CHARACTERIZATION OF OPEN CELLED METALLIC FOAM

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CHARACTERIZATION OF OPEN CELLED METALLIC FOAM

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<tr>
<td>$V_{x,\omega}$</td>
<td>Volume of phase $x$ at realization $\omega$</td>
</tr>
<tr>
<td>$I_{i,x}$</td>
<td>Indicator function at realization $w$ for phase $i$ at location $x$</td>
</tr>
<tr>
<td>$\phi_i$</td>
<td>Porosity of phase $i$</td>
</tr>
<tr>
<td>$S_n(r)$</td>
<td>N-point correlation function for length $r$</td>
</tr>
<tr>
<td>$A_x$</td>
<td>Translated set $A$</td>
</tr>
<tr>
<td>$B_x$</td>
<td>Translated structuring element $(B)$</td>
</tr>
<tr>
<td>$\Delta B$</td>
<td>Input image</td>
</tr>
<tr>
<td>$A \ominus B$</td>
<td>Dilation of $A$ by $B$</td>
</tr>
<tr>
<td>$A^c$</td>
<td>Complement of $A$</td>
</tr>
<tr>
<td>$\bar{B}$</td>
<td>Reflected structuring element</td>
</tr>
<tr>
<td>$A \circ B$</td>
<td>Erosion of $A$ by $B$</td>
</tr>
<tr>
<td>$p$</td>
<td>Pixel location</td>
</tr>
<tr>
<td>$I(p)$</td>
<td>Image $I$ at pixel location $p$</td>
</tr>
<tr>
<td>$h$</td>
<td>Height (calculated from distance map)</td>
</tr>
<tr>
<td>$C_{h,M}$</td>
<td>Catchment basin for minima associated with $M$ with threshold value $h$</td>
</tr>
<tr>
<td>$D_{equiv}$</td>
<td>Equivalent diameter of an irregularly shaped object</td>
</tr>
<tr>
<td>$A_{pore}$</td>
<td>Area associated with a pore</td>
</tr>
</tbody>
</table>
\( D_{\text{equiv}}^{\text{ASTM}} \) Equivalent diameter of an irregularly shaped object calculated using the ASTM standard

\( P_{\text{pore}} \) Perimeter of a pore

\( f_{\text{circ}} \) Shape factor compactness (also referred to as circularity)

\( E \) Shape factor Eccentricity (image processing)

\( \mu_{p,q} \) Central moment of the shape with

\( AR \) Aspect ratio

\( D_{\text{max}} \) Maximum diameter of a particle

\( D_{\text{min}} \) Minima diameter of a particle

\( < D_{p,q} > \) Average mean diameter of the particle size distribution

\( n_i \) Number of occurrences in size bin i

\( D_i \) Mean diameter of size bin i

\( q \) Specific flux vector

\( J \text{ or } -\text{grad}\varphi \) Hydraulic gradient

Permeability calculated using the Kozeny Carman equation

\( F \) Form factor

\( s \) Specific surface area per unit volume

\( K \) Darcy permeability

\( \dot{\vartheta} \) Velocity field

\( \hat{\mu} \) Effective viscosity

\( p \) Pressure
\( \Delta p_j \)  
Change in pressure at pore \( j \)

\( D_j \)  
Pore body \( j \)

\( d_j \)  
Pore throat \( j \)

\( R_j \)  
Hydrodynamic resistance associated with pore throat \( j \)

\( q_j \)  
Flow rate through pore throat \( j \)

\( V_{roi} \)  
Volume fraction of phase of interest

\( A_{ROV} \)  
Area fraction of phase of interesting per field of view

\( s_{planar} \)  
Planar surface area

\( s_{true} \)  
True surface area

\( R \)  
incidence matrix

\( A \)  
Adjacency matrix

\( I \)  
Identity matrix

\( G \)  
modified adjacency matrix

Length of flow path

Nominal length

\( \tau \)  
Tortousity factor

\( EDM \)  
Euclidean distance map

\( MV \)  
Mean volume weighted diameter

\( V \)  
Percent volume change per size bin (for the particle size analyzer data)

