CHARACTERIZATION AND EVOLUTION OF COMPLEX 3D MATERIALS BY
DIGITAL IMAGE ANALYSIS AND CORRELATION

By

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A thesis submitted to the

Graduate School – New Brunswick

in partial fulfillment of the requirements

for the degree of

Master of Science

Graduate Program in Mechanical and Aerospace Engineering

Written under the direction of

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And approved by

New Brunswick, New Jersey

January 2011
The aim of this thesis is to present a methodology to utilize spatial density visualization capabilities of X-Ray Microtomography (\(\mu\)CT) to characterize, and to capture the deformation in complex materials. \(\mu\)CT is a versatile technique, which has been used to non invasively visualize, investigate and quantitatively analyze materials and works on the principle of attenuation of X-Rays as they pass through them. In the first part of this dissertation, we have attempted to employ this 3D imaging technique to study the influence of local density variations along the length and width of a roller compacted Micro Crystalline Cellulose (MCC) ribbon on the local ribbon strength which in turn, potentially alter the mechanical properties (such as tensile strength and hardness) of its end product - a solid pharmaceutical dosage form. Density variations in three cases of ribbons produced a) with no lubrication b) by including Magnesium Stearate lubricant in the excipient and c) by lubricating rolls and screws have been investigated. Heckel and elastic recovery analysis indicate that the local density variations in the roller compacted ribbons drastically impact the mechanical properties of the solid dosage form.
The rest of this dissertation involves the study of micromechanical behavior of open-cell solid foams. Metallic foams are cellular materials of very low densities in comparison to their parent material and are capable of large deformations owing to cellular collapse. Our understanding of their mechanical behavior is restricted to their heterogeneous deformation under uniaxial compression because of its architecture. Even though high speed photography combined with Digital Image Correlation techniques, capture the whole field strain map, they are limited by their capacity to visualize the surface deformation. Since the boundary conditions inside the volume of the sample are different, the surface deformation may or may not be a representative of the deformation pattern in the complete volume of the sample. Assuming plane strain along the direction of applied uni-axial deformation, the full field strain in the sample volume is measured by Digitally Correlating average images extracted from volumetric μCT data (which capture spatial material information from a thin section of the sample volume) with a similar successive image through progressive steps of applied deformation. Strain fields at slices at the surface and those at the interior hare compared. It has been found that regions closer to the free surface are susceptible to higher deformation values in comparison to the core. A foundation has been created to expand this methodology to capture the out of plane deformation in the entire volume.
Acknowledgements

I would like to express my deep gratitude to my advisor Prof. Alberto M. Cuitiño, for his invaluable guidance, advice and enormous patience all through my research work. It has indeed been a privilege to have been in contact with such an extensively knowledgeable and brilliant, yet, ever approachable scientist and teacher.

I would like to thank the entire faculty at Rutgers, especially Dr. Ellis Dill and Dr. William Bottega for delivering wonderful lectures during my coursework, making mechanical engineering an even more interesting concentration to delve into. Special thanks to the staff of the department, Mr. John Petrowski and Ms. Aiesha Jenkins for being ready to help whenever I approached. Thanks to Dr. Pat Buckendahl, for instructing me on the Micro-CT setup without which this work would not have materialized.

I gratefully acknowledge the financial support from the Dept. of Mechanical Engineering at Rutgers and the Engineering Research Center (Structured Organic Particulate Systems).

My extended thanks to my research group and friends in the department, Athanas Koynov, Ilgaz Akseli, Yuri Gulak, Pedro Romero, Alisar Tuncer, Dan Braido and Ram Chandra Murthy Kalluri for their guidance, friendship and the enjoyable time during my stay at Rutgers.

Finally and most importantly, my parents, Lalitha and Ramakrishnan Iyer, for being the sole reason of my existence, and for shaping me up to what I am today.
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Chapter 1

Introduction

1.1 X-ray micro-computed tomography: Determination of the density distribution in complex materials

X-ray micro-computed tomography (µCT) is a volumetric imaging technique which permits imaging of the interior microstructure of materials non-invasively with spatial resolution which can approach that of optical microscopy (1). This method determines the density of a material in space on the principle of attenuation of X-rays when they pass through it (2). The specimen is placed on a rotating stage between a micro-focal poly-energetic cone source producing a conical beam, and a two dimensional charge-couple detector (CCD) based array (Fig 1.1). A phosphor plate is optically coupled with the detector to intensify the image. The image spatial resolution is primarily determined by the X-Ray source focal spot size, the detector element size and the system geometry. Most modern industrial µCT systems are capable of attaining resolutions of less than 50µm. The attenuation values calculated as a function of space obeys the Beer-Lambert’s Law:

\[ I = I_0 \exp(-\mu x) \]

where \( I_0 \) is the incident X-ray intensity, \( I \) is the transmitted X-ray intensity, \( \mu \) is the linear attenuation coefficient (\( cm^{-1} \)) and \( x \) is the sample length. A more general form of the law above can be written by adding the attenuation increment along the direction of ray propagation as:

\[ I = I_0 \exp[-\int \mu(s)] \]
where $\mu(s)$ is the *linear* absorption coefficient at position $s$ along ray $s$. Thus, the central problem of micro-tomography is to assign the correct value of $\mu$ to each position along ray knowing only the values of the line integral for various orientations (1).

$\mu$ varies along the beam and is dependent on the incident energy as well as the sample composition and follows the law of type

$$\mu(x, y, z) = \frac{K \rho Z^4}{E^3}$$

in the photoelectric domain. Here $K$ is a constant, $\rho$ and $Z$, are respectively, the density and atomic number of the material under investigation (3). Radiographs are the projections of a large amount of information on one single plane. Projections (radiographs) in TIFF format are acquired from a range of angles around the object. A complete projection data set is obtained by capturing equiangular spaced views over $180^\circ$. To overcome the complexity involved in the interpretation, $\mu$CT combines information in many radiographs, each at a different orientation of the sample in front of the detector. For small angular steps it is possible to compute the local value of the attenuation coefficient (4). Reconstructions of $\mu$ are usually made on a rectangular array, namely the horizontal reconstructed images, wherein each image element (pixel) has a value of attenuation coefficient ascribed to it. A horizontal 2D BMP image is reconstructed from the projection data by applying Filtered Back Projection (FBP) algorithms – a Fourier based technique (5). However, in order to display it on a screen, it is conventionally rescaled (6). The intensity scale used in a reconstructed CT image is called the CT number and is measured in Hounsfield Units (HU). It is the linear transformation from the original linear attenuation coefficient into one in which water and air are assigned HU values of 0 and 1000 respectively and is defined by (7) :
where $\mu_{water}$ is the linear attenuation coefficient of water (8). It is thus the fractional difference of the linear coefficient in relation to water (6). This is a standardized scale useful for inter-comparison of CT values obtained from different CT scanners and different beam energy spectra. In the reconstructed image the CT value is mapped linearly into a number of values corresponding to the bit depth. In general, higher the local density of a voxel, greater is the CT number and hence brighter the image. The individual slices (horizontal reconstructions) can be stacked up to render a 3D volumetric visualization of the specimen using standard interpolation techniques such as marching cubes or adaptive rendering. Standard X-Ray sources emit rays of polychromatic spectrum with a range of energies which leads to our consideration of beam hardening. Since the attenuation coefficient is dependent on the beam energy, the lower energy rays are preferentially absorbed near the edges of the specimen leading to brighter edges or higher CT values at the boundaries. Prefiltration of the rays is done by passing rays through metallic filters immediately after the source to absorb the lower energy rays. In the reported study, X-ray $\mu$CT measurements were carried out on a high resolution SkyScan-1172 XRCT (SkyScan, Kontich, Belgium). Fig 1.1 illustrates the working principle of $\mu$CT. It also shows the system we have used for our experiments. It is our aim in this dissertation to apply this versatile tool to study and characterize complex materials, in our case, two of them. Chapter 2 describes its application to pharmaceutical materials and Chapter 3 to the study of metallic cellular materials.
Figure 1.1 X-Ray Computer Tomography setup a) Illustration b) Actual
Chapter 2

Characterization of granular pharmaceutical materials

2.1 Background

Pharmaceutical drug substances are commonly administered as tablets or solid dosage formulations, most often orally, and have been existent since the nineteenth century (9). The desired therapeutic activity is achieved by the excipients added to the formulation as well as the substance’s physico-chemical characteristics. Tablet formulations are products made directly by compressing an intricate blend of excipients and Active Pharmaceutical Ingredients (API) (9). To successfully manufacture acceptable pharmaceutical products, the materials require to be adequately sieved, blended, granulated, and dried before the agglomerates are finally compressed (10) so that the final characteristics of the tablets such as, drug dissolution rate, disintegration time, porosity, hardness etc. meet the specified values (9). One of these important characteristics that influence not only the safety, efficacy, stability, homogeneity and viability of the dosage form but also the powder fluidity, processing behavior and compression characteristics during the manufacturing process is the particle size distribution of the excipients, drugs and their blends (11). The optimal particle size objectives are met in the industry by using granulation technology, wherein particle sizes are enlarged by agglomerating small particles into larger ones. The principal methods of granulating pharmaceutical materials are: wet processes, dry processes and other processes. Among them, roller compaction is a continuous and pressure-induced agglomeration unit operation in the dry granulation process in which granules with acceptable compaction properties, flowability, and compositional uniformity are formed from feed material consisting of mixtures of active
and excipient powders, and relies on powder bonding mechanism such as particle rearrangement, elastic and plastic deformation, and particle fragmentation (12) (13). In the roller compaction process (Fig 2.1), powder blend is continuously fed into the feeding zone (slip region), where most of the densification is solely due to the particle rearrangement under relatively small stresses created by the feeding method. The densified powder blend is then gripped and subjected to high pressure at the compaction zone (nip region) between a pair of counter rotating rolls by gravity or a feed screw. The powder is forced towards the narrow gap between the rotating rolls due to the feeding mechanism as well as friction between the material and the roll surface. The extrusion zone (release region) is the final region where the compaction stress is relieved and the compacted material (i.e. ribbon, flake, briquette or sheet) is released (13). At the extrusion zone, most of the elastic recovery/porosity expansion in the compacted material may occur. The ribbon is subsequently milled into granules, and undergoes additional blending and lubrication before being compressed into pharmaceutical tablets.

