand grain crystal the lattice strain tensor expressed according to the global x, y, z coordinate system, the orientation matrix, the spatial coordinates of the centroid of the crystals, the equivalent radius of the crystal and the error between the measured and fitted coordinates of these centroids. It is difficult to make a simple match between 3DXRD measurement to its corresponding particle merely based on these measurements, especially if some sand particles contain several crystals. Hence, an algorithm was developed by the authors to incorporate the SMT images with the 3DXRD measurements to match the measurements to their corresponding particles. The outline of this procedure is described briefly. The SMT images were first processed to generate a binary image of the sand particles using Avizo Fire V9.5 commercial imaging code. Segmented images were then further processed using a segmentation algorithm to separate touching particles, and each particle was labeled with a unique integer. Details of image processing steps and necessary image enhancements are described in Druckrey et al. (2016). Figure 2.5 displays an example labeled image of the specimen at the first load step (4.35% axial strain) alongside with the corresponding 3DXRD measurements. The filled red dots in Figure 2.5b represent the 3DXRD measurements that show the coordinates of the centroids of the crystals. Then the labeled images were used to generate an array that stored the coordinates of all pixels based on the global coordinate system (Figure 2.1b) at the resolution of the 3D SMT images (1.17 μm). Each particle was represented by a cloud of points based on SMT images (Figure 2.6a&b). The labeled image and the point clouds are very similar in concept as a

labeled image is a set of voxels that belong to the particle and have an intensity equal to the label of the particle. However, the point cloud is a set of points, and as a result, mathematical concepts such as distance between two points are defined much easier and less ambiguous when compared with the voxels which are a volume. The coordinates for the pixels comprising individual particles and void spaces were stored in the array. The array also stored the corresponding particle labels for each individual pixel to facilitate the backtracking of the coordinates to particles. Label zero was used to represent void space.

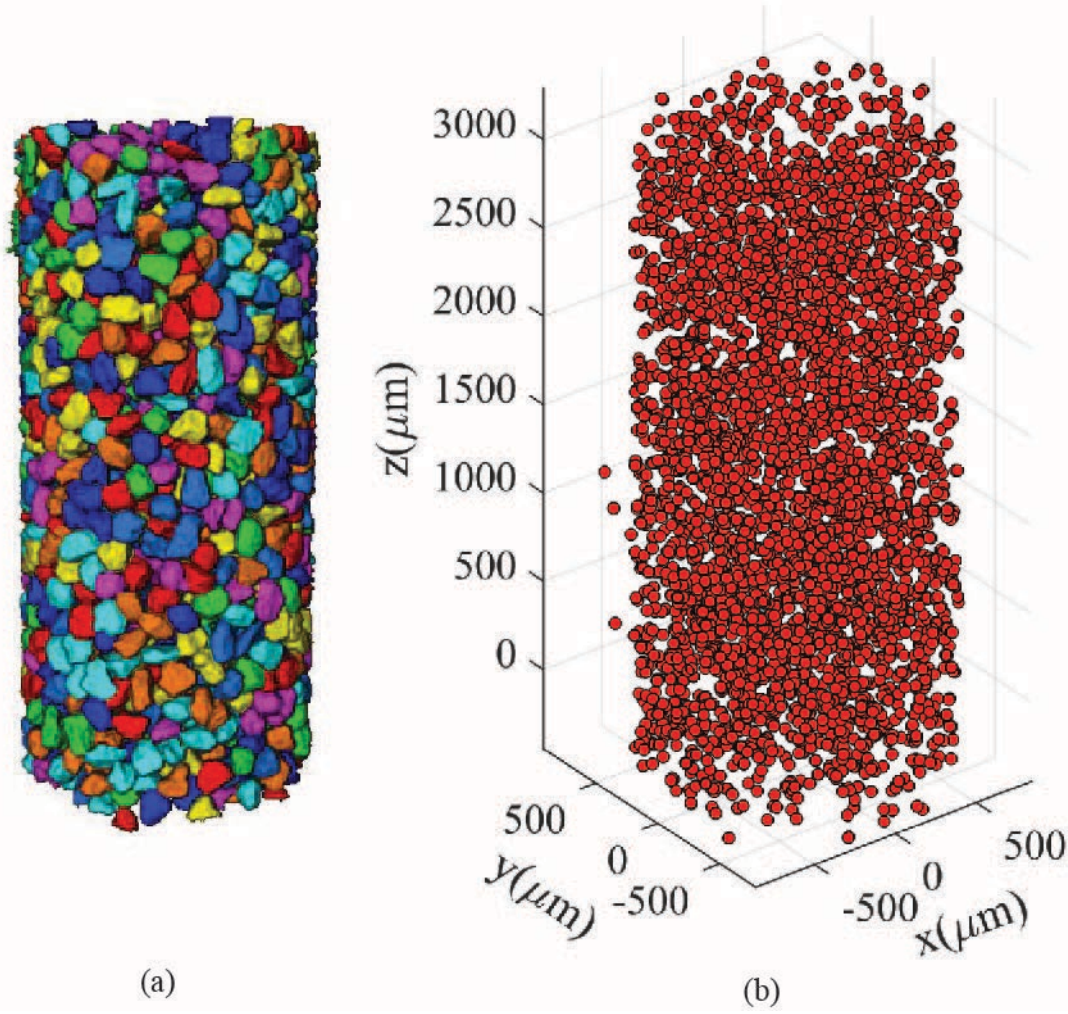


Figure 2.5. (a) labeled SMT image; (b) Coordinates of the 3DXRD measurements (i.e., the red dots represent the 3DXRD measurements, not to be confused with the centroid of particles) at 4.35% global axial strain (numbers are in μm)

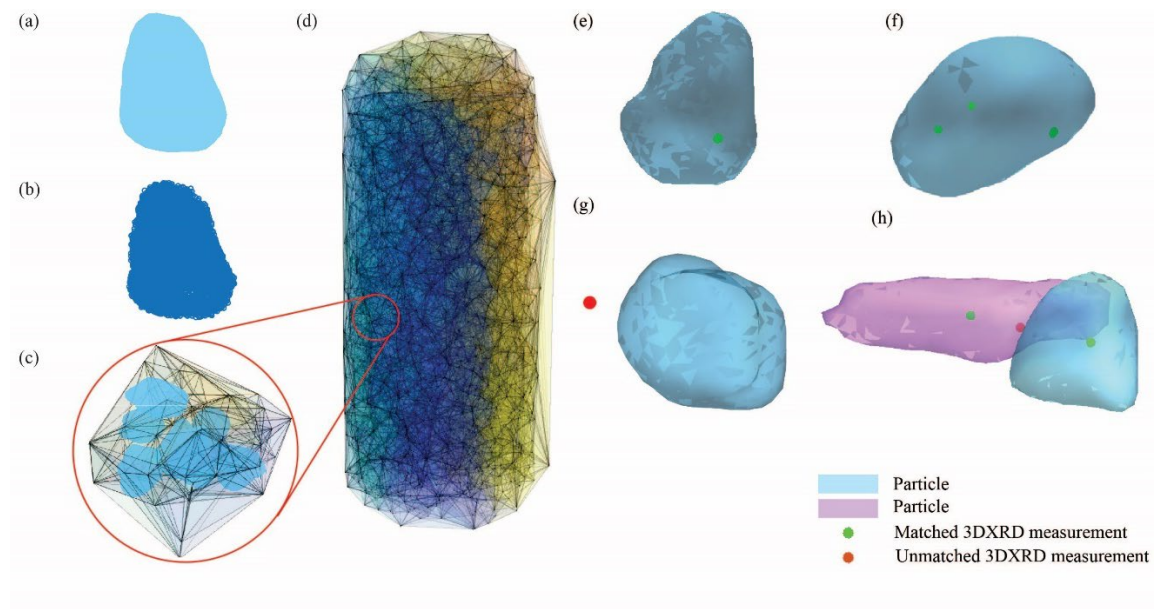


Figure 2.6. (a) Labeled SMT image of an individual particle; (b) point cloud representing the same particle; (c) details of the tessellation; (d) tessellation of the specimen volume using 3DXRD measurement coordinates (not to be confused with the centroid of particles). (e) An example particle with a matched 3DXRD measurement; (f) a particle with multiple matched 3DXRD measurements; (g) a particle with a matched 3DXRD measurement with a distance outside the acceptable accuracy; and (h) matched 3DXRD measurement with multiple particles within the acceptable tolerance.

The coordinates of the centroid of the crystals were used to divide the volume of the specimen into cells using the Delaunay tessellation method (Figure 2.6c&d). The tessellation cells are a combination of tetrahedron cells that uses the centroid of the crystals based on the 3DXRD measurements as their vertices. The cells divide the volume of the specimen into different units. Each cell was used to generate a list of possible crystal matches that are closest to each particle. The same list can be made by creating a list of all 3DXRD measurements with the shortest distance to each point of the point-cloud representing individual particles. However, the tessellation approach is more

computationally efficient as it does not need to calculate the distance between every point in the point clouds and crystal centroids. Moreover, the distance between the vertices of a cell was only computed for particle points within that cell. In the next step, the list of possible matches for an individual particle was investigated to make sure that 3DXRD measurements assigned to a certain particle were closest to the same particle as well (i.e., the distance of the 3DXRD measurement to the particle is smaller than the distance of the same 3DXRD measurement to neighbor particles). The measurements that met this criterion were assigned to the particle while the rest of the measurements sent back to the list containing the unmatched measurements. This method prevented a systematic error where a 3DXRD measurement was assigned wrongfully to a particle just due to the order that the particles were labeled and this procedure was repeated until all the measurements were matched with the particles. Once 3DXRD measurements were matched with the particles, the list of possible matches was filtered to eliminate matches with shortest distances to a particle (i.e., the minimum distance between the measurement and all pixels representing the particle) higher than the accuracy of the measurement coordinates and matches that have multiple neighboring particles within the accuracy of the measurement. Figure 2.6e to f shows an illustrative example of particles and their matched 3DXRD measurements.

The variation in the number of 3DXRD measurements, the total number of particles and the number of matched particles during the experiment are depicted in Figure 2.7. Table 2.1 summarizes the variation of the number of particles, the total number of 3DXRD measurements, the number of Orphan particles, and the number of Orphan 3DXRD

measurements. During each 3DXRD scan, the diffractions from the specimen were analyzed, and the number lattice planes that satisfied the Bragg's condition and were accurately tracked during different load steps (i.e., met the retention factor) were used to measure crystal strains within the particles. The number of such reading differs from one load step to the other (indexing process) and is referred to as the total number of 3DXRD measurements in this study. During the first two load steps (load 0 and 1) the number of particles was consistent and was equal to 2705. The number of particles in the specimen varied slightly during the second, third, and fourth load step for two reasons. First, 11 particles moved outside of the specimen and into the gap between the loading plate and the plastic tube, and as a result, they were omitted from the analysis. Figure 2.3f shows an examples of these particles during the second load step. Moreover, 19 particles fractured during the third load step, which caused an increase in the number of particles during the third and fourth load step. During the fourth load step, to capture all the fragments from the fractured particles, the volume limits during the image processing were decreased and as a result, the number of particles increased to 2738. The number of particles with over-segmentation in the specimen during different loads was smaller than 13 particles during all the loads. There were some particles and 3DXRD measurements that did not have a particle match or a suitable 3DXRD measurement match that are labeled as orphan particles and orphan 3DXRD measurements. The orphan particles are particles that do not have a matched 3DXRD measurement either due to the intensity threshold used to identify the diffraction peaks or because the 3DXRD measurements corresponding to the particle do not meet all matching criteria. The Orphan measurements are measurements that were not

matched with particles either due to the unacceptable distance that the measurement had to the particles (i.e., more than the error of the fitted coordinates) or because they had multiple possible particle matches within the error of the measurement. After the matching process was finished, a merging algorithm was deployed to investigate the internal structure and the orientation of the crystals in individual particles. The algorithm is discussed in the following Section.

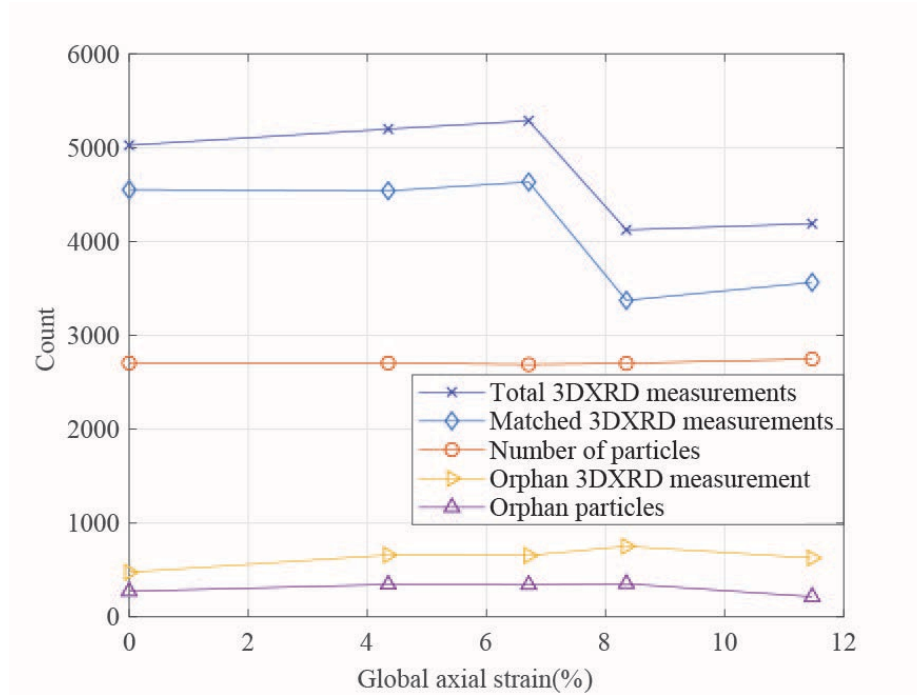


Figure 2.7. Variation of the total number of particles, total number of 3DXRD measurements, number of Orphan particles, and Orphan 3DXRD measurements during different load steps.

Table 2.1. Summary of variation of the total number of particles, the total number of 3DXRD measurements, number of Orphan particles, and Orphan 3DXRD measurements during different load steps

Items	Step 0	Step 1	Step 2	Step 3	Step 4
Number of particles	2705	2705	2689	2701	2738
Total number of 3DXRD measurements	5027	5199	5288	4124	4191
Number of orphan particles	271	344	340	352	213
Number of orphan 3DXRD measurements	474	518	653	751	626

Internal structure, merging algorithm, and stresses within the particles

After matching the 3DXRD measurements with the associated particles, measurements were examined to characterize the crystal structure of individual particles within the specimen. For every 3DXRD lattice strain measurement, a corresponding crystal orientation was determined in terms of a rotational matrix that transformed a local orthogonal coordinate system fitted inside quartz trigonal crystal structure into the global orthogonal coordinate system (c). Local orientations quantified within each particle were then examined to identify the crystal structure of each particle (i.e., single-crystalline or polycrystalline particle). If two 3DXRD readings within a particle have orientations within 1° deviation in direction they were considered as a single crystal (Figure 2.8a). Interestingly, most of the particles with different local orientations exhibited local orientations with 60° , 120° , 180° rotation about the local crystal c-axis. α -quartz has a trigonal crystal structure that exhibits 3-fold symmetry in the c-plane such that the crystal repeats itself every 120° rotation about the c-axis (Figure 2.8b). Therefore, local

orientations with 120° rotations about the c-axis were considered equivalent, and the particle was classified as a single crystal particle.

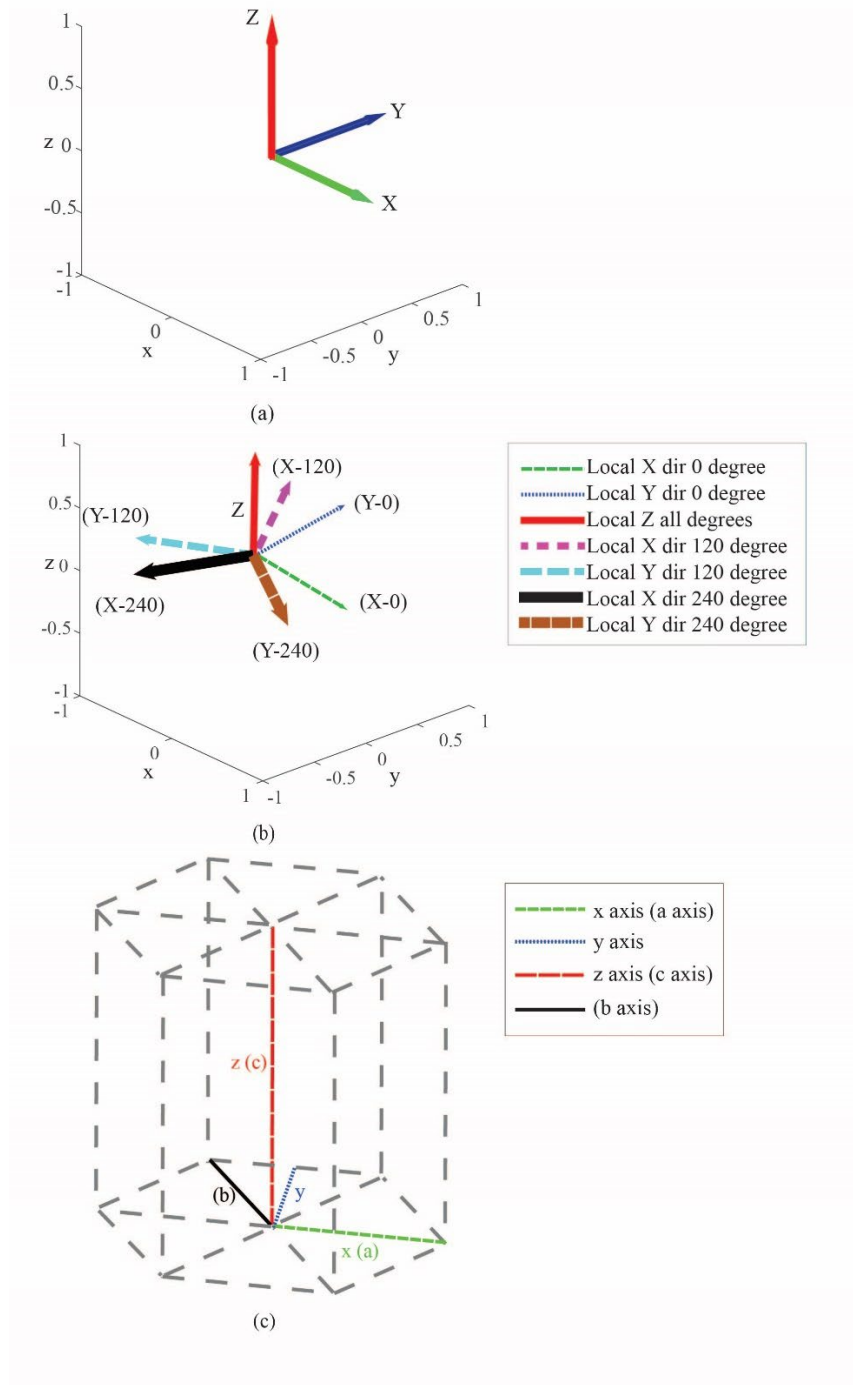


Figure 2.8. The local coordinate system for particles with (a) single crystals; (b) multiple crystals with 0°, 120°, 240° rotations around the crystal c-axis; (c) The local coordinate system fitted inside an α -quartz crystal lattice.

Particles that had multiple 3DXRD reading that did not meet the aforementioned criteria were considered to be polycrystalline. Twinning is very common in quartz, and it develops when separate crystals share lattice and grow with different spatial operations (e.g., reflection, rotation or inversion) with respect to each other. One of the most common twinning structures in quartz is Dauphiné twinning where two inter-grown crystals have similar c-axis, but the a and b axes are rotated 60° about the c-axis (Fron del 1945). Dauphiné twins should be considered as single crystals as they are two intergrown crystals. However, they make multiple diffraction spots due to the rotation of the crystals around the c-axis. Identifying the twins is complicated and requires further investigation; therefore, particles containing twins are considered as polycrystalline particles in this study. During the initial state before loading, 1339 particle out of the 2345 matched particles had single crystalline structures. This number decreased slightly during the first and second load steps (1108 and 1087, respectively) as the total number of 3DXRD measurements increased (from 5027 to 5199 and 5288). The third and fourth load steps were omitted from the analysis as the total number of 3DXRD measurements decreased significantly during these steps (4124 and 4191, respectively).

Once a particle is classified as a single crystal particle, a merging algorithm was implemented to average the measured multiple crystals strains. Lattice strains of similar crystals were averaged based on the volume of the corresponding crystals. After the strains were averaged, they were converted into stresses using Hook's law with the appropriate anisotropic elasticity matrix (C_{ijkl}) for α -quartz mineral (Heyliger et al. 2003). Table 2.2

summarizes the elastic moduli for both isotropic and anisotropic models used in this study. The strains in the global coordinate system were converted into strains in the local crystal coordinate system based on the orientation matrices, and then the strains in the local coordinate system were multiplied by the appropriate elasticity matrix to calculate the stresses in the local coordinate systems. The calculated stresses were transformed back into the global coordinate system. The process is summarized in the following formulas:

$$[\varepsilon'] = [O^T][\varepsilon][O] \quad (2.3)$$

$$[\sigma'] = [C][\varepsilon'] \quad (2.4)$$

$$[\sigma] = [O][\sigma'] [O^T] \quad (2.5)$$

where ε is the strain tensor of a particle in the global coordinate system, ε' is the strain tensor in the local crystal coordinate system and C is the elasticity matrix. σ and σ' are the stress tensor in the global and local coordinates systems, respectively. O is the orientation matrix that converts the local crystal coordinate system into the global coordinate system, and O^T is the transpose of O . Figure 2.9 shows the distribution of stresses (σ_{ij}) prior to and during the experiment at different load steps. Table 2.3 summarizes the variation of stresses within particles during different load steps. The average of the normal stresses (σ_{xx} , σ_{yy} , and σ_{zz}) increased during the experiment except during the third load, as the specimen experienced bulging, rearrangement of the particles, and redistribution of stresses. The standard deviation of the normal stresses increased too. The average value of shear stresses (σ_{xx} , σ_{yz} , and σ_{xz}) was very close to zero during all load steps. However, the standard deviation of the shear stresses increased during all the load steps indicating an increase in the magnitude of shear stresses with loading. The presence of slightly high normal and

shear stresses prior to the loading could have been due to residual stresses that developed during the formation of the crystals or due to a slight difference between the silica crystal lattice parameters in the particles and the strain-free lattice parameters used in MIDAS (hydrostatic stresses can result from errors in the unstrained lattice parameters). Moreover, a variation of the unstrained lattice parameter could happen due to the properties of natural sand. The standard deviation of the normal stresses provides a good estimate of the error in the stresses due to this variation. During the first and second load steps, the normal stresses increased in magnitude with a median value increased from 20 MPa to 30 and 40 MPa, respectively.

Table 2.2. Summary of isotropic and anisotropic elastic moduli for α -quartz (Heyliger et al. 2003).

Isotropic Model	
Elastic properties	Value (GPa)
Young modulus (E)	99.45 (GPa)
Poisson Ratio (ν)	0.06
Anisotropic model	
C_{11}	87.26
C_{22}	87.26
C_{33}	105.8
C_{44}	57.15
C_{55}	57.15
C_{66}	40.35
C_{12}	6.57
C_{13}	11.95
C_{23}	-17.18
Poisson Ratio	
ν_{12}	0.1286
ν_{23}	0.0984
ν_{13}	0.1274

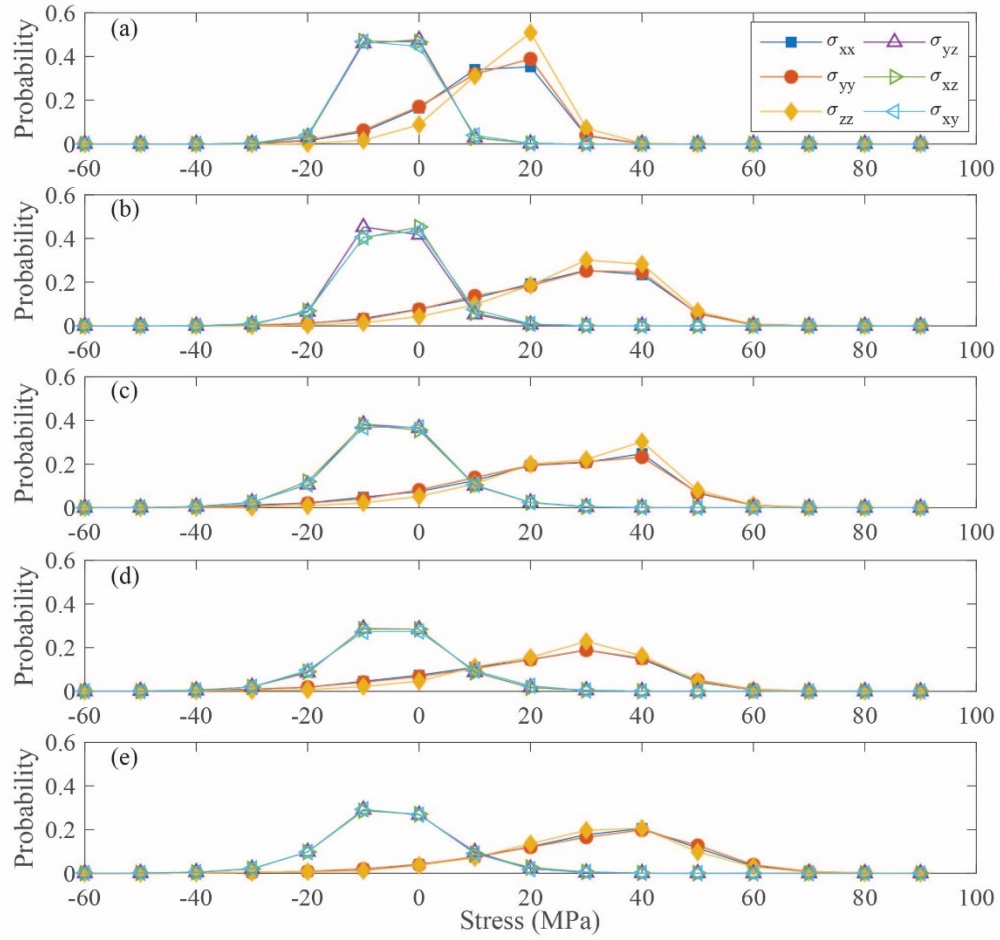


Figure 2.9. Statistical distribution of stresses within individual particles at global axial strains of (a) 0%; (b) 4.35%; (c) 6.71%; (d) 8.35%; and (e) 11.47%

Table 2.3. Summary of the variation of stresses during different load steps.

	Load step 0		Load step 1		Load step 2		Load step 3		Load step 4	
Stresses	mean (MPa)	S _d (MPa)	mean (MPa)	S _d (MPa)	mean (MPa)	S _d (MPa)	mean (MPa)	S _d (MPa)	mean (MPa)	S _d (MPa)
σ_{xx}	16.01	9.891	29.89	19.87	31.18	19.39	26.07	18.97	35.31	17.93
σ_{yy}	16.15	10.29	29.98	18.55	31.18	19.22	27.05	18.91	35.81	18.26
σ_{zz}	20.27	7.903	33.58	13.43	35.07	15.28	30.02	15.81	35.19	16.95
σ_{yz}	0.067	5.231	-0.292	7.021	-0.288	10.29	-0.057	10.36	-0.160	10.70
σ_{xz}	0.029	5.179	0.291	7.077	-0.375	10.25	-0.155	10.55	0.007	11.30
σ_{xy}	-0.023	5.721	0.284	7.525	-0.089	10.70	-0.155	10.92	-0.354	11.17

Characterization for force structures

There is not a unique definition for force structures in the literature; however, the main common thread between all of the methods is that a force structure is a set of connected particles that carry high stresses or have high inter-particle forces between them (Cates et al. 1998; Cates et al. 1999; Howell et al. 1999). Peters et al. (2005) developed an algorithm to characterize the force structures in a granular assembly due to the indentation of rigid rectangular punch into a half medium using 2D DEM. They defined a force structure to be a chain-like series of highly stressed particles (i.e., particles with maximum principal stress higher than the average of the maximum principal stress within all particles) with a length of three or more particles with principal stresses in the direction of the contacts of the particles. The concept proposed by Peters et al. (2005) was modified by the authors to characterize the force structures within the specimen in the present study. This algorithm used the contact network of individual particles (contact between particles, labels of particles in contact, and the normal vector of the contacts) within the specimen during each load step. The contact network used in the algorithm was provided by another code that

was developed by Druckrey et al. (2016). The code identified the contact between touching particles from the SMT images and calculated the normal vector to the plane that best fitted the pixels representing the contact between two particles using the principal component analysis. The accuracy of this method depends on the resolution of the SMT images, the contrast of the particle surfaces, and the morphology of particles. The high resolution of the SMT images in this study ($1.17 \mu\text{m}/\text{pixel}$) compared with the size of the particles facilitated an excellent characterization of particles' boundaries. Moreover, the Ottawa sand used in this study has relatively rounded particles. The contacts between particles were carefully examined visually to validate the quantified contact normal vectors. Figure 2.10 displays a few examples of typical contacts between particles within the specimen and their normal contact vectors that were quantified using the previously mentioned code. Amirrahmat et al. (2018) and Imseeh et al. (2018) used the aforementioned code to characterize the evolution of fabric and variation of average contact number within multiple triaxial specimens. There are other studies that have quantified the contacts in granular materials which are not discussed here to avoid prolonging the discussion and are just referred to (Andò et al. 2013; Cheng and Wang 2018; Jaquet et al. 2013; Vlahinić et al. 2014; Wiebicke et al. 2017) for further details.

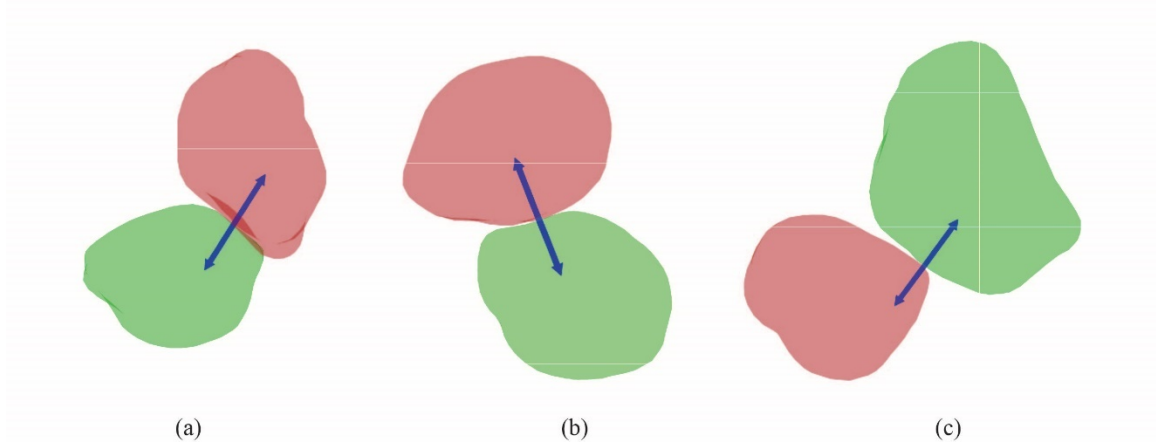


Figure 2.10. Illustrative examples of three random contacts between Ottawa sand particles (the arrow represents the normal to contact vector).