\( MN \)  
Number weighted mean diameter

\( MA \)  
Area distribution mean particle diameter
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Definition</th>
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<tr>
<td>SD</td>
<td>Standard Deviation</td>
</tr>
<tr>
<td>$D^{\text{eff}}$</td>
<td>Effective Diameter</td>
</tr>
<tr>
<td>$A_{\text{total}}$</td>
<td>Total area of all pores within an image</td>
</tr>
<tr>
<td>PDF $x$</td>
<td>The probability density function at size bin $x$</td>
</tr>
<tr>
<td>$CDF(x)$</td>
<td>The cumulative density function at size bin $x$</td>
</tr>
<tr>
<td>$\rho$</td>
<td>The neck radius used to generate the neck between two touching spheres</td>
</tr>
<tr>
<td>$\kappa$</td>
<td>The proportionality constant used in the creation of necks in the sphere packing algorithm</td>
</tr>
<tr>
<td>$r_1$, $r_2$</td>
<td>The radii of the two spheres from which the pore neck is formed</td>
</tr>
<tr>
<td>PMMA</td>
<td>Poly(methyl methacrylate)</td>
</tr>
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</table>
SUMMARY

Open cell metal foams are a type of engineered material can be characterized by high porosity, high strength to weight ratio, tortuous flow paths and high surface area to volume ratio. It is the structure that gives the metal foams the characteristics that make them well suited for many application including heat exchangers. In this work, the structure of open celled metal foams is quantitatively characterized using an image analysis based method in order to enhance evaporative heat transfer of the metal foam increasing fluid permeability. Several image processing algorithms were developed to quantitatively characterize the porosity, surface area per unit volume, fluid permeability and the tortousity of the metal foams from digital images of the cross sections of the material. An algorithm was developed to partion the pore space in the digital images so that individual cells within the structure could also be quantitatively characterized. Tools were also developed to predict the structure of an open celled foam processed using a sacrificial template-based method.

Three samples of commercially available metal foams, with manufacturer provided fluid permeability values and effective pore diameter values obtained from ISO testing standards, were quantitatively characterized using the image analysis. The porosity, surface area per unit volume and tortousity were calculated, and the fluid permeability was calculated using a modified Kozeny Carman equation. The calculated fluid permeability values corresponded with manufacture provided value reasonably well (max error=5.6%).

To quantify changes in the structure of a metallic foam, changing processing parameters of the sacrificial templating method of creating open celled copper foams was
studied. For this templating method a slurry containing certain percentage of binder, micron sized copper powder and sacrificial polymer beads (which act as templates for the pores) is prepared. The slurry is then spun onto a flat copper substrate and dried. The dried sample and substrate are then heat treated in a reducing atmosphere. To study how the structure is changed by the solids loadings composition of the slurry, two different solids loading of copper were processed and the structures of the samples were quantitatively characterized using image analysis. These results indicate as the amount of copper within the sample is decreased, the feature size within the structure decreases, which creates more tortuous pathways and more surface area per unit volume. For the heat treatment study, samples were heat treated to maximum temperatures of 700ºC and 800ºC. The results indicate that as the heat treatment temperature increases, larger pathways are formed but structural features such as porosity and surface area per unit volume are reduced.

In order to predict how the particle size distribution of the sacrificial polymer beads effect the structure of the metal foam, a particle packing algorithm was developed. Particle size distributions of different polymer beads were measured using a laser diffraction based particle size analyzer and image analysis. Then a particle packing algorithm randomly selects particles from this distribution to pack to a desired porosity. The resulting particle packing is expected to simulate the structure of the open celled foam prior to the heat treatment. In addition, a pore neck algorithm was developed to estimate how the interconnected pathways form when the polymer beads are removed during the heat treatment. Several different neck geometries were generated using different κ values used to generate pore neck geometry and the results were compared
against one another using quantitative characterization methods used on the digital images of the real foams. The microstructures were compared using two point correlations functions that were generated for the different digital microstructures. As the $\kappa$ value was decreased, the surface area increased and the morphology of the pore neck became less and less noticeable.