Sheskey et al. (14) studied roller compaction as a viable mechanical process to develop controlled-release matrix tablets. They have indicated that an increase in roll pressure resulted in a decrease in tablet hardness and increase in tablet friability. Farber et al. have reported the reduction in strength of tablets from granulation when compared to those from direct compression (15). Sheskey and (16) Hendren also observed that roller compacted ribbons produced relatively smooth surfaced granules with higher bulk and tap densities in comparison to the ones produced by wet granulation. In accordance to Wikberg and Alderbom’s studies (17), they thus led to production of tablets with increased mechanical strength.
Figure 2.1 Schematic diagram of the roll compaction process (not to scale). Different zones of roller compactor; 1. feed zone (slip region), 2. compaction zone (nip region) and 3. extrusion zone (release region). $\theta$ and $\alpha$ are the entry and nip (gripping) angles, respectively.

It has been also found that the tablets prepared from direct compression had higher crushing strength than those prepared from roller-compacted granules. Despite these several advantages of agglomeration in roll presses, this technique presents some drawbacks such as generation of fines (18) (19), and lack of reworkability/tabletibility (20) (13). Though not greatly affected in case of brittle materials such as lactose, tabletability of plastically deforming materials such as MCC is decreased due to granule size enlargement (21) (22). When ribbons produced using a lab-scale compactor and tablets produced from a uni-axial compression with a compactor simulator were compared, it was found that the ribbons presented much larger variations in the tensile strength and solid fraction values than the tablets (23). This loss of reworkability is often
attributed to work hardening caused by the strengthening of particles due to the high volume of defects or dislocations in the crystal structure (13) (24).

Mechanical and physical properties of a tablet such as elastic properties, mechanical strength, and mass density are closely related to the unit operations employed in its production (e.g., roller-compaction and tableting). The variations of local densities in the ribbons lead to localized ribbon strengths and consequently resulting in the non-uniform size distribution and strength of the produced granules for a given milling operation. In addition, the variation of local density and strength in a ribbon can affect powder flow, mixing, compaction, and physical stability, which, in turn, alter the final dosage form performance. It has been discussed by many researchers that the physical and the mechanical properties of tablets are known to potentially influence the solid dosage form physical stability (due to mechanical defect generation over time), the accuracy of dosage (due to density distribution) and the shelf life (due to residual stresses and defects in the coat and core materials) (16) (25) (26). Therefore, to explore and characterize the density distributions in the roller-compacted ribbons and link this knowledge to the mechanical performance of the compacted pharmaceutical material (i.e., powder-to-tablet) would provide a means to characterize tablet stability, structural integrity and improve tableting quality.

Mechanical and physical property characterization of tablets using destructive (25) (26) (27) (28) (29) and non-destructive techniques (30) (31) (32) (33) has been extensively investigated over the years. However, relatively fewer methods have been dedicated to the investigation of density distribution in roller compacted ribbons and their impact on the performance of final pharmaceutical dosage forms using non-destructive techniques.
With the aid of μCT, it is possible to successfully examine the density variation in soft porous materials like pharmaceutical tablets. Busignies et al. (27) studied the localized density variations in tablets of the form of flat faced cylindrical compacts of microcrystalline cellulose by X-ray μCT. It has been demonstrated that higher density zones were found in peripheral region while lower density zones were located in the middle of the tablets. In a recent study, the local density variations in the roller- compacted ribbons using micro-indentation, X-ray μCT and sectioning methods have been investigated (34). It has been shown that due to the friction between the powder and the feed hopper walls, a higher density was obtained in the middle of the ribbon width when compared to those close to the edges of the ribbon. In this study, we have employed μCT techniques to investigate the variation in local densities in the roller compacted ribbons and by subsequent milling and compaction of these ribbons reveal how they drastically impact the elastic properties of the final dosage form for a given milling condition and particle size distribution. These results were corroborated by non-destructive ultrasonic technique which uses the principle of attenuation of acoustic wave (in the bandwidth of ultrasonic frequency).

2.2 Materials:

In the current study, microcrystalline cellulose MCC (Lot # P208819508, Avicel PH102, FMC Biopolymer, Newark, DE) and magnesium stearate (MgSt) (Lot # V18852, USP, Mallinckrodt, NJ) were employed as the test and the lubricant materials, respectively. The true density of the pure virgin MCC was measured by helium pycnometry (AccuPyc 1330, Micromeritics Instrument Corp., Norcross, GA) in triplicate using fresh samples
each time. In order to investigate the effect of lubrication on the density distribution in the ribbons, three cases were considered. In case 1 (hereafter referred to as C1), MCC was roller-compactd without lubricating the powder, rolls, feed screws (vertical and horizontal) and the feed hopper. In case 2 (hereafter referred to as C2), MCC was prepared by adding 0.5% w/w MgSt and blended for 15 min at 15 rpm (Patterson-Kelley, East Stroudsburg, PA). In case 3 (hereafter referred to as C3), rolls and feed screws were sparingly lubricated with MgSt powder suspended in methanol (5%, w/v). For each case, the masses of powders that was filled into the feed hopper were kept identical, i.e., 2970g of MCC for C1, 2955g of MCC + 15g of MgSt for C2, and 2970g of MCC for C3. All powder materials were used as received, with the exception of MgSt which was pre-screened through a 297µm (48 mesh) screen to minimize agglomeration. In the current study, all measurements were conducted at ambient conditions of 23±2 °C and 38±5% RH.

2.3. Methods

2.3.1. Ribbon preparation

Powders were roller compacted using an instrumented roller compactor (Fitzpatrick, IR520). Fluted rolls of 203mm in diameter and 50mm in width were used for roller compaction. The powders were fed into the compaction zone with vertical and horizontal feed screws through a feed hopper (Fig. 2.1). In all three cases, examined powders was compacted at a roll speed of 10rpm, vertical feed screw (VFS) speed of 100rpm, horizontal feed screw (HFS) speed of 20rpm, roll gap of 2mm and average roll pressures of 15.5MPa, 9.8MPa and 15.1MPa for Case-1, Case-2 and Case-3, respectively. The
ribbons were collected after the first minute of each run for 2 min. The ribbons produced at the beginning and at the end of the roller compaction were discarded. By adjusting the powder feed screw speed the thickness of the produced ribbon was kept approximately constant at 2mm (coefficient of variation (CV) of 3.6%). For the X-ray μCT, nondestructive ultrasonic testing and tableting, a series of 50 ribbons per each case were employed. Same ribbons were used throughout each experiment. The thickness, width, and length of the selected ribbons were measured by a digital caliper (±0.01 mm, Starrett B5000BZ-40/1000, Athol, MA) and the mass was recorded by an electronic balance (±0.1 mg, Fisher Scientific XD-800, Pittsburgh, PA) immediately after roller compaction. The width, thickness and length of the selected ribbons were 49±1mm, 2±0.2mm, and 27±1.1mm, respectively. It is observed that some of the collected ribbons have less width than the roll width (e.g., all Case-2 and some of the Case-1 ribbons). This is attributed to the leakage of loose powder at the ribbon edges which are relatively less compacted. To determine the solid fraction (SF) values of different locations of the ribbons, collected samples were sectioned into three segments of approximately 34mm×2mm×7.6mm as middle and 7.5mm×2mm×7.6mm (Width×Thickness×Length) as left and right edges. Solid fraction was then calculated from the following relationship:

\[
SF = \frac{\rho_e}{\rho_t}
\]

where \(\rho_t\) is the true density of the virgin MCC powder and \(\rho_e\) is the envelope density of each segment which is defined as the ratio of the segment mass to the segment apparent volume, including pores and cavities. The envelope density values of each segment were
measured using envelope density analyzer (GeoPyc 1360, Micromeritics, Georgia, USA). A 25.4 mm internal diameter tube was used for the envelope density measurement. For each ribbon segment, five replicate readings were carried out. A consolidation force of 51 N and a conversion factor of 0.5153 cm³/mm were used. It was observed that for each case SF values of each segment remained reasonably constant during the roller compaction process. The reported envelope density and SF data for each segment are a

The X-ray source was operated at a voltage (U) of 50 kV and a current (I) of 100 µA. The total sample rotation was set at 180° with an interval of 0.6° (i.e., the ribbon was scanned every 0.6°). The spatial resolution was 14.8 µm/pixel for the ribbons considered (i.e., 14.8 µm resolution size in the three directions after image reconstruction). A better effective resolution of the specimen is obtained by positioning it closer to the source, however at the cost of the reduction in field of view. Thus, a tradeoff has to be made between the desired resolution and the presence of the entire object within the field of view. Magnified transmitted projection pictures were detected by a two-dimensional X-ray CCD camera with 1024×1024 pixels resolution 12-bit dynamic range sensor. To minimize the beam hardening artifacts, an Aluminum-Copper plate with a 0.5 mm thickness was placed over the X-ray source to filter the low energy X-rays. Depending on the sample length typical image acquisition times were in the range of 20-25 min. The reconstruction was carried out with the NRecon software (Skyscan, Kontich, Belgium). Quantitative parameters such as thresholding were analyzed using CT-analyser (CTAn) software (Skyscan, Kontich, Belgium). 3D model rendering and viewing were performed using associate program CT-Volume (CTVol) software (Skyscan, Kontich, Belgium).
ImageJ software which is a public domain Java-based image processing program was employed to analyze all the images ([http://www.nih.gov](http://www.nih.gov)).