Particles within force transmission structures carry most of the applied load in granular materials and consequently have higher internal stresses. In order to characterize the force structures within the specimen; initially, a threshold was used to filter highly stressed particles (HSP). The principal stresses within particles were calculated, and the principal stress with the greatest magnitude (i.e., absolute value) was identified for each particle and was labeled as the maximum absolute principal stress (MAPS). Particles with MAPS higher than 75% of the MAPSs (i.e., 75th percentile) in the entire particle population were considered highly stressed particles. In normal distributions, the percentile limits can be related to the average and the standard deviation of the distribution.

$$X = M + Z * S \quad (6)$$

Where X is the percentile limit, M is the average of the distribution, and S is the standard deviation of the distribution. In a normal distribution, the value of Z for the 75th percentile

is 0.625. The values of Z during the four load steps corresponding to the 75% percentile are 0.57, 0.64, 0.61 and 0.66. Figure 2.11 shows the cumulative distribution of the MAPSs during the four load steps. The threshold value (higher than 75% of MAPS) increased during the first two load steps from 52.5 MPa to 57.2 MPa as the magnitude of the stresses increased with loading. However, the bulging of the plastic tube and the fracture of some particles affected the stresses, and the threshold slightly decreased to 55.7 MPa during the third load step. The threshold value increased during the fourth load step to 57.9 MPa.

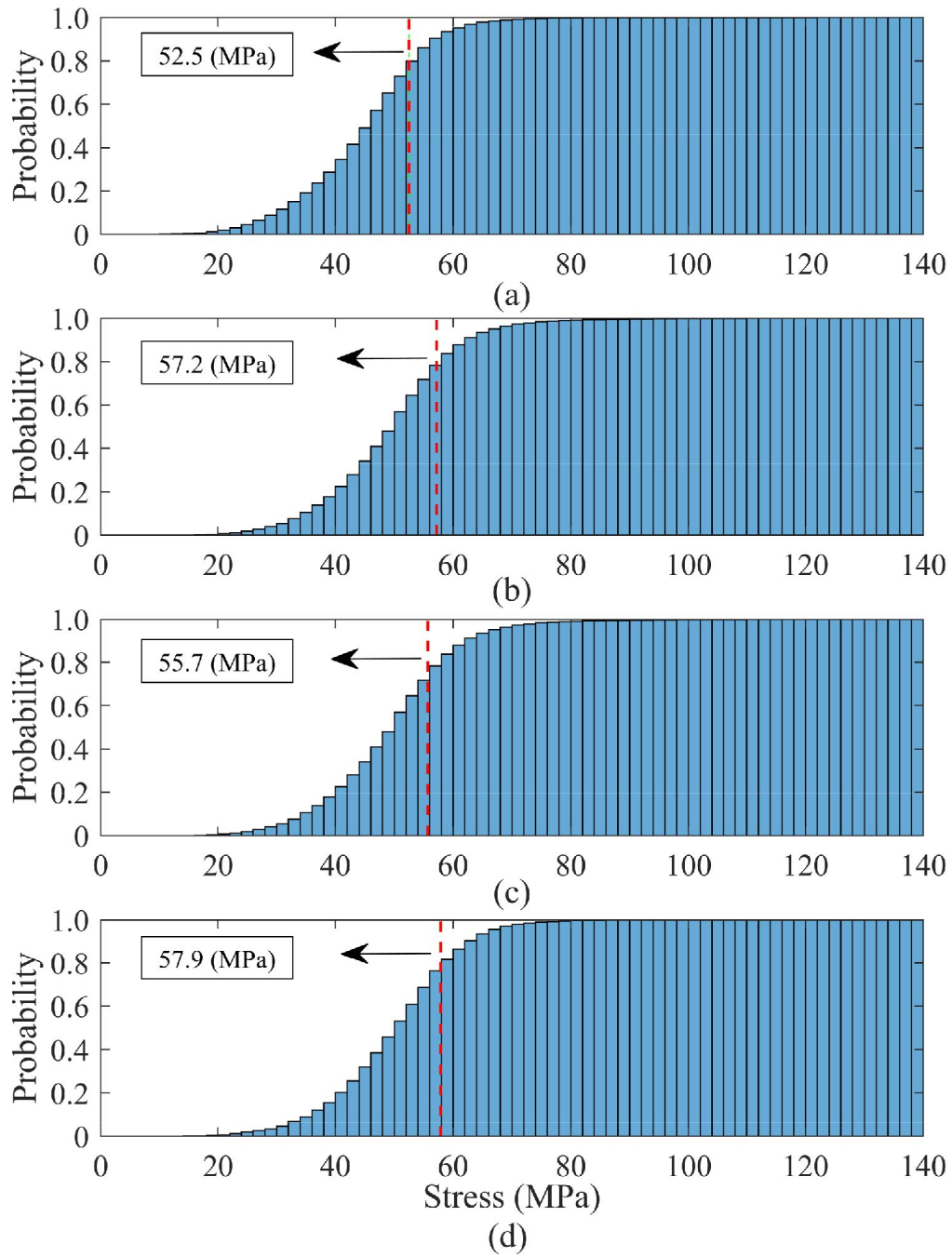


Figure 2.11. Cumulative distribution of the magnitude of MAPSs at global axial strains of (a) 4.35%; (b) 6.71%; (c) 8.35%; and (d) 11.47

The thresholded HSPs were used to create a list of particle pairs that are in contact with each other and have high MAPS. Each pair was investigated individually to determine whether the direction of contact between the pair was in the direction of MAPS in both particles within a tolerance of 30°. Pairs that did not satisfy the aforementioned criterion were removed from the list. The remaining pairs were used to create an adjacency matrix, a square symmetric matrix with zeros and ones as components. A component was one if two particles were in the list previously mentioned and zero if they were not. The adjacency matrix was used in an in-depth-first-search algorithm to identify the connected pairs of particles and form a series of particles. The algorithm started at an arbitrary particle and traversed on connected particles as far as possible before backtracking each step and iterating the traversal for each step of backtracking. Figure 2.12a shows an example of the first-in-depth-search algorithm. The algorithm starts at the first particle and traverses on connected particles to the third particle. Then, the algorithm continues to a particle connected to the third particle arbitrarily (the fourth particle in this example). It continues to traverse on the connected particles until it reaches a particle that does not have a connected particle that has not been traversed (yellow labels). The particles are backtracked until a particle with a new (i.e., not traversed) connected particle is reached (green labels). The new connected particle is traversed (red label) and backtracked (blue labels) until all connected particles are traversed. A more detailed description of in-depth-first-search algorithms is reported by Tarjan (1972). A connected set of particles with a length of more than or equal to three particles were considered as force structures. The in-depth-first-search algorithm was able to detect branching in force structures. Individual structures and

their spatial shape were examined. The structures had two major spatial shapes of clusters and chain-like structures that are referred to as particle clusters and force chains, respectively. An objective definition of particle cluster was necessary to distinguish between the clusters and the force chains. The general shape of the force structures and the proximity of the particles in the structures were examined to characterize force chains and particle clusters. The general shapes of the force structures were categorized using the dimensions of the bounding boxes (i.e., a box with minimum possible dimensions that contains all particles in the force structure). One of the sides of the bounding box is fixed with the direction of loading (z-direction). However, the other two sides can rotate in the plane perpendicular to the z-axis (x-y plane) to best fit the direction of the force structure. The dimensions of the bounding box were measured for each force structure. The lengths of the sides of the bounding boxes were sorted in descending order and labeled as A, B, and C (Figure 2.12b). The proximity of the particles in force structures was investigated through a closeness index. The closeness index was defined for each particle as the number of other particles in the force structure within a distance of $20\text{ }\mu\text{m}$ ($0.15*d_{50}$, Figure 2.12b). Force structures that had bounding boxes with dimensional ratios of A/B and A/C between 1 and 1.5 and their average closeness index of all the particles in the structure was greater than 2 were considered to be particle clusters. Figure 2.12c & d shows an example of the two types of force structures.

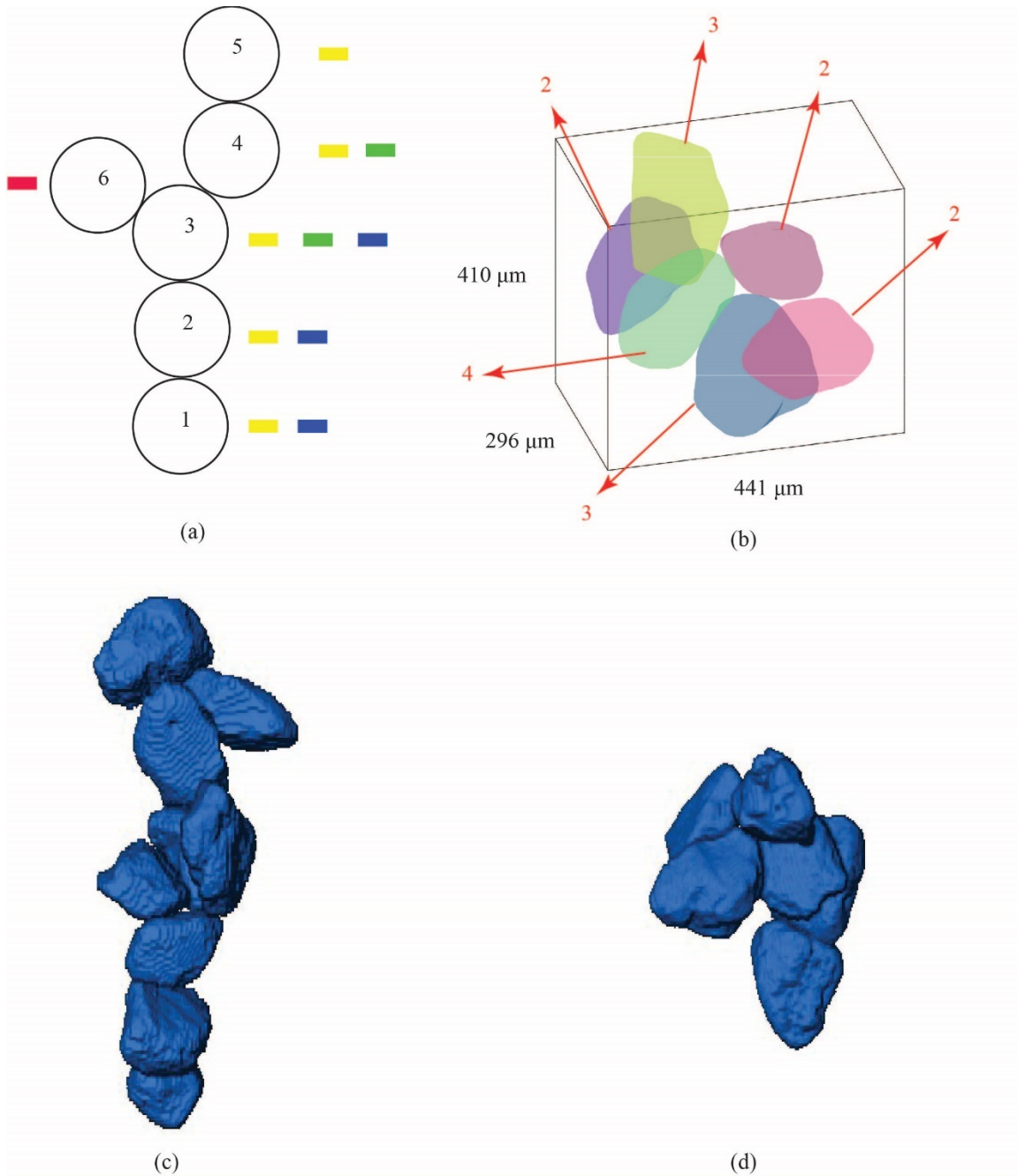


Figure 2.12. Illustrative examples of (a) first-in-depth-algorithm; (b) bounding box and closeness indices of a particle cluster; (c) a force chain; (d) a cluster of particles with the closeness index for each particle.

Figure 2.13 shows the force structures within the specimen at different load steps where the color of each particle represents the magnitude of the MAPS within that particle. The global axial stress during all these four load steps is smaller than 9 MPa but there are particles within the force structures that have stresses as high as 85 MPa. This difference between the global axial stress and stresses within individual particles is attributed to the difference between the stiffness of individual particles and the stiffness of the sand specimen that has void space between particles. If one excludes particles rigid body motion then a small strain within an individual particle can create high stresses within it (Alshibli et al. 2013; Cil et al. 2017). Also, it is reported in the literature that the magnitude of stresses within the particles in a regular consolidation specimen can be much higher than the global stress (McDowell 2002). During the first load step, the force structures were dispersed within the specimen and did not have a particular direction. The structures were mostly in the form of clusters rather than force chains. However, the force structures formed a more concentrated pattern where they were mostly aligned with the loading direction during the second and fourth load steps, and most of the structures were in the form of force chains rather than particle clusters. During the third load step, the plastic tube surrounding the specimen expanded in the middle due to the high stress in the specimen and 19 particles fractured into multiple fragments. The bulging and the fracture of the particles resulted in a decrease in the stresses within some particles, and the force structures had a dispersed pattern similar to the first load step. The fragmentation of highly stressed particles can cause buckling within the force structures which can lead to a more homogenous force structures pattern. Tordesillas et al. (2011) studied the evolution of force structures and in

granular assemblies in a triaxial DEM simulation and showed that buckling of force structures within the localization areas can cause the force structures to have a more accumulated or homogenous pattern. The variation of the load at the top and bottom of the specimen is more visible during the second and fourth load step as well. The external forces are distributed between more particles during the first and the third load step due to the dispersed shape of force structures which masks this difference. In addition, the difference between the value of top and bottom forces increases with loading as well.

Figure 2.14a shows the variation of the number of HSPs and the number of particles within the force structures (FSP). The number of highly stressed particles decreased during the experiment (1086 to 767 particles) which can be attributed to the concentration of force structures as stresses increased. The ratio of FSP to HSP during load steps 2 and 4 decreased as the force structures became more concentrated with increased loading. The ratio of the number of FSPs to HSPs increased during the first and third step where the force structure exhibited a more dispersed pattern. The ratio of the number of FSPs to the HSPs was between 32% to 48% during all load steps. Peters et al. (2005) showed that this value is approximately 50% during the advancing of the punch into the half medium. Radjai et al. (1998) also showed that only 40% of particles with contacts referred to as “strong contacts” was attributed to the force transmission system in a 2D assembly of discs in a box limited by immobile walls and base and loaded due to the movement of the top loading plate.

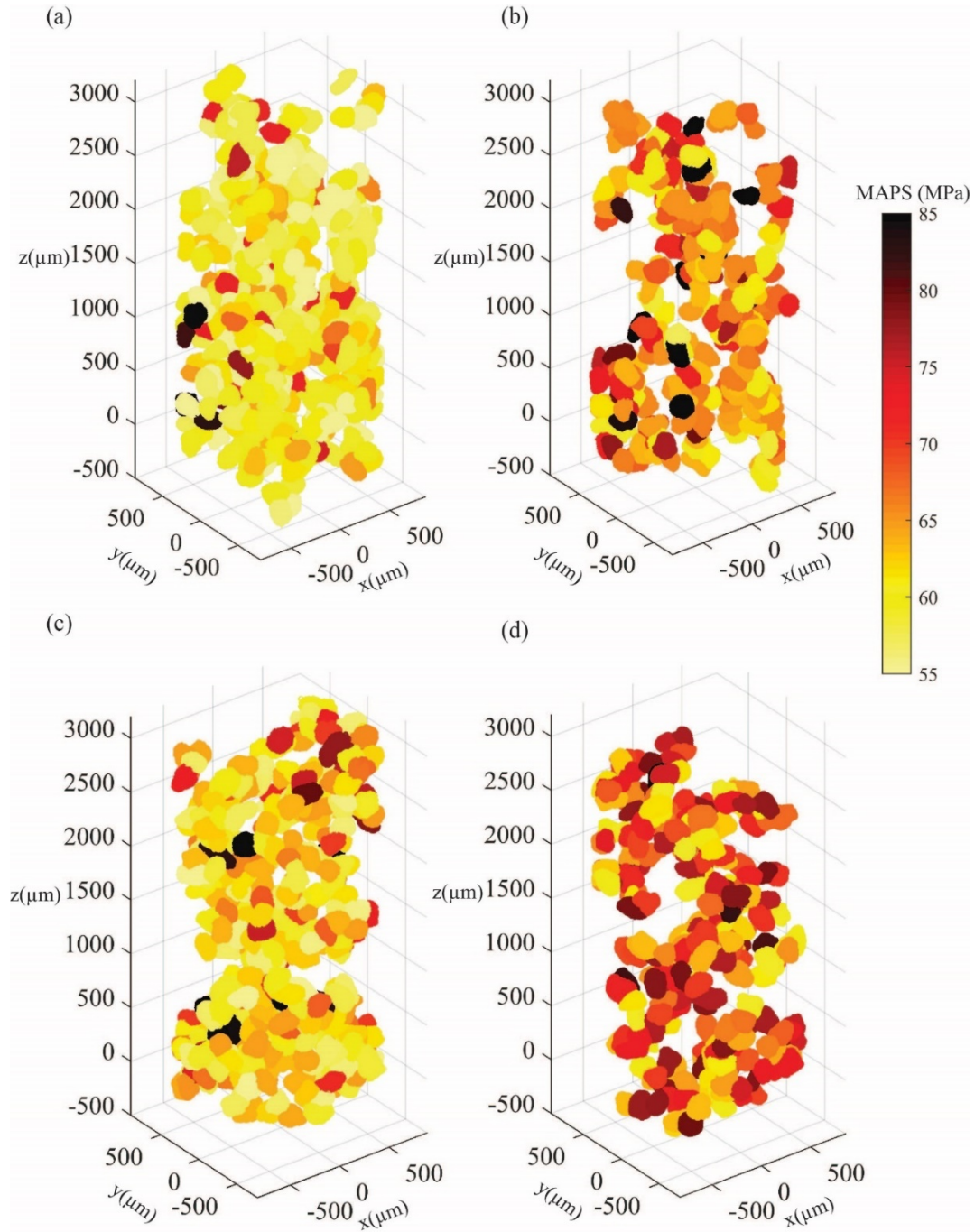
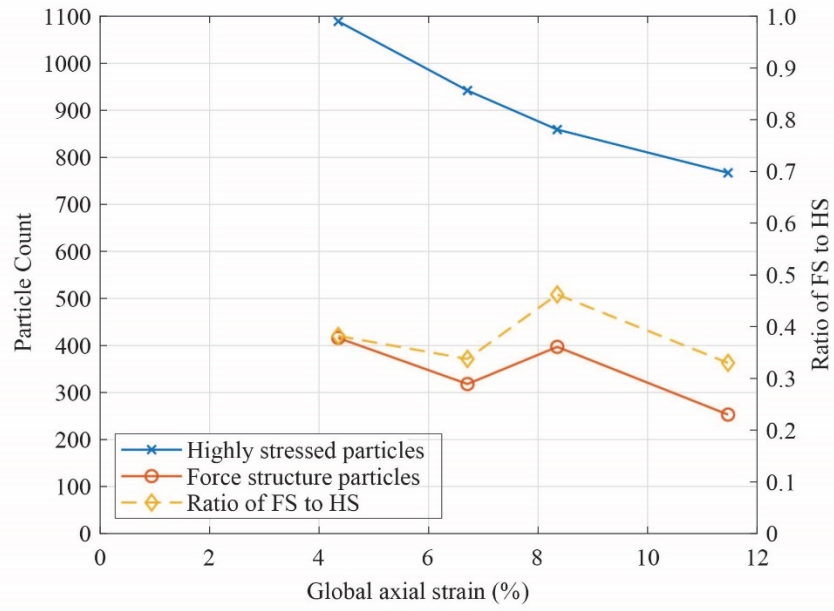
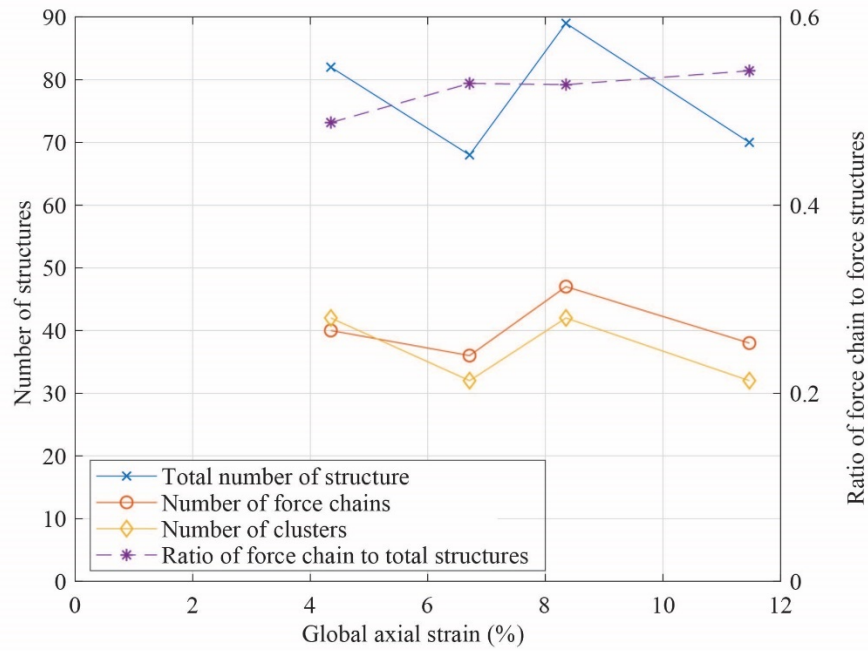


Figure 2.13. Characterized force structures assuming an anisotropic constitutive model for the sand particles at a global axial strain of (a) 4.35%; (b) 6.71%; (c) 8.35%; and (d) 11.47%



(a)



(b)

Figure 2.14. Summary of the variation of (a) HSP and FSP and (b) number of force structures, number of force chains and number of particle clusters during different load steps assuming the anisotropic constitutive model.

The variation of the number of force structures, number of force chains, number of particle clusters, and the ratio of the number of force chains to the number of particle clusters versus the global axial strain is depicted in Figure 2.14b. The total number of structures decreased during load steps 2 and 4 as the force structures formed a more concentrated pattern during these load steps. The ratio between the numbers of force chains to the number of clusters generally increased with loading as the contacts bearing the load aligned with the direction of the loading. However, during the third load, the ratio remained almost the same (0.529 to 0.528) as the tube around the specimen expanded in the lateral direction allowing room for stress relaxation. Makse et al. (2000) used DEM and experimental methods to study random packings of spherical particles and their stress paths under compression. They defined force chains as a series of connected particles which spanned from top to the bottom of the specimen and had inter-particle forces greater than the average of the inter-particle forces. They illustrated that the force chains were sparsely distributed in the specimen under low compression stresses and as the stresses increased, the force chains became more concentrated. They also showed that the variation range of the inter-particle forces decreased as the compression stresses increased. Similar patterns were observed in this study, where the variation range of the stresses decreased during the second and the fourth load step.

Figure 2.15a shows how the statistical distribution of the length of force chains developed as the global axial strain increased. The probabilities were calculated based on the total number of force chains at each load step. The average length of the force chain increased during the first, second and third load steps from 4.17 to 5.36 particle length; then it

decreased slightly during the last step of the test to 5.22 particle length. During load steps 2 and 4, the ratio of the number of chains with a length higher than 6 particles to the total number of chains increased while the ratio of chains with a length under 5 particles decreased indicating that chains were getting longer during these steps. The maximum length of force chains also increased with loading (10 to 12). However, during the third step, the ratio of chains with length less than 5 particles to the total number of chains increased showing that force chains length decreased as the stresses decreased in the specimen during the bulging. Clearly, these lengths were dependent on the assigned allowable angle between the direction of contact and direction of MAPS used in the algorithm, but the trends remained similar when the force structures were characterized using an allowable angle of 45° . Similar studies were conducted by Peters et al. (2005) that reported the dependence of the length of force structures on the angle used in the algorithm. However, the change in the angle would not change the trend of variation in the length of the force chains.

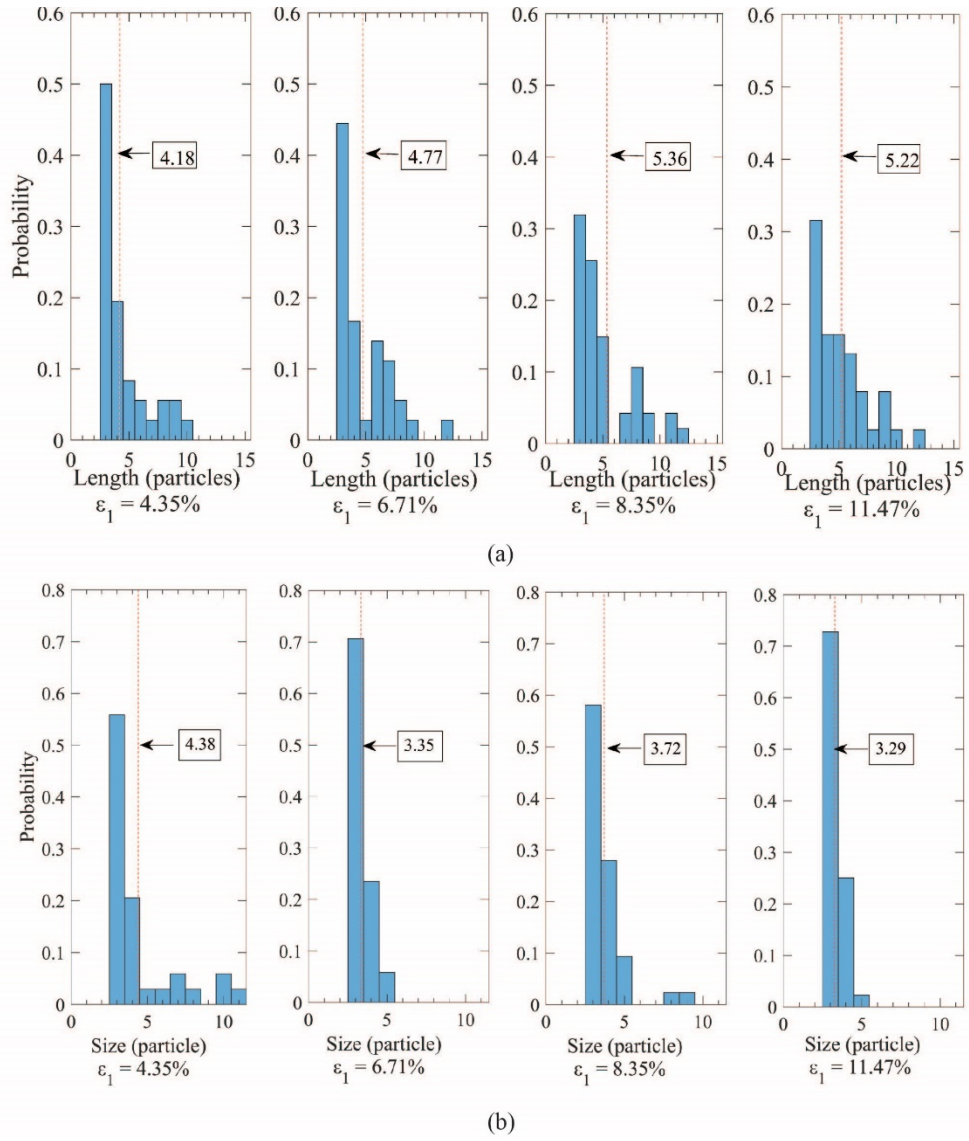


Figure 2.15. Distribution of the length of (a) force chains and (b) the size of the particle clusters during different load steps assuming anisotropic material behavior.

Similar to the force chains, the particle clusters surrounding them change in size during different load steps. The variation of the size of the particle clusters (number of particles forming the cluster) is also shown in Figure 2.15b. The probabilities were calculated based on the total number of particle clusters at each load step. The average size of the clusters varied from 3.29 to 4.38 particles. The particle clusters had a high variation in size during the first load step (3 to 11 particles per cluster). This range generally decreased with loading as in all consecutive load steps after the first step more than 93% of the clusters had a size between 3 to 5 particles. The size of clusters increased during the third load step in comparison with the second load step, and the average of the size increased from 3.35 to 3.52 particles during the third load step. Similar to the length of the force chains, the size of the clusters is dependent on the assigned allowable angle between the contact vector and the direction of the MAPS. However, the change would not affect the trend of variation in the size of the particle clusters.

The packing of the particles and the contact number of the particles can affect the onset and the evolution of force structures within the specimen as they can change the kinematics of individual particles and hence affect the deformation of individual particles as well. The average CN of the specimen increased from 6.08 to 7.10 during the first load step. CN stayed relatively the same (7.09) during the second load step. The CN of the specimen increased to 7.49 and 7.79 during the third and fourth load step. The evolution of force structures was investigated thoroughly to shed the light on the effect of contact number and internal crystal structure on the development of force structures. The contact number of

the particles that are part of force structures were investigated to study the effect of contact number on the transmission of the force and formation of the force structures. Figure 2.16 shows the distribution of the contact number of particles within and outside the force structures during different load steps. The probabilities were calculated based on the number of particles within and outside the force structures at each step. The average contact number at each load step is shown as a dashed red line. The average contact number of particles within the force structures increased during the second and fourth load steps (7.72 and 8.32, respectively). The minimum contact number of particles also increased during those two load steps compared with the first and third load steps while the ratio of particles with contact higher than the average increased as well. The average of the contact number of particles outside the force structures remained almost constant at 6.99 and 6.98 during the first two load steps, respectively. The average increased during load steps 3 and 4 to 7.41 and 7.71, respectively. The range of contact number for particles outside the force structures remained almost constant during different load steps at 2 to 13. The difference of the average contact number between particles inside and outside the force chain increased during load steps 2 and 4 in comparison with the first and third load steps. The particles within the force structures had a higher average contact number during all load steps. This difference decreased during the third step as the specimen relaxed due to the tube bulging.

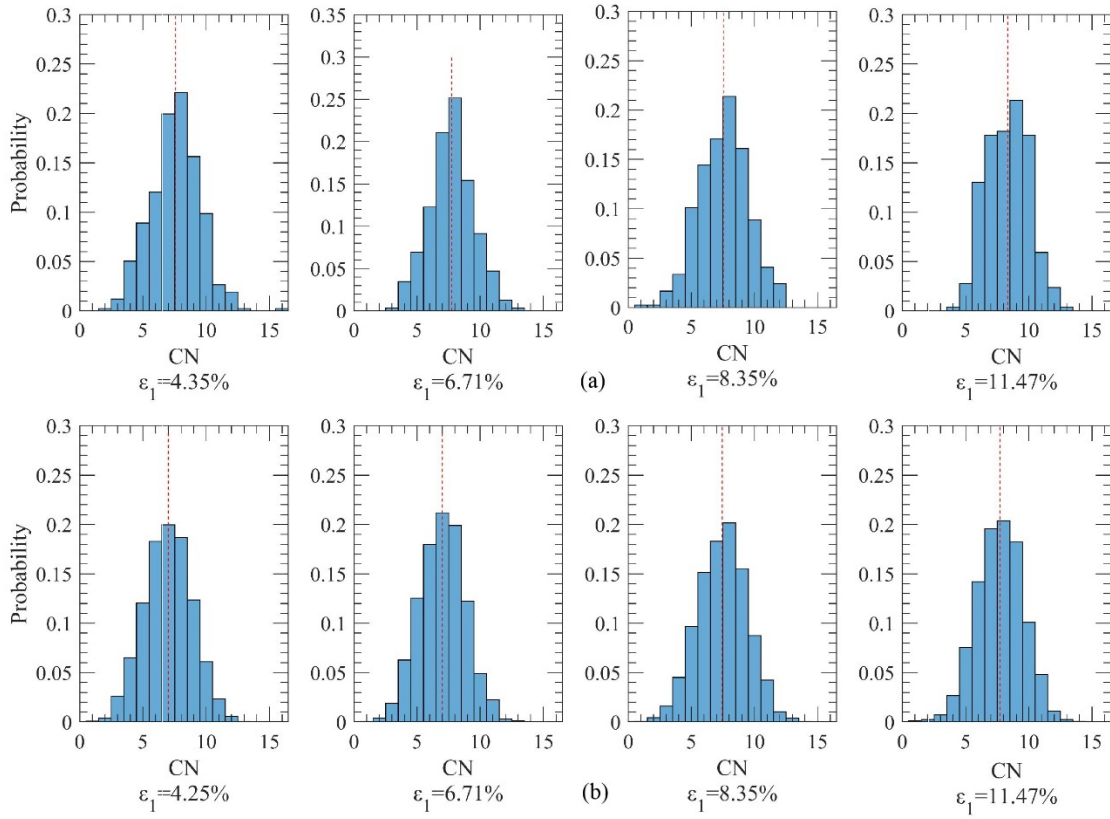


Figure 2.16. Distribution of the contact number (CN) of the particles (a) within the force structures and (b) outside the force structures during different load steps assuming anisotropic material behavior.