A spherical contraction algorithm was also performed on a packed sphere configuration to simulate the copper powder sintering during heat treatment. The resulting digital microstructure was compared to real open celled copper foam using two point correlation functions and quantitative structural characterization. The two point correlation function of the two different structures did not match due to the surface roughness due to the partially sintered copper powder that is not present in the digital microstructure, however both two point correlation functions did exhibit similar long range order behavior.
1.1 Porous Media & Open celled Metallic Foams

Open celled metallic foams are a type of engineered structural porous material that can be characterized by an interconnected pore network with a rigid walled interconnected solid phase. Metallic foams are of practical interest due to their unique structural features such as cell size and shape can be altered based the foam processing parameters, thus allowing for material to be designed to fit a specific application. Currently metallic foams are being used for applications such as mechanical energy absorbers, filters, flow straighteners and heat exchangers (Banhart 2001; M Ashby 2000; M Ashby and T Lu 2003; Banhart and Weaire 2002; Banhart et al. 2001; Boomsma, Poulakakos, and Zwick 2003) The process of modeling materials as porous media has been applied in many different fields of engineering including the oil industry (Jacob Bear 1988), bioengineering and material science. Statistical descriptors of the spatial organization of the pore structure allow for short and long range order of the pore structure to be quantified (Corson 1974; S Torquato 2002; Cohen and Introduction 1982; Meier, Friess, and Steinfeld 2008; Habisreuther, Djordjevic, and Zarzalis 2009)

Before the structure of the metallic foam can be optimized, the structure must be characterized. To characterize the structure of a metallic foam, the techniques used for characterizing porous media will be used. Characteristics such as interfacial area per unit volume and pore size can be estimated from images of the microstructure of the porous materials (Zalc, Reyes, and Iglesia 2004; Mauran, Rigaud, and Coudevylle 2001; Liang 2000a; W Brent Lindquist 2006; S Torquato 2002; James G Berryman and Stephen C

1.2 Fluid Permeability

Many open celled metallic foams are used in applications where the fluid transport properties of the material dictate the critical operating parameters. Although much work has been done to estimate the fluid permeability of porous media (Johnson, Koplik, and Dashen 2006; Auriault 1980; Sung and Yethiraj 2008; J Berryman and S Blair 1986; James G Berryman and Stephen C Blair 1986; Martins et al. 2007; Xu et al. 2008) little work has attempted to characterize the fluid permeability of open celled metallic foams (Dai et al. 2010; Gottfried Laschet et al. 2009; G Laschet et al. 2008; Kopanidis et al. 2010). Most methods of calculating the fluid permeability of open cell metallic foam involve the homogenization techniques (Gottfried Laschet et al. 2009; G Laschet et al. 2008) or pore level simulation of the fluid flow through individual cells (Kopanidis et al. 2010). Both methods of calculating the fluid permeability are computationally expensive. Many studies have used analytical approaches such as the Kozeny Carmen equation to calculate the fluid permeability of the porous media using structural features of the porous material such as surface area per unit volume, porosity and tortousity (James G. Berryman and Stephen C. Blair 1987; Mauran, Rigaud, and Coudevylyle 2001; Liang 2000a; Yu 2002; Rubinstein and S Torquato 2006). In This
work the Kozeny Carman equation was used to estimate the fluid permeability of the metal foams obtained from the quantitative characterization of its structural features.

1.3 Thesis Organization

A brief introduction into metallic foams and the methods of processing them are also discussed in Chapter 2. Image analysis algorithms are introduced along with stereological concepts and calculations used to obtain quantitative information regarding the pore structure are also introduced and different models and methods used to calculate the fluid permeability are introduced and reviewed. The development of the quantitative characterization methods using image analysis are presented in Chapter 3. The development of several algorithms which digitally construct microstructures based upon a given particle size distribution are also described in Chapter 3. The results and discussion of several studies involving the characterization of particle size distributions of sacrificial polymer beads and structural characterization of open celled copper foam and stainless steel filters are presented in Chapter 4. The conclusions and future work presented in Chapter 5.
BACKGROUND

Introduction

In this chapter, metallic foams and methods of processing the metallic foams are discussed. The methods and the principles of many of the image analysis techniques that are associated with the different image acquisition methods used to image the foam’s pore structure of a metallic foam are also discussed. The structural characterization techniques that correspond with the image analysis will be described. Methods used to describe particle size and particle size distributions will also be discussed. The basic concepts behind fluid permeability are introduced and the different methods used to calculate the fluid permeability are reviewed in this chapter.