Table 2.1 Particle size distributions of the milled granules and segment solid fractions of the ribbons used

<table>
<thead>
<tr>
<th>Segment SF</th>
<th>Left Segment of the ribbon</th>
<th>Middle Segment of the ribbon</th>
<th>Right segment of the ribbon</th>
<th>Virgin MCC Powder</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C-1</td>
<td>C-2</td>
<td>C-3</td>
<td>C-1</td>
</tr>
<tr>
<td>Segment SF</td>
<td>0.53</td>
<td>0.45</td>
<td>0.63</td>
<td>0.70</td>
</tr>
<tr>
<td>Particle size dist. (µm) (D(v,0.5))</td>
<td>68.32</td>
<td>71.08</td>
<td>72.55</td>
<td>67.96</td>
</tr>
</tbody>
</table>

2.3.2 Tablet Compaction

Virgin MCC powder and roller compacted granules were compressed using a tableting emulator (Presster; Metropolitan Computing Corp., NJ) to simulate a Fette 2100 (47 stations) press with a press speed of 16.4rpm. A dwell time of 27ms (i.e., the time when the flat portion of punch head is in contact with the compaction roll), corresponding to a production speed of 42400 tablets/h, was used. The ejection angle was set at 5.3 degrees. No pre-compression was used. A set of flat-faced punches (10mm diameter) was used for compression. For mechanical property characterization of each case, a series of 30 tablets from the left, middle, and right segments of the ribbon were produced (hereafter referred to as \(L_i\)-tablet, \(M_i\)-tablet, and \(R_i\)-tablet, respectively, where \(i\) is the corresponding case number). For comparison purposes, using the same compaction settings, tablets were produced with unlubricated sieved virgin MCC powder, sieved virgin MCC powder
blended with MgSt at a 0.5% w/w level for 15 min at 15 rpm and unlubricated sieved virgin MCC powder using a lubricated die and punch surfaces with MgSt suspended in ethanol (5%, w/v). For each case, granules obtained from the left, middle, and right segments were also blended for 15 min at 15 rpm and compacted using the same compaction settings. The compaction profiles for each tablet were recorded. Each powder was compressed to a target solid fraction of 0.85 when possible, which is typical of pharmaceutical tablets (Hancock et al., 2003). The thickness and diameter of tablets were carefully measured by a digital caliper (±0.01 mm, Starrett B5000BZ-40/1000, Athol, MA) and the weight was recorded by an electronic balance (±0.1 mg, Fisher Scientific XD-800, Pittsburgh, PA) immediately after ejection. From these measurements, the volume and packing density of the tablets were determined (Table 2.2).

2.3.3 Analysis of Compression: Heckel analysis

Heckel analysis is the most frequently used method for studying the powder volume reduction process which is based on the assumption that the volume reduction of the powder during compression follows first-order kinetics in which the pores constitute the reactant (35). The kinetics of the process may be described as proportionality between the change in the density with pressure and the pore fraction. The relation was described by the following equation:

$$\ln \left[\frac{1}{1-D}\right] = KP + A$$

(5)

where $D$ is the relative density, $P$ is the applied pressure, $K$ and $A$ are constants that describe the ability of the compact to deform plastically and the ability of the particle movement and rearrangement at low pressures before inter-particulate bonding,
respectively. In the current study, out-of-die Heckel analysis was performed to determine the mean yield pressures of the powders. Out-of-die analysis can be characterized by two phases; (i) the initial curvature that is due to particle rearrangement (Heckel, 1961), particle fragmentation and a decrease in the total porous volume and (ii) linear phase that is attributed to densification and plastic deformation. The slope of the linear region is the Heckel constant ($K$). Hersey and Rees (36) demonstrated that the reciprocal of $K$ represents the mean yield pressure $P_y$ by the expression $P_y = 1/K$. For the out-of-die analysis and elastic recovery measurements, using the virgin powder, blends and granules were compacted in a compression pressure range of 15MPa-125MPa. The corresponding tableting data was evaluated in order to determine the mean yield pressure $P_y$ values.

### 2.3.4 Elastic recovery

The elastic recovery ($ER$) that describes the percentage of axial expansion of a compact is determined according to the following equation:

$$ER = \frac{h_0 - h_f}{h_f} \times 100$$

(6)

where $h_0$ is thickness of the tablet ‘out of die’ and $h_f$ is thickness of the tablet ‘in die’. $h_0$ was measured using a digital caliper (±0.01 mm, Starrett B5000BZ-40/1000, Athol, MA) and $h_f$ is the height of the tablet in the die at maximum pressure which is determined directly from the tableting emulator. All the tablets were tested and mechanically characterized on the same day to reduce effects of aging and ambient conditions on the measurements.
2.3.5. Image Processing

Due to the large volume of data and size of individual image data set, images were resized to reduce computer storage space and improve computational efficiency. Matlab 7.9b (The Mathworks, Natick, MA, USA) software was utilized for the following image processing steps. In the first step, individual images were read into 2D matrices and then stacked up to render 3D volume. Further reduction in data was achieved by cropping individual slices to eliminate most empty voxels. Since, in practice, the X-ray source is non-monoenergetic, despite the use of an Aluminum-Copper filter, it was observed that few beam hardening artifacts of relatively lower grayscale values remain. To prevent their contribution to the calculations, artifacts were eliminated by assigning a zero value for all voxels with values below a cutoff. The choice of this cutoff value was made, visually, by selecting the grayscale value at which almost all the artifacts just vanish. Figure 2.2 depicts the 3D volumetric regeneration of the sectioned ribbon with the characteristic dimensions of 49mm width, 2mm thickness, and 7.6mm length along the $x$, $y$ and $z$-axes. To compute the relative densities of the compacted ribbons a statistical approach has been used. Sample tablets of the same material as the ribbons (MCC) and same thickness as the ribbons were compacted in the tablet emulator in a compression pressure range of 15MPa-140MPa. Each of the tablets were scanned maintaining the exact same scan parameters (i.e., resolution, rotation step, voltage and intensity) as used for the ribbons (Fig. 2.4). The average grayscale value of each of the tablet and relative densities were computed. Fig 2.3 shows the plot of tablet relative density vs. average tablet grayscale value. In agreement with our presumption, the plot is strongly linear ($R^2$
Figure 2.2 Reconstructed X-ray micro-CT images of the actual roller-compacted ribbon with its perspective (a) and side (b) views. Piezoelectric transducers with diameter of 3mm are illustrated during a scan operating in pitch-catch mode (b). Dashed circles are corresponding to the ultrasonically scanned areas. The ribbon thickness is measured as 2±0.1mm.
We have used this plot as the basis of calibration to determine the relative densities of the compacted ribbons by interpolating mean grayscale values of the ribbons.

<table>
<thead>
<tr>
<th>Case No.</th>
<th>Segment</th>
<th>Mass density $\rho$ (g/cm$^3$)</th>
<th>Thickness $h$ (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Case-1</td>
<td>Left</td>
<td>1.32 (0.005)</td>
<td>4.05 (0.008)</td>
</tr>
<tr>
<td></td>
<td>Middle</td>
<td>1.31 (0.008)</td>
<td>4.06 (0.014)</td>
</tr>
<tr>
<td></td>
<td>Right</td>
<td>1.33 (0.002)</td>
<td>4.05 (0.009)</td>
</tr>
<tr>
<td>Case-2</td>
<td>Left</td>
<td>1.31 (0.006)</td>
<td>4.06 (0.014)</td>
</tr>
<tr>
<td></td>
<td>Middle</td>
<td>1.32 (0.009)</td>
<td>4.06 (0.017)</td>
</tr>
<tr>
<td></td>
<td>Right</td>
<td>1.31 (0.011)</td>
<td>4.06 (0.016)</td>
</tr>
<tr>
<td>Case-3</td>
<td>Left</td>
<td>1.31 (0.005)</td>
<td>4.05 (0.016)</td>
</tr>
<tr>
<td></td>
<td>Middle</td>
<td>1.31 (0.003)</td>
<td>4.06 (0.008)</td>
</tr>
<tr>
<td></td>
<td>Right</td>
<td>1.31 (0.007)</td>
<td>4.06 (0.011)</td>
</tr>
<tr>
<td><strong>Unlubricated</strong></td>
<td><strong>Virgin MCC Powder</strong></td>
<td>1.31 (0.012)</td>
<td>4.06 (0.009)</td>
</tr>
<tr>
<td><strong>Lubricated</strong></td>
<td><strong>Virgin MCC Powder</strong></td>
<td>1.32 (0.009)</td>
<td>4.05 (0.007)</td>
</tr>
<tr>
<td><strong>Virgin MCC Powder</strong> (Lubricated tooling)</td>
<td>1.31 (0.006)</td>
<td>4.06 (0.011)</td>
<td></td>
</tr>
<tr>
<td>Case-1 (Blend)</td>
<td>1.32 (0.010)</td>
<td>4.06 (0.012)</td>
<td></td>
</tr>
<tr>
<td>Case-2 (Blend)</td>
<td>1.31 (0.012)</td>
<td>4.06 (0.008)</td>
<td></td>
</tr>
<tr>
<td>Case-3 (Blend)</td>
<td>1.32 (0.007)</td>
<td>4.05 (0.005)</td>
<td></td>
</tr>
</tbody>
</table>

**Table 2.2** Summary of the physical properties of the tablets and roller-compactcd ribbons. Standard deviation in estimate of MPa is included in parenthesis. Tablet solid fraction of 0.85
2.3.6 Measurements and calculations

Data measurements for both methods (X-ray µCT and nondestructive ultrasonics) were taken at discrete sections on the specimen, spanning the entire sample. The selected points for X-ray µCT method were accurately marked for nondestructive ultrasonic data collection. Same data points were used for both nondestructive measurements. Each section of inspection is a representative cylinder with a diameter equal to that of the piezoelectric transducer (i.e., 3mm) with its axis along the thickness (y-direction). Measurements were taken at 30 equidistant inspection points so as to form a rectangular matrix with 3 rows (along the length, z-direction) and 10 columns (along the width, x-direction). The computed mean CT value for all voxels within a cylinder is the representative of the density for that particular section. A comparative study of these mean CT values of each of these sections reflects the local density variation of the ribbon.