Force bearing contacts were also investigated in this paper. Force bearing contacts are defined as contacts between particles that are all within the force structures. Figure 2.17 shows the distribution of the number of force-bearing contacts (FBCN) during the loading of the specimen. The FBCN is significantly lower than the total contact number of particles within the force structures during all load steps indicating the presence of particles that provided support around the load bearing particles but were not part of the structure themselves. These particles are very influential to the formation of the force structures even

though they were not part of the structures themselves as they supported the force bearing particles and shaped the force structures by restricting the kinematics of particles surrounding them. The distribution of the force bearing contacts indicated that during the second and fourth load steps, the particles in the force structures had lower force bearing contacts. The lower number of FBC indicated a decrease in the branching of the force structures and an increase in the formation of the force chains.

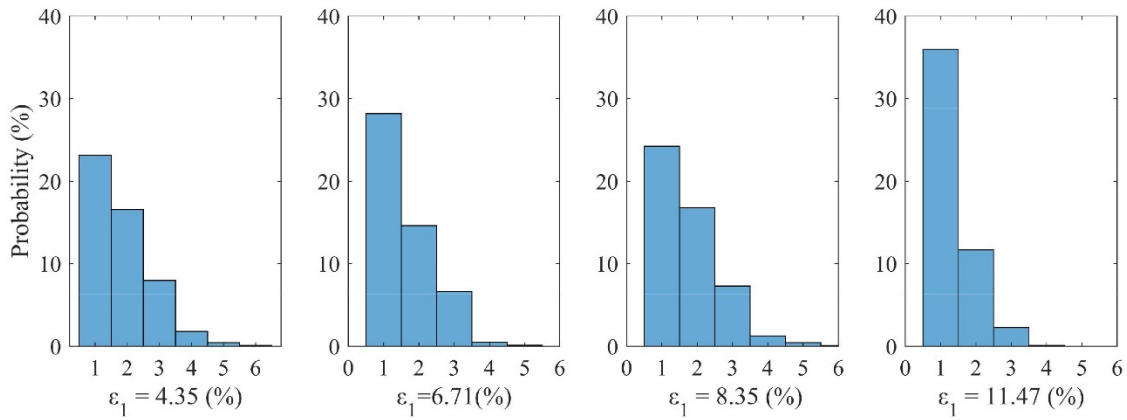


Figure 2.17. Distribution of force bearing contacts during different load steps assuming anisotropic material behavior.

Conclusions

The lattice strains and stresses of individual particles and force transmission structures within a natural Ottawa sand specimen consists of 2705 particles subjected to a 1D confined compression loading were characterized using SMT images and 3DXRD techniques. The followings conclusions are drawn from this study:

- 1- The highly stressed particles were not all part of the force structures within the specimen. The ratio of force structure particles (FSP) to the highly stressed particles (HSP) was between 32% and 46% during different load steps.
- 2- The number of structures was higher during the initial load step where the stresses were not fully developed to a high value and during the third load step when the specimen experienced relaxation due to the bulging of the specimen in the middle section. The number of structures decreased during the second and fourth load step as the stresses increased with loading and the structures formed a more concentrated pattern and the stresses were distributed more uniformly between the particles within the force structures.
- 3- The force structures had two main spatial shapes named as force chain and particle cluster. The force chains generally spanned from the top of the specimen to the bottom, transmitting the compressive forces on the boundaries of the specimen. The particle clusters alongside the other highly stressed particle outside the force structures surrounded these force chains and provided the necessary support for the chains. The variation of the number of the two structures indicated that as the loading progressed the contact vectors between the particles aligned in the direction of the loading, the number of force chains increased to a number higher than the number of particle clusters. The number of particle clusters was higher than the force chains during the first step where the structures were not fully formed and also during the third step where the specimen experienced relaxation due to the bulging of the specimen.

- 4- The length of the force chains increased during the second, and the fourth loading step as the contacts aligned with the loading direction and the structures formed more concentrated patterns. On the other hand, the size of the particle clusters decreased during the second and fourth load step. The size range of the particle clusters narrowed during the second and fourth loading step as well. The evolution of the particle structures indicates the formation of concentrated column-like structures that are responsible for the transmission of the forces from different boundaries. The particle clusters alongside with other highly stresses particles provide the necessary support for the force chains.
- 5- Particles within the force structures had a higher average contact number in comparison with the particles outside the structures. The difference between the two averages increased during the second and fourth load step as the stresses developed within the specimen. The contact number of the particles affects the distribution of stresses within particles as greater number of contacts restricts the translation and rotation of the particle and increases the likelihood of stress concentration within the particles.
- 6- The particles in contact with particles within the force chain were not all force bearing contacts. The number of force-bearing particles was between 1 to 5 particles. This number decreased as the stresses increased during the second and fourth step and shape of the structures changed from clusters to force chains.

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CHAPTER 3
INFLUENCE OF PARTICLE-SCALE PROPERTIES ON FRACTURE
BEHAVIOR OF SILICA SAND

A version of this Chapter was recently submitted for possible publication by Siavash Amirrahmat and Khalid Alshibli to Granular matter Journal

My contribution to this paper is:

- 1- The analysis of the data and creating the framework for data analysis (matching algorithm, force chain algorithm, analyzing the 3D images, quantifying the particles' stresses, quantifying the kinematics and other particle scale properties, studying the effect of particle-scale properties on the fracture of the particles, Using RRelief algorithm to rank the predictors)
- 2- Majority of the writing and the Figures

Abstract

The distribution of stresses within individual granular particles subjected to 1D confined compression is affected by various particle-scale properties such as morphology, kinematics, position, crystal structure, and the interaction of the particles (i.e., the local contact network). A thorough examination of the effects of particle-scale properties on the variation of stresses within individual particles and the fracture of particles in granular materials is essential for both the theoretical analysis and modeling of engineering behavior of granular materials. In this study, 3D synchrotron micro-computed tomography (SMT) and 3D x-ray diffraction microscopy (3DXRD) techniques (non-destructive) were concurrently used to quantify the variation of internal particle stresses, as well as the aforementioned particle-scale properties of a specimen composed of Ottawa sand that was subjected to a 1D confined compression. The effects of the particle properties on the variation of particles' internal stresses and the fracture of particles were studied in detail. The importance of the particle-scale properties based on their effect on the variation of particle stress was ranked using a feature selection algorithm called RReliefF. The particle properties that were deemed to be significant (kinematics, contact number, the relative orientation of the local contact network with respect to the loading direction, and position) by the feature selection algorithm were investigated further to uncover their influence on the internal particle stresses. In addition, the

effects of the particle-scale properties on the fracture of the particles were examined. The fracture of the sand particles proved to be a complex phenomenon that was controlled not only by particle-scale properties but also by the interaction between the particles. The relative orientation of the fracture surface in the fractured particles was examined to assess the effect particle shape and local contact network of the particles on the fracture of the particles.

Introduction

The distribution of stresses within individual granular particles subjected to external loading is affected by various particle-scale properties such as morphology, kinematics, position, crystal structure and the interaction of the particles (i.e., the local contact network). A comprehensive insight into the effects of particle properties and local contact networks on the variation of stresses within individual particles can potentially have a significant impact on both the theory and modeling of granular materials as experimental measurements can be used to calibrate and validate particle-scale constitutive models. Moreover, quantification of in-situ internal particle stresses has proven to be elusive even with novel methods such as Neutron diffraction and 3D x-ray diffraction (3DXRD) microscopy as these methods are complex and have limited-user access. Further understanding of the relationship between particle-scale properties and their internal stresses offers an easier alternative to develop predictive models that use particle-scale properties as an input. The focus of the current paper is to synthesize experimental and numerical literature about quantifying the variation of particle stresses and the effect of particle-scale properties on these stresses and to propose a statistical analysis that uses unique experimental measurements as input to investigate the effects of particle scale

properties on variation of stresses in a specimen composed of natural silica sand subjected to 1D confined compression.

Drescher and De Jong (1972) used photo-elastic material to characterize the effect of incremental displacement of the particles and their rotation on the variation of internal particle stresses within an assembly of 2D photo-elastic disks subjected to a shear load. Zhang et al. (2010) studied a 2D assembly of photo-elastic particles to investigate the effect of contact number and strain localization on the variation of the stresses within the 2D disks. In recent years, 3D x-ray computed tomography (CT) has been widely used to characterize the *in-situ* kinematics, local contact network, and the strain field of granular materials (Amirrahmat et al. 2019; Desrues and Andò 2015; Druckrey et al. 2018; Hall et al. 2010; Jarrar et al. 2018). SMT and CT imaging techniques have proven to be an efficient method to quantify the kinematics and contact network of individual particles, but they cannot quantify the deformation, and internal strains (or stresses) within the particles. Numerical methods such as discrete element method (DEM) and finite element method (FEM) have been used commonly in the literature to characterize the particle stresses within granular materials. For example, Tordesillas (2007) studied the particle-scale mechanics of buckling and shear banding in dense assemblies of circular particles subjected to quasi-static biaxial compression using DEM. Zhang et al. (2017) used DEM to investigate the variation of force chains and local void ratio during quasi-static biaxial loading and dynamic impact loading. Turner et al. (2016) employed FEM to investigate the variation of internal particles stresses in a sand specimen to identify the effect of the particles' contact number (CN) and discretization (meshing) parameters employed in the

FEM model. Imseeh and Alshibli (2018) also used FEM to investigate the force transmission, variation of internal particle stresses and fracture of individual particles in an assembly of 35 sand particles subjected to a confined 1D compression loading. They also investigated the effect of the CN of the particles on the variation of the principal stress within the sand particles.

The variation of internal particle stresses and force transmission mechanisms in granular materials is studied extensively in the literature. However, not all of the studies are discussed here in detail and just are merely referred to in this paper to avoid prolonging the discussion (Brodu et al. 2015; Cates et al. 1998; Cates et al. 1999; Delaney et al. 2010; Hall and Wright 2015; Hurley et al. 2014; Hurley et al. 2016; Majmudar and Behringer 2005; Makse et al. 2000; Peters et al. 2005; Radjai et al. 1996; Radjai et al. 1999; Tordesillas et al. 2010). Even though numerical models have been used to quantify the variation particles' internal stresses and characterize force transmission structures, there are still limitations on the size of the numerical simulations, especially if the morphology of the granular materials are included in the models (Kawamoto et al. 2018). Moreover, there is a need for 3D experimental results to validate the aforementioned models in the literature. A comprehensive study of the variation of particles' internal stresses and the effect of particle-scale properties on the stresses can significantly enhance such models.

Neutron diffraction techniques have been employed by a few studies to measure global and lattice strains within assemblies of granular materials by defining a volume gauge and measuring the lattice strains within that volume (Hall et al. 2011; Penumadu et al. 2009; Wensrich et al. 2012). 3DXRD is another non-destructive technique that has been used in

recent years to quantify lattice strains within individual particles in assemblies of granular materials. Hall et al. (2011) used radiograph scans and 3DXRD to study the evolution of lattice strain in individual particles in an assembly of Ottawa sand particles. Alshibli et al. (2013) and Cil et al. (2014) used 3DXRD and SMT imaging to quantify the lattice strain within particles of a specimen composed of three ASTM 20-30 Ottawa sand particles. Hall and Wright (2015) employed 3DXRD and x-ray CT imaging techniques to quantify the lattice strain of synthetic sub-spherical single crystal particles in an assembly of 96 particles. The CT images and the lattice strains particles were used together to study the internal stresses and the kinematics and the particle-scale structure of the specimen during a 1D compression of particles. Hurley et al. (2016) utilized CT imaging and 3DXRD to quantify the inter-particle forces in an assembly of 77 spherical quartz particles and characterize the variation of stresses within the assembly. Cil et al. (2017) used 3DXRD and SMT images to investigate the evolution of lattice strains of individual particles in an assembly of 35 particles of natural Ottawa sand. Hurley et al. (2018) used CT imaging techniques and 3DXRD to study and quantify the lattice strains and mechanical behavior of angular particles (i.e., particles created by chiseled and creating fragments out of a single-crystal block) in a specimen composed of 313 particles subjected to 1D compression. Amirrahmat et al. (2020) used SMT images and 3DXRD to characterize the force chains and study their evolution in an assembly of 2705 Ottawa sand particle subjected to 1D confined compression loading.

Another critical facet of the mechanics in granular materials is the fracture of particles and its effects on the constitutive behavior of the material. It changes the particle size

distribution, morphology, and affects the density of the specimen. It also affects dilatancy behavior, linear compression line, the plastic yielding in granular materials, and the evolution of force chains and mechanisms of force transmission (Bolton 1986; Coop 1993; Cooper 2011; Felice and Gupta 2009; Harireche and McDowell 2003; Iskander et al. 2015; McDowell and Bolton 1998; Peters et al. 2005). A thorough understanding of in-situ particle fracture mechanisms offers valuable insight into these effects. Different particle-scale properties such as morphology, kinematics, and local contact network of individual particles have been linked to the fracture of particles. There are many FEM and DEM simulations and experimental studies that have investigated the in-situ fracture behavior of single particles and assemblies of particles under different loading conditions using CT images techniques and post-fracture assessment of the fragments (Amirrahmat et al. 2019; Cil and Alshibli 2014; Cooper 2011; Druckrey et al. 2018; Druckrey and Alshibli 2016; Hurley et al. 2018; Karatza et al. 2017; Turner et al. 2019; Zhao et al. 2015).

In this paper, SMT images and 3DXRD measurements have been used to quantify the variation of lattice strains and stresses within the particles of a specimen composed of 2705 natural silica sand particles that was subjected to 1D confined compression. The concurrent use of SMT and 3DXRD techniques provided unique measurements of kinematics, local contact networks, and the internal stresses within the particles. Moreover, SMT images are used to identify the fracture mechanism of particles during the experiment as well as to characterize different factors regarding their fracture mechanisms such as shape and spatial orientation. The effects of particle-scale properties and the particles' interaction (i.e., local contact network of individual particles) on the variation of internal

stress within the particles are investigated in this paper. A feature selection method is used to rank the particle-scale properties based on the effect they had on the particles' stress field. The fracture of the particles and the effect of particle scale properties on their fracture are also studied in detail.

Experiment

A confined 1D compression experiment was conducted on a specimen of natural silica sand known as F75 Ottawa sand, mined from Ottawa, Illinois, and marketed by US Silica Company and has a specific gravity of solids of 2.65. The particles with grain size between US sieve #100 (0.149 mm) and sieve #140 (0.105 mm) were used in this study. The sand is composed of 99% α -quartz mineral and has been used widely by the geotechnical community in the USA. Figure 3.1 a & b show a 3D rendering and a schematic view of the apparatus used in this experiment. It consists of an acrylic specimen mold, a plastic tube insert (housing the sand particles), and a stepper motor to apply the compressive load on the specimen (Figure 3.1b). The acrylic mold was fabricated by drilling a 2 mm hole through the center of a solid cylindrical acrylic rod that has a diameter of 15 mm. The inner surface of the drilled hole is rough and can cause substantial friction forces with sand particles. In order to offer a smoother surface to interact with the sand particles, a plastic tube with an inner diameter of 1.650 mm was inserted inside the acrylic mold. The particles were then deposited inside the plastic tube using dry pluviation technique with the help of a small aluminum funnel, specially fabricated for this purpose. The acrylic mold provides the necessary confinement for a k_0 stress condition during the experiment. The sand specimen had a height of 3.720 mm and an initial density of 1.41 g/cm³ (void ratio of 0.879)

prior to loading. In order to measure the applied axial load at the top and bottom of the specimen (to quantify the friction between the sand particles the plastic tube), two load cells with resolutions of 0.35 N and 0.65 N were used in this experiment at top and bottom of the specimen. A stepper motor was used to apply axial compression at the top of the specimen at a constant displacement rate of 0.2 mm/min. The specimen was compressed to four pre-defined global axial strains of 4.35%, 6.71%, 8.35%, and 11.47%, and the loading was paused at each strain to acquire SMT and 3DXRD scans.

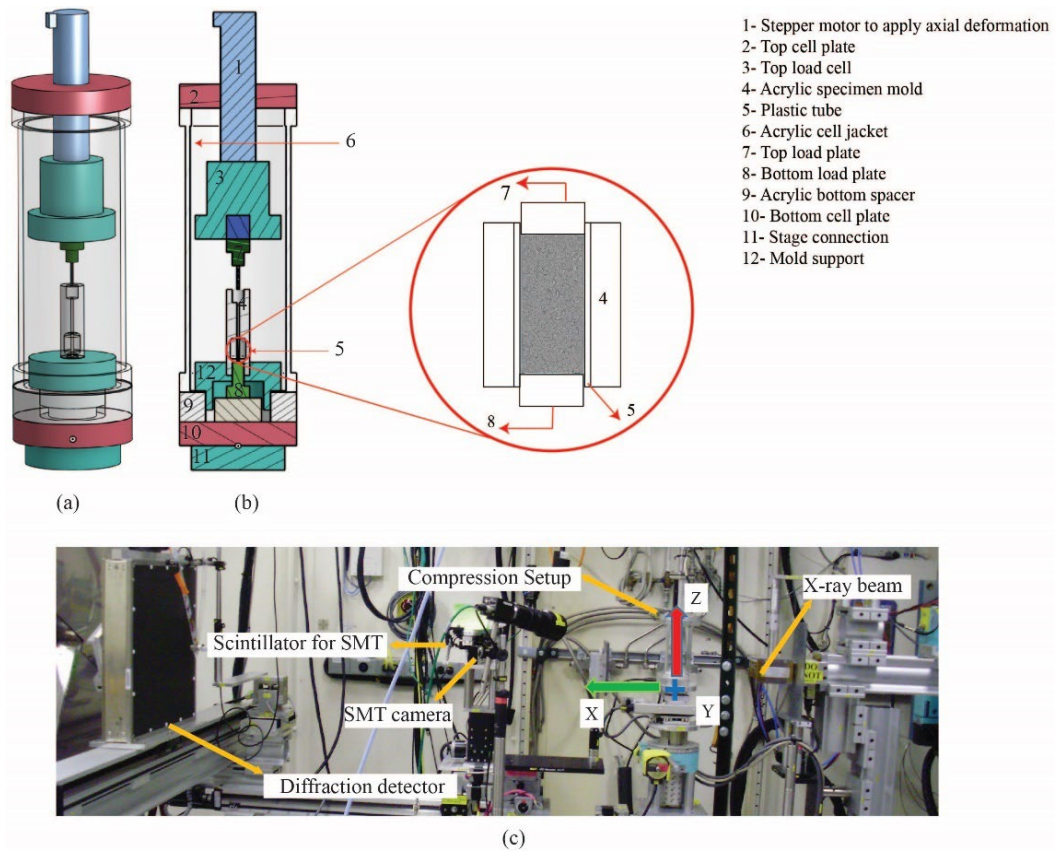


Figure 3.1. (a) 3D rendering of the experiment (b) Schematic of the 1D compression apparatus (c) Experimental setup at the APS beamline 1-ID.

SMT imaging and 3DXRD SCANS

The SMT and 3DXRD scans were acquired at beamline 1-ID of the Advanced Photon Source (APS), Argonne National Laboratory (ANL), Illinois, USA. The high precision setup provided by the beamline allowed the specimen to translate along any direction (Figure 3.1c) and rotate about the vertical axis and permitted tip-tilt alignments. The setup is described in more detail in Benda et al. (2016). The x-ray beam defined the horizontal x-axis; the rotation axis defined the z-axis and was carefully aligned vertically and perpendicular to the x-axis. The third orthogonal direction is denoted as the y-axis and matches a right-hand coordinate system. The axis of the cylindrical specimen and thus the loading direction was aligned with the z-axis. The SMT detector was mounted on a horizontal translation stage that allowed the reproducible motion of the detector between the specimen and the diffraction detector. The attenuated x-ray beam passed through a single crystal scintillator, and the radiographic images were captured using a 1960 x 1080 pixel resolution PointGrey Grasshopper3 CMOS camera. The scans were acquired using a 2 mm wide and 1.1 mm high x-ray box beam that had an energy of 61.32 keV. X-ray SMT scans were acquired using incremental rotations of 0.2 for the total rotation of 360° and an exposure time of 0.25 seconds resulting in a total of 1800 frames. The SMT scans were reconstructed with a spatial resolution of 1.17 $\mu\text{m}/\text{pixel}$ which are referred to as SMT images for simplicity.

The 3DXRD scans were also acquired during the same load steps using the same setup and x-ray beam. For each load step, four scans, each with a height of 0.95 mm, were collected and saved to cover the entire height of the specimen. The beam size was 1.3 mm $\times$ 0.7 mm

to ensure a consistent beam intensity through the height of the scan for accurate measurements of lattice strains. The 3DXRD scans were acquired at an angular rotation interval ($\Delta\omega$) of 1° for a total rotation of 360° . An amorphous silicon flat-panel detector (GE Revolution 41RT, (Lee et al. 2008)) with the dimensions of $410 \text{ mm} \times 410 \text{ mm}$ was positioned at a distance of about 1392 mm from the specimen. It has a resolution of 2048×2048 pixels with a pixel size of $200 \times 200 \text{ }\mu\text{m}$. Prior to loading, SMT images and 3DXRD scans were acquired for the initial state of the specimen. Then, the specimen was compressed until the target global strain was reached (i.e., 4.35%, 6.71%, 8.35%, or 11.47%), and loading was paused to acquire the next scan.

Data analysis and results

The relationship between the global axial displacement and the axial force and the variation of global void ratio versus the axial stresses during scans are depicted in Figure 2.2a& b. The axial force increased steadily during the experiment except during the relaxation periods once the loading was paused to acquire SMT and 3DXRD scans. The friction between the particles and the plastic tube surface was quantified as the difference between the readings of the top and bottom load cells. The friction between the plastic tube and particle remained relatively the same during the experiment (3.0 N during the 1st load step and 3.7 during the 4th load step). The slight increase in the friction forces is due to the change in the packing of the particles and the lodging of some particles into the inner surface of the plastic tube. Figure 2.2b depicts the variation of the void ratio versus the global axial stress where the stress points marked by diamonds are calculated based on the axial loads during the scans after the relaxation phase was finished. The void ratio of the

specimen changed substantially during the 1st load step as the specimen was in a loose state. However, after the 1st load step, the variation in the void ratio was much smaller when compared with the 1st load step as particles moved closer to each other, resulting in a denser state. The specimen began to bulge during the 3rd load step due to higher lateral pressure on the inner walls of the plastic tube, and the void ratio increased despite the axial compression of the specimen. In order to characterize bulging in the specimen, the profile of the middle section of the plastic tube wall at different loads was superimposed on each other. The left boundaries of the plastic tube (from the middle axial section) at different loads were superimposed on top of each other, and the right boundaries were plotted in Figure 2.3g to show the bulging of the specimen. The SMT images offered an excellent visual aid to assess the kinematics and rearrangement of the particles, while the 3DXRD measurements provided measurements of the deformation and lattice strains of individual particles.

Figure 2.3 depicts a central axial SMT slice before the loading and at different load steps of the experiment. The specimen was in a loose state prior to the loading, and the particles rearranged during the early stages of the experiment to form a denser packing (Figure 2.3b & c). The plastic tube expanded during the third and 4th load step due to the high lateral pressure against the tube walls (Figure 2.3d & e). Most of the bulging happened during the 3rd load step (Figure 2.3g). The fracture of particles was studied using the SMT images. The first particle fracture happened during the 3rd load step, where 19 particles fractured into multiple fragments. More particles fractured during the 4th load step (187 particles). The majority of fractured particles were close to the top and bottom of the specimen

adjacent to the loading plates. The fractured particles were identified using the 5th SMT scan at an axial load of 37.5 N and a global axial strain of 14.03%. The fracture of particles affected the analysis of the 3DXRD data as each sand particle disintegrated into multiple fragments which made it challenging to track diffraction spots of a large number of fragments; therefore, this study is limited to the behavior of the particles during the first four load steps, and the 5th step was omitted from the analysis as the ratio of fragmented particles to the total number of particles increased rapidly.

Quantifying the kinematics

The relative rotation (referred to as rotation for simplicity) and relative translation (referred to as translation for simplicity) of individual particles in the specimen were quantified from the SMT images using a script developed by the authors. Initially, the SMT images were processed to generate a binary image of the particles using Avizo Fire V9.6 commercial imaging code. The binary images were processed using a segmentation algorithm to separate touching particles within the binary image. The segmented particles were labeled using unique integers (label image). Figure 3.2a shows an example of a labeled image. The details of the image processing steps are not discussed in length to avoid prolonging the discussion and the reader is referred to Druckrey et al. (2016) for more details. Figure 3.2(b to d) show the 3DXRD measurements (i.e., the centroid of the crystals) and the matching results which will be discussed in detail in the next section. The labeled images and the aforementioned script are used to track individual particles during different load steps. The script compares two SMT images (consecutive load steps) to find and track individual particles within them based on their position and morphology (i.e., volume, surface area, and length of principal axes). A neighborhood is defined for each particle within the first image based on their position (i.e., the centroid of the particle), and the script searches for a possible counterpart particle within that neighborhood in the second image. The particles with the closest morphology (i.e., the least difference in volume, surface area, and length of principal axes) would be considered as paired particles within different load steps.

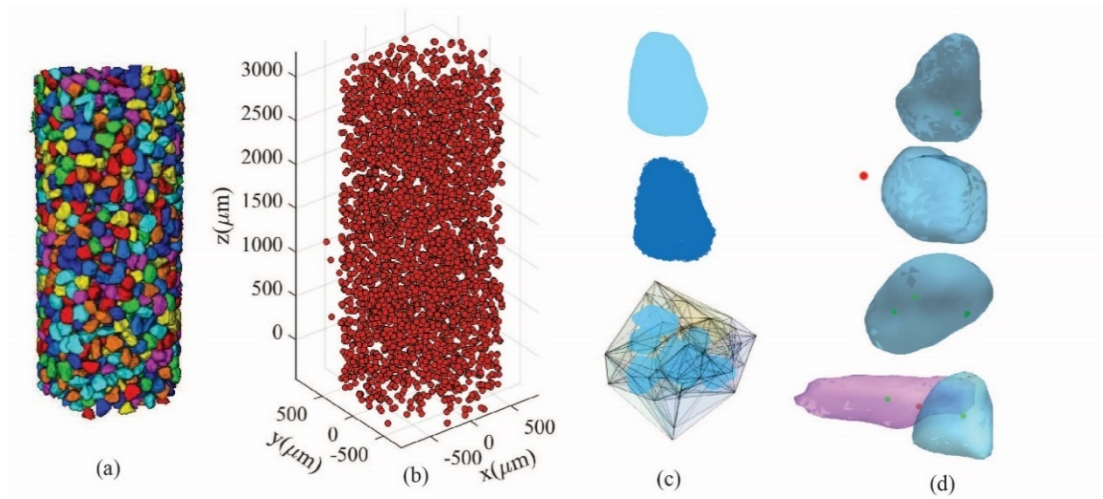


Figure 3.2. an example of (a) labeled image (b) 3DXRD measurements (i.e. the centroid of the measurements not to confused with the centroid of particles); (c) 3DXRD measurements' matching steps; (d) different outcomes during the matching process (erroneous matches are illustrated using red dots).

The change in position of the centroid of these paired particles is considered as the translation of particles, and also the angles between the principal axes of the particles were considered as the rotation angles of the particles. The particle size of Ottawa sand used in this experiment has a very narrow range of morphology of the particles, and it was necessary to use stringent tolerances for the acceptable differences in the morphology of the particles during different loads steps to avoid pairing wrong particles during different loads steps with each other. An iterative algorithm was used to increase the tolerance from 1% of the initial value of volume, surface area, and the length of principal axes to 7% of the aforementioned values. These values were selected by examining the paired particles for possible wrong pairs. The number of wrong matches increased drastically from 21 to 47 particles as the tolerance changed from 7% to 8%. The number of paired particles during

the 1st load step was 2040 as the particles had higher translation and rotation and it was difficult to track the particles with a high degree of certainty. However, this number increased to 2634, 2377, and 2341 particles during the 2nd load step to the 4th load step, respectively. The details of this pairing algorithm are discussed in Druckrey et al. (2017). Jarrar et al. (2018) and Amirrahmat et al. (2019) employed the aforementioned script to quantify the kinematics (translation and rotation) of the particles in sand specimens and illustrated the robustness of the algorithm. Figure 3.3a displays the evolution of the translation of the particles normalized with respect to the global compression of the specimen during different load steps. The particles exhibited higher translation values during the 1st load step as the specimen was in a loose state prior to the loading. The translation of the particles decreased with the compression of the specimen as the particles formed a denser packing. Figure 3.3b shows the variation of rotation angle (maximum rotation angle) of the particles during the experiment where particles exhibited higher rotation angle values during the 1th load step as the specimen was in a looser state compared with other load steps. The rotation of particles decreased with the compression of the specimen as the particles formed a denser packing that resulted in reducing the rotation of the particles. However, a few particles showed higher rotation values during the 3rd load step as the bulging initiated within the specimen.

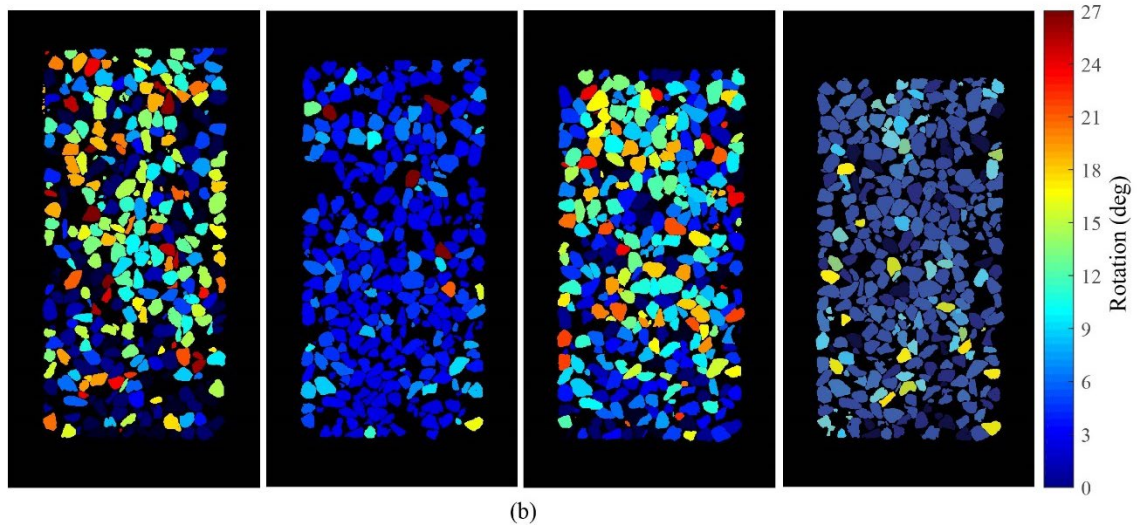
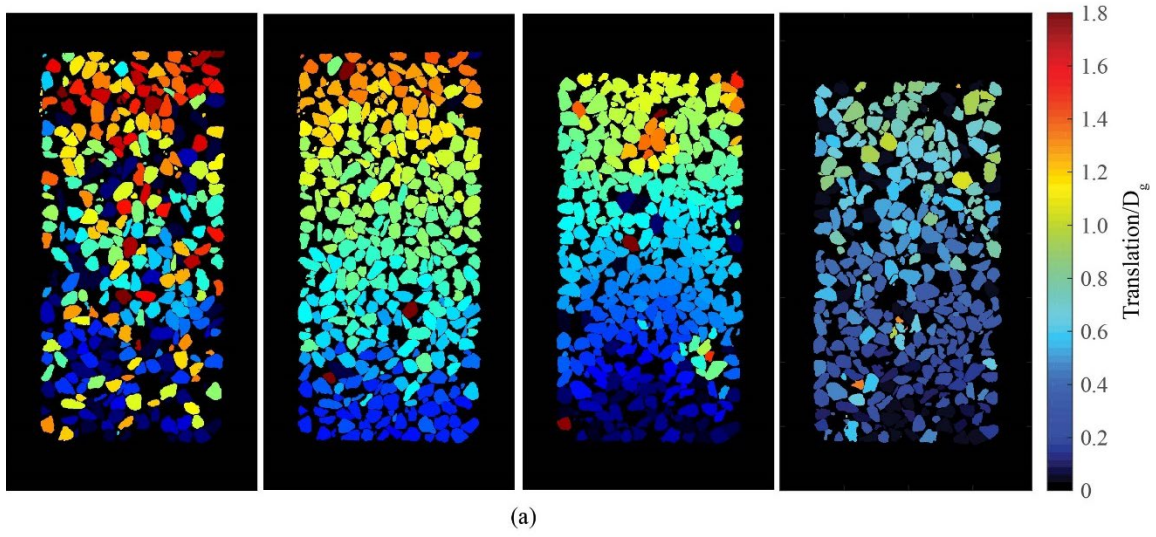


Figure 3.3. (a) The translation of the particles within the middle slice of the specimen during different load steps normalized with respect to the global compression of the specimen; (b) The rotation of the particles (in degrees) within the middle axial slice of the specimen during different load steps.

