2011

Measurement of spatial variation of density of compacted powder using synchrotron microtomography

Yi Yang
Louisiana State University and Agricultural and Mechanical College, yyang23@tigers.lsu.edu

Follow this and additional works at: https://digitalcommons.lsu.edu/gradschool_theses
Part of the Civil and Environmental Engineering Commons

Recommended Citation
https://digitalcommons.lsu.edu/gradschool_theses/2234

This Thesis is brought to you for free and open access by the Graduate School at LSU Digital Commons. It has been accepted for inclusion in LSU Master's Theses by an authorized graduate school editor of LSU Digital Commons. For more information, please contact gradetd@lsu.edu.
MEASUREMENT OF SPATIAL VARIATION OF DENSITY OF COMPACTED POWDERS USING SYNCHROTRON MICROTMOMOGRAPHY

A Thesis

Submitted to the Graduate Faculty of the Louisiana State University and Agricultural and Mechanical College in partial fulfillment of the requirements for the degree of Master of Science in Civil Engineering in

The Department of Civil and Environmental Engineering

By

Yi Yang
B.S., Wuhan University, Wuhan, China 2009
May, 2011
This thesis is dedicated

To my father Hongyu Yang
To my mother Yapin Yu
And
To my family and friends
ACKNOWLEDGEMENTS

I would like to thank Louisiana State University and National Science Foundation for funding this research. I would also like to thank Professor Khalid Alshibli for guiding me and sharing his wealthy experience and knowledge to further my career.

Special thanks go to my Master thesis committee members: Dr. Clinton Willson and Dr. Guoping Zhang for the valuable discussion and guidance. Very special thanks to Dr. Karsten Thompson and Pradeep Bhattad of Louisiana State University for giving me an access to their computer program. Special thanks to Dr. Alsidqi Hasan for his previous lab work, advices, useful information given during the research.

Thanks also to fellow students and good friends including Jian He, Xuan Kong, and Mehmet Burak Cil, Md Haque for their help in the pursuit of my Master’s Degree.

Last but not least, I would like to express my gratitude to my parents and my family for their support and love for me to further my career.
# TABLE OF CONTENTS

ACKNOWLEDGEMENTS ....................................................................................... iii

LIST OF TABLES ................................................................................................ vi

LIST OF FIGURES ............................................................................................... vii

ABSTRACT ........................................................................................................... ix

CHAPTER 1: INTRODUCTION ............................................................................. 1
  1.1 Background .................................................................................................. 1
  1.2 Problem Statement .................................................................................... 2
  1.3 Thesis Objectives ....................................................................................... 3
  1.4 Thesis Outline ........................................................................................... 4

CHAPTER 2: LITERATURE REVIEW ................................................................. 5
  2.1 X-ray Computed Tomography .................................................................... 5
  2.2 Synchrotron Tomography Scanning ............................................................ 6
  2.3 Density Calibration .................................................................................... 8
  2.4 Image Segmentation .................................................................................. 8
  2.5 Representative Elementary Volume .......................................................... 10
  2.6 Density Measurement .............................................................................. 11

CHAPTER 3: MEASUREMENT OF DENSITY VARIATION ................................ 14
  3.1 Introduction ................................................................................................ 14
  3.2 Experiment Preparation ............................................................................ 14

CHAPTER 4: SCANS ANALYSIS AND RESULTS .......................................... 27
  4.1 Introduction ................................................................................................ 27
  4.2 Compaction Configuration I ..................................................................... 27
    4.2.1 Density Map ...................................................................................... 27
    4.2.2 Row Density ..................................................................................... 34
    4.2.3 Column Density ............................................................................... 37
    4.2.4 Compaction Stress Versus Strain Curves .......................................... 39
    4.2.5 Density Histogram ........................................................................... 41
  4.3 Compaction Configuration II .................................................................... 43
    4.3.1 Density Map ...................................................................................... 43
    4.3.2 Row Density ..................................................................................... 48
    4.3.3 Column Density ............................................................................... 50
    4.3.4 Compaction Stress Versus Strain Curves .......................................... 51
    4.3.5 Density Histogram ........................................................................... 52

CHAPTER 5: CONCLUSIONS AND RECOMMENDATIONS ............................... 55
  5.1 Conclusions .............................................................................................. 55
  5.2 Recommendations .................................................................................... 56
LIST OF TABLES

3.1 Specification of the aluminum powder.................................................................15
3.2 Summary of scans..................................................................................................22
4.1 Density map and respect density value for axial strain of 50%..............................32
LIST OF FIGURES

2.1 Schematics of CT system.................................................................6
2.2 Photo of SMT setup.................................................................7
2.3 (a) Example of CT image before the segmentation process...............9
2.3 (b) Example of CT image after the segmentation process ...........10
3.1 SEM Image of Ampalloy AMB 2712 Aluminum alloy powder ...........14
3.2 Schematic of Configuration of the two dies used to compact the powder ....16
3.3 Details of the die .................................................................16
3.4 Details of steel top punch ......................................................17
3.5 Details of bottom punch #1 ...................................................17
3.6 Details of steel bottom punch #1 ............................................18
3.7 Details of steel bottom punch #2 ............................................18
3.8 Details of steel spacer #1 .....................................................19
3.9 Details of steel spacer #2 .....................................................19
3.10 Photo of compaction experiment at Argonne National Laboratory ....20
3.11 Flowchart of analysis of CT image ........................................23
3.12 Fraction theoretical density against side length for ten REV locations ...25
4.1 Density Map at 0% axial strain along x- and y-axes.......................28
4.2 Density Map at 5% axial strain along x- and y-axes.......................28
4.3 Density Map at 10% axial strain along x- and y-axes .....................29
4.4 Density Map at 15% axial strain along x- and y-axes .....................29
4.5 Density Map at 20% axial strain along x- and y-axes .....................29
4.6 Density Map at 25% axial strain along x- and y-axes .....................30
4.7 Density Map at 30% axial strain along x- and y-axes .....................30
4.8 Density Map at 35% axial strain along x- and y-axes .....................30
4.9 Density Map at 40% axial strain along x- and y-axes ........................................31
4.10 Density Map at 45% axial strain along x- and y-axes .........................................31
4.11 Density Map at 50% axial strain along x- and y-axes .........................................31
4.12 Compaction response curve for Compaction configuration I ...............................33
4.13 Row statistics of average density taken from compaction configuration I ............35
4.14 Column statistics of average density taken from compaction configuration I .........37
4.15 Stress and strain relationship of compaction configuration I .................................40
4.16 Density histogram of compaction configuration I .................................................41
4.17 Density Map at 0% axial strain along x- and y-axes .............................................44
4.18 Density Map at 3% axial strain along x- and y-axes .............................................44
4.19 Density Map at 6% axial strain along x- and y-axes .............................................45
4.20 Density Map at 9% axial strain along x- and y-axes .............................................45
4.21 Density Map at 12% axial strain along x- and y-axes .........................................46
4.22 Compaction response curve for Compaction configuration II ...............................47
4.23 Row statistics of average density taken from compaction configuration II ...........49
4.24 Column statistics of average density taken from compaction configuration II .......51
4.25 Stress and strain relationship of compaction configuration II ..............................52
4.26 Density histogram of compaction configuration II .................................................53
ABSTRACT

This research considers density change in powder metallurgy when compacting a powder blend in a die. In order to provide insights into particle behavior during compaction, atomized aluminum powders with an average particle size of 100 µm were used in two different compaction configurations. The compaction dies are cylindrical in shape: compaction configuration I displays an upper punch, a die, a lower punch, and a spacer. Compaction configuration II displays an upper punch, two dies, a lower punch, and a spacer with different geometry. The Synchrotron Microtomography scans were conducted on both compaction configurations. For compaction configuration I, the scan were acquired at axial strains of 0%, 5%, 10%, 15%, 20%, 25%, 30%, 35%, 40%, 45% and 50%. The scans for compaction configuration II were acquired at axial strains of 0%, 3%, 6%, 9% and 12%.