![Graph showing the correlation between relative densities and gray scale values](image)

**Figure 2.3** Correlation between relative densities of the calibration tablets made with different compression pressures and the grey scale values.
Figure 2.4 X-ray micro-CT images of the calibration tablets compacted in a compression pressure range of 15MPa-140MPa.
Figure 2.5 Density distribution across the width of the ribbon for Case-1 (a), Case-2 (b), and Case-3 (c) obtained from X-ray micro-CT.
2.4 Results and discussions

2.4.1 Heterogeneous density distribution in a roller-compacted ribbon

X-ray micro-computed tomography techniques to determine the local density variations in the roller-compacted ribbons were utilized. Close examination of X-ray µ-CT images for C1 ribbon reveals that there is an intensive high density region in the middle of the ribbon width and length and relatively lower density regions at the left and right edges (Fig. 2.5). This indicates that the powder in the middle was compressed more than that close to the edges which leads to a relatively high density zone at the middle and low density region at the edges of the ribbon and is attributable to the presence of friction (absence of lubrication) at the feed zone between the powder and side walls (Fig 2.5). The side wall friction inhibits the powder at the edges from moving downwards as it is gripped into the nip region; consequently the powder at the edges is less compressed while powder at the middle is highly compressed. These results are also confirmed by ultrasonic testing where the Times of Flight were correspondingly in the reverse order. However variations in density in the case of lubricated powder (C2), and in the case of unlubricated powder with lubricated rolls and feed screws (C3) are much less observable and the ribbons are compacted more homogeneously. Due to the minimized effect of the particle-particle and particle-side wall friction caused by the reduction of inter-particulate bonds by the presence of MgSt, flowability of MCC increases and compaction pressure decreases. In the case C3, powder to tooling friction is minimized despite the existence of inter-particulate friction. Thus, even though flowability is reduced, the powder still undergoes a more uniform compaction.
2.4.2 Heckel Analysis and tablet Mechanical Properties

The ability of a material to undergo plastic deformation is often defined by the mean yield pressure \( P_y \) which is inversely proportional to the Heckel constant \( (K) \). In order to determine the compression characteristics of powders and the roller-compacted granules, the compression data were fitted according to the Heckel equation. To estimate the mean yield pressure \( (P_y) \) values of the materials out-of-die analysis was performed. For the sake of reference, we have included Young’s modulus values measured from the non-destructive ultrasonic technique (33), which was another associated independent study performed on the same samples. Values of \( P_y \) recorded in Table 2.3 are plotted in Fig. 2.7 as a function of the solid fraction values. In general tablets prepared with granules made from the left and right segments of the ribbon exhibit lower \( P_y \) values in comparison with the tablets prepared from the middle of the ribbon and indicate that the granules obtained from the left and right segments in exhibit faster onset of plastic deformation during tableting compared to the middle segment. These results are however more pronounced in case of C1. C2 has comparatively modest increase in yield pressure \( P_y \) and are as a consequence of lesser plastic deformation due to the presence of MgSt film which reduces intergranular bonding. In the case of C3 granules display plastic deformation with a small increase in \( P_y \) and indicate work hardening. Thus, it is evident that granules produced from sections of higher density (i.e. higher SF) have undergone work hardening, and hence are difficult to plastically deform any further. This alters the compression behavior of the powder bed and results in significantly lower tablet strengths.
2.4.3 Elastic recovery

The elastic recovery (ER) is useful as a measure of the disruptive effects of elastic deformation, in other words, it provides an insight into the elastic deformation undergone by the powder bed during compression. Elastic recovery occurs during decompression phase and results in the rupture of interparticulate bonds formed during compression. Excessive elastic recovery after compression could strongly reduce the intergranular bonding which could cause a drastic decrease in tablet mechanical strength. In Fig. 2.6, for C1 tablets, it is shown that the differences between the ER values of L_1-, M_1-, and R_1-tablets become more pronounced above a compression of 40MPa suggesting that the high tendency of M_1-tablets to recover elastically. This large elastic recovery of M_1-tablets contributes to weaker inter-granular bonding and lower tensile strength values as tabulated in Table 2.3. Relatively higher values of the mean yield pressure of granules obtained from the middle segment also support the increase in the ER values for M_1-tablets since higher the mean yield pressure the more difficult onset of plastic deformation during compression. In addition, at higher compression pressures the still slight increase and decrease of the ER values of the C1 tablets indicates that the reorganization of the material was not totally completed. In contrast, ER values of C2 and C3 tablets were relatively stable above a compression pressure of 65MPa. Close examination of the ER values reveals that C2 tablets axially expand to a larger extent than those of C1 and C3 tablets. This could be attributed to the incorporation of MgSt into the MCC powder which forms a film of MgSt on the surface of the granule. Due to the presence of this film the intergranular bonding may significantly reduce and this can result in the accumulation of the stored elastic strain within the granules that will be
released upon the removal of the rigid constraints (*i.e.*, upper punch and die wall). This mechanism can act like a loaded-spring and hence cause an elastic spring-back as the compaction force is relaxed during decompression and ejection phases which will weaken the tablet mechanical strength.

Table 2.3 Summary of the elastic properties, tensile strength and mean yield pressures of the tablets. Standard deviation in estimate of GPa (for E) and MPa (for $\sigma$ and $P_y$) is included in parenthesis. Tablet solid fraction of 0.85.

<table>
<thead>
<tr>
<th>Case No.</th>
<th>Segment</th>
<th>Young’s Modulus E (GPa)</th>
<th>Mean Yield Pressure $P_y$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Segment solid fraction</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Case-1</td>
<td>Left</td>
<td>0.53</td>
<td>2.67 (0.21)</td>
</tr>
<tr>
<td></td>
<td>Middle</td>
<td>0.70</td>
<td>1.28 (0.09)</td>
</tr>
<tr>
<td></td>
<td>Right</td>
<td>0.57</td>
<td>2.34 (0.16)</td>
</tr>
<tr>
<td>Case-2</td>
<td>Left</td>
<td>0.45</td>
<td>0.84 (0.04)</td>
</tr>
<tr>
<td></td>
<td>Middle</td>
<td>0.48</td>
<td>0.82 (0.08)</td>
</tr>
<tr>
<td></td>
<td>Right</td>
<td>0.46</td>
<td>0.80 (0.07)</td>
</tr>
<tr>
<td>Case-3</td>
<td>Left</td>
<td>0.63</td>
<td>1.71 (0.17)</td>
</tr>
<tr>
<td></td>
<td>Middle</td>
<td>0.66</td>
<td>1.69 (0.08)</td>
</tr>
<tr>
<td></td>
<td>Right</td>
<td>0.65</td>
<td>1.66 (0.15)</td>
</tr>
<tr>
<td>Unlubricated Virgin MCC Powder</td>
<td>n/a</td>
<td>3.78 (0.13)</td>
<td>84.33 (3.6)</td>
</tr>
<tr>
<td>Lubricated Virgin MCC Powder</td>
<td>n/a</td>
<td>3.54 (0.11)</td>
<td>81.37 (2.1)</td>
</tr>
<tr>
<td>Virgin MCC Powder (Lubricated tooling)</td>
<td>n/a</td>
<td>3.66 (0.14)</td>
<td>83.66 (2.4)</td>
</tr>
<tr>
<td>Case-1 (Blend)</td>
<td>n/a</td>
<td>1.52 (0.13)</td>
<td>97.54 (1.9)</td>
</tr>
<tr>
<td>Case-2 (Blend)</td>
<td>n/a</td>
<td>0.81 (0.07)</td>
<td>91.23 (2.4)</td>
</tr>
<tr>
<td>Case-3 (Blend)</td>
<td>n/a</td>
<td>1.69 (0.09)</td>
<td>94.36 (2.7)</td>
</tr>
</tbody>
</table>
Figure 2.6. Elastic recovery profiles for Case-1 (a), Case-2 (b), and Case-3 (c) tablets prepared from the left, middle, and right of the ribbons.
Figure 2.7 Yield pressure and Young’s modulus of compacted tablets of for each of the three cases as a function of ribbon solid fraction.
2.5 Conclusions

We observed that the roller compaction granulation process produced ribbons with localized density variations. These local density variations determined the localized ribbon strengths and the size distribution of the produced granules for a given milling operation. This, in turn altered the mechanical performance of the final dosage form. In this study, we evaluated the density distributions in the ribbons of microcrystalline cellulose and the impact of these variations on the elastic properties of the compacted solid dosage forms using nondestructive X-ray micro-computed tomography techniques.

Three different cases for roller compaction were considered; Case (1) no lubrication, Case (2) lubricated powder, and Case (3) lubricated feed hopper, feed screws, and rolls. µCT captured the differences in density at various segments of the test ribbon, namely, left, middle and right segments; substantial difference in the localized ribbon density along the width and length of the ribbon was found. For the unlubricated case, high density regions were found at the middle of the ribbon while relatively low density regions at the edges. For lubricated powder and lubricated tooling cases, more homogenous ribbon density was obtained. Subsequent milling and compaction of these ribbons revealed that variation in local densities in the ribbons drastically affects the elastic properties of the final tablet for a given milling condition and particle size distribution. In addition, out-of-die Heckel analysis and elastic recovery of these tablets were also studied. These results were corroborated by non-destructive ultrasonic methods.
Chapter 3

Characterization of Metallic Cellular Materials

3.1 Metallic Foams

Cellular solids are formed by repetitive arrangement of cells made up of an inter-connected network of solid struts (open cell) or plates (closed cell) (35). They occur commonly in many natural systems such as wood, cork, bones, honeycomb, coral etc. and man-made substances such as cakes, packaging materials, ships, aircrafts etc.

![Examples of cellular solids](image)

**Figure 3.1** Examples of cellular solids a) open cell polyurethane, b) closed cell polyethylene, c) nickel, d) copper, e) zirconia f) mullite g) glass h) polyether with both open and closed cells (35)
They have widespread applications due to its physical and mechanical properties such as a) Thermal insulation (disposable coffee cups, building, rocket boosters) b) Packaging (polystyrene, polyethylene wraps and sponges), c) Structural (sandwich panels) d) Buoyancy (fishing boats) and e) Others (filtration, vibration damping, dielectric properties, heat exchangers, catalysts).