Analysis of 3DXRD measurements

3DXRD measurements were used to quantify the lattice strains within the particles in the specimen. The incident monochromatic x-ray beam diffracted on the crystals of the

particles of the specimen and the diffracted intensity was measured on the detector for those lattice planes which satisfied the geometrical condition defined by Bragg's law:

$$2d_{hkl}\sin\theta_{hkl} = n\lambda \quad (3.1)$$

where λ is the wavelength of the incident beam, n is a positive integer, and θ_{hkl} is the angle of the incident beam related to the lattice planes denoted by h, k , and l Miller indices with distance d_{hkl} (Figure 3.4a). As the specimen rotated during the 3DXRD scan, the lattice planes within the scanned volume came into Bragg condition and could be observed on the Debye–Scherrer rings on the diffraction detector (Figure 3.4b). The precise position (Figure 3.4c) of the diffraction spots were used to quantify the lattice strains within individual particles by evaluating the change in d_{hkl} according to Bragg's law. The diffraction analysis was performed using the MIDAS code (Sharma et al. 2012). In this analysis, after the calibration procedure, first, all diffraction spots and their corresponding crystal grains were determined. This procedure is called indexing. Then the diffraction spot positions measured from the same crystal grains were used for fitting its orientation (3 parameters), center of mass position (3 parameters), and lattice strain tensor (6 parameters) simultaneously in a least mean square sense through a 12-dimensional search for the solution of a highly over-determined equation system resulting from the precise measurement of the diffraction geometry and application of Bragg's law. The method is based on the principle that the diffraction spots measured in different directions and at different specimen angles are a direct measure of a certain projection of the strain of the crystal. Knowing the precise position of the specimen and the diffraction spots provides many samplings of the strain in various projections both in the laboratory and the crystal frame, which makes this procedure successful for strain tensor evaluation. Hence measuring the ε_{hkl} strain perpendicular to a specific (hkl) lattice plane can be obtained using the following formula:

$$\varepsilon_{hkl} = \frac{d_{hkl}^0 - d_{hkl}}{d_{hkl}^0} \quad (3.2)$$

where the parameter d_{hkl} is calculated from Bragg's law, d_{hkl}^0 is the corresponding value to the unstrained lattice prior to the loading. In this notation, compressive strains were considered to be positive. The procedure to calculate lattice strains is discussed briefly here to avoid lengthening the paper. For further details about the setup calibration aspects, calculation, and accuracy of the lattice strains, the reader is referred to the literature (Alshibli et al. 2013; Borbely et al. 2014; Cil et al. 2017; Moscicki et al. 2009).

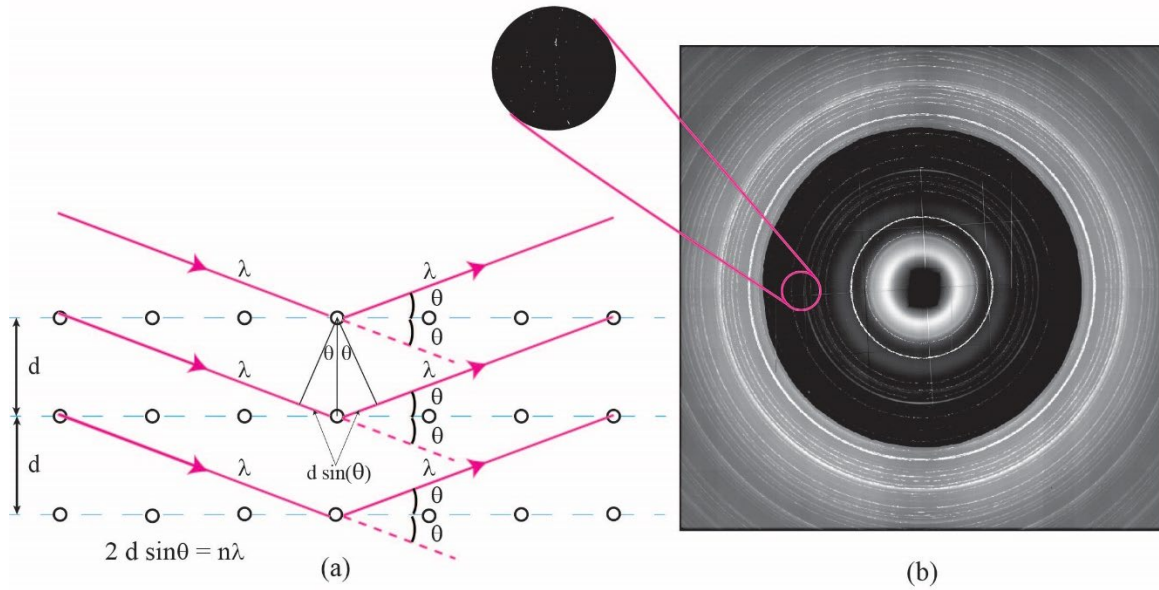


Figure 3.4. (a) Schematic illustration of Bragg's diffraction; (b) Debye Scherrer rings for all projections during the initial step prior to the loading and Diffraction peaks for the focus area

Matching Algorithm

A script developed by the authors was employed to match the 3DXRD measurement to their corresponding particles (i.e., this script matched the measured lattice strains within a crystal to the particle that was composed of that crystal.). The output file of MIDAS (program calculating the crystal strains) lists the lattice strain tensor expressed according to the global x, y, z axes, the orientation matrix, the spatial coordinates of the centroid of the crystals, the equivalent radius of the crystal and the error between the actual and fitted coordinates of the centroids for the crystals forming the particles within the experiment. Figure 3.4b illustrates the 3DXRD measurements as points at their corresponding crystal centroid coordinates. It is difficult to make a simple match between 3DXRD measurements and their corresponding particles merely based on these measurements, especially as some sand particles contained multiple crystals. The aforementioned script uses the labeled images, coordinates of the 3DXRD measurements, and the error associated with coordinates of the 3DXRD measurements to match each measurement to its corresponding particle. The algorithm is described here briefly. Initially, the SMT images are used to convert the voxels representing a particle into a list of points with coordinates associated with them (Figure 3.2c) as it is mathematically easier to define concepts such as distance between two points rather than between a point and a voxel (i.e., distance of a point to volume is both ambiguous and mathematically complex). The specimen is then divided into tetrahedral cells using the 3DXRD measurements (i.e., the centroids of the crystals are used to divide the space of the specimen into cells) using a tessellation algorithm (Figure 3.2c). A list of possible matches (3DXRD measurements with the shortest distance to all

the points representing each particle) was created based on the cells that each particle occupied (Figure 3.2d). The shortest distances between each possible match and the particles were compared to ensure that the matches were closest to the previously matched particles and to avoid a systematic matching error where a measurement is matched with the wrong particle just due to the order of the labeling in the labeled image.

A similar image with different labeling sequences was used to test the matching algorithm, and the matching results were similar among the two labeling sequences. Once the list of matches was finalized, the distances of the matches to the particles were examined to avoid erroneous matches. These erroneous matches included 3DXRD measurements outside the tolerance associated with the measured coordinates of the centroid of the crystal and also measurements with multiple possible particle matches. Figure 3.2d illustrates the different possibilities during the matching process. The correct matches are shown with green dots, while erroneous matches are shown with red dots. Most of the particles in the specimen had single 3DXRD measurements matched with them. However, there were some particles that had multiple 3DXRD measurements matched with them. The number of particles with matches varied during the experiment. The variation in the number of 3DXRD measurements, the total number of particles, and the number of matched particles during the experiment are summarized in Table 3.1.

Table 3.1. Summary of variation of the total number of particles, the total number of 3DXRD measurements, number of orphan particles, and orphan 3DXRD measurements during different load steps

Items	Step 0	Step 1	Step 2	Step 3	Step 4
Number of particles	2705	2705	2689	2701	2738
Total number of 3DXRD measurements	5027	5199	5288	4124	4191
Number of orphan particles	271	344	340	352	213
Number of orphan 3DXRD measurements	474	518	653	751	626

During each 3DXRD scan, the diffractions from the particles were analyzed, and the lattice planes that satisfied the Bragg's condition and were accurately tracked (indexing process) during different load steps (i.e., met the retention factor) were used to measure crystal strains within the particles. The number of such readings differed from one load step to the other and was referred to as the total number of 3DXRD measurements in this study. Moreover, there were some particles and 3DXRD measurements that did not have a particle match or a suitable 3DXRD measurement match. They were labeled as orphan particles and orphan 3DXRD measurements, respectively. The orphan particles were particles that did not have a matched 3DXRD measurement either due to the intensity threshold used to identify the diffraction peaks or because the 3DXRD measurements corresponding to the particle did not meet all matching criteria. The Orphan measurements are the ones that were not matched with particles either due to the unacceptable distance that the measurement had to the particles (i.e., more than the error of the fitted coordinates) or because they had multiple possible particle matches within the error of the measurement.

The strain within the particles with multiple matched measurements was averaged based on the volume of the measurements. After the lattice strains were averaged (volumetric average), they were converted into stresses using Hook's law with the appropriate anisotropic elasticity matrix (C_{ijkl}) for α -quartz mineral (Heyliger et al. 2003). Table 3. 2 lists the elastic moduli for the anisotropic model used in this study. The strains in the global coordinate system were converted into strains in the local crystal coordinate system based on the orientation matrices associated with the crystals, and then the strains in the local coordinate system were multiplied by the appropriate elasticity matrix to calculate the stresses in the local coordinate systems. The calculated stresses were transformed back into the global coordinate system. The process is summarized as follows:

$$[\epsilon'] = [O^T][\epsilon][O] \quad (3.3)$$

$$[\sigma'] = [C][\epsilon'] \quad (3.4)$$

$$[\sigma] = [O][\sigma'][O^T] \quad (3.5)$$

where ϵ is the strain tensor of a particle in the global coordinate system, ϵ' is the strain tensor in the local crystal coordinate system, and C is the elasticity matrix. σ and σ' are the stress tensor in the global and local coordinates systems, respectively. O is the orientation matrix that converts the local crystal coordinate system into the global coordinate system, and O^T is the transpose of O . The details of the matching process, and the calculations related to the averaging of the strains and stresses within the particles are reported in (Amirrahmat et al. 2020). Figure 3.5 shows the variation of principal stresses within the particles before the loading and during different load steps. Normal stresses generally increased during the experiment except during the 3rd load step as the specimen began to bulge, which caused the rearrangement of the particles and redistribution of the stresses. The shear stresses within the particles remained relatively small in magnitude during the

experiment (i.e., the average remained close to zero). However, the standard deviation of the shear stresses increased (i.e., the range of shear stresses increased in magnitude). Table 3.3 summarizes the variation of the average and standard deviation of the stresses within the particles.

Table 3.2. Summary of isotropic and anisotropic elastic moduli for α -quartz

Isotropic Model	
Elastic properties	Value (GPa)
Anisotropic model	
C11	87.26
C22	87.26
C33	105.8
C44	57.15
C55	57.15
C66	40.35
C12	6.57
C13	11.95
C23	-17.18
Poisson Ratio	
ν_{12}	0.1286
ν_{23}	0.0984
ν_{13}	0.1274

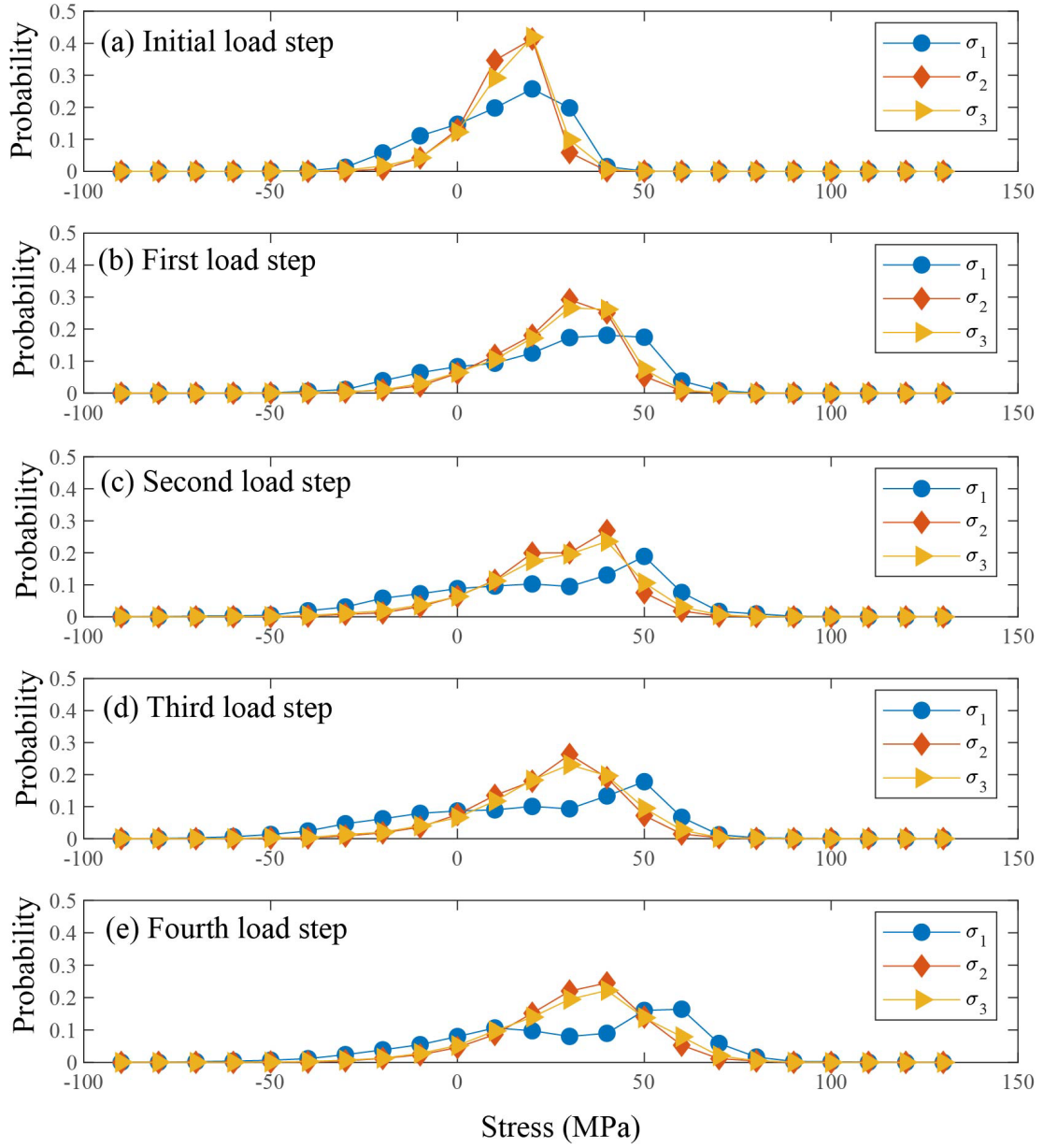


Figure 3.5. Statistical distribution of principal stresses within individual particles at global axial strains of (a) 0%; (b) 4.35%; (c) 6.71%; (d) 8.35%; and (e) 11.47%

Table 3.3. Summary of the variation of stresses during different load steps.

Stresses	Initial load step		1 st load step		2 nd load step		3 rd load step		4 th load step	
	mean (MPa)	S _d (MPa)	mean (MPa)	S _d (MPa)	mean (MPa)	S _d (MPa)	mean (MPa)	S _d (MPa)	mean (MPa)	S _d (MPa)
σ_{xx}	16.01	9.891	29.89	19.87	31.18	19.39	26.07	18.97	35.31	17.93
σ_{yy}	16.15	10.29	29.98	18.55	31.18	19.22	27.05	18.91	35.81	18.26
σ_{zz}	20.27	7.903	33.58	13.43	35.07	15.28	30.02	15.81	35.19	16.95
σ_{yz}	0.067	5.231	-0.292	7.021	-0.288	10.29	-0.057	10.36	-0.160	10.70
σ_{xz}	0.029	5.179	0.291	7.077	-0.375	10.25	-0.155	10.55	0.007	11.30
σ_{xy}	-0.023	5.721	0.284	7.525	-0.089	10.70	-0.155	10.92	-0.354	11.17
σ_1	18.94	9.25	32.79	18.83	35.23	19.36	29.73	19.59	36.01	20.44
σ_2	17.93	10.65	31.25	19.03	30.41	17.27	29.11	17.84	35.85	18.30
σ_3	15.94	15.78	30.40	22.88	30.32	17.57	24.38	30.54	34.45	20.39

Definition of particle scale properties

The variation of stress fields within particles and the mechanics of force-transmission in granular materials is influenced by various particle-scale properties. Investigating the effect of these properties such as position, morphology, fabric, crystal structure, and kinematics of the particles on the variation of stresses can provide an important insight into the mechanics of granular materials. The particle-scale properties investigated in this study include the 3D spatial location of the particle from the bottom of the specimen normalized with respect to the initial height of the specimen (H), the distance of the particle from the central axis (loading direction) of the specimen normalized with respect to the initial radius of the specimen (r), the volume of particles normalized with respect to the maximum volume of the particles (Vol), the surface area of particles normalized with respect to the maximum surface area of the particles (S), the principal length (diameter) of particles normalized with respect to the maximum principal length (PL), the relative orientation of the principal length with the direction of loading (PLD), number of crystals forming each particle (NCR), the relative orientation of the crystals (after averaging process) with respect

to the loading direction (DCR), the deviation of the contact network of each particle with the direction of loading (DC), the number of the contacts (CN), the translation of the particles normalized with respect to the global compressive displacement of the specimen (D), and the rotation of the particles normalized with respect to the maximum rotation of the particles in each step (R).

It is essential to investigate these particle properties for possible correlations among themselves since highly correlated variables can influence any statistical models describing the variation of the internal stress field within the particles.

Figure 3.6 displays the correlation coefficients of the particle properties. The volume, surface area, and principal length of the particles were highly correlated with each other as they are all related to the size of the particle. The spatial location of the particles and their contact numbers were reversely correlated, suggesting that particles at the bottom of the specimen had higher CN compared with the particles at the top of the specimen (i.e., the specimen had a denser packing at the bottom compared with its top). The translation of the particles was correlated to their spatial location, indicating that particles at the top of the specimen had higher translations compared with particles at the bottom of the specimen. Volume, surface area, and the principal length of the particle were correlated with the number of crystals within particles, indicating that larger particles were more likely composed of more crystals. The direction of the contacts and the direction of the crystals were also correlated with the number of contacts and crystals, respectively, as more contacts or crystals would promote a higher chance of deviation from the loading direction. The CN of the particles was correlated with the size of particles (i.e., the volume, the

surface area, and the principal length) as well since larger particles have higher contact numbers. Most of the aforementioned particle properties were numeric variables (i.e., continuous) except the spatial location (bottom, middle, top), the number of crystals, and the number contacts, which were categorical variables.

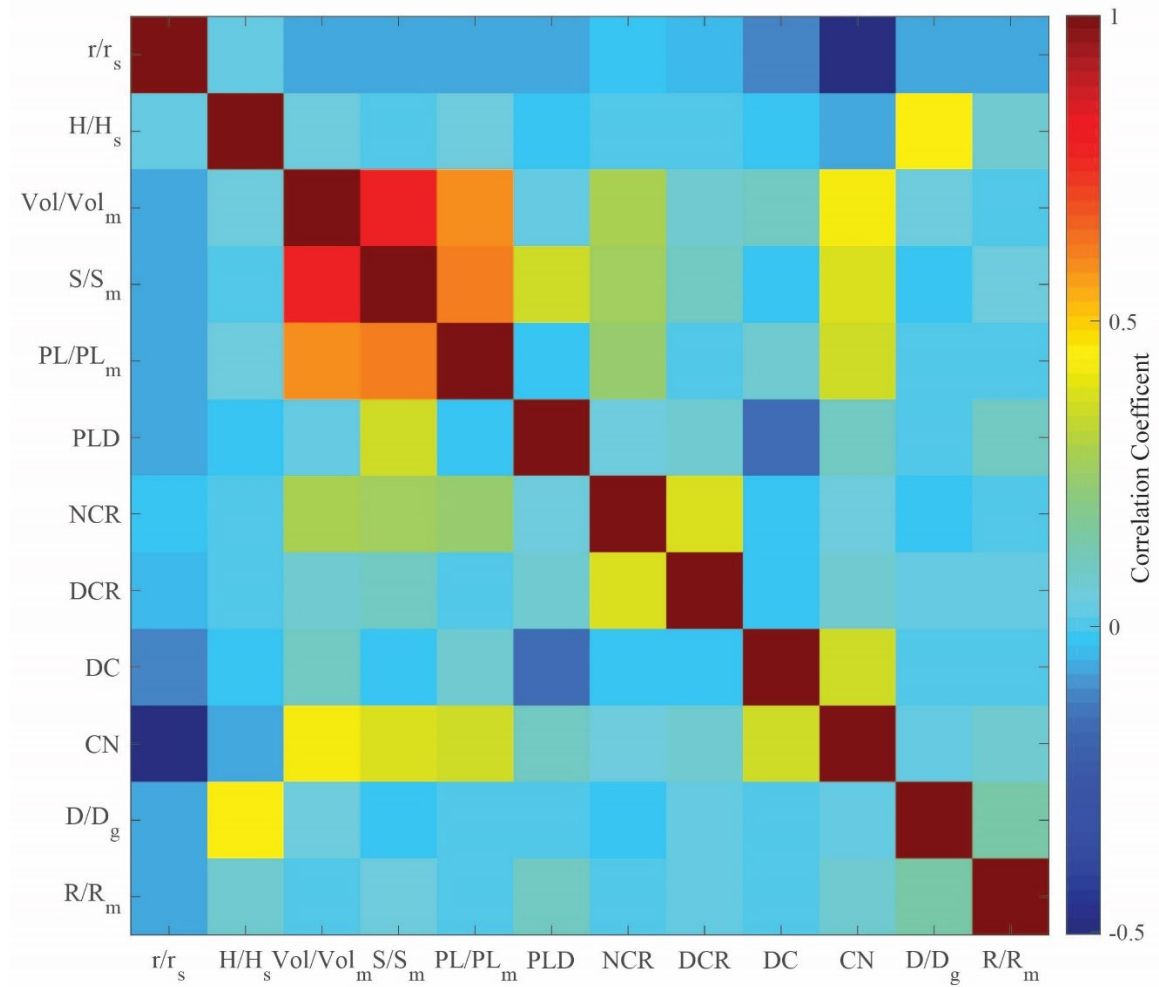


Figure 3.6. The correlation coefficient between different particle-scale properties during the 1st load step

Feature selection

A feature selection algorithm called RReliefF was employed in this study to identify the important particle-scale properties on the variation of the internal particle stress field as a preliminary statistical analysis. RReliefF is one of the feature selection algorithms from the family of Relief algorithms. Relief Algorithms are feature selection algorithms based on the nearest neighbor method which are used to rank the importance of different predictors (particle-scale properties) explaining a response field (variation of maximum absolute principal stress or MAPS for simplicity). As many feature selection problems, a high number of the predictors affects the accuracy of a created model since some of these predictors create noise in the response field (MAPS) and provide very little useful information. Relief algorithms were originally designed to select important features in binary (i.e., hit or miss) response fields (Kira and Rendell 1992; Kira and Rendell 1992). Unlike other heuristic measures to rank importance of properties, Relief algorithms do not assume independence of the properties (i.e., no interactions between the properties) which makes it more appropriate in problems with possible property interaction. Robnik-Šikonja and Kononenko (1997) expanded the original Relief algorithm to RReliefF for problems with continuous response field (i.e., regression problems). RReliefF is not limited to two-class problems and is more robust compared with the original Relief algorithm in dealing with noisy response fields. The RReliefF algorithm is briefly explained here and the readers are referred to Robnik-Šikonja and Kononenko (1997) and Kononenko (1994) for more information about the algorithm.

RReliefF algorithm ranks the importance of the predictors using a k-nearest neighbor method by assigning a weight to each predictor. The algorithm finds the k-nearest neighbors to a reading and rewards the predictors that give different values to responses that have different predictor values and penalizes predictors that give similar values to responses with different predictor values. The algorithm uses intermediate weights to calculate the final weights. The intermediate weights include W_{d_y} , W_{d_j} , and $W_{d_y \wedge d_j}$. W_{d_y} is the weight of having different response value, W_{d_j} is the weight of having different values for a predictor (F_j), and $W_{d_y \wedge d_j}$ is the weight of having both a different response and a different predictor F_j . Initially, these weights were set to 0 and then the algorithm recalculates each weight value through an iterative method. The following Equations define the framework of the method:

$$W_{d_y}^i = W_{d_y}^{i-1} + \Delta_y(x_r, x_q) \cdot d_{rq} \quad (3.6)$$

$$W_{d_j}^i = W_{d_j}^{i-1} + \Delta_j(x_r, x_q) \cdot d_{rq} \quad (3.7)$$

$$W_{d_y \wedge d_j}^i = W_{d_y \wedge d_j}^{i-1} + \Delta_j(x_r, x_q) \cdot \Delta_y(x_r, x_q) \cdot d_{rq} \quad (3.8)$$

$$\Delta_y(x_r, x_q) = \frac{|y_r - y_q|}{\max(y) - \min(y)} \quad (3.9)$$

$$\Delta_j(x_r, x_q) = \begin{cases} 1, & \text{if } F_{jr} = F_{jq} \\ 0, & \text{if } F_{jr} \neq F_{jq} \end{cases} \quad (3.10)$$

$$d_{rq} = e^{-\left(\frac{\text{rank}(x_r, x_q)}{\sigma}\right)^2} \quad (3.11)$$

$$W_j = \frac{W_{d_y \wedge d_j}}{W_{d_y}} - \frac{W_{d_j} - W_{d_y \wedge d_j}}{m - W_{d_y}} \quad (3.12)$$

where $W_{d_y}^i$ and $W_{d_y}^{i-1}$ are the response weights at iteration $i-1$ and i , $W_{d_j}^i$ and $W_{d_j}^{i-1}$ are the predictor weights at iteration $i-1$ and i , and y_r and y_q are the response value for the two selected near neighbors. The importance factor d_{rq} is used to control the effect of nearest neighbors based on their nearness rank (i.e., $\text{rank}(x_r, x_q)$) and σ value (scaling factor), which is equal to 50 in this study. W_j is the calculated weight corresponding to predictor j . This method is susceptible to the number of nearest neighbors used to calculate the weight of the predictors. Using too few or too many neighbors can cause over-fitting or under-fitting in the model where a model either cannot predict the values correctly (under-fitting) or it is overly sensitive to the change associated with the predictors (over-fitting). In this study, different neighborhood sizes were used to assess the sensitivity of the model to the size of the neighborhood. Figure 3.7 shows the variation of weight of the particle properties with the different number of neighbors (5 to 25) and the models is stable (i.e., the weights do not change much with different numbers of neighbors). Kira and Rendell (1992) introduced a significance threshold where a predictor would be significant if the weight is higher than $\frac{1}{\sqrt{\alpha m}}$ where α is the probability of accepting an irrelevant predictor as relevant (selected to be 5% in this study) and m is the number of iterations (in this case the number of particles or 2705). However, they showed that this threshold is very conservative, and smaller weights are used during preliminary studies routinely. Due to the nature of the Equations used in this method, the algorithm works for either discrete predictor fields (classification problem) or numerical variables (regression problem) and will not work for both types of predictors together. Moreover, a high correlation between the predictors affects the accuracy of the model. An RReliefF model was employed to rank the importance of a selection of the numerical predictors that did not exhibit high correlations (volume, PLD, DCR, DC, translation, and rotation). The rest of the predictors were examined separately to assess their influence on the variation of MAPS within the particles. Figure 3.7 summarizes the variation of the weights of the particle-scale properties during the 1st load step

using different nearest-neighbor sizes (5 to 25 neighbors). Translation, rotation, and the direction of the contacts were identified as the important predictors using the RREliefF model. These properties, along with discontinuous properties such as the number of contacts, and the spatial location of the particles in the specimen (i.e., top, middle, and bottom) were examined in more detail in the next section. The rest of the particle properties such as radius, volume, the direction of crystals, number of crystals, surface area, and principal length are not considered any further as they did not exhibit any meaningful effects on the variation of MAPS.

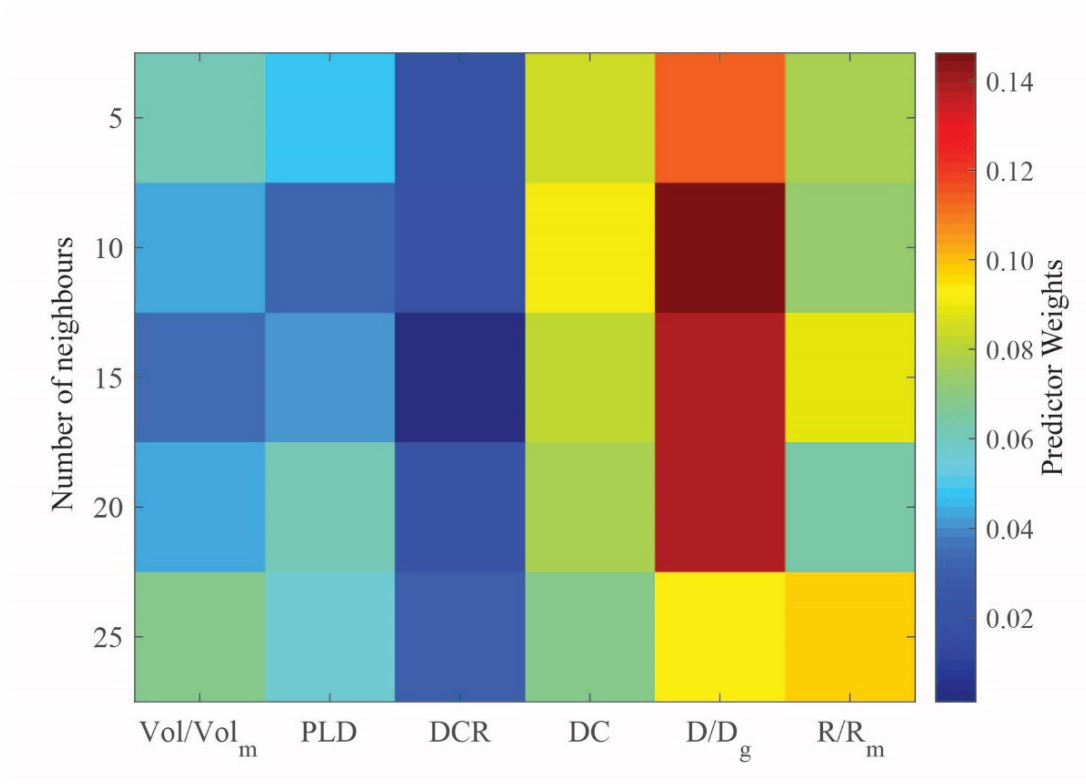


Figure 3.7. The weight of particle-scale properties during the 1st load step using different numbers of neighbors.