After scanning these compacted powders, the images were analyzed by means of computer codes that were written using Interactive Data Language (IDL) program. Programs of density map and image segmentation were used to analyze the image. Then, the spatial density maps and histograms of density variation were developed. Based on this data, the study generated curves of row statistical density, column statistical density, and compaction response in order to understand density change in compacted powder.

This study revealed that Synchrotron Microtomography can be successfully applied to examine density variation and track microscopic deformations of aluminum powders under compaction. The density distribution within the compaction die was controlled by several factors, such as constitutive behavior of powder, friction interaction between powder and die wall, geometry of compaction die, and sample filling condition.
Springback represents an important factor for influencing density distribution, especially during the scanning process. Moreover, the effect of Springback cannot be eliminated and can only be decreased in powder compaction. The aluminum compacts exhibit considerable side-to-side asymmetry which agrees with previous research findings. Finally, the generation of void gap between layers indicates the inefficiency in transmitting forces during compaction.
CHAPTER 1

INTRODUCTION

1.1 Background

Powder is a dry, bulk solid composed of a large number of very fine particles that may flow freely when shaken or tilted. Examples of powders include metallic powders, cosmetic powders, Lunar, and Martian regolith, as well as many pharmaceutical products. Due to their importance to industry and the medicine and earth sciences, many researchers have studied powders in great detail.

Powder metallurgy (PM) is a highly evolved method of manufacturing reliable net shaped components by blending elemental or pre-alloyed powders together, compacting this blend in a die, and sintering or heating the pressed part in a controlled-atmosphere furnace to bond the particles metallurgically. Some manufacturing process using PM involve four major steps: powder and lubricant mixing, compacting powders into appropriate shapes in closed dies to produce green compacts, sintering the compacts at elevated temperature and finally, post-sintering secondary operation (Chtourous et al. 2002; Mori et al. 1999). The PM industry has had relatively effective modeling applied to the die filling and compaction process of powder which could provide information on the macroscopic behavior of the powder assembly such as powder movement, density distribution and the shape of the compact during and after compaction. Thus the powder medium is considered as a continuum that undergoes large elastic-plastic deformation. Details on cold compaction process are referred to Ariffin and Ihsan (1995), where the numerical modeling of the compaction, relaxation, ejection and emergence phases have been developed, and validated by experiments.
An increased demand for light-weight components, primarily driven by the need to reduce energy consumption in a variety of societal and structural components, leads to the use of aluminum. Aluminum PM offers components with exceptional mechanical and fatigue properties, low density, corrosion resistance, high thermal and electrical conductivity, excellent machinability, a good response to a variety of finishing processes, and are competitive on a basis of cost-per-unit volume. In addition, aluminum PM parts may be further processed to eliminate porosity and improve bonding yielding properties to compare favorably to those of conventional wrought aluminum products.

1.2 Problem Statement

Particle behavior defines the porous scale and macroscopic flow deformation response of dense, dry, particulate material. PM industrial processes may benefit from an improved understanding of particle behavior. Powder compaction, also known as powder pressing, is the process of compacting metal powder in a die through the application of high pressures. Typically, the tools are held in a vertical orientation, with the punch tool forming the bottom of the cavity. The powder is then compacted into a shape and ejected from the die cavity. Based on loading conditions applied to the powder in a die, an extensive review of materials under compression is needed to image and track the particle translation and rotation that will most likely occur during the powder compaction process.

Many researchers acquired a density measurement using x-ray computed tomography (CT). CT is an imaging method, employing tomography created by computer processing. Digital geometry processing is used to generate a three-dimensional (3-D) image of the inside of an object from a large series of two-dimensional x-ray images, taken around a single axis of rotation.
An x-ray CT has emerged as a powerful, non-destructive, 3-D imaging technique to study the internal fabric of particulate materials. The technique consists of measuring the attenuation of an x-ray beam for a number of different paths through the tested object, and mathematically extracting the density of each point from a plane of the object. The 2-D density map constitutes a CT slice. By creating slices, a 3-D volumetric image of the object may be constructed. This technology can be used to study particle behavior in metallic powders.

1.3 Thesis Objectives

The main objective of this study is to image the compaction process of the particles in the powder compaction to better understand the powder behavior for potential application in the powder metallurgy. Specific aluminum powders were compacted, using two different compaction configurations. The scanning process involves investigations at macro and micro scales. The scanning images were analyzed by using computer codes written by an IDL program. The thesis provides insight with a better understanding of the deformation and compaction of the aluminum powders. This experimental work used state-of-the-art technique to compare results with the latest findings in order to establish a better and integrated approach regarding particle behavior in powder compaction. The thesis has the following objectives:

- To assess the potential of CT system to investigate the particle behavior in the aluminum powder compaction;
- Develop an integrated approach to investigate the particle behavior and quantify the CT scan images.
- Evaluate the factors that contribute to the spatial variation of the density maps of compacted powders.
• Identify and quantify problems associated with powder compaction such as the
  generation process of void gap and side to side asymmetry of compacted powders.

1.4 Thesis Outline

The thesis begins with an introduction to powder-relative industries and research
technology development. A literature review then presents an overview in the research
development of CT on powder compaction and image analysis technology. A description of how
to conduct the measurement of the density variation of compacted powder using Synchrotron
Microtomography is presented in Chapter 3. The descriptions of the apparatus for testing and
specimen preparation are presented in Chapter 3. A summary of the geometry of compaction die
and the analysis flowchart is also presented in Chapter 3.