Figure 3.2 Application of cellular materials grouped according to the degree of "openness" needed and whether the application is more functional or structural (36)

Metallic foams are manufactured in a variety of methods which broadly fall into one the following techniques depending on the state the material is in viz. Metal vapor (vapor deposition), Liquid metal (direct foaming with gas/blowing agents, spray forming, casting etc.), Powdered metal (sintering hollow spheres, gas entrapment, slurry foaming, extrusion, sintering of powder etc) and Metal ions (electrochemical deposition) (36).

Apart from the degree to which cells are open or closed, a distinction resulting in inherently different characteristics of the foams, the most important structural
characteristic of a cellular solid is its relative density, $\rho^*/\rho_s$ (the density, $\rho^*$, of the foam divided by that of the solid of which it is made, $\rho_s$) (35). Other characteristics affecting the mechanical properties of a foam are, its porosity, which is the fraction of its pore space, or simply, $(1 - \rho^*/\rho_s)$, and, the cell shape, as opposed to the cell size. The directional properties are controlled by the shape anisotropy ratios $R_{12}$ and $R_{13}$. Equiaxed cells, however, result in isotropic properties of the foams. Crucial cell wall properties include the solid density, $\rho_s$, Young’s modulus, $E_s$, yield strength $\sigma_{ys}$, fracture strength, $\sigma_{fs}$, and the creep parameters (37). Non material properties influencing the mechanical characteristics of the foam include the strain-rate, temperature, anisotropy and multi-axial loading. Compressive deformation mechanisms have been well characterized in polymeric foams. Metallic foams have recently been studied extensively due to their potential applications. Deformation mechanisms in foam structures are associated with specific foam structures and cell morphologies which themselves are depend on fabrication methods. The macroscopic stress/strain behavior of open-cell foam is shown below (Fig 3.3):
Figure 3.3 Macroscopic Stress vs. Strain response of an open-cell Aluminum foam

At low stresses they exhibit linear elastic behavior, followed by a plateau region, truncated by a regime of steep rise in stress, resulting from densification (35). The relationship between the elastic moduli to the bending stiffness of cell walls (in case of closed cell) or struts (for open cell) has been established. Expressions for the moduli \( E^* \) and strengths of foams \( \sigma_{pl}^* \) as functions of their relative density \( \rho^*/\rho_s \) have been obtained using dimensional analysis (37) and are given by:

\[
\frac{E^*}{E_s} = C_1 \left( \frac{\rho^*}{\rho_s} \right)^2 \quad \frac{\sigma_{pl}^*}{\sigma_{ys}} = C_2 \left( \frac{\rho^*}{\rho_s} \right)^{3/2}
\]

where \( E_s, \sigma_{ys} \) are the Young’s modulus and strength of the solid cell wall material. \( C_1, C_2 \) are constants related to the cell geometry.

The stiffness exhibited by commercially available metallic foams is considerably lower than ideal foam. Stiffness knockdown factors typically range between 2 to 50 (35) (38) and reasons are commonly attributed to morphological defects (39) (40). These microstructural morphological defects were observed to primarily cause the bending rather than stretching of the cell walls (41). The significant degrading morphological effects include curved or wrinkled cell walls (40) (39) (42) and cell shape as opposed to size (43) at relatively high volume fractions. There is some inconsistency in the reporting of failure mechanisms in closed-cell foams. While Thornton and Magee (44) observed uniform collapse of cells over the entire sample, Bart-Smith et al (45) reported heterogeneous deformation by the initiation and propagation of deformation bands, approximately one cell thick, at levels much below the peak stress (on the order of 1/3). With the elevation of stress, their number density increases, followed by a plastic
collapse. Multiple adjacent or isolated band formations are observed until densification is attained. Previous work has been done to visualize the plastic response of cellular materials by surface deformation measurements, using Digital Image Correlation (46) (47), as well as X-Ray computed tomography (48), (49).

To monitor such macroscopic phenomena as well as evolution of mechanisms at cell level, most theoretical strategies are corroborated by experimental techniques. Surface deformation has been measured by optical microscopy the Digital Image Correlation technique is utilized. Complemented by X-Ray Micro Tomography, its application is extended to compute and visualize deformation in the complete volume. In this study we have applied these techniques to estimate average deformation in the volume. Assuming strains to be mostly planar, we have correlated 2D average extracted slices close to and below the free surface. While DIC using high speed photography of the surface undergoing deformation has been studied in detail (46) (47), our knowledge about the evolution of strain in the volume is very limited. Given that the constraints at the interior are different those at the surface, the surface strain map may or may not be representative of the mechanics at the core. It would hence be interesting to investigate the extent of cell collapse and the transition from the exterior to the interior. To our knowledge a study of how the strain field varies at the free surface from that in the core has never been done before. At the first stage we make an attempt to achieve this aim by assuming strains to be planar and apply Digital Image Correlation methods to the cross-sectional layers extracted from the volumetric μCT data to measure the strain maps in the volume. As we shall see in the results that follow that this methodology is very effective. However, it would still fail to capture the 3D volumetric displacements because
of our assumption above. An expansion of this methodology would be to develop full field 3D DIC procedure (Chapter 4).

3.2 Materials

The metallic foam specimens used in this work were Duocel 6101 – T6 aluminum alloy foam with open-cell architecture (ERG Materials and Aerospace, Oakland CA). The specimens had 6% relative density. The average cell size is 40 pores per inch (ppi) which implies the mean cell sizes are 0.508mm. The cells are, for all practical purposes, equiaxed. Small samples of dimensions 20x20x17.5 mm$^3$ have been cut from company provided samples of dimensions 25.5x25.5x38 mm$^3$ for all experiments presented in this work. The macroscopic response of the material is approximately isotropic. Therefore it can be expected that deformation mechanisms will be similar in foams with different structures and cell morphologies despite similar loading conditions. Hence the stimulus to study the elastic-plastic deformation in metallic foams (50).

3.3 Methods

3.3.1 In situ axial compression set-up

The uniaxial compression tests were conducted using a specifically built in-situ set-up that allowed being placed in the X-ray permitting the foam samples to be scanned under load. The entire set-up is made of extruded acrylic cylinder (A), $\rho=1162$ kg/m$^3$, (McMaster-Carr) and is extremely transparent to X-Rays. The cylinder (OD=2”, ID=1.75”, length 1.5”) is sealed on both ends with fixed (stationary platen) and removable circular discs (C and B) made of the same material and is very similar to conventional systems. A thumb screw (E) (5/16”-18) attached on the removable circular
disc (B) transmits fixed displacement to foam via a circular Teflon (Virgin Electrical Grade PTFE) platen (D), \( \rho = 357 \text{ kg/m}^3 \). Teflon imparts very low friction to both, the foam as well as the fluted end of the screw. Axial strain and thereby the deviator stress is applied using the manually driven thumb screw with a pitch of \( 1/18'' \) (1.41mm). Incremental strains resulting from a quarter revolution (0.353 mm axial displacement) were applied to the specimens in between each step of tomographic data acquisition. The dimensions of the apparatus had to be selected in order to be accommodated within the limited space of the Micro-CT setup and permitted manual vertical compression only.

![Schematic and actual photo of the in-situ compression setup](image)

**Figure 3.4** Schematic and actual photo of the in-situ compression setup

### 3.3.2 Data Acquisition

For this study, X-ray µCT measurements were carried out on a high resolution SkyScan-1172 XRCT (SkyScan, Kontich, Belgium). X-Ray source power was set
corresponding to 60kV voltage and 148\(\mu\)A current. This setting was maintained for each progressive step of deformation. Projection images were acquired at incremental rotation steps of 0.6°. Image resolution and camera pixel settings were 34.9 microns and (1k x 0.5k) respectively. That is, each captured set of tomographic data consisted of a vertical stack of 500 square horizontal images of side 1000 pixels each of dimension 34.9 microns. Beam hardening artefacts were reduced by using a 0.5 mm Aluminum plate. Standard image acquisition times were in the order of 11 minutes.

### 3.3.3 Image Processing

Images were reconstructed using compatible NRecon software (Skyscan, Kontich, Belgium). CT-Analyser (CTAn) software (Skyscan, Kontich, Belgium) was used to derive quantitative parameters and constructing 2-D and 3-D visual models of the specimens. It uses simple global or adaptive methods for processes like thresholding and segmentation. To view and manipulate 3-D surface rendered models the associate program CT-Volume (CTVol) software (Skyscan, Kontich, Belgium) was used. It is possible to visualize tangible aspects of the 3-D structure of the object. Data manipulation is performed using Matlab 7.9b (The Mathworks, Natick, MA, USA) software. Collective reconstructed data is read into 2-D matrices and stacked up to render 3D volumetric data. Further processing of the images using Image Correlation techniques require the capture of deformation features in the 2D images.
Figure 3.5 Shadow Projection Image of a foam and extracted reconstructed slices at three z-locations (px-150,300 and 450) corresponding to (5.25, 10.50 and 15.60 mm) from the lower platen.
Figure 3.6 Illustration indicating process of volumetric data acquisition for a sample Al foam undergoing progressive deformation between scans from undeformed to densified states. Foams are 3D stl visualizations from acquired data. Deformation is expressed as engineering strain.
However, vertical compression only captures images of the planes perpendicular to the deformation plane. The 3D volumetric data is rotated by 90° and 2D BMP images are extracted from the volume to render visualization of the deformation planes. Blank voxels (voxels not occupied by specimen material) are cropped to reduce computer storage space and increase computational efficiency. Extracted images are thereafter processed using image matching technique known as Digital Image Correlation. As described later in Section 3.3.3.1, DIC requires a random intensity distribution in the images in order to converge successfully. Often, speckle patterns, are artificially introduced by spraying contrasting paint on the sample surface to create randomness of the distribution. Large regions of uniformity hinder convergence or lead to wrong results. In case of a foam sample, because of the inherent randomness in the occurrence of the material in space, our task is easier. Because of the lack of sufficient features, attempts to match two consecutive extracted images from the volume before and after applied deformation, each one slice thick (34.9µ), fail. An average image, extracted by projecting 150 consecutive slices (5.235mm) thickness, at each progressive step of deformation, provides just sufficient features for the routine to converge. Posts processing of data from DIC, plotting and graphical visualizations have been done in Matlab. 3D volumetric generations as well as rendering have been done in Matlab as well as Tecplot 360 (Tecplot, Inc., Bellevue, WA, USA).
3.3.3.1 Measurement Approach: Digital Image Correlation (DIC)

DIC is an experimental, computer vision based, non-contact full field strain measurement technique. It is a “fuzzy” measuring method that works on the principle of comparing one digital image of the displaced/deformed surface with the original one using a mathematically well defined correlation function over a (i) correlation window of the digital image (Local DIC) or (ii) using the entire image or its subsection (Global DIC).