Effect of particle-scale properties on the variation of stress within particles

The kinematics of the particles (translation and rotation) affects strains and stresses within particles. The inter-particle forces at contacts within a bulk of granular material can cause particles to either translate, rotate, or deform (which causes internal stresses within the particle). Moreover, the local contact network of individual particles influences the distribution of inter-particle forces, kinematics of the particles, and can alter internal stresses within particles. The location of the particles with respect to the boundaries of the specimen can affect the evolution of stresses within particles as well. The effects of the aforementioned particle properties on the evolution of stresses within the particles are discussed in this Section.

Figure 3.8 shows the variation of MAPS versus the translation of particles normalized with respect to the global compression of the specimen. During the 1st load step, particles exhibited a wider range of translation compared with other load steps, and MAPS showed a more uniform distribution. Highly stressed particles (i.e., particles with MAPS higher than 60 MPa) had a smaller translation (< 0.8) compared with the rest of the particles during the 2nd through 4th load steps. The distribution of stresses smaller than 60 MPa was more uniform when it was compared with the MAPS above 60 MPa. In other words, translation was more accurate as a predictor for particles with higher MAPS (above 60 MPa) than particles with lower MAPS (less than 60 MPa). This accuracy also increased in particles with a higher translation (> 1) as the range of variation of stresses decreased within these particles (between 35 to 65 MPa). This was especially evident in the 3rd and 4th load steps.

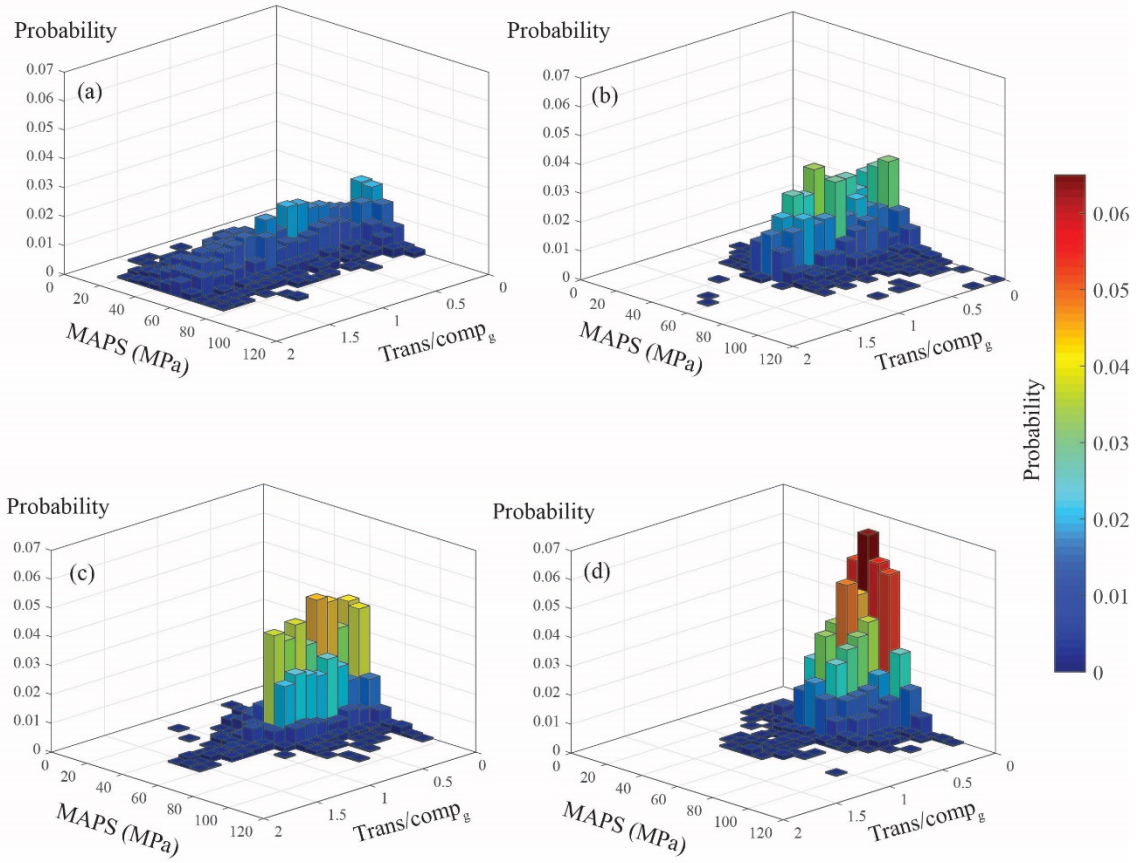


Figure 3.8. The variation of MAPS within particles versus their translation normalized with respect to global compression at the global axial strain of (a) 4.35%; (b) 6.7%; (c) 8.35%; and (d) 11.47%.

Figure 3.9 displays the variation of MAPS versus the rotation angle of the particles normalized with respect to the maximum rotation angle of all particles within the specimen at each load step. It is evident (especially in the 2nd load step) that particles with higher MAPS exhibited smaller rotation angles which is similar to observation related to the influence of particles' translation. However, the distribution of rotation versus the MAPS of the particles in other load steps has a nearly uniform distribution (rotation can be used to identify possible highly stressed particles, but the accuracy of predicted stresses based

on rotation is reduced in the lower range of MAPS). Comparing the effect of rotation and translation also suggests that the translation is a better predictor of MAPS within the particles in this experiment as the distribution of MAPS with respect to the rotation of the particles is closer to a uniform distribution. It should be noted that the dominance of translation compared with rotation in affecting the MAPS within the particles is dependent on the loading (e.g., triaxial or 1D compression) and the deformation of the specimen as well and the role of translation and rotation might be reversed.

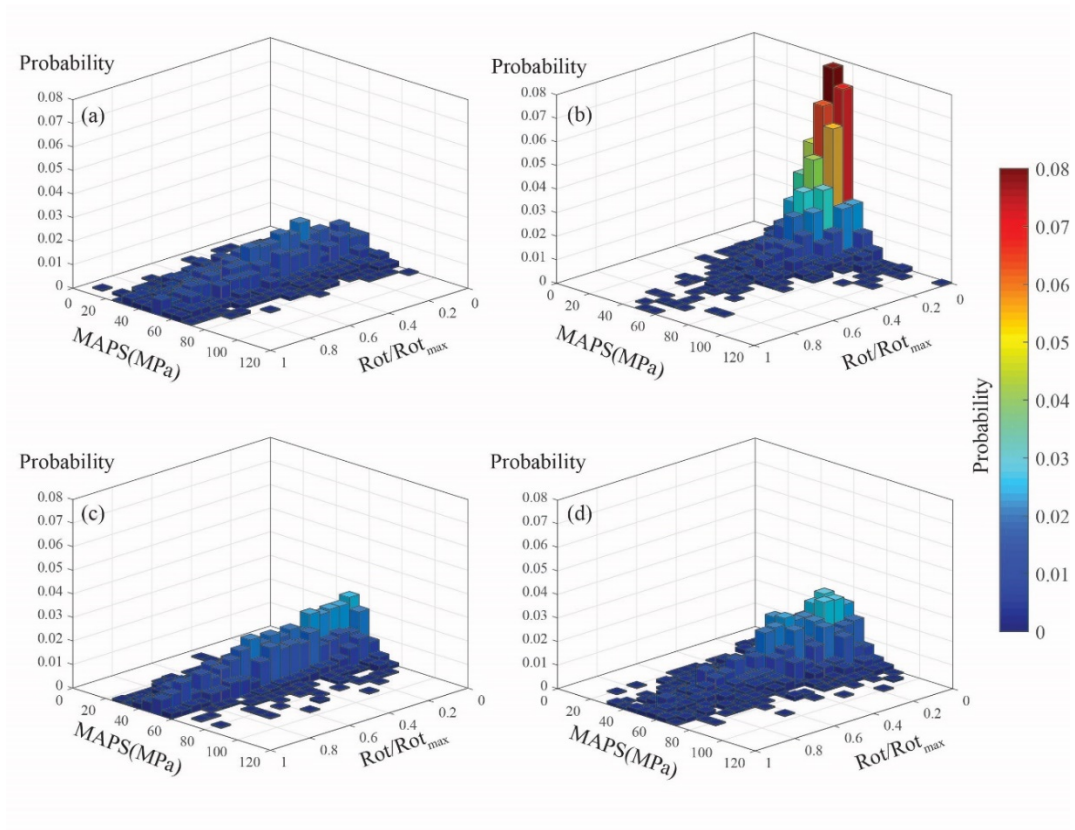


Figure 3.9. The distribution of MAPS versus rotation angle of the particles normalized with the maximum rotation angle of the particles within the specimen at global axial strain (a) 4.35%; (b) 6.71%; (c) 8.35%; and (d) 11.47%.

The variation of MAPS versus the cosine of the maximum angle between the contacts and the direction of loading (i.e., deviation from the loading direction) is illustrated in Figure 3.10. Particles with higher MAPS (above 60 MPa) had contacts that were more aligned with the direction of loading. The range of variation in the magnitude of the stresses decreased as the particles' contacts deviated more from the direction of loading (between 40 to 60 MPa).

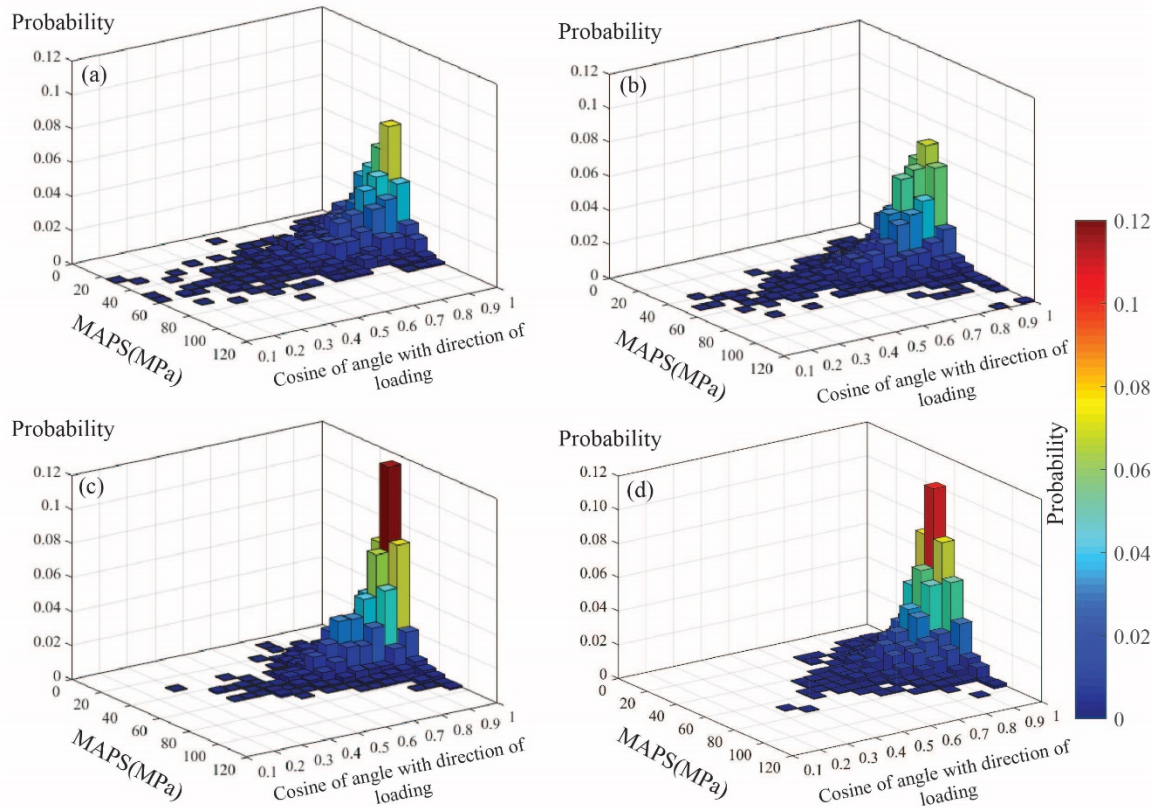


Figure 3.10. The distribution of MAPS versus the cosine of the maximum angle of the contacts made with the direction of loading (i.e., deviation from the loading direction) at a global axial strain of (a) 4.35%; (b) 6.71%; (c) 8.35%; and (d) 11.47%.

Figure 3.11 illustrates the distribution of MAPS versus the CN of the particles during different load steps. During the 1st loading step, the stresses within the particles decreased as the CN of the particles increased except for particles with CN = 10 to 11 that exhibited a local maxima. During the 2nd load step, particles with mid-range CN (CN = 5 to 9) and particles with a high CN (CN = 9 to 13) showed an increase in the stress while particles with a low CN = 2 to 5 showed a decrease compared with the 1st load step. Particles with mid-range contact numbers exhibited a decrease in stress while other particles with a low and a high CN remained relatively the same during the 3rd load step. The increase in stresses during the 4th load step was most evident in particles with a mid-range CN as well. The stresses within particles with a high CN increased more when compared with particles with a mid-range CN through the experiment. Also, the stresses within particles that have a mid-range CN increased after the particles with a high CN (compare the mid-range CN particles during the 2nd load step with the 3rd and 4th load step in Figure 3.11b,c, and d). In summary, Figure 3.11 suggests that the stresses in the highly confined particles (high CN) generally increased before the mid-range and low CN particles. The increase in the stresses of high CN particles was higher in magnitude in comparison with other particles as well. Moreover, particles with mid-range and low CN were more prone to changes in stresses due to any loading and unloading (in this case, bulging caused unloading) compared with high CN particles. Turner et al. (2016) and Imseeh and Alshibli (2018) used FE models to model the variation of particle stresses due to compression. Both papers showed that the magnitude of the principal tensile stress decreases in particles with higher CN. This is true in this study for particles with mid-range CN as they show more variation in MAPS during

different load steps. However, particles with high CN exhibited higher values for MAPS compared with particles with lower contact number as they had less degree of freedom for translation and rotation. The particles in Turner et al. (2016) and Imseeh and Alshibli (2018) had looser packing especially in the later stages of compression compared with the specimen reported in this paper (i.e. their maximum contact number was 10 and 8, respectively).

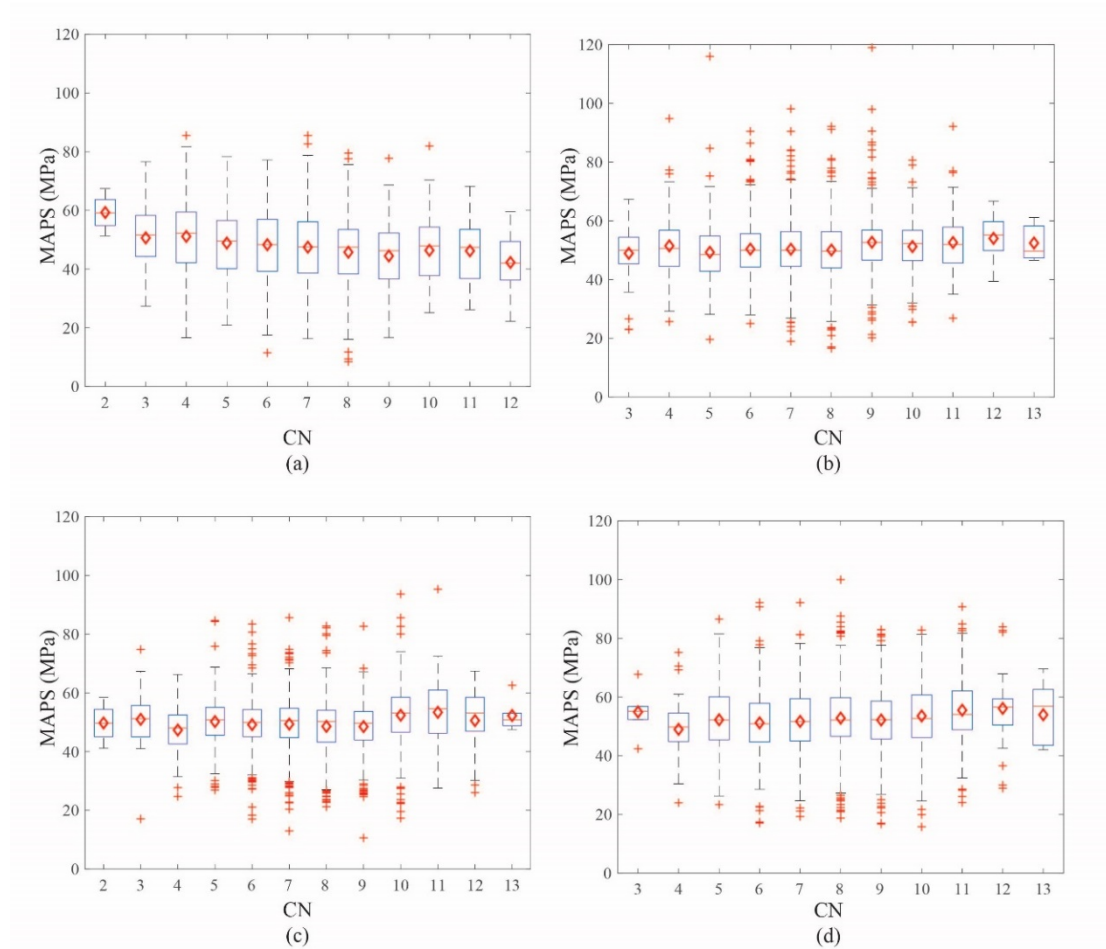


Figure 3.11. The distribution of MAPS versus the CN of the particles at a global axial strain of (a) 4.35%; (b) 6.71%; (c) 8.35%; and (d) 11.47%.

The variation of MAPS versus the spatial location of the particles along the height of the specimen is shown in Figure 3.12. It is evident that during the 1st load step, the stresses within the bottom of the specimen was significantly higher than the middle and top of the specimen. During the 2nd load step, the stresses in the middle and at the top of the specimen increased and got closer to values of stresses within particles at the bottom of the specimen, but the bottom particles still had the highest stresses. During the 3rd load step, as the specimen began to bulge in the middle, the stresses within the particles decreased and became nearly uniform. As loading continued, stresses within the bottom of the specimen increased during the 4th load step.

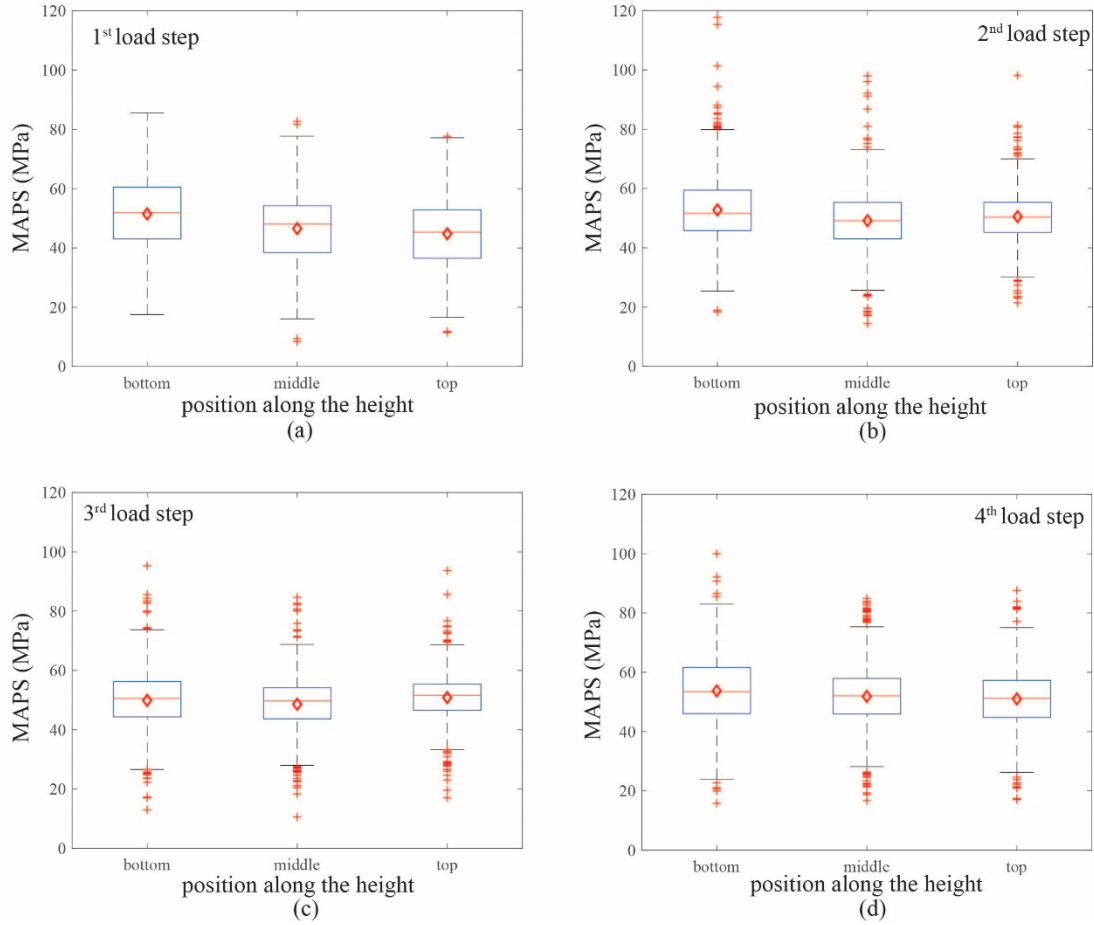


Figure 3.12. The distribution of stress at the bottom, in the middle and at the top of the specimen at global axial strain (a) 4.35%; (b) 6.71%; (c) 8.35%; and (d) 11.47%.

Effect of particle scale-properties on the fracture of sand particles

The fracture mode of particles is rather a complex phenomenon that is influenced by particle scale-properties and the nature of interactions between particles. The effects of particle-scale properties such as spatial location, size, translation, rotation, CN, and local contact network of fractured particles are studied to expose possible relationships between these properties and the fracture of particles. The evolution of both hydrostatic and deviatoric stresses within these particles was also studied for possible correlations between the stresses and the fracture of the particles.

The fractured particles were initially identified from the experiment using the SMT images. 19 particles fractured during the 3rd load step and 187 particles fractured between the 4th and 5th load steps (the fractured particles were identified using the 5th load step image at 37.5 N axial load and 14.03% axial strain). The fractured particles were tracked throughout the experiment and 111 particles had stresses associated with them at all load steps. Figure 3.13 displays two examples of the fractured particles. The first particle fractured during the 5th load step whereas the second particle fractured during the 3rd load step followed by a further fracture during the 5th load step. Figure 3.13a & g shows the variation of maximum principal stress within the two particles during the experiment. The maximum principal stress within the first particle exhibited a continuous increase until particle fracture during the 5th load step (Figure 3.13a & f). The second particle is one of the 19 particles that fractured during the 3rd load step. The magnitude of maximum principal stress increased during the first two load steps, and then it decreased as the particle fractured during the 3rd load step (Figure 3.13g & k). Then, the stress within the particle increased during the 4th loading step and the particle fractured again during the 5th load step as well (Figure 3.13m). The effect of internal stresses within the particles was examined by assessing the variation of hydrostatic and the deviatoric stresses within the particles. The hydrostatic and deviatoric stresses are defined as:

$$\sigma_h = \sigma_{ii}/3 \text{ (summation intended)} \quad (3.12)$$

$$\sigma_D = \sqrt{(\sigma_1 - \sigma_2)^2 + (\sigma_2 - \sigma_3)^2 + (\sigma_1 - \sigma_3)^2} \quad (3.13)$$

where σ_h is the hydrostatic stress, σ_{ii} are the diagonal components of the stress tensor, σ_D is the deviatoric stress, σ_1 , σ_2 and σ_3 are the principal stresses within particles.

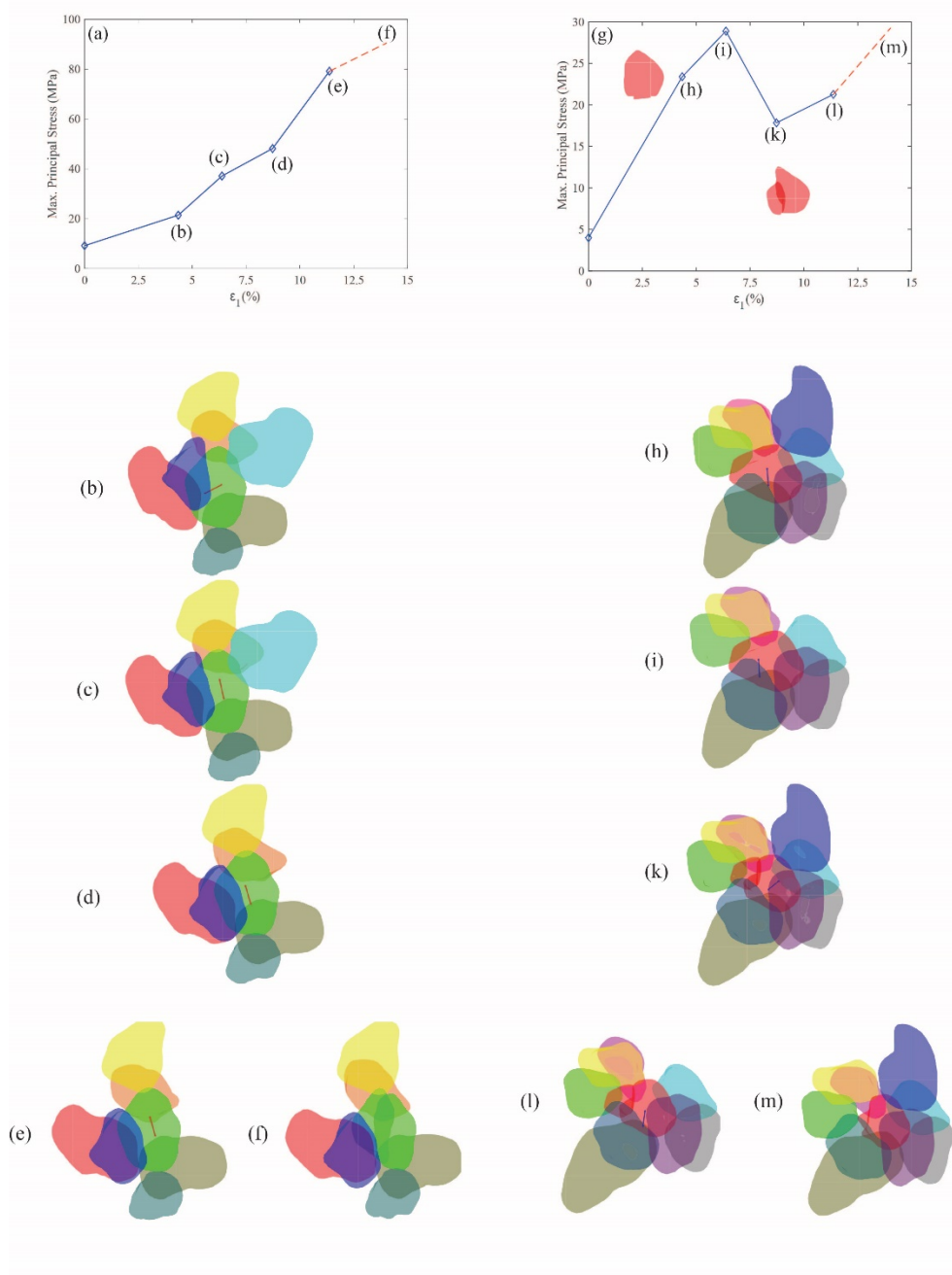


Figure 3.13. Evolution of two example particles from the initial state up to the fracture of the particles during the 5th load step (the dashed line indicates that a fracture happened at the 5th load step but the 5th load step is removed from the analysis).

Figure 3.14 depicts the variation of the hydrostatic stress versus the deviatoric stress within particles one load step prior to their fracture (i.e., second load step for particles that fractured during the 3rd load step and for the rest particles during the 4th load step). The stress magnitudes within the fractured particles vary widely and internal particle stresses cannot be an accurate parameter to identify particles that have a high probability of fracture. This wide variation range is due to the different internal structures of particles (i.e., the change in the number of crystals in different particles as well as the presence of defects in some of the particles).

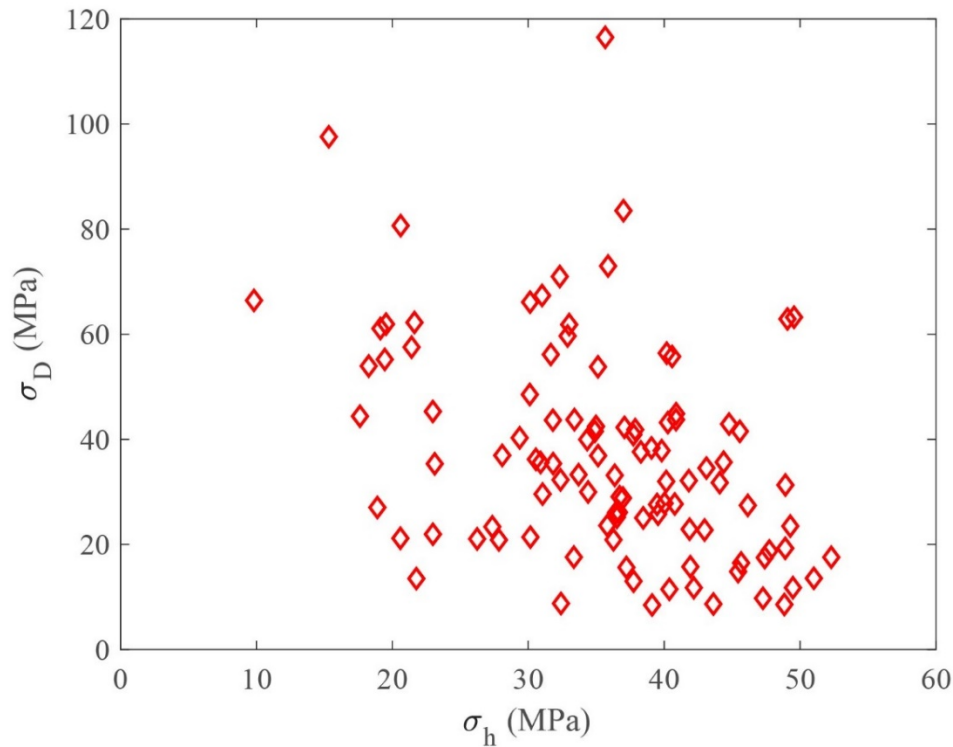


Figure 3.14. The variation of the hydrostatic stress versus deviatoric stress within the particles that fractured one load step prior to their fracture.

Figure 3.15a shows the variation of the average translation of particles versus the global axial strain of the specimen. The fractured particles exhibited smaller translation in comparison with the rest of the particles. However, the difference between the fractured and intact particles decreased as the loading progressed. A similar pattern is evident in the rotation of fractured particles (Figure 3.15c) in comparison with the rest of the particles as the inter-particle forces can either cause translation/ rotation of the particles or deformation within the particles that will eventually lead to fracture. The effect of the local contact network of the particles on their fracture was also investigated where the average contact numbers of the fractured particles were compared with the intact particles as shown in Figure 3.15b. Initially, the fractured particles had similar ACN as the rest of the particles, but the ACN of the fractured particles increased during the latter load steps of the experiment. The higher CN of the particles limits the translation and rotation of the particles and changes the distribution of inter-particle forces and eventually affects the fracture potential of particles. Hurley et al. (2018) used 3DXRD and CT images to study the fracture of single-crystal sapphire particles (spherical) under 1D compression. They showed that particles with smaller CN have a higher chance of fragmentation. However, this fact might not be completely correct in particles with arbitrary morphology as the contact forces will not be coplanar (i.e., do not meet at the center of mass of the particle). This effect is clearly evident as the ACN of particle that fractured increased with the compression of the specimen. The effect of the orientation of these local contact networks relative to the direction of the fracture was also examined, which will be discussed later in this Section. The spatial location of particles within the specimen and relative position to

the boundary of the specimen were studied as one of the possible particle properties that can affect the fracture of the particles. Figure 3.15d depicts the normalized position of the particles (spatial location and radius from the central axis of the specimen). Most of the fractured particles were close to the top and bottom load plates and the plastic tube wall around the specimen rather than the center of the specimen and away from the loading plates.