Chapter 4 discusses the results of experimental work, which include the results of two
compaction experiments using two die configurations. The results include density maps, row
density and column density plots, density distribution histograms, compaction response diagrams.
Chapter 5 briefly presents the conclusions based on what discussed in Chapter 4, and the chapter
also provides recommendations for future research.
2.1 X-Ray Computed Tomography

X-ray CT is an imaging method employing tomography, created by computer processing. Digital geometry processing generates a 3-D image of the inside of an object from a large series of 2-D x-ray images, which are taken from a single axis of rotation. X-ray slice data are generated, using an x-ray source that emits an x-ray which passes through a rotating object, with x-ray detectors are positioned on the opposite side (Figure 2.1). Many data scans are progressively taken as the object is rotated at certain angular increments. These are combined by mathematical procedures known as topographic reconstruction. The data are arranged in a matrix of memory, and each data point convolves with its neighbors, by means of a seed algorithm, using Fast Fourier Transform techniques. This dramatically increases the resolution of each voxel (volume pixel). Essentially, a process known as back projection then reverses the acquisition geometry and stores the result in another memory array. This data may then be displayed, photographed, or used as input for further processing, such as in multi-planar reconstruction.

The CT system acquires the 3-D image of a scanned object by projecting the specimen onto a detector by creating a cross-sectional slice. Then the specimen is rotated to acquire another projection. The whole specimen usually consists of more than one slice, which will be collected by a computer. The whole specimen volume may be created by compiling the entire slices stack together to form a 3-D image.
2.2 Synchrotron Microtomography Scanning

In order to acquire higher resolution images, a synchrotron x-ray was introduced into CT, because of its higher brightness and intensity. A synchrotron x-ray is state-of-the-art technology that can be used for imaging, which yields excellent resolutions. The synchrotron accelerator generates electromagnetic radiation ranges from infrared to hard x-ray. As a synchrotron facility, electrons are usually accelerated by a synchrotron, and then injected into a storage ring, in which they circulate, producing synchrotron radiation, but without gaining further energy. The radiation is projected at a tangent to the electron storage ring to be captured by beamlines. These beamlines may originate either at bending magnets, which mark the corners of the storage ring, or by insertion devices, which are located in the straight sections of the storage ring.

Hasan (2009) wrote about these CT scans, acquired using a Sector 13-BMD synchrotron microtomography beamline at the Advanced Photon Source (APS) of Argonne National Laboratory (ANL), Illinois, USA (Figure 2.2). According to Hasan (2009), “The synchrotron facility produces intense, monochromatic, continuous, highly collimated beams of hard x-ray
with energy ranges from 10 eV to nearly 100 keV. Hence, it is capable of yielding 3D images with a higher resolution. Throughout scanning, x-ray beam strikes and penetrates the scanning object. Some of the x-ray energy is attenuated by the specimen and some x-ray energy is transmitted through the specimen. The transmitted x-ray beam is converted to a visible light by a scintillator and captured by an optical Charged Coupled Device (CCD) camera system. The light captured by the camera is basically an image projection of the specimen at one angle. In order to reconstruct a complete scan, several image projections of the sample were taken at different angles from 0° to 360°.”
2.3 Density Calibration

Density calibration is usually performed in order to correct the difference in sensitivity that exists between different materials. The CT system can detect the composition of material inside of the scanned object in order to determine the density of the material when density calibration is sought. Generally, Density calibration can be performed according to the ASTM guidelines (ASTM, 2003). For example, densities of known materials such as Aluminum, Graphite and Acrylic can be calculated using the CT numbers which has a linear relation between their individual attenuation coefficients, retrieved from national Institute of Standards and Technology (NIST) (Hubbell and Seltzer, 1997). Therefore, for other materials that scanned in CT system, CT and linear attenuation could be known through interpolation or extrapolation of density.

2.4 Image Segmentation

In computer vision, segmentation refers to the process of partitioning a digital image into multiple segments (sets of pixels, also known as superpixels). The goal of segmentation is to simplify and/or change the representation of an image into something that is not only more meaningful, but also is easier to analyze. Image segmentation is typically used to locate objects and boundaries (lines, curves, etc.) in images.

More precisely, image segmentation is the process of assigning a label to every pixel in an image, such that pixels with the same label share certain visual characteristics. The result of image segmentation is a set of segments that collectively cover the entire image, or a set of contours extracted from the image (see edge detection). Each of the pixels in a region is similar
with respect to some characteristic or computed property, such as color, intensity, or texture. Adjacent regions are significantly different with respect to the same characteristic(s).

Reconstructed 3D images from the CT system contain noise, which may cause difficulties in segmenting/separating the two phases (i.e., particles and void space). Using a certain threshold value in segmenting the image might produce misclassification errors, which are common to the two univariate populations (Oh and Lindquist, 1999). Bhattad et al. (2010) wrote a Fortran program to implement the Indicator Kriging method. In the program, voxels (cubic pixels) were assigned either 1 or 0 value to particles and voids, respectively. However, the images still contained residual noises in forms of ‘islands’ (voxels belong to particles trapped within void voxels) and ‘holes’ (noise voxels belong to voids trapped within particles). The islands and holes are usually isolated (stay unconnected) from their groups and are relatively very small in size. IDL codes were developed to remove the noise inside the image. After removing islands and holes, the images were visually compared with the raw image. Several trials were performed to obtain good results.

Figure 2.3 (a) Example of CT image before the segmentation process
2.5 Representative Elementary Volume

Representative Elementary Volume (REV) consists of the physical dimensions in the CT image representing a physical property (e.g., density). In order to calculate the volume properties in the CT images, a representative volume must be determined. One approach to determine the volume is by calculating the REV as the minimum volume size in the CT image that represents a physical property (e.g., density). The REV must show that the property calculated above the minimum volume is nearly constant.

The characterization of the REV of a continuum was studied in recent imaging technique advances such as an x-ray computed tomography. Bear (1972) and Dullien (1979) focused on the characterization of REV size. The difficulties associated with the measurement and identification of the REV size may be found in real media. Some measurements relating to the REV of glass beads (Culligan et al. 2004) and of Ooid sand (Al-Raoush and Willson 2005) were found recently, by using a cubical elementary volume. A numerical simulation of particle assemblies was also made to determine the REV for relevant properties (Stroeven et al. 2004; Ostoja-
Razavi, et al. (2007) presented a systematic technique to quantify the characteristics of REV in granular materials. The technique uses x-ray CT imaging techniques to examine the presence of REV. A 3-D interactive imaging-processing program was developed to process the 3-D CT images and to choose the REV. The effect of the shape and size of the particles, specimen porosity, and location of REV center were examined by using different specimens of spherical glass beads, Silica sand, and Ottawa sand.

This thesis will describe an interactive computer program, developed to study density variation within image by means of an increasing voxel size, drawn from images of the powder. The calculation was repeated by increasing the physical size, starting from two voxels. When the calculated density becomes nearly constant in ten locations, then the minimum volume size may be determined from measurements.