First proposed in 1982, as automated approach for computing surfaces strains and displacements (51) (52), it was thereafter formulated in six-dimensional space (two rigid displacements $U$, $V$, and four deformation gradients $U_x$, $U_y$, $V_x$, $V_y$ representing...
deformation by a first-order Taylor series approximation) in order to study 2D experimental solid mechanics. The coefficient was optimized using a “two parameter” (search for two parameters at each iteration while keeping the rest constant) iterative technique (52). Discrete image information was rendered continuous by interpolation (bilinear (52) or bicubic (53)) techniques to obtain continuous data and subpixel accuracy. Newton-Raphson’s derivative based iterative scheme was applied (53) to DIC in 1989 to enhance the computational efficiency. However, to overcome its drawbacks of sensitivity to initial values and local extrema a coarse-fine multi-grid search strategy was adopted (53). This multi grid approach not only added robustness to the search, but was also capable of conducting searches in higher dimensional spaces. Lu and Cary (54) introduced seven new parameters \( U_{xx}, U_{xy}, U_{yy}, V_{xx}, V_{xy}, V_{yy} \) including one for the grayscale value offset) to the formulation (54). This increased the accuracy of strain measurements in case of inhomogeneous and large deformation situations.

Despite its general purpose of surface strain measurement tool, its applications have expanded its versatility due to its attributes of adjustable spatial resolution, measurements at various length scales from macroscopic to microscopic levels, and extreme experimental environments. It has the merits of not requiring any surface preparation or a complicated optical arrangement. In fact DIC has the advantage of applicability to images captured by a high speed camera, SEM (55), STM or X-Ray CT (49) to measure not only the surface deformation but also in the volumetric sense.

In our work, we have used the Local DIC method, in the sense of correlating one pair of the windows in the original and deformed images, respectively, and averaging the displacement field over the correlating windows through a specific interpolation routine,
to compute the average in-plane displacement field for representative image for a small thickness of the sample foam. In this case, the whole field displacement measurement, continuous in most cases, is achieved pixel-wise, and is really a local thing. Only the central point (node) of the correlation window, with the help of its neighboring points, is considered. However for the extension of DIC to 3D measurement, we have chosen the Global approach wherein we incorporate the assembling technique of *Finite Element Method*. In this case correlation is achieved by multi-variable global optimization.

3.3.3.2 Formulation of DIC:

DIC technique analyses a pair of digital images (planar or volumetric) prior to, and after application of incremental deformation. Information is contained in the images in the form of distribution of gray values, denoted by $F(X)$ for the undeformed image, and $f(x)$ for the deformed one in terms of their respective local coordinate systems $X$ and $x$. The *basic assumption*, that holds almost true in most DIC’s application, is that the field characterizing the object, such as, composition, transparency, reflectivity and, in the case of X-Ray Tomography, the local density, at each spatial point is preserved by the application of deformation field, thereby maintaining a one to one correspondence between the gray distribution on the image corresponding to the deformed and undeformed states. Thus DIC can be looked upon as an unconstrained optimization problem where, by means of optimizing a merit function, a field pair is correlated to solve for the deformation relationship between them (56).
3.3.3.3 Merit function:

A least square type cross-correlation merit function, originally proposed by Knauss and Vendroux (57), and implemented by Wang and Cuitiño (56) is defined as

\[
\chi^2 = C[\mathbf{x}(\mathbf{X})] = \frac{\int_{V} \left( \frac{f[\mathbf{x}(\mathbf{X})] - F(\mathbf{X})}{\sigma(\mathbf{X})} \right)^2 d^3x}{\int_{V} F^2(\mathbf{X})d^3x}
\]

where the integration is performed over the material volume \( V \), the standard deviation of the fields at each point, and the denominator also remains constant for a specified volume. Removing both the terms from the equation, the expression for the merit function is reduced to

\[
\chi^2 = C[\mathbf{x}(\mathbf{X})] = \int_{V} (f[\mathbf{x}(\mathbf{X})] - F(\mathbf{X}))^2 d^3x
\]

The optimizer is efficiently achieved using a derivative-based iterative procedure that requires the computation of first (Jacobian) and second (Hessian) order derivatives for a given interpolation scheme.

3.3.3.4 Local DIC:

Local DIC mathematically translates, rotates and deforms the reference subimage centered on the nodes distributed on the field of interest till the deformed subimage is obtained. The displacement in the correlation window (subimage), is interpolated based on information at its central point ‘c’. The Eulerian \( \mathbf{x}(\mathbf{X}_p) \) coordinates in terms of Lagrangian coordinates \( \mathbf{X}_p = \mathbf{X}_c + \Delta \mathbf{X}_p \) is,

\[
\mathbf{x}(\mathbf{X}_p) = \mathbf{x}_c + \mathbf{u}(\mathbf{x}_c) + \left[ \frac{\partial \mathbf{u}}{\partial \mathbf{x}} \right]_{\mathbf{x}_c} \Delta \mathbf{X}_p
\]
where ‘p’ represents the neighboring points around ‘c’. The displacement u(Xc) has three components along three dimensions and its spatial gradient matrix has nine components.

Denoting the vector p as
\[
p = \left[ u_c \ v_c \ w_c \ u_{c,x} \ u_{c,y} \ u_{c,z} \ v_{c,x} \ v_{c,y} \ v_{c,z} \ w_{c,x} \ w_{c,y} \ w_{c,z} \right]^T
\]
the local mapping function is now written as
\[
x(X) = x(X_c, \Delta X, p)
\]
The merit function thus degenerates to
\[
\chi^2 = C(p) = \int_{v_{\text{window}}} \left\{ f[x(X_c, \Delta X, p)] - F(X_c, \Delta X)^2 \right\} d^3 \Delta X
\]
which is minimized by the Newton-Raphson iterative formula
\[
p_{n+1} = p_n - H_n^{-1} J_n
\]
The first derivative (Jacobian) J and the simplified second derivative (Hessian) H are evaluated as (56)
\[
J = \frac{\partial \chi^2}{\partial p} = \frac{\partial C(p)}{\partial p} = 2\left\{ f[x(X_c, \Delta X, p)] - F(X_c, \Delta X)^2 \right\} \frac{\partial f[x(X_c, \Delta X, p)]}{\partial p} d^3 \Delta X
\]
\[
H = \int_{v_{\text{window}}} 2 \left( \frac{\partial f[x(X_c, \Delta X, p)]}{\partial p} \otimes \frac{\partial f[x(X_c, \Delta X, p)]}{\partial p} \right) d^3 \Delta X
\]
Despite being formulated as an unconstrained optimization problem, in reality the mapping function x(X) is bounded by the requirement of satisfaction of continuity condition. Except for the case of appearance of cracks, continuity helps to interpolate the values at neighboring points based on given local information.
3.3.4 Finite Strain Calculations

In summary, the result of minimizing the Local Digital Image Correlation coefficient, essentially, is to computationally measure the displacement field $u$ by mathematically mapping $x$ onto $X$ and is given by their difference. The partial differential of the displacement vector with respect to the spatial co-ordinates yields the Lagrangian displacement gradient tensor. Thus,

$$ u = x - X $$

$$ \nabla_x u = I - \frac{\partial X}{\partial x} = I - H $$

where $H$ is the spatial deformation gradient tensor $\frac{\partial x}{\partial x}$ which is the inverse of the material deformation gradient tensor $F = \frac{\partial x}{\partial x}$ (58). Deformation gradient in material coordinates is then computed by inverting

$$ F^{-1} = H = I - \nabla_x u $$

The right stretch tensor, resulting from the polar decomposition of the deformation gradient $F$ is expressed in the form

$$ U = C^{1/2} = (F^T F)^{1/2} $$

$C$, a positive definite symmetric tensor is the right Cauchy strain tensor, and is a measure of deformation at a material point. Biot ($E_B$) and Green-Lagrange ($E_{GL}$) strain tensors are calculated directly from $U$ and are given by (59):

$$ E_B = U - I \quad E_{GL} = U^2 - I $$

$U$ is a symmetric tensor and is equivalent to the local engineering strain (60).
3.4 Results and Discussions