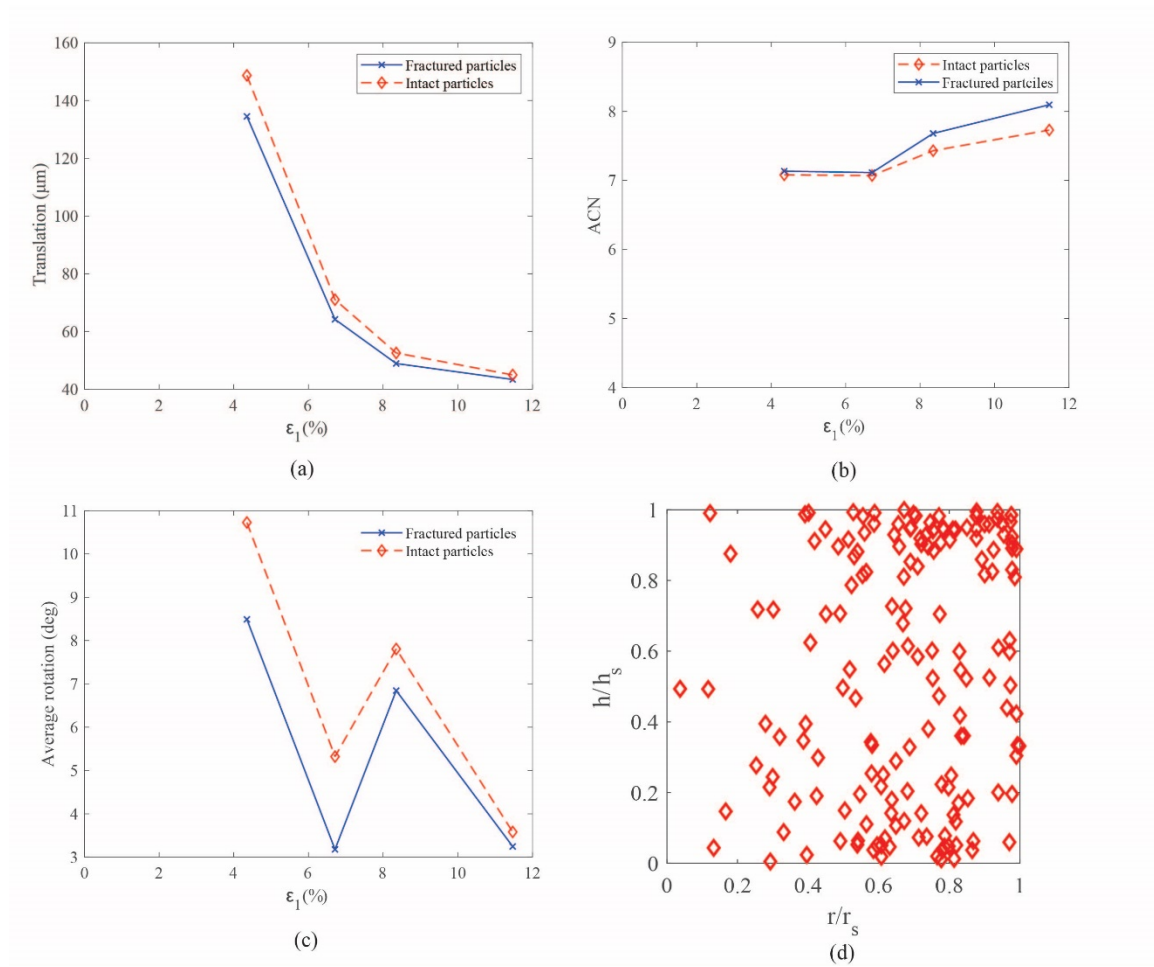


Figure 3.15. The effects of (a) translation; (b) ACN; (c) rotation; (d) and position of fractured particles.

The relative orientation of the fracture surface compared with the direction of maximum and minimum principal length of the particle (shape) and the eigenvector of the local fabric tensor of individual particles (weighted and unweighted) were examined to assess the effect of loading direction on the fracture of particles. The orientation of the fracture surfaces needs]ed to be characterized as a first step. The fracture surfaces of the particles were examined using SMT images. They were divided into three types; linear, slightly curved, and curved surfaces. Figure 3.16a shows an example of a linear surface where the red dashed line represents the characterized fracture surface. The surfaces were characterized by defining two vectors (V_1 and V_2) on each surface and the normal vector of each surface (N) was calculated by finding the outer product of V_1 and V_2 . Figure 3.16b shows an example of slightly curved surface where the surface of the fracture is mostly linear and has a minimal curvature. The white dashed line in Figure 3.16b compares the mathematically characterized fracture surface with the actual fracture surface of the particle. The characterized fracture surface matched with the actual surface closely except for a small area to the left side of particle. However, there were particles with curved fracture surfaces that could not be described as one surface (i.e., curved). The curved surfaces had two different types. Some of them were composed of more than one linear surface (Figure 3.16c), and others were composed of multiple curved surfaces (Figure 3.16d). 83% of the fractured surfaces were either linear or slightly curved and could accurately be described by a single surface Figure 3.16e).

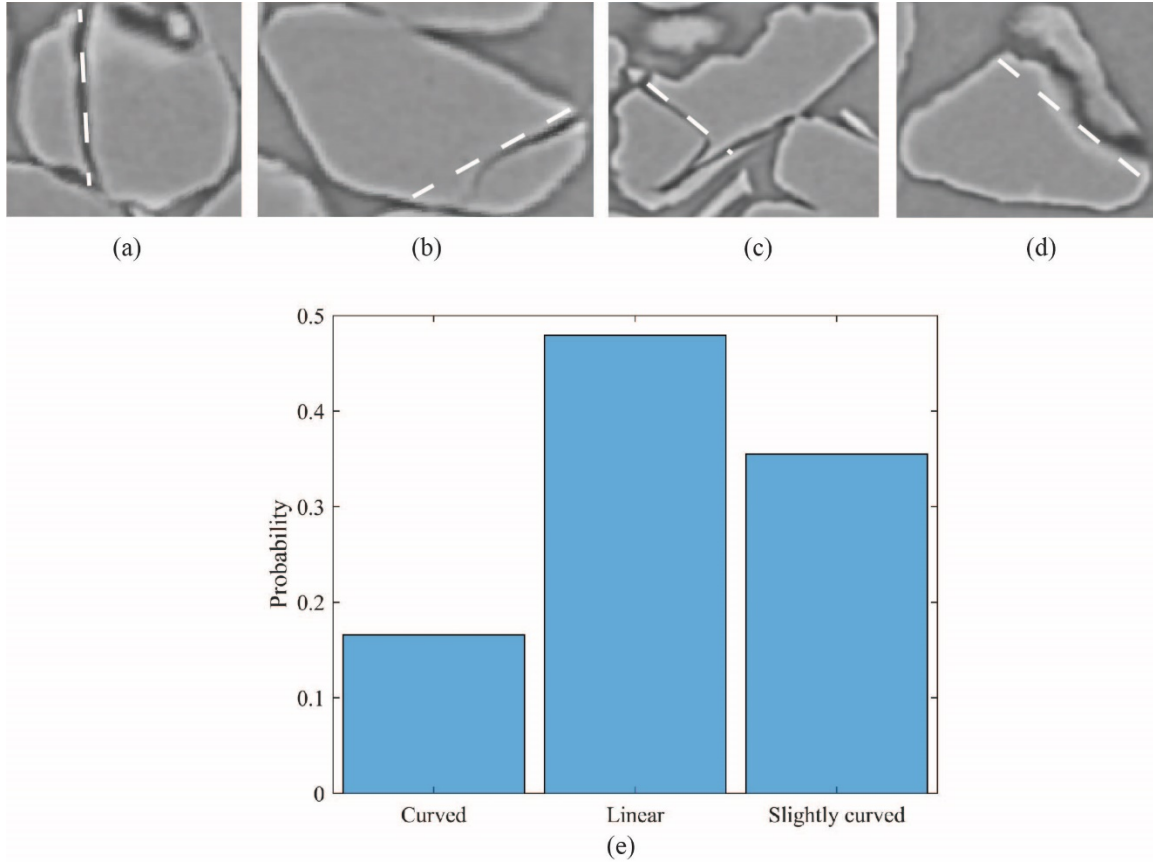


Figure 3.16. (a) to (d) Definition of fracture surfaces and the shape of the fracture surfaces (e) probability of different modes of fracture.

Figure 3.17a shows the relative orientation of the fractured surface of the particles with respect to the direction of the minor principal length of the particle. The normal to the surface made an average angle of 60.38° with the direction of minimum principal length as most of the fractures aligned with the shortest dimension of the particles rather than the longest dimension. The effect of the fractured particles' local contact network on the orientation of the fracture surface within the particles was examined by comparing the direction of the normal vector of the fracture surfaces to the eigenvector of the weighted and unweighted fabric tensor of the particle contacts. The weighted and unweighted fabric tensor of the local contacts for each particle is defined as:

$$F_{ij} = \frac{1}{2N_c} * \sum_{c=1}^{N_c} f_i^c f_j^c \quad (3.14)$$

$$\widetilde{F}_{ij} = \frac{1}{\langle W_n \rangle} * \sum_{c=1}^{N_c} W^c f_i^c f_j^c \quad (3.15)$$

where F_{ij} is the i^{th} and j^{th} component of the fabric tensor. N_c is the number of contacts and f_i^c is the i^{th} component of the normal vector for contact c . $\widetilde{F}_{ij}$ is the i^{th} and j^{th} component of the weighted fabric tensor W^c is the weight of contact vector c (i.e., the stress in the other particle in contact c) and W_n is the average of the weight of all contact vectors. The variation of the angle between the fracture surface normal vector and eigenvector corresponding to the maximum eigenvalue of the weighted and unweighted fabric tensor of each particle are presented in Figure 3.17b & c. The normal to the fracture surface makes, an average angle of 55.47° with the direction of the loading as the fracture surface is more aligned with the eigenvector of the loading direction, and the fracture of the particle is due to the tensile stress formed perpendicular to the loading direction. This angle increases to 61.10° once the contacts are averaged using a weighted average based on the stress within the particles in contact. Figure 3.17 suggests that fracture of the particles (especially the shape of the fracture) is rather a complex phenomenon as it is not only related to the kinematics of the particles and the inter-particle properties such as CN of the particles but also the location of the contacts and shape of the particles as well.

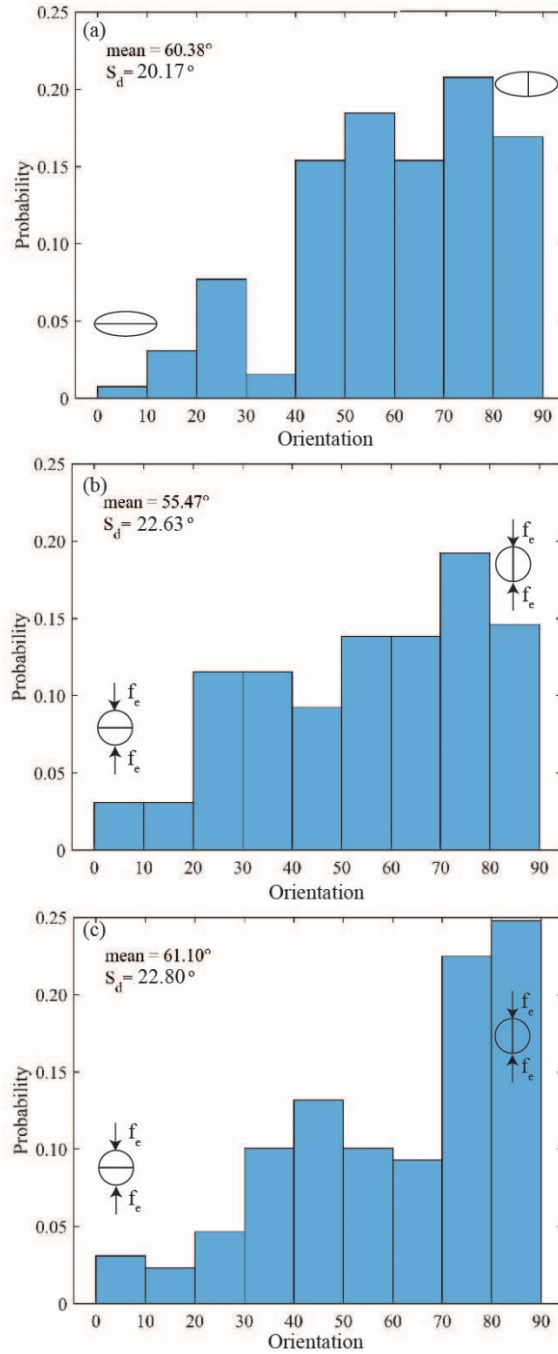


Figure 3.17. Distribution of the angle the fracture surface normal with respect to the direction of (a) the minor principal length; (b) eigenvector of the unweighted fabric (principal loading direction); (c) eigenvector of weighted fabric (the schematics show the direction of fracture at the two ends of the spectrum).

Conclusions

SMT images and 3DXRD were used to investigate the variation of particle stresses and the effect of particle-scale properties and interaction between the particles on the stresses within the particles in a 1D confined compression experiment of a specimen composed of natural silica sand particles. The paper presents a new insight into the variation of particle stresses and fracture of sand particles based on unique experimental measurements, which can provide critical 3D measurements for both theory and modeling of granular materials. A feature selecting algorithm (RReliefF) was employed to rank the importance of different particle properties on the variation of stresses within particles. Moreover, effect of each property was studied individually as well. The fracture of particles was investigated to shed the light on the effects of particle properties and the relative orientation of loading on the fracture of particles. The following conclusions are drawn from the paper:

- 1- The evolution of stresses within the particles are affected by different particle-scale properties across different length-scales. Particles that exhibited smaller translation, smaller rotation, and higher contact numbers experienced higher stresses. Moreover, particles with contacts that aligned with the direction of the loading had higher stresses as well. These effects of particle properties were more evident in particles with stresses higher than the average stresses of all particles within the whole specimen. However, the effect of these particle scale properties would relatively fade away as the stresses within particles with values smaller than the average stress showed a more uniform distribution versus the aforementioned particle-properties (i.e., particle scale properties would become less accurate as predictors for particle stress).
- 2- Similar to the variation of the stresses within particles, the fracture of the particles demonstrated dependencies on particle-scale properties. The particles that fractured on average showed smaller translation and rotation compared with the intact (unfractured) particles. However, the two averages (translation and rotation of fractured and intact particles)

converged together as the compression progressed to higher strains. On the other hand, the average contact number of the particles that fractured were similar to the average of the whole specimen during the first two load steps. Fracture particles exhibited higher average contact number compared with intact particles in the later load steps of the experiment.

- 3- The variation of the stresses within the particles that fractured demonstrated that internal stresses cannot be used as an accurate parameter to predict fracture of particles as the fractured particles exhibited a wide range of variation of internal stresses.
- 4- The local contact network of the particles (i.e., the principal direction of loading) and the shape of the particles affected the fracture mode of particles. A careful evaluation of the fractured particles showed that the normal vector of the fracture surfaces tends to align more with the direction of the major principal length of particles and the direction perpendicular to the weighted and unweighted direction of the loading.

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CHAPTER 4
CHARACTERIZING THE CONTACT NETWORK OF SHEARD SAND
PARTICLES USING COMPLEX NETWORK ANALYSIS

A version of this chapter was submitted for publication in PHYSICA A:Statistical mechanics and its application by Siavash Amirrahmat and Khalid Alshibli

My contribution to this publication was:

- 1- Analysis of the data, analyzing the 3D SMT images, characterizing the evolution of subgraph centrality of the contact networks, examining particle effects on the variation of the subgraph centrality
- 2- Most of the writing and Figures

Abstract

Characterizing the effects of the particles' interaction and the influence of the fabric of granular materials is one of the main challenges in studying the constitutive behavior of granular materials. The evolution of the fabric of granular materials and its response to applied stresses have been investigated extensively in the literature. Contact number is one of the most common metrics that is used to study the evolution of the structure of granular materials subjected to external loading. However, it is a limited metric as it only incorporates the effect of the particles that are in immediate contact with a specific particle and cannot characterize the evolution of the fabric of granular materials at meso-scale. A new metric that can incorporate the effect of particles in direct contact with a specific particle, as well as other particles within its vicinity, can characterize the evolution of the fabric of the granular materials much better. Subgraph centrality (SC) is introduced as a complex network property that describes the change in the number of closed cycles in a network and a new metric that can characterize the contact network of the particles at particle-scale and meso-scale. 3D Synchrotron micro-computed tomography images

(SMT) and SC were used to characterize the evolution of the fabric in five specimens, which were composed of two different types of silica sand particles, subjected to axisymmetric triaxial loading. The effects of the specimens' initial density, confining pressure, kinematics of the particles, and particle morphology on the evolution of the contact network of the particles was investigated. The evolution of four node structures as one of the underlying fabric structures within the specimen was investigated to illustrate how the structure of the specimens was evolving and causing the change in the SC of the particles. The variation in the average subgraph centrality of the specimens was connected to their volumetric strain to demonstrate the relationship between the change in the contact structure of the particles and the constitutive behavior of sheared sand.

Introduction

The constitutive behavior of granular materials is highly influenced by particle-to-particle interaction and fabric of the material. Fabric is defined as the arrangements of particles, particle groups, and associated void space. One of the main challenges in characterizing the constitutive behavior of granular materials is understanding the response of their fabric to external stresses and strains. The evolution of the structure of granular materials has been investigated extensively in the literature. One of the common metrics that has been used to quantify the fabric of granular materials is the contact number of the particles in an assembly of particles (Al-Raoush 2007; Amirrahmat et al. 2018; Jang and Frost 2000; Kahn 1956; Kuhn 1999; Makse et al. 2000; Rothenburg et al. 2004). The contact number of particles is an essential metric to characterize the local fabric of granular materials at different points within the material, and it is easy to quantify as well. However, it is limited

as it fails to capture the effect of particles that are surrounding a specific particle but are not in direct contact with it. It is limited to particle-scale and cannot capture the effect of surrounding particles at meso-scale. Having a new metric that can offer information about particles both at particle-scale and meso-scale can significantly enhance the ability of such a metric to capture the response of a granular material to external loading.

Network theory is the mathematical analysis of the structure of systems that are represented by networks. The networks in this context are comprised of nodes (different subjects) and links that connect them. These links can represent different types of relationships between the nodes. Complex networks have been used recently to study the evolution of the contact network of the particles and their evolution. Some studies have employed complex network properties to investigate the change in force structures within specimens as well as the packing of the particles during different external loadings. Smart and Ottino (2008) studied the evolution of fabric in 2D granular assemblies at the particle's mesoscale and global scale. They studied three different phenomena, namely pressure variance in disperse packings, the effect of contact loops on the stability of granular packings, and heat transfer in granular packings. Arevalo et al. (2009), Arevalo et al. (2010), and Arevalo et al. (2010) used molecular dynamics simulations and complex network properties such as contact number, clustering coefficient, and node distance to investigate the evolution of packing in a 2D assembly of disks subjected to an isotropic compression. Tordesillas et al. (2010) employed complex network properties to study the evolution of force chains and force cycles in an assembly of 2D spheres constrained to move along a plane subjected to a variety of compression and penetration external loads.

Moreover, complex network analysis has been employed to study the conformation within different specimens and expose the effect of these conformations on the contact network and the force structures within specimens. Walker and Tordesillas (2010) studied the topological evolution in dense 2D granular packings from a complex network perspective. They studied the properties of the contact network of the particles such as degree, clustering coefficient, and centrality (i.e., importance) measurements. The relationships between the buckling of force structures, shear bands, and nonaffine deformation of the assembly with the evolution of the aforementioned network properties were investigated as well. Tordesillas et al. (2012) examined the variation of cluster conformations in an assembly of 1568 bidisperse sphere disks subjected to cyclic shear under constant volume. Walker and Tordesillas (2012) characterized the rearrangement of particles in a granular assembly subjected to biaxial compression. They summarized the rearrangement of the particles in the evolution of the properties of the contact network of the particles as well as the evolution of the building structures of the assembly. Arevalo et al. (2013), and Arevalo et al. (2013) used complex network properties to investigate the difference in the evolution of the fabric of 2D granular packing subjected to tapping and regular compression loads. Shi et al. (2014) used statistical complex network properties to assess the percolation of nano-rods and their effect on the macroscopic conducting behavior of synthetic materials. Walker et al. (2014) studied the macroscopic response of photo-elastic disk assemblies subjected to prolonged cyclic pure shear under uniform compression. They used the clustering coefficient of the particles to expose the variation of the cyclic patterns that provide the support and stability for the force structures in the specimens. Walker et al.

(2015) explored the structural transition of fabric in an assembly of near-frictionless granular material by tracking the transition of the 3D conformations formed by the contact network of the particles subjected to uniaxial compression. They showed how different properties such as clustering coefficient of the contact network evolved with loading. They also tracked the evolution of the prevalent conformations with loading to show how these small fundamental structures change with loading and unloading. Walker et al. (2015) quantified the variation in the number and shape of the motifs and super-families (Milo et al. 2002; Xu et al. 2008) to study the topological change in the contact network of 3D triaxial experiments. The evolution of the super-families and motifs (i.e., small structural patterns formed frequently) of the contact networks were investigated to show how these fundamental structures evolve during the shearing and how they contribute to the change in stability of the force transmission structures and micro shear bands.

This paper utilizes complex network analysis and contact network of 3D triaxial experiments, which were characterized based on synchrotron micro-computed tomography (SMT) images (Druckrey et al. 2016) to investigate the evolution of the fabric of the specimens composed of two different types of sand with different morphologies, initial densities, and confining pressures. The paper adopts the subgraph centrality concept (Estrada and Rodríguez-Velázquez 2005) in complex network theory to provide another metric that can characterize the variation in fabric both at particle-scale and meso-scale. The evolution of subgraph centrality of the particles and the average subgraph centrality of the specimens during the experiments were investigated in detail to study micro and macro behavior of the fabric of the specimens with different initial density and confining pressure.

The effect of strain localization into shear bands on the contact network of the particles within the specimens was investigated through comparing the variation of the subgraph centrality of the particles versus their relative particle displacement gradient, which was defined in (Amirrahmat et al. 2018; Druckrey et al. 2018). The effect of morphology (the ratio of the major principle length to the minor principle length of the particle) on the variation of the subgraph centrality of the particles was examined as well. The variation of average subgraph centrality of the specimens was connected to the volumetric strain of the specimen to link an abstract mathematical property of the contact network to a solid mechanics property of the specimens and to show how the variation in the structure of the contact network of the particles contributes to the change in the volume of the specimen. Moreover, the evolution of the four-node structures (i.e., structures that are repeated with high frequency in the contact network of the particles) was investigated to expose the structural change in the contact network of the particles with loading.

Experiments

Two different types of sand with different morphologies (Alshibli et al. 2015) were used in the experiments. Columbia grout sand (GS40) and F35 Ottawa sand (F35) with a mean particle size (d_{50}) of 0.36 mm were used to represent different types of silica sand with varying angularity and roundness. Only the size fractions between US sieve #50 and #40 (0.297 mm to 0.429 mm) were used in this experiment to eliminate the particle size effect on the behavior of the specimens. The average micro-scale morphology indices of the particles such as sphericity index (I_{sph}), roundness index (I_R) and root mean square texture (R_q) of the particles were quantified to provide a baseline for comparison between the

morphology of the particles and are summarized in Table 4.1. The morphology indices are defined in (Alshibli et al. 2015), and they were measured based on the 3D SMT images of the particles and optical profiler. The morphology indices are defined as:

$$I_{sph} = \frac{V_p}{V_s} \quad (4.1)$$

$$I_R = \frac{A_p}{4\pi(\frac{d_s+d_I+d_L}{6})^2} \quad (4.2)$$

$$R_q = \sqrt{\frac{1}{MN} * \sum_{i=1}^M \sum_{j=1}^N Z_{ij}^2} \quad (4.3)$$

Where V_p is the volume of particle, V_s is the volume of the sphere with a diameter equal to the shortest length of the particle. d_s , d_I , and d_L are the shortest, intermediate, and longest diameter of the particles, A_p is the surface area of an individual particle. Z_{ij} is the surface height of a specific pixel in the optical interferometer image with respect to a datum (reference mean plane) while M and N are the number of pixels in the x and y directions. I_{sph} compares the shape of the particle to a sphere, and values close to 1 indicate particles with shapes closer to a sphere. I_R quantifies the presence of asperities and corners, and values close to 1 indicate particles with little to no corners. R_q measures the standard deviation of the height of the asperities of the particles. F35 particles had a mean I_{sph} of 1.872 and I_R of 0.956 while the GS40 specimens had a mean mean I_{sph} of 1.674 and I_R of 0.924. The GS40 sand particles had more spherical shapes and were more angular compared with the F35 sand particles. F35 sand particles had a higher surface texture compared with the GS40 particles (R_q of 2.084 and 1.923, respectively). In this study, the effect of particle shape on the variation of SC of the particles using the form of the particles

defined as the ratio of the major principal length of the particle to the minor principal length of the particles (a/c).

Table 4.1. Summary of the experiments

Experiment	Initial void ratio	D_r (%)	I_{sph}	I_R	R_q (μm)	σ_3 (kPa)	Resolution (μm /voxel)
F35-D-400kPa	0.586	91	1.872	0.959	2.084	400	11.18
F35-D-15kPa	0.603	83				15	11.14
F35-L-15kPa	0.663	49				15	8.16
GS40-D-400kPa	0.720	89	1.674	0.924	0.381	400	8.16
GS40-D-15kPa	0.721	89				15	11.14

A mini-triaxial apparatus described in (Druckrey and Alshibli 2014; Hasan and Alshibli 2012) was used to conduct the experiments. A stepper motor was employed to shear the specimens at a constant displacement rate of 0.2 mm /min. A load cell with a capacity of 400 N and an accuracy of 0.1 N located within the test cell was used to record the axial load during the compression of the specimens. A constant confining pressure (σ_3) was provided using air pressure. The specimens were prepared to have a diameter of about 10 mm and a height of 20 mm. Five axisymmetric triaxial experiments were conducted on loose and dense dry specimens at two different confining pressures of $\sigma_3 = 15$ and 400 kPa (Table 4.1).

3D SMT images were acquired once the confining pressure was applied to the specimens before the compression of the specimen. The images were acquired in Beamline 13MD of the advanced photon source (APS) at Argonne National Laboratory. The experiment was paused at different compression stages, and radiograph images (900 images with a rotation increment of 0.2°) were acquired. The images were reconstructed to create 3D SMT images

at different compression steps. The resolution of the images is provided in Table 4.1. The reconstruction process is discussed in more detail in (Rivers 2012; Rivers et al. 2010), and the readers are referred to them for more information. The incremental kinematic behavior and the contact network of the particles during the compression of the specimens were characterized using the SMT images, which are discussed in the next section.

Image processing, particle kinematics, and contact network

The SMT images were employed to quantify the kinematics of the particles and to characterize the contacts between particles. The kinematics of the particles were used to expose the micro shear bands in specimens, while the particles' contacts were used to quantify the contact network of the specimens. In this Section, the images processing steps are summarized, and then the process of quantifying the kinematics and the contacts are described briefly. The discussions regarding the image processing steps are kept short as they are not the main topic of this paper. However, references, where these processes are discussed in detail, are provided for further reference.

Image processing

The SMT images captured from the experiments and the Avizo Fire software was used to do the preliminary image processing steps such as anisotropic filtering, segmentation, and labeling. The details of these preliminary steps are provided in (Druckrey et al. 2016). The labeled images were then used to quantify incremental kinematics of the particle between different load steps (i.e., target axial strain) based on the change in the 3D position of the centroid and orientation of the particle at each load step. The contacts between the particles

were also identified using the labeled images using a code developed by the authors. The details of the process of characterizing the contacts and also the kinematics of the particles are provided in (Alshibli et al. 2017; Druckrey and Alshibli 2014; Druckrey et al. 2016). Amirrahmat et al. (2018) and Imseeh et al. (2018) used this code to quantify the variation of the fabric in mini-triaxial experiments, which showed the robustness of the code in characterizing the contact between the particles. The characterized contact network between the particles and kinematics of the particles were used to quantify the relative particle displacement gradient of the particles (RPTG) a concept defined by (Druckrey et al. 2018) to expose the micro shear bands and localization of shear strain the specimens. RPTG was defined as the magnitude of the relative translation vector of a particle with respect to the particles that are in contact with it.

The contacts between the particles were also used to characterize the contact network between the particles as a graph consisting of nodes and connections where the nodes represent the particles, and the connections represent the contacts between the particles. Figure 4.1 illustrates the contact network of the first 1500 particles in the F35-D-400kPa experiment prior to the loading. The first 1500 nodes out of 26748 of the graph were isolated just to provide a clear visual representation as including all 26748 nodes would have resulted in a confusing graphical representation of the data. The structural properties of these contact networks and the evolution of the network with the compression of the specimens were investigated. The network properties that were investigated in this study are discussed in detail in the next section.

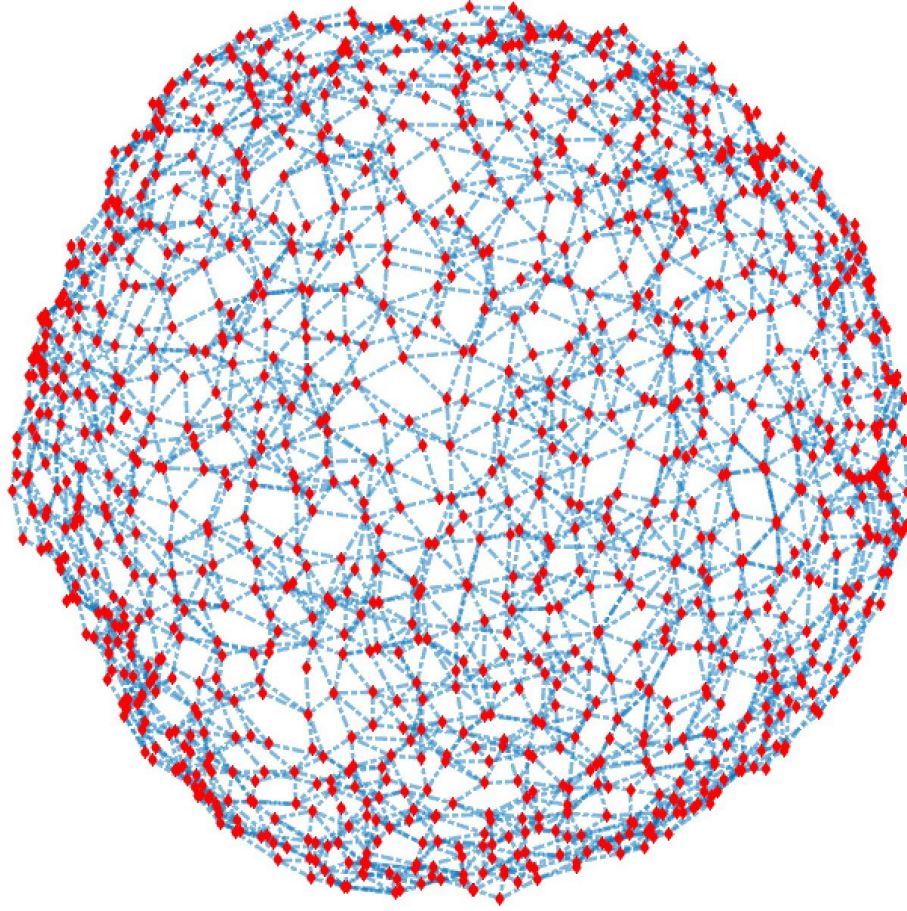


Figure 4.1. The graph representation of the contact network of 1500 particles in the F35-D-400kPa specimen prior to the loading

Contact network and network properties

The contact network described in the previous Section is an unweighted and undirected graph. One of the main ways to describe such graphs is the adjacency matrix.

$$\begin{cases} A_{ij} = 1 & \text{if nodes } i \text{ and } j \text{ are connected} \\ A_{ij} = 0 & \text{if nodes } i \text{ and } j \text{ are not connected} \end{cases} \quad (4.4)$$

where A_{ij} is the i^{th} and j^{th} component of the Adjacency matrix. The adjacency matrix can be used to quantify different structural properties of the graph, such as the degree, clustering coefficient, and subgraph centrality. The degree of a node in a graph is one of the most common and fundamental properties of a graph where it shows the number of nodes that are connected to that node. The degree of a contact network is known as the contact number of the particles. The contact number of the particles has been commonly used in the study of the evolution of the packing of the granular materials, as it is straightforward to quantify. However, it is limited only to the particles that are in direct contact with a specific particle and does not reflect the change in the structures around the particles.