2.6 Density Measurement

Density variations are usually introduced in the process of die compaction, which is unit operation-employed in powder metallurgy, ceramics, and other industries. Many methods have been previously used to estimate internal density variation (Kamm, et al. 1949, Kuczynki and Zaplatynskyj, 1956, Reed, 1988, Rajab and Coleman, 1985, Garino, 1995,). This led to a widely accepted schematic of a density map, formed during die compaction of ceramic powders. These are believed to arise because of frictional forces, particle morphology, and other powder characteristics. Density variations in PM industry are inherent to the manufacturing process and are important because these may lead to differences in local properties or a mechanical response, such as a post compaction operation (Lowell and Shields, 1984).
Train (1957) first conducted a density measurement of powder compacts in the early 1900s. The researcher also presented techniques based on differential machining, hardness tests, or an x-ray shadow of lead grids placed in the compact. Macleod and Marshall (1977) presented autoradiography experiments, using ceramic compacts that possessed natural radioactivity. The density distribution patterns were discussed in the context of die wall friction. Phillips and Lannutti (1996) developed a normally accepted methodology to quantify the density map. In CT, the transmitted intensity of a collimated beam of radiation is measured after passage through an object and related to density, according to Lambert’s law of absorption. These data are reconstructed into an internal density map.

Lin and Miller (2000) presented preliminary experimental findings of pore structure in 3D, using x-ray microtomographic techniques. The findings showed a detailed description of fluid flow, as well as transport in the filter cake structure. Kong and Lannutti (2000) employed an x-ray CT to monitor the compaction of spray-dried alumina. Local density changes and compaction curves showed that the well-known stage I-II transition indicates an alteration in the direction of transmitted pressure through the uppermost layer. Simulations reproduced the compaction procedure, while providing a clear explanation of the density distribution development. Burch (2001) reviewed techniques developed for the quantitative non-destructive mapping of density variations in material. These techniques were implemented on TOMOHAWK-AEA Technology’s PC-based CT system. Results are also obtained from the application of those techniques to the measurement of a density variation within powder compacts, both in the green or sintered states of P/M industry.

Li et al. (2001) provided quantitative characterization of part-to-part variations in bronze compaction and contrasted the compaction behavior with previous work on porous, spray-dried
agglomerates. The researchers found the density distribution patterns to be qualitatively and quantitatively dissimilar. Different mechanisms appear to dominate the transmission of stress through assemblies of solid metal particles versus that of spray dried agglomerates. Sinka et al (2003) applied an x-ray CT to measure the material density distribution in pharmaceutical tablets. For a particular material and x-ray energy, x-ray attenuation is approximately proportional to material density; density maps in tablets manufactured under controlled conditions also are presented.
CHAPTER 3

MEASUREMENT OF DENSITY VARIATION

3.1 Introduction

This chapter describes the materials and apparatuses used in the experimental work. The chapter also presents details of procedures used to prepare the specimens and the compaction experiments, and provides a description of the analyses procedures.

3.2 Experiment Preparation

Atomized Aluminum powder prealloyed 3.8Cu-0.75Si-1.0Mg was used in the experiments. The powder is manufactured by U.S. Ampal Inc., Flemington, NJ, and has an average particle size of 100 μm. Figure 3.1 shows an SEM image of the powder, while Table 3.1 lists the vendor, composition, mesh size, and density of the powder.

Figure 3.1 SEM Image of Ampalloy AMB 2712 Aluminum alloy powder
**Table 3.1 Specification of the Aluminum powder**

<table>
<thead>
<tr>
<th>Vendor</th>
<th>Ampal</th>
</tr>
</thead>
<tbody>
<tr>
<td>Powder</td>
<td>AMB2712</td>
</tr>
<tr>
<td>Composition</td>
<td>Al-3.8Cu-0.75Si-1.0Mg</td>
</tr>
<tr>
<td>Mesh</td>
<td>-100/+325</td>
</tr>
<tr>
<td>Pycnometer density (g/cm³)</td>
<td>2.7146</td>
</tr>
<tr>
<td>Apparent density (g/cm³)</td>
<td>1.22</td>
</tr>
<tr>
<td>Tap density</td>
<td>1.51</td>
</tr>
</tbody>
</table>

In this thesis, two types of compaction die configurations were used in the compaction experiments. Compaction configuration I has an upper punch, a die, a lower punch, and a spacer. Compaction configuration II has an upper punch, two dies, a lower punch, and a spacer. The schematics of the two compaction configurations are depicted in Figure 3.2. In this experiment, the die is made of aluminum. The punch is made of steel and can apply a high axial pressure on the powder without breakage. The spacer was used to keep the punch by accurately applying pressure in the axial direction without eccentricity. The geometry of all compaction toolsets is shown in Figures 3.3 through 3.9.
Figure 3.2 Schematic of configuration of the two dies used to compact the powder

Figure 3.3 Details of the die
Figure 3.4 Details of steel top punch

Figure 3.5 Details of bottom punch #1
Figure 3.6 Details of steel bottom punch #1

Figure 3.7 Details of steel bottom punch #2
Figure 3.8 Details of steel spacer #1

Figure 3.9 Details of steel spacer #2
The CT scans were conducted on two die configurations. The powder was poured into the die, using a funnel to achieve a uniform filling, because the homogeneity of the filling affects the spatial density distribution. The dies were especially fabricated to conduct the experiment. The specimen must be small enough to obtain higher resolution of the CT scans. In addition, the die must be light-weight to enable a mounting on the stage of the CT scanner and must rotate freely to acquire the CT scans. Figure 3.10 shows a photo of the compaction experiments. A computerized loading frame applied the load at an axial strain rate of 1%/min. Finally, it was scanned. A data acquisition system acquired the signal from both the load cell and the displacement sensor.

Figure 3.10 Photo of compaction experiment at Argonne National Laboratory
3.3 Experimental Procedure

Specimen I is cylindrical in shape, with an initial volume of $4.99 \times 10^{-4}$ in$^3$ and an initial density of 76.0 lb/ft$^3$. The specimen was loaded in the axial direction up to a maximum load of 98.5 lb, at a constant strain rate of 1%/min. Loading was paused at 5% strain increments up to 50% axial strains in order to acquire the SMT scans. Specimen II, with a larger initial volume of $9.07 \times 10^{-4}$ in$^3$ and initial density 97.2 lb/ft$^3$, was loaded and reached to a maximum strain of 12%. Loading paused at 0%, 3%, 6%, 9%, and 12% nominal axial strain for scanning. Table 3.2 presents a summary of scanning information for the two compaction configurations.

After scanning the powders, the images were analyzed, utilizing the Interactive Data Language (IDL) program. Computer codes were written by the author, using IDL to process and analyze the CT images. Figure 3.11 presents a flowchart describing the steps of image analysis. The IDL code reconstructed images at different axial strain levels to render SMT images. The author developed the IDL code to calculate the density of each REV size. The analysis focuses on developing a spatial variation of density map for both configurations, and illustrates the compaction mechanism of aluminum powder by comparing various density maps under different strains.

The CT scanning process yields the raw data of image. The height of the specimens was too large to scan in one image; therefore, three scans (labeled as A, B, and C) were acquired for each configuration. An IDL program was developed to truncate the overlap area between adjacent scans. The overlap scan should be cropped in order to combine the residual parts to create a new, whole volume file.
Table 3.2 Summary of scans.