Displacement fields generated by the minimization routine are obtained for each point on a regular rectangular mesh. As a first step to the visualization, the data is refined by applying a smoothing spline routine, on the Matlab (7.9.0) software, which returns a B-form (basis spline) function for the noisy displacement data. Fig 3.8 depicts the vector plot of the displacements within the correlation window, which itself is a subset of the region of the image containing the foam. Displacements along the direction of compression are plotted at the nodal points of the mesh. Correlation for all compression steps is performed on a fixed windows size, corresponding to the initial size of the foam. However, calculations at each step, for the displacement and strain fields are made taking into account, only the regions encompassing the foam. Obviously, the number of nodal points, at each progressive step varies linearly with the size of the foam is reduced at each step of compression. The direction of applied deformation is vertical and from the top towards the bottom. The bottom end of the window is adjacent to the lower platen and is held stationary at all times. Displacement data is generated for multiple layers in order to compare the deformation mechanics at the surface and the interior of the foam. A reduction in the size of the displacement vector from the top to the bottom indicates a greater displacement of points on the top compared to the ones on their below. This indicates a downward compression. Our observations for all the three layers at 25, 150 and 300 pixels are similar and consistent with the applied deformation. Also consistent with our applied boundary condition, points adjacent to the stationary lower platen show negligible displacement.
Figure 3.8b Displacement fields for layer 150 (150 px/5.23mm from the surface) at 4.2%, 14.7%, 25.2% and 37.8% applied engineering strain respectively. Both axes represent pixel count.
Figure 3.8c Displacement fields for layer 300 (300 px/10.47mm from the surface) at 4.2%, 14.7%, 25.2% and 37.8% applied engineering strain respectively. Both axes represent pixel count.
Figure 3.8a Displacement fields for layer 25 (25 px/872 μ from the surface) at 4.2%, 14.7%, 25.2% and 37.8% applied engineering strain respectively. Both axes represent pixel count.
Fig 3.9 shows the contour plot of the strains at the same layers for at 4.2%, 14.7%, 25.2% and 37.8% applied engineering strain during the compression experiment and gives more insight into the micromechanics. The contours clearly reflect the heterogeneity in the deformation process of the foam. At 4% applied engineering strain, when the deformation is still in the elastic regime, the response is mostly homogeneous and the foam behavior is similar to a regular solid material. Localized bands start appearing at 8% applied strain. Despite negligible strains in some regions of the volume, deformations in the existing bands intensify with progressive deformation, while new bands are formed elsewhere in the volume. Differences are noticed in the appearance of the band as we investigate layers at the surface and at the core. Layers closer to the free surface indicate the appearance of the band earlier than the ones at the core. Though not clearly perceptible in a contour map, they can be better understood by the study of deformation histograms (discussed later).
Figure 3.9a Biot Strain fields contour for layer 25 (25 px/872 μ from the surface) at 4.2%, 14.7%, 25.2% and 37.8% applied engineering strain respectively. Both axes represent pixel count. Legend shows values in compressive strain.
Figure 3.9b  Biot Strain fields contour for layer 150 (150 px/ 5.23mm from the surface) at 4.2%, 14.7%, 25.2% and 37.8% applied engineering strain respectively. Both axes represent pixel count. Legend shows values in compressive strain.
Figure 3.9c Biot Strain fields contour for layer 300 (300 px/10.47mm from the surface) at 4.2%, 14.7%, 25.2% and 37.8% applied engineering strain respectively. Both axes represent pixel count. Legend shows values in compressive strain.
To verify the accuracy of our measurement we need to compare our computed values with some known/measurable quantity. In our case the obvious quantity to compare would be the applied or the engineering strain. We have computed the average Biot strains in the volume and plotted it with the actual applied strains on Fig 3.9. Since Biot strains are representatives of the applied strains, the values should and do coincide closely to the applied strains. Points are fitted with a linear curve and the closeness of fit measured by the correlation coefficient ($R^2$ values) is given in the Table 3.1 below. Fig. 3.12a, b, c show the values at three different slices at 25,150 and 300 pixels from the surface. The slope of the straight line fit very closely matches with the applied engineering strain of 2.1% at each progressive step. The values of average strains match closely to the applied value at the surface and seem to diminish by a little as we progress to the interior.

<table>
<thead>
<tr>
<th>Layer</th>
<th>Slope (Avg Biot Strain) (%)</th>
<th>Correlation Coeff ($R^2$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>25</td>
<td>-2.188</td>
<td>.9988</td>
</tr>
<tr>
<td>150</td>
<td>-2.017</td>
<td>.9859</td>
</tr>
<tr>
<td>300</td>
<td>-1.913</td>
<td>.9901</td>
</tr>
</tbody>
</table>

Table 3.1
Figure 3.10 Average Biot strain value plot as a function of time step for layers a) 25 b) 150 and c) 300 pixels from the surface. Y axis represents Strain
Figs 3.11 show the histogram distributions of the strains for each of the three abovementioned layers, at progressive deformation steps. In an ideal case of homogeneous deformation, we would expect all the nodal points to strain equally and simultaneously, thus exhibiting a Dirac-delta type distribution. Conversely, for a heterogeneous deformation we would witness a flatter histogram. In our case the deformation begins nearly homogeneously, and with time becomes heterogeneous evident from the flattening of the histograms. Further, a leftward shift of the bins confirms an increase in average strain. Appearance of isolated peaks indicates regions undergoing high strains and hence corresponds to localized band formations. Differences in the histogram patterns are explicit as we move from the surface to the core and become more pronounced as the applied strain increases.

Histograms at a given layer at a particular strain step (25px, 150px, 300px from the surface), invariably, are flatter, the closer they are to the surface. Layers closer to the surface also exhibit more peaks than the others, causing the distribution to appear more multimodal. This implies the creation of more deformation bands at the surface. All the above observations indicate that layers closer to surface are susceptible to higher strain values compared to the core for a given constant applied strain.

Another method to visualize and compare the strains in the exterior to the interior would be to obtain and plot the scattered strain data in the entire measured volume at a given strain step. However, the numerousness of the slice count in any direction makes the computation not only tedious but also computationally expensive. We have hence, obtained scattered strain field data for a selected number of slices and interpolated these to the volume using linear methods available in Matlab software. We have plotted the
iso-surface of volumetric strain in the Fig 3.12. They represent the volume of the foam at
the initial (4%) and final (42%) of stages of compression displaying iso-surfaces at strain
values of 1.5% and 32.6% respectively. These values have been selected randomly for the
ease of visualization of the deformation pattern and capture important aspects of the
mechanics. Fig 3.12c shows the conical iso-surfaces observed at every step of
deformation (similar to the shapes shown in the insets of 3.12a,b). These cones have their
base on the free surface and vertex towards the core and are representatives of disparities
in strain values at the surface and the core and indicate how cells or struts with lesser
confinement or restrictions undergo higher strains. These are in conjunction with our
expectations and intuition.

3.5 Energetics of deformation in the interior and the exterior

In order to validate our observations in the previous section, we introduce a simple model
for open-cell solid foam subject to uni-axial compression. We begin by assuming a the
microstructure as a simple network of bars, a regular cell comprising of five struts
converging at a vertex. In a real case, these numbers may vary and the equations
presented hence could be modified according to the situation. This model without
oversimplified, and has been used before (48) but serves our purpose to capture the
essence of the micromechanics of the foam. We compute the strain energy in the cell due
to the application of a small displacement $\delta$ at the end of the semi-strut in the rise
direction (Figure 3.13). Like previous models we assume that the bars do not buckle, and
deform purely by member stretching and pure bending at the vertex. We impose
constrains, so that due the displacement $\delta$ of the end of the strut 1, the vertex is displaced
by a distance $u$, resulting in the change of angle between bar 1 and each of 2-5, while the
Figure 3.11.1 Histogram plots for layer 25 (25 px/872 μ from the surface) at a) 4.2%, b) 14.7%, c) 25.2% and d) 37.8% applied engineering strain respectively. Horizontal axes represent Biot Strain value.
Figure 3.11.2 Histogram plots for layer 150 (150 px/ 5.23mm from the surface) at a) 4.2%, b) 14.7%, c) 25.2% and d) 37.8% applied engineering strain respectively. Horizontal axes represent Biot Strain value.
Figure 3.11.3 Histogram plots for layer 300 (300 px/10.47mm from the surface) at a) 4.2%, b) 14.7%, c) 25.2% and d) 37.8% applied engineering strain respectively. Horizontal axes represent Biot Strain value.
Figure 3.12 Plot of strain iso-surface in the foam volume a) at 4% applied strain displaying 1.5% strain value b) at 42% applied strain displaying 32.6% strain iso-value. Insets show zoomed in section of the volume. c) Illustration showing the shape of iso-surfaces at the exterior
ends of bars 2-5 remain fixed. We then use simple geometry to compute the stretches \( \varepsilon_1, \varepsilon_{2-5} \) in the bars 2-5. The strain energy thus generated due to this compression is due to the contribution of stretch in bar 1, resulting in the stretch in the bars 2-5 and due to the bending between bar 1 and 2-5. For the sake of simplicity, we assume that the contribution to the strain energy due to the much smaller inter-angular change between bars 2-5 is smaller than the other components of energy.

**Figure 3.13** Illustration showing a) typical 5 bar foam structure b) undergoing deformation due to uniaxial compression stretch in the rise direction c) Deformation process due to applied displacement \( \delta \) causing vertex to undergo displacement \( u \) resulting in the change in the length of arms from \( L \) to \( L' \) and the angle from \( \alpha \) to \( \beta \)
We geometrically arrive at the relations for the normalized stretches $\tilde{\varepsilon}_{1,ax}$, $\tilde{\varepsilon}_{2-5,ax}$ and the change in angle $\phi = \alpha - \beta$ respectively as a function of the normalized applied displacement $\tilde{u} = \frac{u}{L}$ and the known parameter $\alpha$ and define a normalized energy function $\tilde{\Omega}(\tilde{u})$ given by

$$\tilde{\Omega}(\tilde{u}) = \frac{\Omega}{E L^3} = K_{ax} \left( \tilde{\varepsilon}_{1,ax}^2 + \sum_{i=2}^{5} \tilde{\varepsilon}_{i,ax}^2 \right) + K_{be} \sum_{i=2}^{5} \tilde{\varepsilon}_{i,be}^2$$

The constants $K_{ax}$ and $K_{be}$ scale with geometrical parameters $\kappa$ and $\kappa^2$ where $\kappa$ radius to length ratio $\frac{r}{l}$ of the bars. Assuming the constant of proportionality to be $O(1)$ and the contribution of each of the bars 2-5 to be equal due to similar geometry and mechanical properties the final normalized energy functional is as

$$\tilde{\Omega}(\tilde{u}) = (\tilde{u} - \delta)^2 + n^2 \left[ \sqrt{\left( \frac{\tilde{u}^2}{\tilde{u}^2 + 1 - 2.\tilde{u}.\sin(\alpha)} \right)} - 1 \right]^2 + \kappa^2 \left[ \tan^{-1} \frac{\tilde{u}.\cos(\alpha)}{1 - \tilde{u}.\sin(\alpha)} \right]^2$$

The three terms in the order of their appearance are the contributions from the stretch of bar 1, bars 2-5 and the change in angle $\phi$. The constant $n$ accounts for the number of bars which in a general case in the case of a cell in the interior is 4. In order to compare the potential energy in a cell at the surface of the foam, we have to negate the contribution due to more struts. We thus assume a value of $n=2$ in the case of a exterior cell. We are interested in finding a configuration which effects the minimization of the energy functional $\tilde{\Omega}$ resulting in obtaining the relation $\delta(u)$. A parametric plot of energy functional against the input displacement $\delta(u)$ at the tip of bar 1 clearly follows the expected non-convex shape which is typical of physical systems with inherent heterogeneous spatial field associated with specific processes (Figure 3.14).
Fig. 3.14 Parametric plot of Energy functional against applied deformation. Slope of tangents indicate yield stress value at band formation.