The clustering coefficient is one of the complex network properties that can be used to characterize the evolution of the most basic structure (cycles of 3) with networks (Newman 2003). Clustering coefficient is a metric that quantifies the number of cycles of size 3 (i.e., triangle structures) in graph which ranges from 0 to 1. A node with a zero clustering coefficient has neighbors that are not linked to each other at all (loose packing), and a node with a one clustering coefficient has neighbors that are all linked together (dense packing). The clustering coefficient is a better metric compared with the contact number (degree) in characterizing the meso-scale structure of the particles, but it is still limited to the structures of size 3. There have been efforts in extending the notion of clustering coefficient to cycles of size four (Lind et al. 2005), but these metrics are limited in the same sense.

Another complex network property that has been used to characterize the evolution of structures in networks is the Subgraph centrality of the nodes. Subgraph centrality of a

node is a measurement of the number of cycles with all lengths that go through that specific node, which can be a great extension to the notion of node degree (contact number) and clustering coefficient that were introduced previously. Subgraph centrality is defined as:

$$SC = \sum_{i=0}^{\infty} \frac{C_i}{i!} \quad (4.5)$$

Where C_i represents the number of cycles with length i and SC represent the subgraph centrality. Subgraph centrality includes the effect of particle contacts (C_2), clustering coefficient (C_3) and cycles of higher length. The value of subgraph centrality is controlled mostly by the number of shorter cycles since the value of $i!$ in the denominator in Equation 5 increases rapidly with the increase of the length of the cycles. This value can be calculated by the diagonal entries of the powers of the adjacency matrix as the entries of the A matrix to the power of n (A_{ij}^n) indicate the number of closed paths from node i to node j . So subgraph centrality of a node can be defined based on the powers of the adjacency matrix as:

$$SC(i) = \sum_{n=0}^{\infty} \frac{A_{ii}^n}{n!} \quad (4.6)$$

Where $SC(i)$ represents the subgraph centrality of node i and A_{ii}^n represents the i^{th} diagonal entry of the A^n matrix. Subgraph centrality of individual nodes can also be calculated based on the eigenvalues and eigenvectors of the adjacency matrix as well (Estrada and Rodríguez-Velázquez 2005).

$$SC(i) = (\mu(i))^2 * e^{\lambda_i} \quad (4.7)$$

Where λ_i is the i^{th} eigenvalue of the adjacency matrix, and $\mu(i)$ is the i^{th} component of the eigenvector associated with λ_i . Figure 4.2 illustrates a sample network and Table 4.2

summarizes the number of closed walks of different lengths for each node as well as the value of their subgraph centrality. The computation time of the subgraph centrality based on Equation 7 is dependant on the size of the adjacency matrix as finding eigenvalues of a very large matrix is computationally expensive. In this study, the adjacency matrices are in the order of 25000×25000 to 40000×40000 . The subgraph centrality of the nodes is calculated based on Equation 6 and a recursive method. At each iteration, the diagonal components of different powers of the adjacency matrix are added to the subgraph centrality, and the change in the average SC of the whole graph is examined to see if meaningful changes are detected in the average subgraph centrality of the graph associated with the specimen. Figure 4.3 shows the variation of the estimate of the subgraph centrality for the F35-D-400kPa specimen with 26748 particles. The average subgraph centrality of the particles remained relatively constant after the C_{18} cycles were included in the calculations. A similar method was used to calculate the subgraph centrality of the other specimens as well.

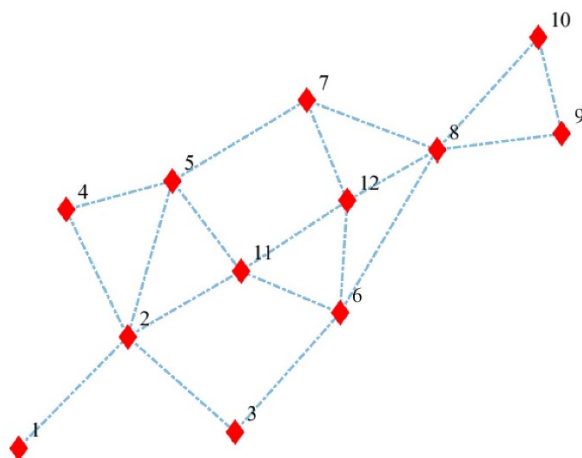


Figure 4.2. The graph representation of a sample network

Table 4.2. Summary of the number of closed walks and the SC of the nodes of a sample network

Node	C1	C2	C3	C4	C5	C6	C7	SC
1	0	1	0	5	4	37	64	1.8183
2	0	5	4	37	64	348	876	7.0438
3	0	2	0	13	12	120	240	2.9049
4	0	2	2	13	54	115	296	3.3396
5	0	4	4	30	66	363	910	6.2094
6	0	4	4	33	72	363	10.60	6.5291
7	0	3	2	23	46	241	706	4.7636
8	0	5	6	39	90	396	1186	7.8407
9	0	2	2	9	20	72	212	3.0460
10	0	2	2	9	20	72	212	3.0460
11	0	5	4	37	78	408	1170	6.8497
12	0	4	6	34	94	388	1286	7.1874

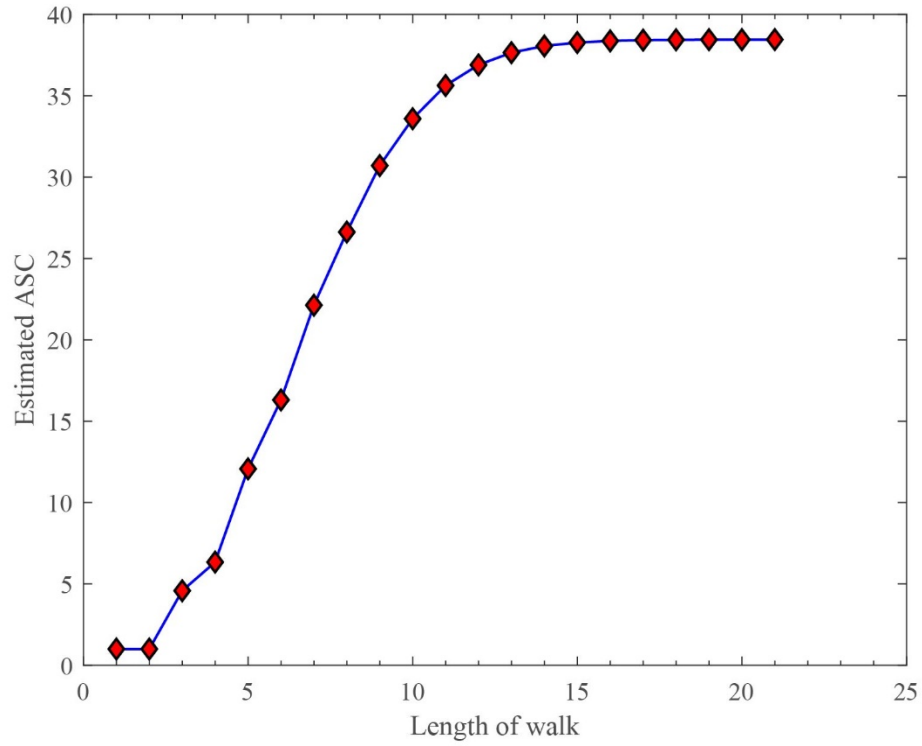


Figure 4.3. Variation of average SC of F35-D-400kPa specimen prior to the loading based on the maximum length of walk used in the calculations

Results

The axial strain (ε_1) and the compressive loads of the specimens were recorded and variation of the principal stress ratio ($PSR = \sigma_1/\sigma_3$) versus ε_1 curves are shown in Figure 4.4a. The volume change of the specimens was also calculated based on the SMT images, and the variation of the volumetric strain (ε_v) versus ε_1 of the tested specimens are depicted in Figure 4.4b. At high confining pressures ($\sigma_3 = 400 \text{ kPa}$), both specimens exhibited a gradual increase in the PSR leading into a peak state, which was followed by a softening phase. The F35-D-400kPa specimen reached a peak value of 3.9 at $\varepsilon_1 = 5.1\%$, followed

by a softening phase. The GS40-D-400kPa had a peak PSR value of 3.8 at a $\varepsilon_1 = 5.0\%$ followed by a small amount of softening. The specimens tested at low confining pressures ($\sigma_3 = 15 \text{ kPa}$) did not exhibit any peaking state in the variation of the PSR where the PSR gradually increased reaching an asymptotic value as ε_1 increased. The 400 kPa confining pressure experiments exhibited an increase in the volume after a small initial contraction. However, the 15 kPa experiments exhibited dilation throughout the experiment.

The evolution of the SC of the specimens and the effect of the specimen's initial density, confining pressure, particle kinematics, and the shape of the particles are discussed in this section. The evolution of four node substructures as one of the most basic structures forming the granular materials' contact networks is studied to illustrate how shearing affects the basic structure of the sand specimens.

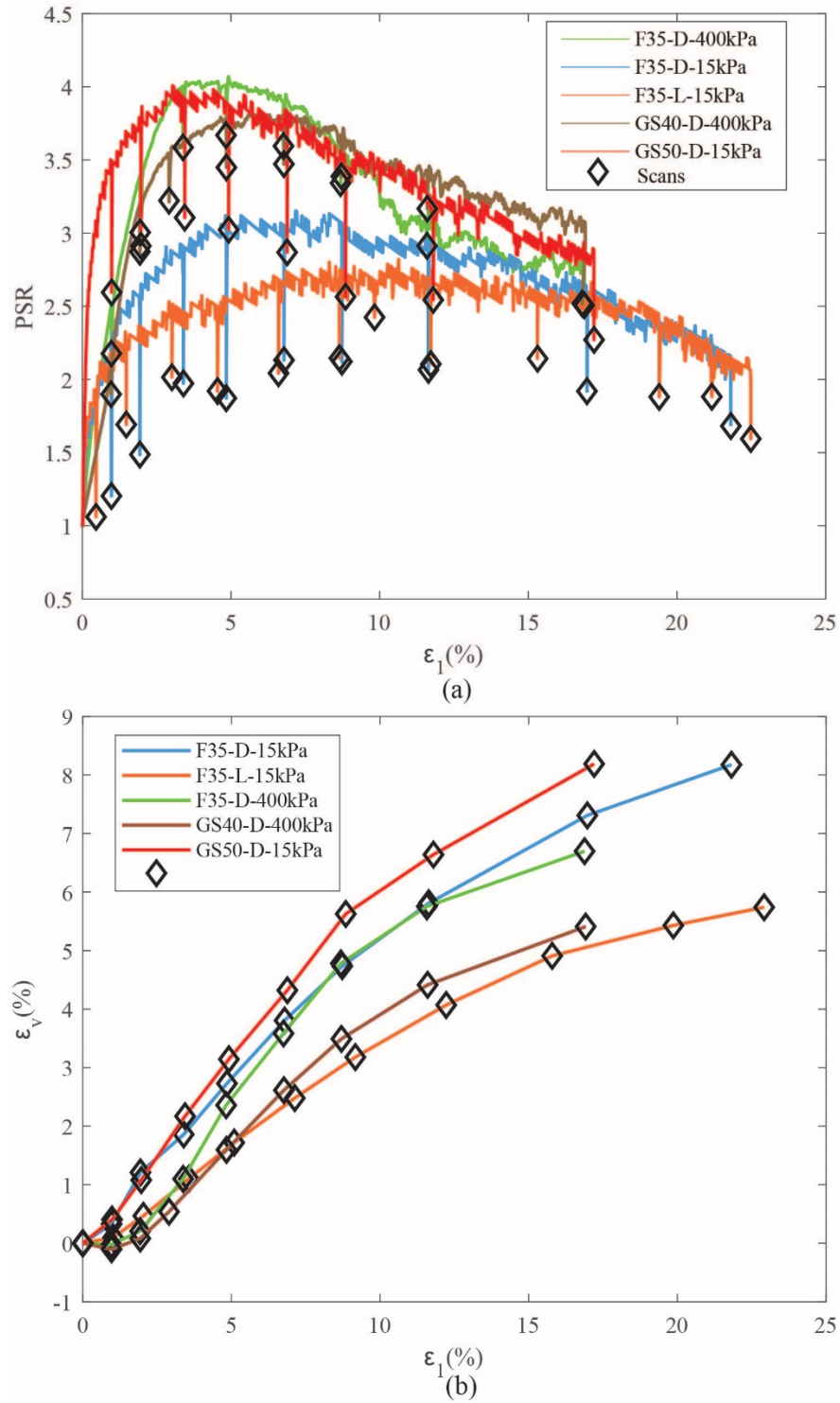


Figure 4.4. a) Principal stress ratio (PSR) versus the axial strain of the specimens (b) volumetric strain versus axial strain of the specimens

Effect of specimen density and confining pressure

Figure 4.5 illustrates the evolution of average SC of the F35 and GS40 specimens with different initial density and confining pressure. The dense specimens tested at high confining pressure (GS40-D-400kPa and F35-D-400kPa) exhibited an initial increase in the values of SC until ε_1 reached 1% when both specimens showed a slight contraction that caused the particles to form a closer packing (i.e., forming more contacts) and subsequently have higher subgraph centrality. The value of the average SC of the specimens decreased after the initial peak as the specimens began to dilate and break contacts between particles. Initially, the average SC decreased at a higher rate (higher dilation), but this rate decreased for both specimens, and the value of the average SC of the particles reached an approximately constant value towards the end of the experiments. However, the other less dense experiments tested at a low confining pressures did not exhibit any peak states as the specimens dilated throughout the experiment causing the decrease in the values of the average SC. Similar to the other specimens, the low confining pressure and the low initial density specimens are associated with a gradual decrease in the values of the SC as the specimens dilated and the particles formed looser structures. Moreover, the rate of change in the average SC of the particles decreases with loading and the average SC of the particles reaches a relatively constant value.

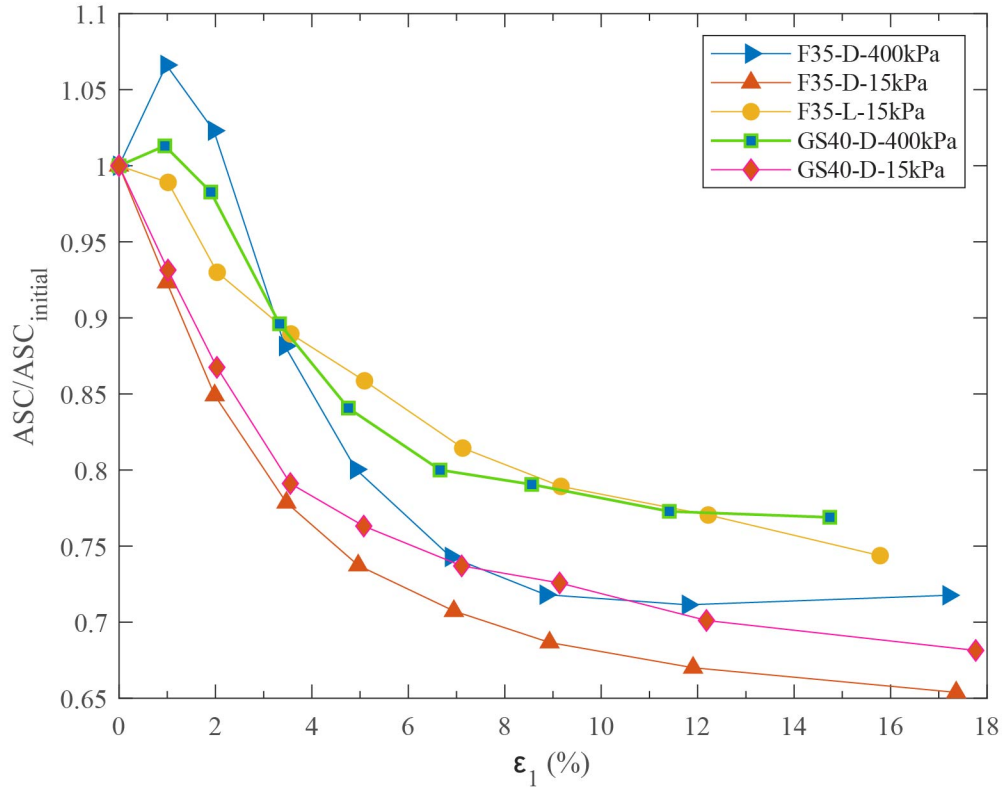


Figure 4.5. Variation of average SC of the specimens with versus axial strain

The variation of the subgraph centrality of individual particles in different experiments at different load steps was investigated to expose the effect of initial density and confining pressure of the specimens on the evolution of the SC of the particles. Figure 4.6 shows the variation of the SC of individual particles within the middle cross-section of the F35-D-400kPa at different axial strains. Particles initially have a relatively uniform SC. Then SC value increased slightly during the first load step ($\epsilon_1 = 1\%$) as the specimen contracted. However, as shearing continued, the SC of the particles in the middle of the specimen started to decrease. The particles with lower SC form a distinct diagonal pattern that goes

through the middle of the specimen. This pattern matches with the pattern of shear bands developed within the specimen. Figure 4.6j shows the shear band formed at $\epsilon_1 = 11.7\%$ characterized by (Amirrahmat et al. 2018). The SC of the particles outside of the shear band changed much less compared with the particles within the shear band as these particles moved as rigid wedges during the shearing of the specimen while the particles within the shear bands rotated during the shearing which broke contacts between the particles. The pattern of the areas where the SC decreased in the specimens matches closely with the pattern of the shear band in the specimen. Figure 4.7 shows the evolution of the SC of the individual particles in the middle cross-section of the GS40-D-400kPa experiment. Similar to the F35-D-400kPa experiment, the SC of the GS40 specimen particles decreased within the shear band and the remained relatively constant outside of the specimen.

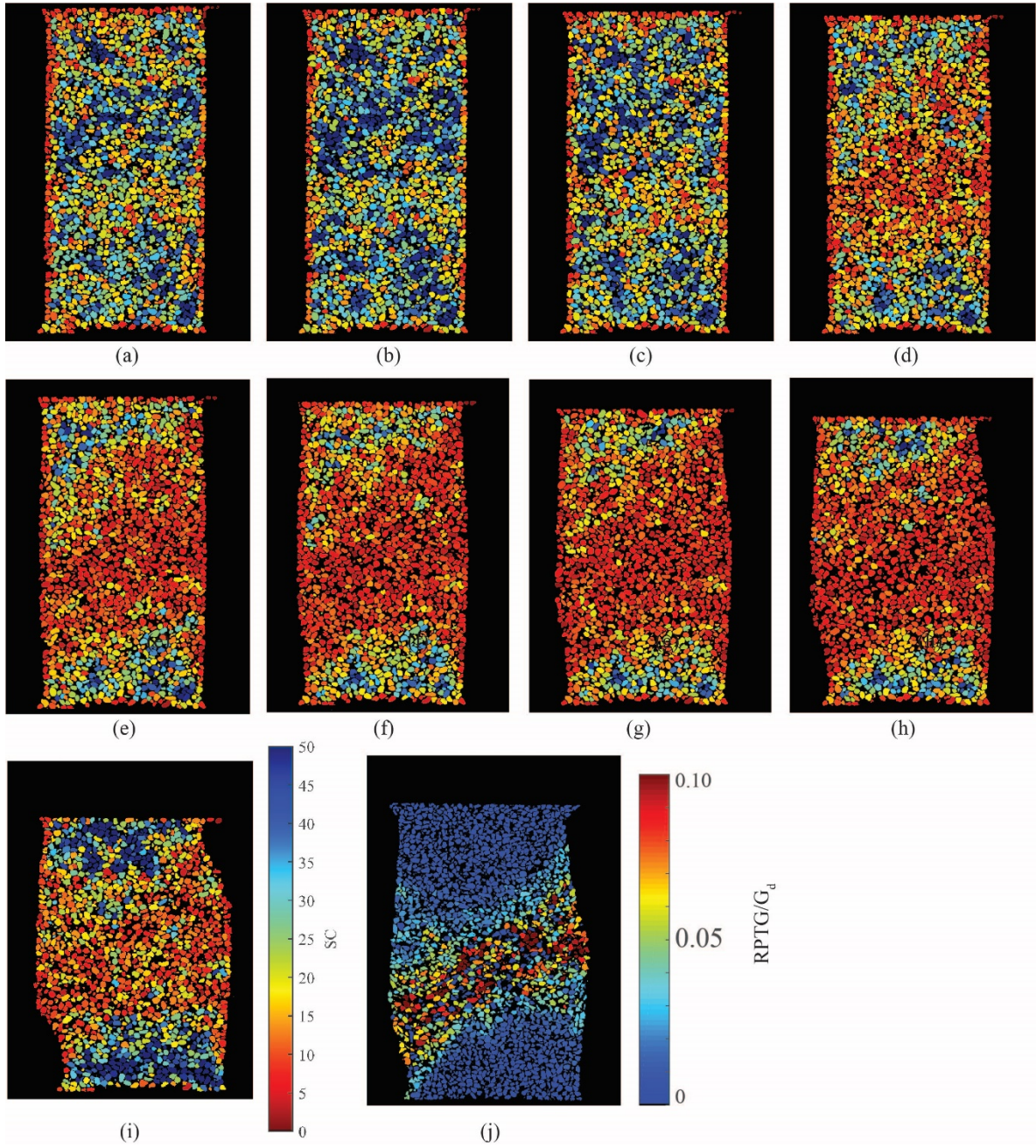


Figure 4.6. Variation of the SC of F35-D-400kPa at (a) $\epsilon_1 = 0\%$ (b) $\epsilon_1 = 0.98\%$ (c) $\epsilon_1 = 1.96\%$ (d) $\epsilon_1 = 3.44\%$ (e) $\epsilon_1 = 4.91\%$ (f) $\epsilon_1 = 6.88\%$ (g) $\epsilon_1 = 8.84\%$ (h) $\epsilon_1 = 11.79\%$ (i) $\epsilon_1 = 17.2\%$ (j) RPTG of the specimen at $\epsilon_1 = 11.79\%$ normalized with respect to incremental global displacement

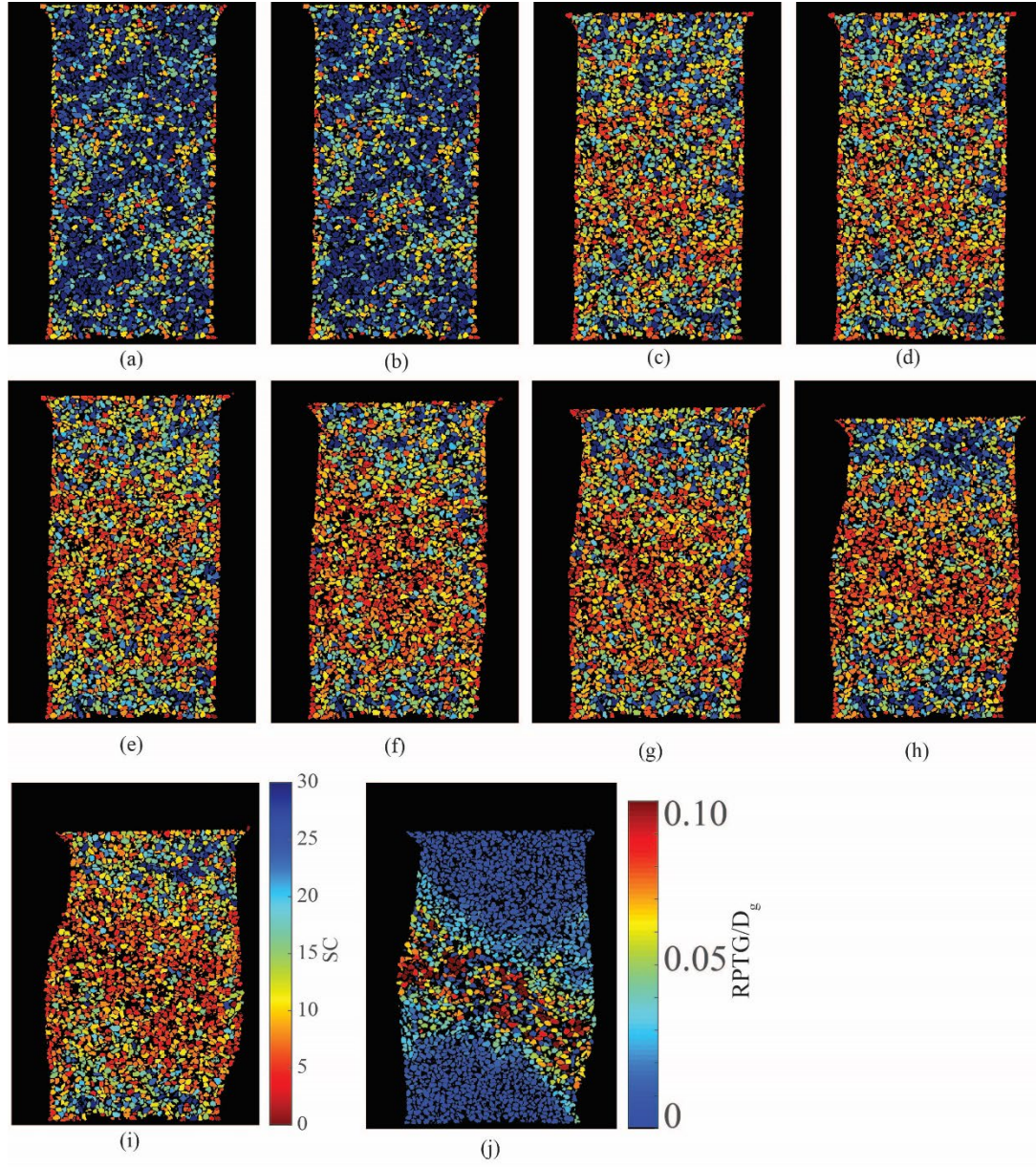


Figure 4.7. Variation of the SC of GS40-D-400kPa at (a) $\varepsilon_1 = 0\%$ (b) $\varepsilon_1 = 0.95\%$ (c) $\varepsilon_1 = 1.92\%$ (d) $\varepsilon_1 = 3.32\%$ (e) $\varepsilon_1 = 4.75\%$ (f) $\varepsilon_1 = 6.55\%$ (g) $\varepsilon_1 = 8.55\%$ (h) $\varepsilon_1 = 11.41\%$ (i) $\varepsilon_1 = 14.74\%$ (j) RPTG of the specimen at $\varepsilon_1 = 11.41\%$ normalized with respect to incremental global displacement

The variation of SC of the particles of the dense specimen sheared under low confining pressure (F35-D-15kPa) and the loose specimens (F35-L-15kPa and GS40-D-15kPa) are presented in Figures 4.8 through 4.10, respectively. It is evident that particles that exhibited a decrease in the SC formed a more dispersed pattern when compared with the F35-D-400kPa and the GS40-D-400kPa experiments. Only small conical areas at the top and the bottom of the specimens remained intact during the experiments. Figure 4.8j, Figure 4.9j, and Figure 4.10j show the shape of shear bands within these specimens as they were characterized by (Amirrahmat et al. 2018) at axial strains of 17.38%, 12.18%, and 12.22% respectively. The shearing pattern in the loose and dense specimens sheared under low confining pressure had a more dispersed pattern as multiple shear bands were formed within these specimens—the pattern formed by particles with lower SC values matched closely with the pattern of the shearing zones.