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum Powder CERAC A-1208</td>
<td>I</td>
<td></td>
<td>4.99E-04</td>
<td>2.20E-05</td>
<td>4.41E-02</td>
<td>1.50E+05</td>
<td>98.5</td>
<td>50</td>
<td>27</td>
<td>4.96</td>
<td>0% strain, 5% strain, 10% strain, 15% strain, 20% strain, 25% strain, 30% strain, 35% strain, 40% strain, 45% strain, 50% strain</td>
</tr>
<tr>
<td>(grain size = less than 149</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>µm</td>
</tr>
<tr>
<td>µm)</td>
<td>II</td>
<td></td>
<td>9.07E-04</td>
<td>5.10E-05</td>
<td>5.62E-02</td>
<td>1.50E+05</td>
<td>33</td>
<td>12</td>
<td>27</td>
<td>4.96</td>
<td>0% strain, 3% strain, 6% strain, 9% strain, 12% strain</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
3D reconstructed volume images (one specimen image consists of few stacks of subvolume)

Stitch scans of images (IDL program)

Determine REV (IDL program)

Convert volume files to ASCII files (IDL program)

Open SSH secure shell and connect to Pelican computer system

Process image segmentation and remove the noise using the following analysis programs:
1. ik.in and ik3d.exe (indicator Kriging)
2. removeih.in and removeih (remove noise)

Use the computer program Ik.in and Ik3d.exe to determine the threshold values

Use the computer program Removeih.in and removeih to remove the noise inside the image

Repeat the above process. Use output of the above process as input. Flip the input data between voxel label to search for grain/void and voxel label to replace. save and exit. in the prompt type ./removeih

Download all the segmented file to locate file folder and transfer to volume file, stitch those files to obtain a whole sample file (IDL program)

Use "Mapping" (IDL program) to convert images into density maps

Figure 3.11 Flowchart of analysis of CT images
After compiling the entire scan volume file, the REV size should be determined before the image segmentation. Due to the difficulties associated with the measurement, as well as the characterization of microstructure, the existence and size of a REV has remained largely conjectural. A systematic method was used to examine the characteristics of the REV, using x-ray computed tomography images. Generally, an original image of 0% axial strain level was used to determine REV. The threshold value was obtained from the image histogram. In this experiment, the minimum threshold value was 600 and the maximum threshold value was 700. The radius of the kriging window was 3. The threshold values were used for all compaction images, assuming the REV would not change too much during compaction.

A computer program was developed to investigate density variation within an image with increasing voxel sizes. The calculation was repeated by increasing the size of the volume, starting from two voxels. Figure 3.12 depicts the relationship between the density and the side length of REV for 10 random locations within the original image. It shows that the density becomes nearly constant, when the side length is larger than 79.36 μm/pixel (n=16). Thus a REV size volume of 79.36 μm × 79.36 μm × 79.36 μm was used to quantify the density variation of the powder.

After determining the REV, all image volume files were first converted to ASCII files using the IDL program and then uploaded to the LSU high performance computer called Pelican. The process required a 34 nodes IBM Series (Power 4, 5 & 5+) cluster running the AIX operating system. 30 IBM Power 5 and Power5+ 575 nodes are coupled with high performance switch interconnects suitable for parallel computing, while others are isolated machines suitable for interactive use or the execution of sequential programs. Pelican was designed to allow machines to change roles with user-demands change and with more available computational
resources. Generally, the SSH secure shell, as a network protocol, permits data to be exchanged between network devices, especially on Linux and Unix-based systems. All ASCII files were written by IDL code and were uploaded to Pelican via OpenSSH software to conduct the analysis.

![Graph](image)

Figure 3.12 Fraction theoretical density against side length for ten REV locations

For many applications, it is desirable to produce a ceramic material that contains a minimum of open and closed porosity. If this ceramic could be densified completely to contain no open or closed porosity, it would consist only of a mixture of solid phases. This pore-free condition would represent the maximum bulk density achievable for the specific composition, referred to as the theoretical density. Theoretical density is often used as a standard against which one may compare the actual bulk density achieved for a material. For example, if a material contains 10% porosity, it would be defined as 90% of theoretical density. Therefore, the fraction theoretical density is 0.90. The theoretical density may be measured directly by the pycnometer method. In this experiment, a CT image was used to calculate the porosity and then the theoretical density.
%TD = \frac{\text{bulk density}}{\text{theoretical density}} \times 100

Porosity = 100\% - \%TD

In the high performance computer system of Pelican, computer codes called ik3d.exe and removeih.exe were used to process image segmentation, which in turn yields a noise-cancelling image. Finally, the image was processed using an IDL Mapping code to plot the density map. In addition, results were exported to a data file that may be opened using Microsoft Excel for further evaluation.
CHAPTER 4

SCANS ANALYSIS AND RESULTS

4.1 Introduction

The geometry of the two configurations of compaction was described in Chapter 3. The results of the compaction are presented in this chapter. Both experiments were prepared and tested using the same procedure. To better comprehend the density distribution of powder compaction, the definition of density will be specified in terms of overall average bulk density, row statistics of average density (row density), and column statistics of average density (column density). Further, the density variation of both compaction configurations will be discussed and compared.

4.2 Compaction Configuration I

4.2.1 Density Map

The results of the compaction configuration I at compression strains of 0% to 50% are shown in Figures 4.1 through 4.11. For each axial strain level, a central axial section with a thickness of one REV size was taken to perform the analysis. The overall density maps were divided into cross-section volumes along x- and y-axes for each strain level. Figures 4.1 through 4.11 show the density maps from the CT analysis of compacted aluminum samples, which depict a considerable density variation among the eleven scans; therefore, each scan is unique.
Figure 4.1 Density Map at 0% axial strain along x- and y-axes

Figure 4.2 Density Map at 5% axial strain along x- and y-axes
(a) Density map along x-axis                                          (b) Density map along y-axis

Figure 4.3 Density Map at 10% axial strain along x- and y- axes

(a) Density map along x-axis                                          (b) Density map along y-axis

Figure 4.4 Density Map at 15% axial strain along x- and y- axes

(a) Density map along x-axis                                          (b) Density map along y-axis

Figure 4.5 Density Map at 20% axial strain along x- and y-axes
Figure 4.6 Density Map at 25% axial strain along x- and y-axes

Figure 4.7 Density Map at 30% axial strain along x- and y-axes

Figure 4.8 Density Map at 35% axial strain along x- and y-axes
(a) Density map along x-axis                                         (b) Density map along y-axis

Figure 4.9 Density Map at 40% axial strain along x- and y-axes

(a) Density map along x-axis                                         (b) Density map along y-axis

Figure 4.10 Density Map at 45% axial strain along x- and y-axes

(a) Density map along x-axis                                         (b) Density map along y-axis

Figure 4.11 Density Map at 50% axial strain along x- and y-axis
Table 4.1 Density map and respect density value for compaction strain of 50%
The CT images were then divided into additional cube elements (16 pixels × 16 pixels × 16 pixels). The computer codes that were described in Chapter 3 were used to calculate the density values for each REV. The results were automatically saved in an Excel file. Table 4.1 depicts an example of the density map of 50% axial strain and gives the respective density values. Each specimen was divided into 25 columns and 24 rows. The values in Table 4.1 match the associated density values in the graph. Therefore, one can easily visualize the density variations at different locations for a certain compaction stage. For example, in the right side image shown in Table 4.1, the element of the first row and first column matches 0.97 in Table 4.1. Therefore, one can easily track any specified volume for a specific density.