There is a very good agreement with our model and experimental observations. There are two unique points in each curve through which a tangent may pass denoting the start and of band formation. The slopes of the tangent indicate the constant stresses during the band formation process. Clearly band formation at the interior occurs at higher stress values and much later in time compared to that at the surface. Both imply that ease of cellular collapse is more, at cells closer to the surface in comparison to the ones at the core.
Chapter 4

Future Work and Conclusion

4.1 Formulation of 3D Global DIC

In this chapter we discuss how we could combine µCT and DIC methodologies as before but by expanding the concept to the entire volume by disregarding our assumption of plane strain. In the previous methodology, upon tracking a single slice of the volume, we would observe that, while most of the significant features are retained in the plane, sections often disappear by moving to the adjacent slice and sometimes reappear with progressive deformation. This indicates the presence of out-of-plane deformation and cannot be captured by the method described in Chapter 3 and hence was one of the reasons that we had to implement averaging of images over the volume. By expanding the DIC methodology to 3D, however, will help us achieve this aim. Unlike the case of Local DIC, we have attempted to adapt a Global approach, based on Zhan’s thesis (63) wherein, instead of correlating a pair of sub-images centered on the nodes, assembling is incorporated using a finite element approach in three dimensions.

The volume of measurement is meshed into 10 node tetrahedral elements with their nodes on the boundaries. Correlation is obtained by using a multi-variable global minimization. Assuming deformation to be homogeneous within each element, the sub-elemental displacements of each pixel is computed through the interpolation of nodal displacements. If \( \mathbf{X}_p \) denotes the pixel position in reference image, \( \mathbf{X}_n \) is the nodal position, and \( \mathbf{U}_n \) represents the nodal displacements, then the position of a pixel in the deformed image is given by,
\[
x(X_p) = X_p + \sum_{n} N_n(X_p)U_n
\]

\(U_n\) has 30 components corresponding to displacements of the 10 nodes in three dimensions for a quadratic tetrahedral element and is represented by vector \(p_e\),

\[
p_e = [u_1 v_1 w_1 u_2 v_2 w_2 \ldots u_{10} v_{10} w_{10}]
\]

The shape functions \(N_n(X_p)\) for the 10-node tetrahedral element have the form (61),

\[
\begin{align*}
N_1(X_p) &= \zeta_1(X_p)[2\zeta_1(X_p) - 1], \quad N_5(X_p) = 4\zeta_1(X_p)\zeta_2(X_p) \\
N_2(X_p) &= \zeta_2(X_p)[2\zeta_2(X_p) - 1], \quad N_6(X_p) = 4\zeta_1(X_p)\zeta_3(X_p) \\
N_3(X_p) &= \zeta_3(X_p)[2\zeta_3(X_p) - 1], \quad N_7(X_p) = 4\zeta_1(X_p)\zeta_4(X_p) \\
N_4(X_p) &= \zeta_4(X_p)[2\zeta_4(X_p) - 1], \quad N_8(X_p) = 4\zeta_2(X_p)\zeta_3(X_p) \\
N_9(X_p) &= 4\zeta_3(X_p)\zeta_4(X_p), \quad N_{10}(X_p) = 4\zeta_2(X_p)\zeta_4(X_p)
\end{align*}
\]

where

\[
\begin{bmatrix}
\zeta_1(X_p) \\
\zeta_2(X_p) \\
\zeta_3(X_p) \\
\zeta_4(X_p)
\end{bmatrix} = 
\begin{bmatrix}
1 & 1 & 1 \\
X_1 & X_2 & X_3 \\
Y_1 & Y_2 & Y_3 \\
Z_1 & Z_2 & Z_3
\end{bmatrix}^{-1}
\begin{bmatrix}
1 \\
X_p \\
Y_p \\
Z_p
\end{bmatrix}
\]

in which \((X_1,Y_1), (X_2,Y_2), (X_3,Y_3), (X_4,Y_4)\) and \((X_p,Y_p)\) are the coordinates for the corner nodes and \(X_p\) respectively numbered counter clockwise. The mapping function for entire region is written as

\[
x(X_p) = x(X_n,X_p,p)
\]

where \(p\) indicates the global displacement vector with a dimension thrice the number of nodes. The least squares merit function \(\chi^2\) as in Local DIC is defined as in (56),

\[
\chi^2 = C(p) = \sum_{p=1}^{M} \left\{ f[x(X_n,X_p,p)] - F(X_p) \right\}^2
\]
where, again, $F(x_p)$ and $f[x(x_p)]$ are grayscale field functions corresponding to the reference and deformed image respectively.

One problem with the calculation of the displacement fields is that of discreteness of data. Because of the unavailability of gray level data in between the pixel, in order to calculate continuous displacements on a sub-pixel level, interpolation techniques need to be used. Similar to the bicubic interpolation scheme used for 2D Global DIC technique we have implemented a local tricubic interpolation scheme based on the Lekien and Marsden’s paper (65). This routine uses prescribed functional values and computed derivatives (using finite difference methods) only in the neighboring cells of the concerned voxel to interpolate the value at that location by enforcing a $C^1$ continuity on each of the 8 corners of the cubical voxel. The resulting tricubic form of the interpolating grayscale function value on the elements is

$$f(x, y, z) = \sum_{i,j,k=0}^{3} a_{ijk} x^i y^j z^k$$

The correlation of the volumetric images at each step and the visualization process will certainly require additional computational resources, but will definitely give us a greater insight into the micromechanics in the entire volume of the foam.

It would also be interesting to diversify this technique to a variety of other applications such as to study the initial consolidation of pharmaceutical powders during the compaction process by the inclusion of marker materials, delamination process in multi-layer materials, deformation in composites (in fact, deformation in any material exhibiting density gradients), to name a few.
4.2 Conclusion

X-Ray tomography is a versatile 3D imaging tool which captures density variations in a material. Utilizing this capability of µCT technique in addition to image processing methods we have characterized and evaluated the evolution of strain in metallic open cellular materials. We have used 2D DIC methods to investigate and compare the evolution of strain field at the free surface of the foam to that in the core of the Aluminum foam specimen undergoing progressive deformation. We have visualized the heterogeneity in the deformation pattern in the foams and have evaluated how there is a transition in the pattern as we move from the surface to the interior.

We have established the layers closer to the surface undergo a greater deformation and are susceptible to higher strains and band formations which reduces as we progress to the core assuming plane strain scenario.

We have also laid a foundation for studying the true volumetric strain negating the plane strain assumption for prospective study of the micromechanics involved in foam deformation.
References


16. The effects of roll compaction equipment variables, granulation technique and HPMC polymer level on a controlled-release matrix model drug formulation.  

17. Compression characteristics of granulated materials. IV. The effect of granule porosity on the fragmentation propensity and the compactibility of some granulations.  


19. Multiple compaction of microcrystalline cellulose in a roller compactor.  

20. Effect of recompression on the properties of tablets prepared by dry granulation.  

21. Insensitivity of compaction properties of brittle granules to size enlargement by roller compaction.  

22. Reduced tabletability of roller compacted granules as a result of granule size enlargement.  

23. Simulation of roller compaction using a laboratory scale compaction simulator.  

24. Effect of recompression on the properties of tablets prepared by dry granulation.  

25. The strength of bilayered tablet.  

26. Relationships between the modulus of elasticity and tensile strength for pharmaceutical drugs and excipients.  
27. Compaction of crystallographic forms of pharmaceutical granular lactoses. II: 
Compacts mechanical properties. V. Busignies, et al. s.l. : European Journal of 


29. Micro-scale measurement of the mechanical properties of compressed 
pharmaceutical powders. 1: The elasticity and fracture behaviour of microcrystalline 
cellulose. B.C. Hancock, Clas, S.D. and Christensen, K. s.l. : International Journal of 

30. Prediction of tablet hardness and porosity using near-infrared diffuse reflectance 
spectroscopy as a nondestructive method. M. Donoso, D.O. Kildsig and E.S. Ghaly. s.l. : 

31. Content uniformity and tablet hardness testing of intact pharmaceutical tablets by 
near infrared spectroscopy - A contribution to process analytical technologies. M. Blanco 

32. Non-destructive acoustic defect detection in drug tablets. I. Akseli, Mani, G. and 

I. Akseli, C. Libordi and C. Cetinkaya. s.l. : Journal of Pharmaceutical Innovation., 

34. Characterisation of density distributions in roller-compacted ribbons using micro-
indentation and X-ray micro-computed tomography. A.M. Miguélez-Morán, et al. s.l. : 

35. Density-pressure relationships in powder compaction. R.W. Heckel. s.l. : Trans 

36. The Effect of Particle Size on the Consolidation of Powders during Compaction. 
Analysis Conference.

37. L.J. Gibson and M.F. Ashby. Cellular Solids: Structure and properties. Cambridge : 

38. Manufacture, characterization and application of cellular metals and metal foams. 