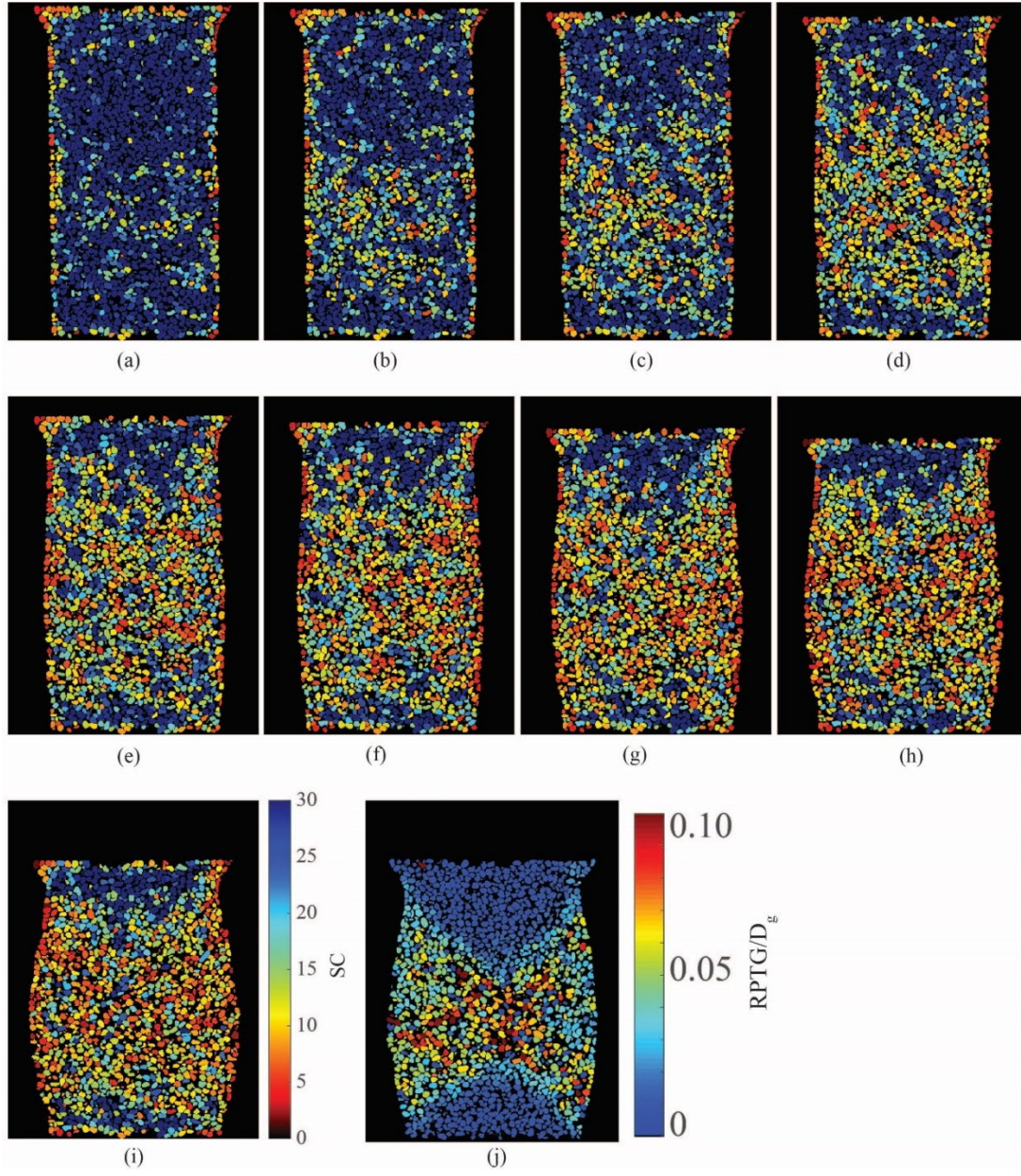


Figure 4.8. Variation of the SC of F35-D-15kPa at (a) $\varepsilon_1 = 0\%$ (b) $\varepsilon_1 = 0.99\%$ (c) $\varepsilon_1 = 1.98\%$ (d) $\varepsilon_1 = 3.47\%$ (e) $\varepsilon_1 = 4.96\%$ (f) $\varepsilon_1 = 6.94\%$ (g) $\varepsilon_1 = 8.92\%$ (h) $\varepsilon_1 = 11.90\%$ (i) $\varepsilon_1 = 17.36\%$ (j) RPTG of the specimen at $\varepsilon_1 = 17.36\%$ normalized with respect to incremental global displacement

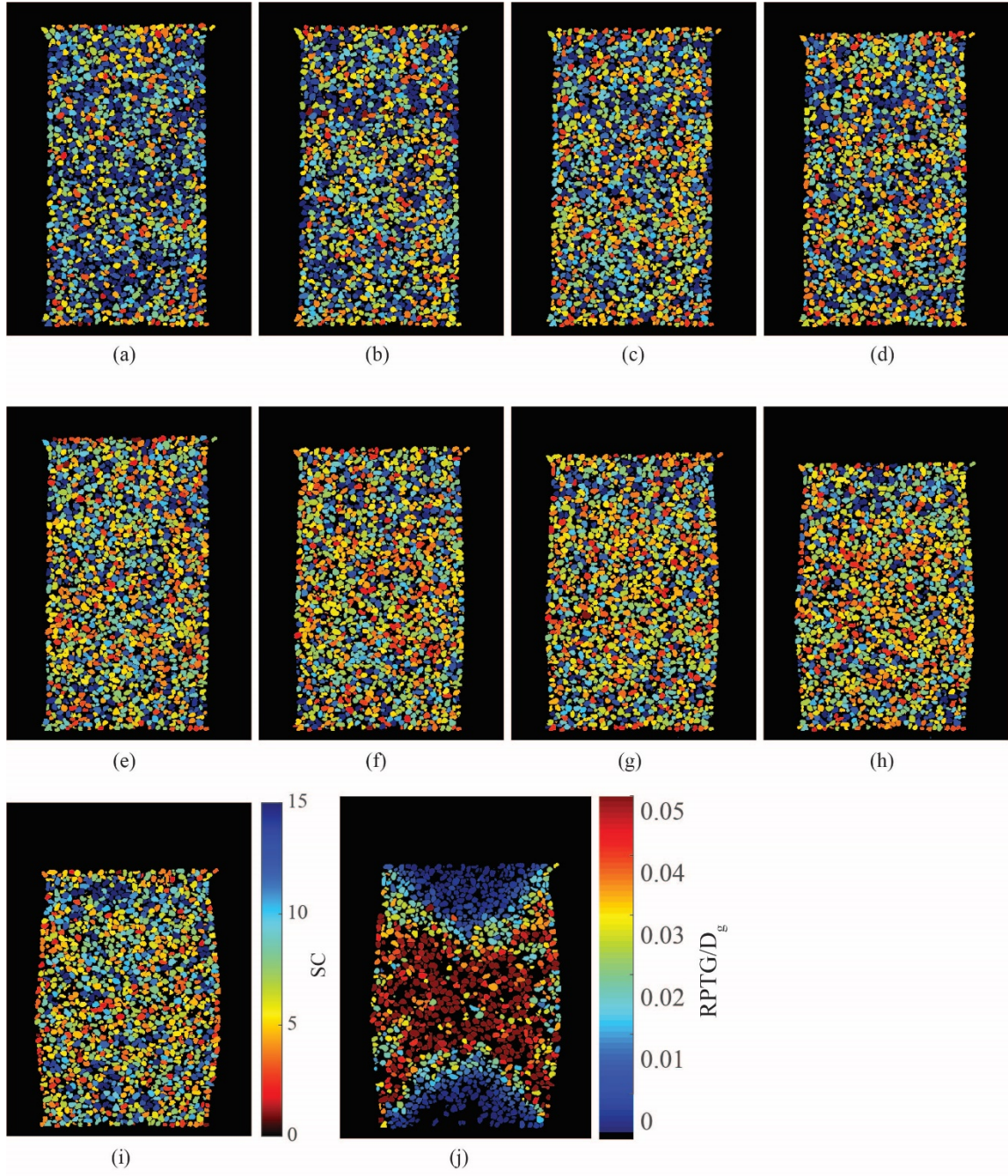


Figure 4.9. Variation of the SC of F35-L-15kPa at (a) $\varepsilon_1 = 0\%$ (b) $\varepsilon_1 = 1.01\%$ (c) $\varepsilon_1 = 2.03\%$ (d) $\varepsilon_1 = 3.56\%$ (e) $\varepsilon_1 = 5.09\%$ (f) $\varepsilon_1 = 7.12\%$ (g) $\varepsilon_1 = 9.16\%$ (h) $\varepsilon_1 = 12.22\%$ (i) $\varepsilon_1 = 15.78\%$ (j) RPTG of the specimen at $\varepsilon_1 = 12.22\%$ normalized with respect to incremental global displacement

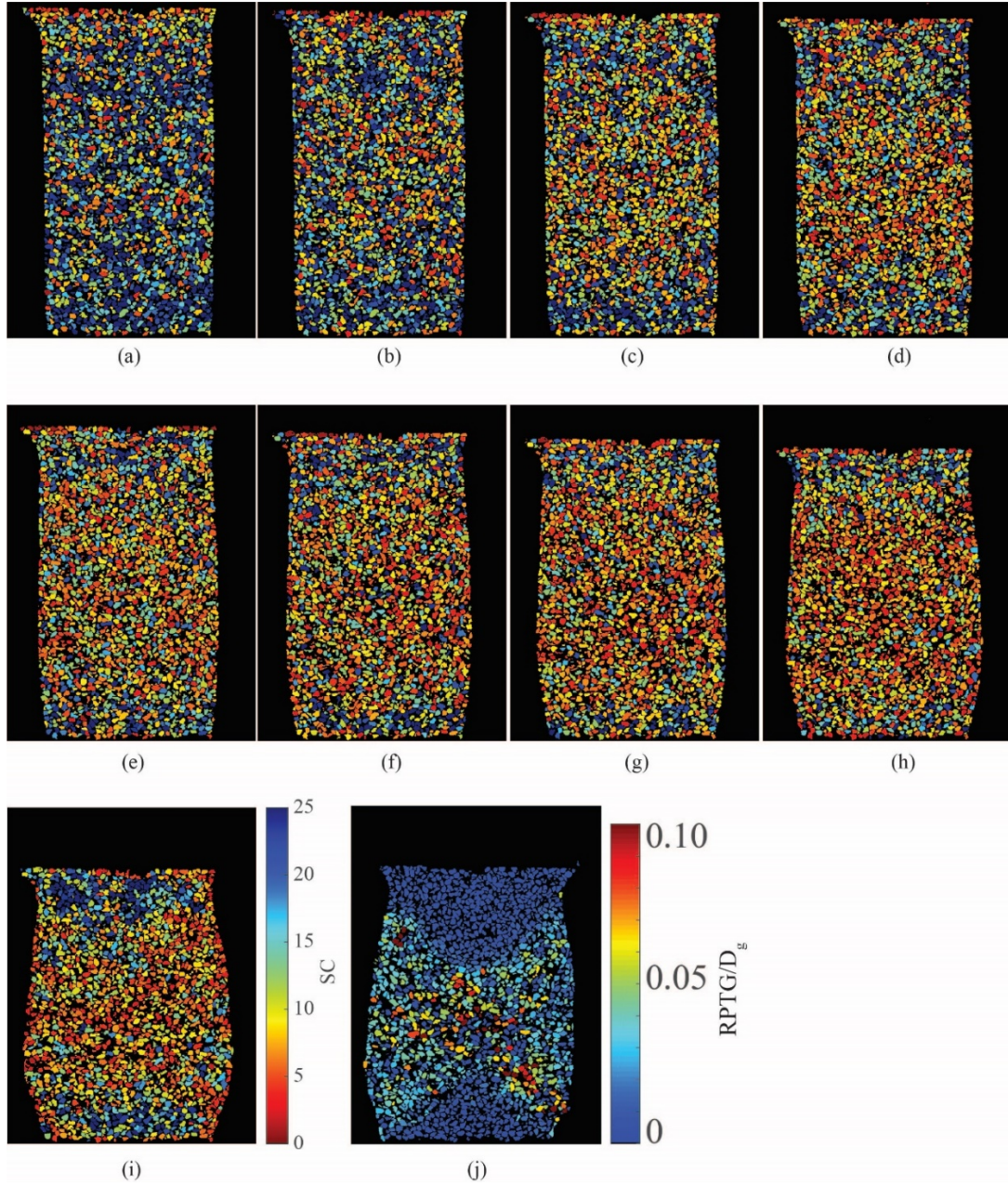


Figure 4.10. Variation of the SC of GS40-D-15kPa at (a) $\varepsilon_1 = 0\%$ (b) $\varepsilon_1 = 1.01\%$ (c) $\varepsilon_1 = 2.03\%$ (d) $\varepsilon_1 = 3.55\%$ (e) $\varepsilon_1 = 5.07\%$ (f) $\varepsilon_1 = 7.10\%$ (g) $\varepsilon_1 = 9.13\%$ (h) $\varepsilon_1 = 12.18\%$ (i) $\varepsilon_1 = 17.77\%$ (j) RPTG of the specimen at $\varepsilon_1 = 12.18\%$ normalized with respect to incremental global displacement

To take a closer look into the variation of the SC of the particles within the shear bands, the relationship between the SC and the RPTG of the particles normalized with respect to the global axial displacement ($\text{Comp}_{\text{global}}$) used to expose the micro shear bands in (Amirrahmat et al. 2018)) of the specimens was investigated. Figure 4.11 shows the variation of SC of the particles versus their normalized RPTG values for F35-D-400kPa experiment at $\varepsilon_1 = 11.74\%$. Each box plot shows the variation of the SC of the particles that exhibited an RPTG within a specific range (the labels represent the middle point of that range). The outliers within the boxplot are defined as particles outside of the lower and upper adjacents. The upper and lower adjacents are defined as:

$$U, L = q_2 \pm 1.57(q_3 - q_1)/\sqrt{n} \quad (4.8)$$

Where q_2 is the median, q_3 , q_1 are the 75th and 25th percentile, respectively, and n is the number of observations. The particles that had the lower RPTG values showed higher SC values as the contacts of the particles outside of the shearing zones remained intact, and as a result, the SC of these particles remained relatively constant. Similar patterns were detected in the other specimens regardless of their initial density and confining pressure. The ratio of particles with higher RPTG was higher in loose experiments as the shear bands formed more dispersed patterns. Only the results for the F35-D-400kPa specimen is provided here to avoid repetition.

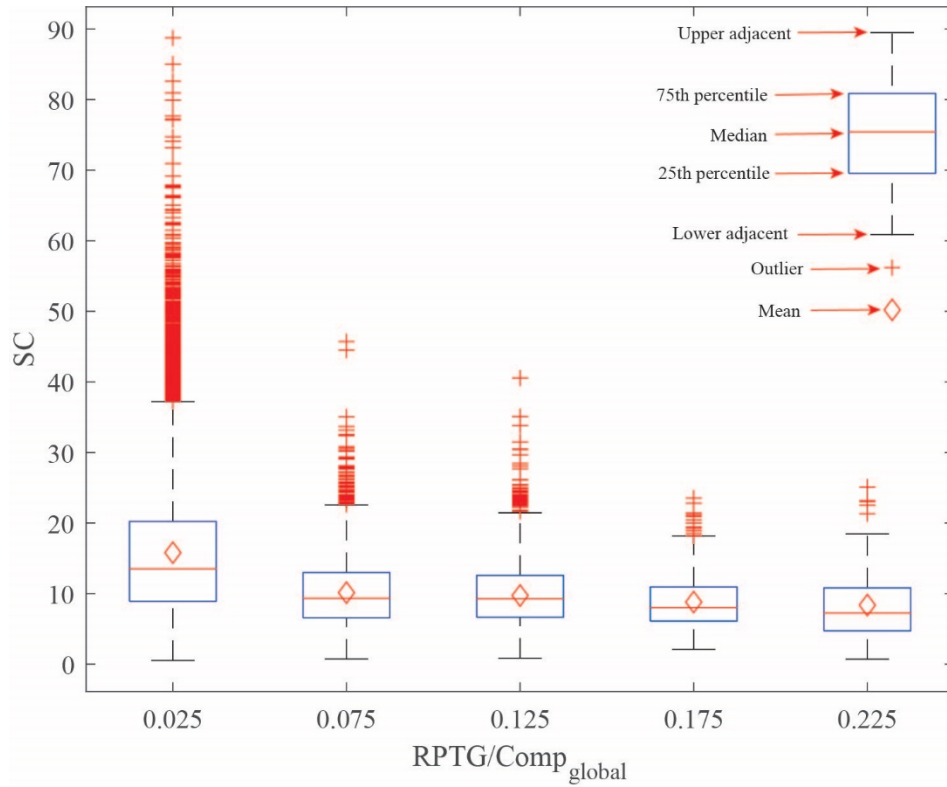


Figure 4.11. Variation of the SC of the F35-D-400kPa at axial strain of 11.79% versus the RPTG of the particles normalized with respect to global displacement

Effect of particle morphology

It has been shown that morphology of a particle affects the contacts that are formed by that particle (Amirrahmat et al. 2018; Ng 2004; Pena et al. 2007; Ting et al.). Figure 4.12 shows the variation of the SC of the particles versus their form (i.e., the ratio of the maximum and minimum principal lengths of the particles) for F35-D-400kPa specimen at $\varepsilon_1 = 11.74\%$. The box plots represent the variation of the SC of the particles within a certain range of form (the middle point represents the center of that range). All the box plot definitions are similar to Figure 4.11. The particles with higher form exhibited higher values of subgraph

centrality. Particles with higher form values have a more elongated shape and make higher contacts with particles in their surroundings and, as a result, have a higher chance of forming a higher number of closed cycles in the contact network of the particles. Moreover, the value of centrality is controlled mostly by the smaller cycles, which is directly affected by the contact number of the particles as well.

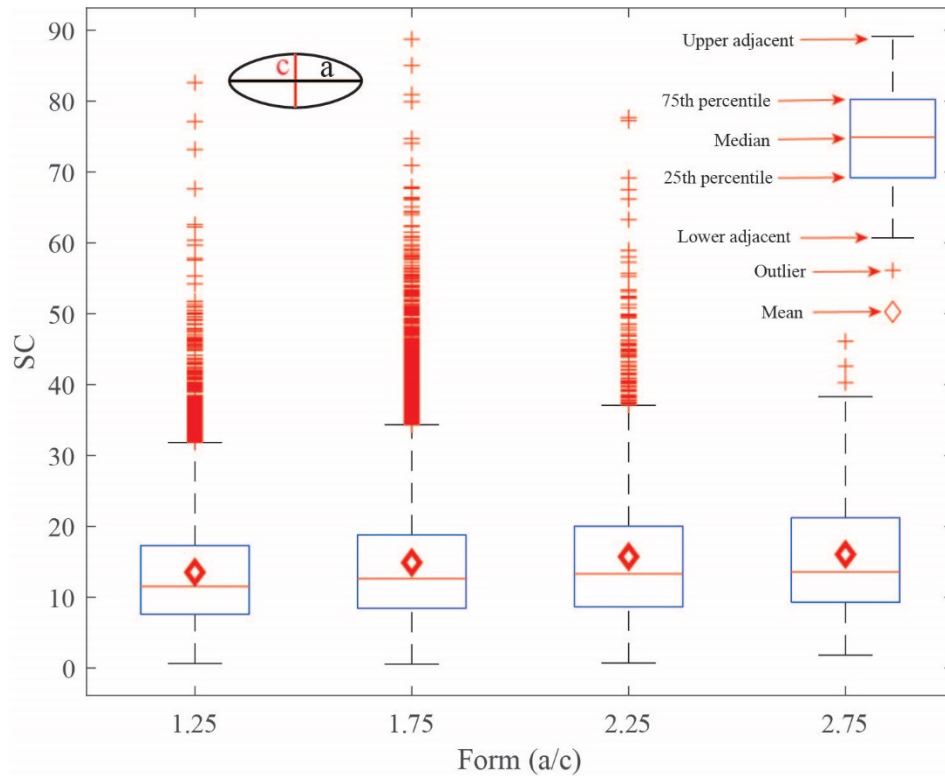


Figure 4.12. Variation of the SC of the F35-D-400kPa at axial strain 0f 11.79% versus the form of the particles

Evolution of the structures in the contact network

The evolution of the structures in the sand specimens was investigated by tracking the variation in the number of different particle structures of size four (i.e., composed of four

particles) as one of the main components forming the contact network. Tracking the evolution of these structures can expose the effect of the shearing on the contact network of the specimens and the mechanism in which it changes the SC of the particles. The variation of the structure of size four was quantified using the algorithm defined by (Itzhack et al. 2007). Figure 4.13 illustrates the variation of the 6 possible structures of size four during the shearing of the F35-D-400kPa. The structures are ordered from the least compact (ID1) to the most compact (ID6). During the shearing, the contacts between the particles were lost, and structures with high compaction (ID3 to ID6) transformed in structures with lower compaction such as ID1 and ID2. The transformation of the particles from the compact structures to the loose structures can cause a decrease in the SC of the particles during shearing.

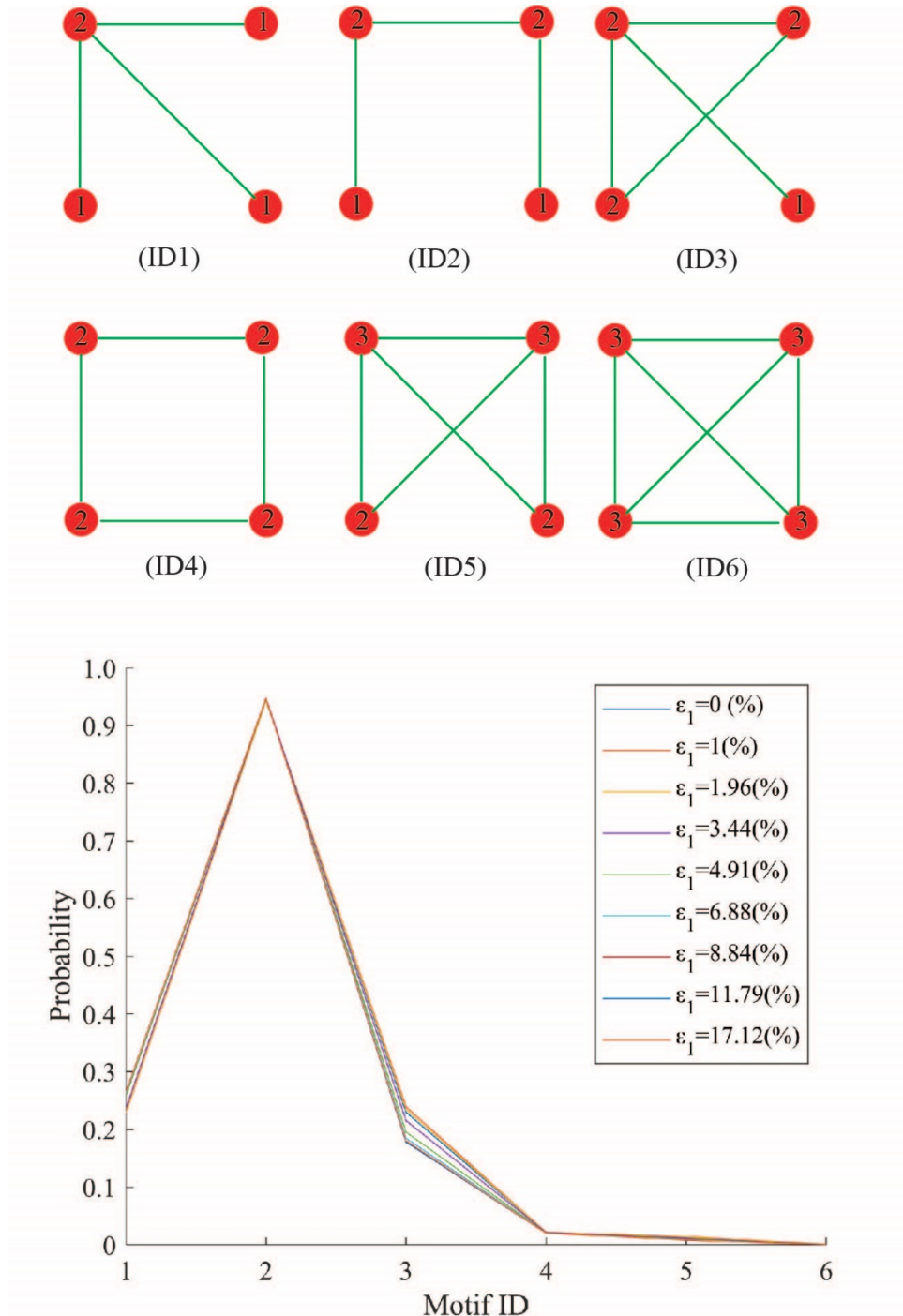


Figure 4.13. Variation of the number of four node structures during the compression of the F35-D-400kPa specimen

Relationship between average SC and volumetric strain

Even though SC of the particles is successful in characterizing the evolution of the contact network of the particles, it is necessary to connect the SC to a mechanical property of granular materials. Figure 4.14 shows the variation ASC of specimens normalized with respect to the initial ASC of the specimens versus ε_v of the specimens. A polynomial equation of the second degree fits very well with data ($R^2 = 0.91$), demonstrating the close relationship between the ASC of the particles and ε_v . It should be noted that the coefficients in this relationship are dependent on the grain size distribution of the specimens. However, this dependency does not affect the inherent relationship between SC and volumetric strain of granular materials as SC characterizes the evolution of the structure of granular materials, and the change in this structure is causing the volumetric change of the material.

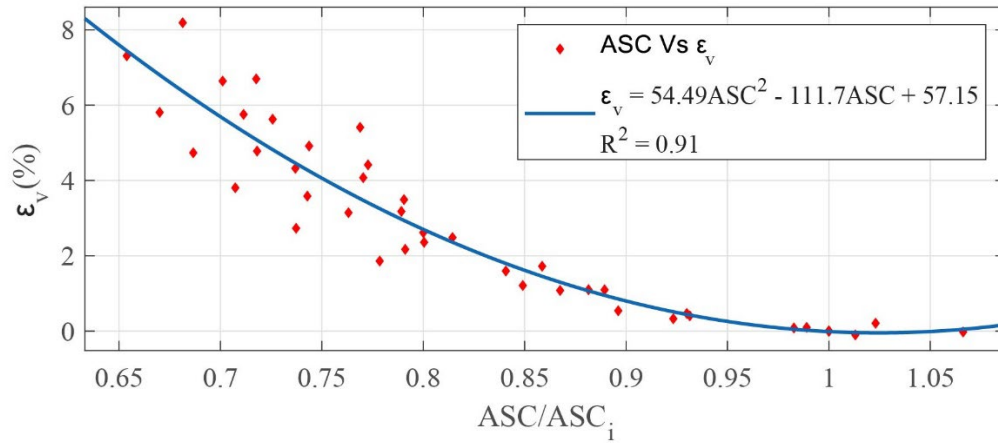


Figure 4.14. Variation of average SC of the specimen versus their volumetric strain (%)

Conclusions

Subgraph centrality (SC), a complex network property, was used in this paper as a new metric to characterize the evolution of the contact network of particles in five triaxial specimens composed of two different types of sand subjected to triaxial loading with different initial densities and confining pressures. The effect of the specimen density, confining pressure, kinematics, and morphology of the particles on the variation of the SC were investigated. In order to provide a better understanding of the mechanism in which SC changes due to the shearing, the variation of the number of the structure of the size four was investigated. Moreover, SC was correlated to the volumetric strain of the specimens to provide a better physical understanding between the change in the structure of the particles and the mechanical behavior of the material. The following is a summary of the important conclusions of this study.

- 1- SC was introduced as a new metric to characterize the evolution of the contact network of the specimen and to enhance current metrics such as contact number, clustering coefficient used to characterize the evolution of the contact network of granular materials. SC of the particles provides information about the particles that are directly in contact with a specific particle, as well as the interactions of that particle at the meso-scale. Moreover, SC can be quantified readily with the same information required for characterizing the contact number of the particles and requires no more experimental data.
- 2- SC can be used to identify the shear localization areas within the specimens as the particles within shear bands break their contacts, and their SC decreases during the experiment. The particles that exhibited high RPTG during the experiment had lower values of the SC, which

emphasizes the effect shearing mechanism in granular materials on the variation of SC of the particles.

- 3- The particles with elongated shapes had a higher value of SC as they formed more contacts compared with more rounded particles and, as a result, had more closed walks associated with them.
- 4- The variation of the four-node structures during the experiment showed how more compact structures turned into looser structures with shearing and caused the change in SC of the specimens.
- 5- The change in the contact network structure of particles affects the volume that is occupied by a specific particle and its neighbors. The change in SC of the particles is correlated with the change in the volumetric strain of the specimen to illustrate this effect. A polynomial fit of the second-order fitted the data closely.

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CHAPTER 5

CONCLUSIONS, RECOMMENDATIONS AND IMPACT

Conclusions

The following is a summary of the major findings:

- 1- The force structures had two main spatial shapes named as force chain and particle cluster. The force chains generally spanned from the top of the specimen to the bottom, transmitting the compressive forces on the boundaries of the specimen. The particle clusters alongside the other highly stressed particle outside the force structures surrounded these force chains and provided the necessary support for the chains. The variation of the number of the two structures indicated that as the loading progressed, the contact vectors between the particles aligned in the direction of the loading, the number of force chains increased to a number higher than the number of particle clusters. The number of particle clusters was higher than the force chains during the first step, where the structures were not fully formed and also during the third step, where the specimen experienced relaxation due to the bulging of the specimen.
- 2- The length of the force chains increased during the second and the fourth loading step as the contacts aligned with the loading direction, and the structures formed more concentrated patterns. On the other hand, the size of the particle clusters decreased during the second and fourth load steps. The size range of the particle clusters narrowed during the second and fourth loading step as well. The evolution of the particle structures indicates the formation of concentrated column-like structures that are responsible for the transmission of the forces from different boundaries. The particle

clusters, alongside with other highly stressed particles, provide the necessary support for the force chains.

- 3- Particles within the force structures had a higher average contact number in comparison with the particles outside the structures. The difference between the two averages increased during the second and fourth load step as the stresses developed within the specimen. The contact number of the particles affects the distribution of stresses within particles as a greater number of contacts restricts the translation and rotation of the particle and increases the likelihood of stress concentration within the particles.
- 4- The evolution of stresses within the particles is affected by different particle-scale properties across different length-scales. Particles that exhibited smaller translations, smaller rotation and higher contact numbers experienced higher stresses. Moreover, particles with contacts that aligned with the direction of the loading had higher stresses as well. These effects of particle properties were more evident in particles with stresses higher than the average stresses of all particles within the whole specimen. However, the effect of these particle scale properties would relatively fade away as the stresses within particles with values smaller than the average stress showed a more uniform distribution versus the aforementioned particle-properties (i.e., particle scale properties would become less accurate as predictors for particle stress).
- 5- Similar to the variation of the stresses within particles, the fracture of the particles demonstrated dependencies on particle-scale properties. The particles that fractured on average showed smaller translation and rotation compared with the intact (unfractured) particles. However, the two averages (translation and rotation of fractured and intact

particles) converged together as the compression progressed to higher strains. On the other hand, the average contact number of the particles that fractured were similar to the average of the whole specimen during the first two load steps. Fractured particles exhibited a higher average contact number compared with intact particles in the later load steps of the experiment.

- 6- SC was introduced as a new metric to characterize the evolution of the contact network of the specimen and to enhance current metrics such as contact number, clustering coefficient used to characterize the evolution of the contact network of granular materials. SC of the particles provides information about the particles that are directly in contact with a specific particle, as well as the interactions of that particle at the mesoscale. Moreover, SC can be quantified readily with the same information required for characterizing the contact number of the particles and requires no more experimental data.
- 7- SC can be used to identify the shear localization areas within the specimens as the particles within shear bands break their contacts, and their SC decreases during the experiment. The particles that exhibited high RPTG during the experiment had lower values of the SC, which emphasizes the effect shearing mechanism in granular materials on the variation of SC of the particles.
- 8- The change in the contact network structure of particles affects the volume that is st by a specific particle and its neighbors. The change in SC of the particles is correlated with the change in the volumetric strain of the specimen to illustrate this effect. A polynomial fit of the second-order fitted the data closely.

Limitations and recommendations for future work

The frameworks developed in this dissertation and the techniques employed in this study were designed to be as general and robust as possible. However, certain limitations exist for each process, and those limitations are discussed here. Moreover, detailed recommendations future investigations are provided as well.

The contact network of the particles was characterized using SMT images and the accuracy of the contact normal vectors quantified for each contact between two particles is dependant on the resolution of the of the SMT images as the normal vector is resultant of the principal compenent analysis of the pixels of two particles that are in contact with each other. The accuracy of the principal component analysis increases with higher number of connected pixels. The accuracy of the internal particle lattice strains and stresses are dependant on the the accuracy of the matching algorithm and the accuracy of the measured lattice strains. Different phenomena, such as fragmentations of the particles, can affect the accuracy of this process.

The framework developed in this study can be applied to different specimens with different types of loading such as triaxial loading to characterize the force transmission structures in more generalized loadings and study how different particle kinematics due to shearing can affect the development of the force structures with in the particles. A study of the effect of particle-scale properties on the variation of stresses within the particles in specimens subjected to more generalized loading such as triaxial loading where particles have more freedom to have different kinematics compared to 1D confined compression can provide a better understanding about the effect of particle kinematics on the variation of the stresses.

Moreover, a prediction model that can provide an estimate of internal particle stresses based on particle-scale properties can significantly help current numerical models. Such a model requires more data from generalized loading conditions.

Even though SC is introduced as a new metric to characterize the contact network of the particles, it should not be considered as a replacement for current metrics. SC is affected by the contact networks around an individual particle and, as a result, is not as effective as the contact number or clustering coefficient in characterizing localized effects. Moreover, quantifying SC is much more computationally expensive compared with the contact number or clustering coefficient, and it requires an in-depth knowledge of complex network analysis. In this study, the evolution of structures of size 4 (i.e., four particles connected to each other) was investigated. The methods used to count the 4 node structures are based on mathematical estimation algorithms (the size of the network is too big to count precisely). Quantifying the number of higher-order conformations has proven to a formidable mathematical task. Some studies are investigating new estimation algorithms for higher-order structures. Once these studies are done, a broader study can investigate the evolution of 3D conformations of higher orders in contact networks. Moreover, such a study can investigate how such conformations turn into each other, break into small conformations, or even evolve into bigger conformations.

Impact

Characterization of force structures is a classic physics problem that has been addressed since the 19th century when Heinrich Rudolf Hertz investigated the interaction between two similar objects that were in contact under compression. This mechanism has been

investigated using numerical models extensively since it is easier to characterize the interparticle forces and contact network of particles in numerical models. However, all these models need to be validated using experimental studies. Current experimental studies have looked into characterizing the force transmission mechanism using different methods such as computed tomography, neutron diffraction, and 3D x-ray diffraction microscopy. However, all of these studies are restricted to specimens with a limited number of particles as well as synthetic materials with similar crystal structures. In this study, a framework was developed to characterize the force structures within a specimen composed of natural sand. This framework is independent of the size of the specimen as well as the material of the particles. It can be applied to any type of granular materials composed of crystalline materials. Characterization of interparticle forces and particles' internal stresses have proven to be very difficult experimentally as they require very complicated experimental methods. This study attempted to provide a link between internal particle stresses and other particle-scale properties such as kinematics, contact number, and direction of contact, which are easier to be quantified.

In the second part of this study, complex network analysis was used to provide a new metric for characterizing the evolution of the contact network of particles. Subgraph centrality was used as a new metric to study the evolution of the contact network of the particles. This new metric provides information about the contact network of the particles both in particle-scale as well as the mesoscale. Moreover, the same information required to quantify the contact number and the clustering coefficient of the particles can be used to quantify the subgraph centrality of the particles. Subgraph centrality can replace contact

number and clustering coefficient in all studies which are trying to characterize the evolution of the contact network.

VITA

Siavash Amirrahmat was born in Tehran, Iran, in 1990. He earned a bachelor's degree in civil engineering at the University of Tehran. He traveled to the United States to pursue his graduate studies in civil engineering at New Mexico Institute of Mining and Technology on the evolution of fabric of the direct shear sand specimens. He earned an MS degree in August of 2016. He moved to Knoxville, TN, to continue his graduate studies in the field of geotechnical engineering in the Fall of 2016. He wrote his thesis about the evolution force structure within sand specimens. He earned a Ph.D. in geotechnical engineering in May of 2020.