The overall density is defined as the average density of all density values in a specified scan. In this case of REV size volume, the sum of all density values divide by the number of REV cubic will yield the overall density. The compaction response curve (overall density vs. axial strain) then can be generated as depicted in Figures 4.12.

![Figure 4.12 Compaction response curve for compaction configuration I](image-url)
The compaction response curve relates the percent average density of the compact to a specific compaction axial strain level. Figure 4.12 indicates that the slope found in the first five scans varies slightly. Initially, the filling procedure of sampling may lead to the occurrence of some high void space in the top layer; however, the vertical load is not transmitted to the particles beneath the layer. The variation of density at the initial five scans cannot be eliminated, due to the properties of the powder materials and the compaction process. Particles began to move into the void space when the curve initiated a 20% axial strain, after which the compaction response curves became almost linear during the last six scans. At this stage, there was no room for particles to slide, since the particles establish multiple contacts with one another and plastically deform as compaction continues.

4.2.2 Row Density

The row density is defined as the average density of all density values in each row of a specific scan. Since the height of a specimen changes during the compaction process, the number of REV cubic in each row is different. For the specimen before compaction, the height is 40 REV or 3.17 mm. When a specimen is compressed to a 50% axial strain, the number of cubic in rows decreases to 24 REV or 1.905 mm, almost half the height of the specimen before compaction. Figure 4.13 depicts row density statistics, showing a variation in the average density of the specimen for eleven axial strain conditions.

Referring to Figure 4.13, the x-axis represents the fraction theoretical density, which may be calculated using the method mentioned in Chapter 3, while the y-axis represents the distance from the bottom of specimen toward the top. In a small axial strain condition such as 0%, 5%, and 10%, the average density of each layer is almost constant in the overall average powder.
When the particles are filled into the die, a high void space appears around the particles, causing a slight contact between them. In turn, the void makes room for particles to move in closer as the compaction continues. After particles move into the void space, even more contact occurs between particles, which results in the plastic deformation of particles, as well as less sliding and rolling.

Figure 4.13 Row statistics of average density taken from compaction configuration I
More conspicuously, a large void layer appears near the bottom of specimen during the initial scans. There are no previous research conclusions about the occurrence of the void layer. Only a few researchers mentioned that a void layer at the beginning of compaction would result from the process of filling the powder. Referring to the curve of 0% axial strain in Figure 4.13, the void layer occurs in the layer at about 0.793 mm above the bottom of the specimen, with an average theoretical density of 0.43, which is 22% less than the overall theoretical average density (0.55). As compaction continues to a 5% axial strain, the void layer moves downward toward the location, which is closer to the bottom of the die. The average theoretical density increases to 0.46, which is 17% less than the overall theoretical average density (0.55). Although the void layer still exists when the axial strain reaches 10%, the location is still the same. At 15% axial strain, the void layer disappears and the average theoretical density is identical to the overall average density.

As the compaction process continues, the disappearance of this layer or any relatively large void space is probably due to the clearance between the punch and the die wall, as particles move into the voids that generate during the filling process. In other words, the stress chain between the particles may not be efficiently transferred to the particles beneath, due to the particles’ ability to move into protected zones between the particles during low axial strain conditions. This also explains why force chains are not efficiently transferred to the particles beneath, because of the particle ability to move into protected zones between particles during a low axial strain condition. This may also serve as an important reason why high density zones and low density zones occur at different locations in the same scan.
### 4.2.3 Column Density

The column density is defined as the average density of all values in each column for a specific scan. The diameter of the specimen does not change during the compaction. Therefore, the width of the curves represents the diameter of the specimen, which is 2 mm or 40 REV sizes. Figure 4.14 depicts the column density statistics, showing a variation in average density of the specimen for eleven axial strain conditions.

This study notes that Figure 4.14 shows typical column statistics of average theoretical density variation from left to right for a specific scan. The top curve in Figure 4.14 represents the highest theoretical density. Further, the column density increases as the axial strain increases. Referring to Figure 4.14, when the axial strain is smaller than 15%, the respective density curves increase in overlapping zones and thus intercept one another. The overlapping zones concentrate...
at the center of specimen, and the density of the zone near the die wall shows a high degree of variation.

For the curves of 5% and 10% axial strain, the average theoretical density of column at the center (from 600 μm to 1400 μm) exhibits a great similarity. In contrast, the average density near the die wall (0 to 500 μm, 1500 μm to 2000 μm) varies greatly. Those zones have density values far from the overall average column densities. The differential density is attributed to the mechanism of the specimen filling procedure, as well as the boundary effects of the die wall and advancing punch. At the beginning of the filling process, the powder is more compacted at the center than near the die wall. Therefore, compaction of the powder, the center has no high void space to eliminate; therefore, the density does not change substantially. However, since there are some voids when filling powders near die boundaries, the elimination of a large void will lead to higher differential densities.

The differential density plots for the aluminum powder compaction shows irregular behavior, with the densities on one side differing from the other side. This quantifies the high side-to-side asymmetry of density in the zones of the compaction trends in Figure 4.14. The trends show a higher side-to-side asymmetry when the axial strain is high. In contrast, the curves of small axial strain are typically symmetrical in shape. Although the average density in each column is relatively uniform, the maximum and minimum column density clearly reveals high side-to-side asymmetry in the density profiles.

As the aluminum powder is composed of highly dense solid particles, each particle is significantly more resistant to the deformation than loosely packed particles during the initial
stages of compaction. Therefore, compaction force is transmitted both vertically and horizontally through particle contacts in the uppermost layer, located under the punch.

The randomly generated characteristics of this thin layer logically dominate the symmetry of force transmission until a relatively high axial strain is reached. For loosely-packed particles, pressure applied by the punch will be easily distributed horizontally by a generalized deformation of this uppermost layer. More laterally uniform force distribution provides a more uniform advance into the un-compacted powder. This provides a logical explanation for the high, lateral asymmetry in the compaction curves.

4.2.4 Compaction Stress Versus Strain Curves

A computerized loading actuator applies a constant displacement in controlled loading for both configurations during the compaction process. Displacement and load data were acquired from a load cell and displacement transducer during compaction. The stress and strain curves are generated and based on load and displacement measurements. The curves show the relationship between displacement and loading when the compaction is in different levels; the curves also present the typical phenomenon in powder compaction.

Figures 4.15 shows the relationship between the stress and strain obtained from the compaction test. Generally, a specimen was removed from the loading system during the scanning process, in order to send the specimen to the CT system to get the image. Once the scanning process was complete, the specimen was compressed to the next strain level. In this process, stress relaxation occurs in the uppermost layer of the specimen, which can be observed in Figure 4.15. The vertical line in the figure indicates that more load needs to be applied to achieve the stress level, before pausing the load. As the load is released, the elastic energy allows for a small expansion of the compacted powder, known as springback.
Springback is usually described as a dimensional change of compact, formed from granular materials in the axial or the radial direction when the applied load is removed. In general, a small amount of springback is necessary to allow the compact to separate from the punch. A large amount of springback may be associated with cracking, delamination, and end capping. In Figure 4.15, the springback is smaller at the small axial strain condition than that at the high axial strain. At the low axial strain condition, the breaking points cannot be observed and the displacement that occurs in the process is less than 1.2 mm. Here, the springback is only due to the necessary separation between punch and the powder. When the axial strain reaches 30%, the displacement occurring in the springback is 2.4 mm, which is larger than the displacement at a small axial strain condition. This result is largely due to the cracking of particles in the compaction and end capping effect.

Figure 4.15 Stress and strain relationship of compaction configuration I
4.2.5 Density Histogram

The density distribution may be represented using a histogram of fraction theoretical density, plotted against relative frequency. Numerical access to the density distributions is summarized graphically, using the overall average densities, row densities, and column densities. The density histograms and the CT images may be combined in the analysis of a compact. The relative positions and magnitude of the histogram peaks may be used as an indicator of the location and severity of the density distribution. Inspection of peak location in the density reveals that the peak in the graph corresponds to the overall average theoretical density and the width of the distribution curve defines the density distribution. In the histogram, relative measurements can be used to quantify the density distribution by determining relative changes in the peak location and magnitude.

Figure 4.16 Density histogram of compaction configuration I
Figure 4.16 was generated from the data of the density values to plot density histogram for the aluminum powder compaction. The relative percentage of values of density distributions following compaction are represented in Figure 4.16. Increased axial strain apparently leads to an increased compact uniformity. As the axial strain increases, the density gets closer to the peak values. The largest width of curves and data scatter occurs at 0% axial strain, since the powder at low axial strain condition has much less contact and interaction than those at high axial strain. As the compaction continues, the width of the curves narrows and the overall average density becomes higher.

The characteristic shape is muted in the averaged plot due to minor variations in the maximum and minimum density values, as well as the small bin size used for the histograms. As the axial strain changes, the histogram shape reflects the internal changes taking place. Increases in the width of the density distribution do not occur uniformly; tails can form in the distribution, which would increase the width. The averaged distribution in Figure 4.16 displays considerably different characters. The curve of 0% axial displays a much wider low-density shoulder. As compaction continues to 20% axial strain, a bimodal distribution develops in which distinct high and low density peaks are always visible in the single data set distributions. This also proves that the transmission of effective stress is not continuous during the compaction, especially at the beginning of the compaction. Then, the curve of the 30% axial strain does not greatly decrease in the width, yet the curve does indicate that more high density values occur. For a curve of 50% axial strain, the high and low density peaks are approximately combined in magnitude, and a distinct density peak is visible.
4.3 Compaction Configuration II

As for the compaction configuration II, a different die with a smaller cylindrical spacer beneath, as well as an anther lower punch, were used. Apparently, there are many more interactions between the particles near the bottom of the die as the lower punch advances upward. Therefore, besides the particles in the uppermost layer, the particles in the bottom also experience an upward force. The stress chain in both compaction configurations is completely different, which leads to different density maps. In this section, the density maps and discussions of the comparison with the compaction configuration I is presented.

4.3.1 Density Map

For compaction configuration II, the compaction starts from 0% axial strain to 12% axial strain with 3% interval for scan. The total of five scans may not be strong enough to come to a conclusion for the powder compaction. However, the special configuration of compaction still may prove some conclusions made regarding compaction configuration I. Moreover, due to the special geometry of compaction configuration II, the effect of L/D ratio and springback may be detailed in the following discussion.

For compaction configuration II, there was no considerable variation in the density map, when an axial strain changed from 0% to 12%. Due to the size of the powder particles and the geometry of the compaction die, the relatively low axial strain condition did not lead to a high density variation. However, some interesting phenomenon could be still observed in the density maps. There was always a void layer near the interception zones of the upper spacer and lower spacers.
Figure 4.17 Density Map at 0% axial strain along x- and y-axes

Figure 4.18 Density Map at 3% axial strain along x- and y-axes
(a) Density map along x-axis

(b) Density map along y-axis

Figure 4.19 Density Map at 6% axial strain along x- and y-axes

(a) Density map along x-axis

(b) Density map along y-axis

Figure 4.20 Density Map at 9% axial strain along x- and y-axes
The void layer does not completely disappear even when compaction level reaches a strain of 12%. Detailed analysis will follow. Another interesting observation is that density values of specific zones around the lower spacer do not change too much during the compaction process. This might be due lack of the invalid transmission of effective stress from top to bottom and the particles filling procedure. Since small strain axial strain condition could not make sure the pressure transfer from the top to the bottom, since some particles begin to establish contact with each other in the uppermost layer or void layer continues in the specific area when the compaction goes to the level. Fortunately, the void layer could be easily observed and the compaction process of void layer also could be tracked from the density map.
The compaction response curve was also plotted for compaction configuration II and shown in Figure 4.22. From 0% to 3% axial strain, the overall theoretical density is almost the same. However, from 3% to 6% axial strain, the overall density sharply changed from 0.559 to 0.583. In the first stage, the stable overall theoretical density is probably due to the stress that might not efficiently transfer to particles in the bottom of the die. Therefore, the void layer remains in the interception zones of the two spacers. On the other side, since the effective stress does not effectively transfer to the bottom, the particles in the zones near the punch undergo a larger stress. As the level is reached, particles primarily undergo a particle rearrangement, due to the powder breakup and crushing controlled by the strength of the powders. However, the limitation of this plot could not address how stress transferred within the powders, and how much particles deformed, compared to the initial status.
4.3.2 Row Density

Row density reflects the statistical average density of each layer from the top to the bottom of the compaction die. For compaction configuration II, the height of the compactions set is 5.4 mm or 68 REV, which is almost twice as long as that of compaction configuration I. Since the compaction strain levels reached only 12% in this compaction configuration, the height decreased to 4.8 mm. Figure 4.23 depicts the row statistics, showing variation in average density from the bottom to top of the cylinder samples for five axial strain conditions.

The x-axis represents the fraction theoretical density, calculated using the method mentioned in Chapter 3, and the y-axis represents the distance from the bottom of the specimen toward the top. In the zones near the bottom of the die, the average density of each layer is almost the same and does not change during the compaction. In the following layer (160 μm, 400 μm, 800 μm, 1200 μm, 1360 μm point in the y-axis), the curves move in the same direction, which means the average density of the specified row shows a great similarity. This proves again that there is no effective transfer of stress to the bottom of the die. At the top zones of the die, when closer to the upper punch, the density variation is higher; yet the variation occurs randomly with no uniformity.

Furthermore, the void gap occurs near the interception zones of the upper and lower spacers. The ratio of length over diameter (L/D) is nearly 6.614 for compaction configuration II, which is much higher than 1.447 for compaction configuration I. This higher ratio contributed to the generation of the void gap in the middle zone. In earlier L/D effect research, the density distribution is quite different, due to the L/D effect. The larger the L/D effect is, the wider the range of density variation. Thus, the larger L/D compact always has a broad, bimodal distribution.
At the beginning of compaction, the void layer occurs near a layer of 1600 mm with an average density of 0.28, which is 49% away from the overall density (0.55). The void layer remains in the same place, when the compaction reaches an axial strain of 3%. This indicates
that the stress does not transfer to the lower area, and that internal force was applied more to the upper zones than to the gap layer. Therefore, the bottom zones have no changes during the first compaction stage. As the compaction continues to an axial strain of 6%, the void layer begins to shrink and moves downward to the 1432 mm point, which is slightly lower than the original height. This may be caused as the stress begins to transfer to the bottom; some voids were filled at the second compaction stage. With continued compaction, the void gap changes to a smaller size and continues moving downward. Thus, the efficiency of the transformation of stress becomes apparent as some particles begin to move into a void area or to establish contact with other particles. The average theoretical density of the void layer also increases sharply to 0.5, which is close to the overall average theoretical density.

4.3.3 Column Density

The column density reflects the average density of all values in each column for a specific scan. The diameter of specimen does not change during the compaction. Therefore, the width of the curves represents the diameter of the specimen which is 2 mm or 40 REV sizes.

Figure 4.24 shows the column statistics of an average density variation of five scans. For 0% and 3% axial strains, both curves have the same magnitude and tendency in each peak and valley. This coincidence proves that the stress transformation in first compression does not work. For the whole five scans, the fluctuations in the points of 480 mm, 640 mm, and 720 mm, through 1600 mm of x axis shows great similarity. A large variation of column density only occurs at the top or bottom zones of the compaction die. The large changes of the density in the uppermost layer are due to a direct contact with punch, which leads to particle movement and
Figure 4.24 Column statistics of average density taken from compaction configuration II

particle arrangement, thereby crushing deformation. The differential changes in the bottom zones may be caused by the powder preparation and filling process. Since the generation of gap makes the stress transformation functionally invalid, the particles in the bottom zones does not have enough particles interaction.

4.3.4 Compaction Stress Versus Strain Curves

Compaction stress and strain curves represent the loading process of compaction, based on load and displacement measurements. One can find a basic tendency of springback phenomenon and end-capping effect. Moreover, the spatial variation of the overall average density may be tracked in the plot.

Curves of compaction configuration II have the same vertical line as the curves of configuration I. At both the beginning and end of compaction, it is not easy to find the vertical line. In the middle stage or after compaction reaches a certain level, the springback is strong, which indicates the elastic energy of the compacted powder expansion is the largest of any other
stage. Normally in this stage, a large amount of springback is relative to cracking and deforming among particles.

Figure 4.25 Stress and strain relationship of compaction configuration II

4.3.5 Density Histogram

Since the die geometry of compaction configuration II is quite different than that of compaction configuration I, the density histogram shows a characteristic shape that is also quite different. The characteristic shape of compaction configuration II is mostly bimodal distribution. In statistics, a bimodal distribution is a continuous probability distribution with two different modes. Distinct peaks appear in the probability density functions. The occurrence of bimodal distribution is largely influenced by the L/D effect, because it could generate the gap layer which divides the die into two compaction zones. The high peak always indicates the overall density as
well as where the most REV density will occur. The low peak reflects the variation in the gap status.

Figure 4.26 Density histogram of compaction configuration II

The averaged distribution in Figure 4.26 also displays a considerably different behavior when compared to results of compaction configuration I. The curve of the 0% axial strain is narrow, with a very slight low-density shoulder. There is only one peak in this curve; the shoulder is not wide enough, similar to the curve in configuration I. The original distribution shape may be attributable to the powder preparation and filling process. For the curve of a 3% axial strain, a bimodal distribution has developed in which distinct high and low density peaks are always visible in a single data set distributions. A great single peak appears in the curve, which means more REV size density, suggesting an approach to an average density as compaction continues. As the compaction proceeds to a 6% axial strain, the main peak
disappears and a much lower peak appears in the curve. In this compaction stage, the particles in various places form a local densification and appear to have more local densification areas. Even when the compaction proceeds to a 12% axial strain, the behavior exhibits minimal changes. If the compaction of configuration II can reach a higher level of axial strain, either all of the densification areas or the high or low densification areas will disappear, and the density of all REV size volumes will be close to the overall average density, forming a major peak.
CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

This thesis utilizes Synchrotron Microtomography (SMT) in order to better understand the compaction behavior of aluminum powders. Two configurations of compaction dies were used in the experiments scanned at APS. IDL algorithms were developed to render the scan images and to perform quantitative analysis. In general, SMT can be successfully applied to examine density variation and to track microscopic deformations of aluminum powders under compaction. SMT provides efficient, quantitative access to internal density distributions during compaction. Comparison of density maps at different strains levels may be used to investigate spatial variation, thus providing critical measurements that may be used to calibrate constitutive models to predict the behavior of compacted powder.

The main factors that influence the density distribution of compacted powder are a) the friction interaction between powder and die wall; and b) the geometry of die and punches. The following conclusions are drawn:

• The density distribution within the compaction die was controlled by several factors. The sample preparation and initial randomness of filling show a profound influence on the uniformity. The initial punching of ram can result in a different distribution of density within the die, which can be observed from the density maps.

• Springback is another important factor which influences density distribution, especially during the scanning procedure. Since the release of stress results in the release
of elastic energy stored in the powders, the deformation of particles changes. This result has an influence on the density distribution.

- The aluminum compacts show a considerable side-to-side asymmetry. Although the average density in each column and row is relatively uniform, the maximum and minimum column density reveals clearly a greater side-to-side asymmetry in the aluminum compacts. When a high axial strain level is reached, the density distribution begins to exhibit great side-to-side symmetry.

- The generation of a void gap between layers indicates an inefficiency regarding the transmission of stress during the compaction. The occurrence of this layer is probably due to the clearance between the ram and die wall at the top. Stress may be inefficiently transferred to these particles, because of their ability to move into the protected area between the wall and the advancing punch.

5.2 Recommendations

Future research recommendations are as follows:

- Investigate the effect of sample filling procedures to determine a method to reduce the influence on the density distribution and springback effect.

- The different scales of compaction die could be tested, especially when length to diameter ratios are large. The L/D effect should be examined and analyzed.

- A higher compaction strain level should be achieved in order to test particle behavior at high strain condition under compaction.
REFERENCES


VITA

Yi Yang was born in September, 1986, in ShaoXing. Then, he moved to HuBei Province with his family and began his study there from elementary school to college. He finished his high school in 2005 and went to one of the top ten universities-Wuhan University in China. He spent four years there and earned his bachelor degree both in civil engineering and economics. After graduation from college, he received full graduate research assistantship to begin his study in geotechnical engineering program in the Civil and Environmental Engineering Department at Louisiana State University, Baton Rouge, United States, in August 2009. He conducted research under the supervision of Dr. Khalid Alshibli and received his master degree in May 2011.