1.4 Characteristics of Foam

Because effectively describing structure of a metallic foam is critical in determining its unique properties, which are different from the properties of its constituent material. A “foam” must be clearly defined. According to Banhart, a foam is defined as a dispersion of gas bubbles in a liquid (Banhart 1999). To minimize the free energy of the liquid-gas interface, foams have a distinct morphology that can be characterized by thin films (or lamellas) which separate the space into a shape fill polyhedral referred to as a cell (see Figure 0.1 Image of soap bubble foam (Anon)) (Studart et al. 2006). If the liquid portion of the foam were to solidify, a “solid foam” would result. However, to call a metallic foam a solid foam is incorrect because the morphology of the cells within the metallic foam is dependent on the method by which is made. If the metallic foam is processed using a template, the cells within the metallic
foam should have a morphology similar to the shape of the template rather than the shape resulting from surface area minimization. Thus, metal foams are in reality “cellular solids”. However a common convention is to describe the metallic “cellular solids” as “open celled metal foams” or “cellular metal foams”, though technically, the cellular solids do not have a structure characteristic of true foams.

Figure 0.1 Image of soap bubble foam (Anon)

1.5 Metallic foams

Metallic foams are a type of material consisting of a metal phase containing dispersion of pores. An open celled foam consists of a structure such that the metal phase is contained only on the edges of a cell such that the cells connect one other through open faces (Gibson and MF Ashby 1999) (see Figure 0.2a). The structure of a closed celled foam is one where the faces of the cells within the structures are separated from one another with cell faces (Gibson and MF Ashby 1999) (see Figure 0.2b). Some metallic foams can contain both partially open and partially closed cells and their morphology is dependent on the method by which the foam is processed.
The structural characteristics commonly used to describe a metallic foam include its cell topography, relative density, cell size, cell type and cell shape. Quantifying these characteristics requires some imaging of the microstructure (M Ashby 2000). Metallic foams can be treated as consolidated porous media, with the solid phase corresponding to the metal phase and the space that the cells occupy corresponding to the pore space.

There are many different methods of processing metallic foams, typically starting from one of the following precursor: liquid metal, solid metal in powder foam, metal vapor or metal ion solution (Willson 2005; Meier, Friess, and Steinfeld 2008; G Laschet et al. 2008; W.B. Lindquist and Venkatarangan 1999; Große et al. 2008; Liang 2000). Of these methods, most of the commercial methods of processing rely initially on the liquid phase e.g. bubbling gas through molten metal (typically aluminum), stirring a foaming agent into a molten alloy and controlling pressure during cooling (M Ashby and T Lu 2003). In this work, a sacrificial templating method of processing open celled metal foams is studied. For this method, a slurry containing certain percentage of binder, micron sized copper powder and sacrificial polymer beads (which act as templates for the pores) is prepared. This slurry is then spun onto a flat copper substrate and dried. The dried sample is then heat treated in a reducing atmosphere. During the heat treatment process, the polymer bead template is thermally extracted; leaving behind an open celled porous structure with micron sized pores.
1.6 Image Acquisition Methods

This section discusses the different methods used to analyze a porous material to obtain information regarding its pore structure and organization. Most of the methods described in the section result in either a two or three dimensional representation of the structure of the porous material.

Three dimensional methods of imaging the microstructure are typically either tomographic or magnetic resonance techniques. (Liang 2000; Willson 2005; Meier, Friess, and Steinfeld 2008; G Laschet et al. 2008; Große et al. 2008) Tomography produces a three dimensional image of the materials structure using high energy emission. X-rays, gamma rays, and electrons are some of the sources used to create these three dimensional images. Several studies have used conventional x-ray tomography to image the pore structure of porous media (Willson 2005; Meier, Friess, and Steinfeld 2008; Jones et al. 2003). The resolution, however, of images produced from conventional x ray tomography is limited to the micron per pixel range. Imaging of materials with micron-scaled features or smaller requires methods of imaging with a greater resolution such as...
synchrotron x-ray tomography, which provides a sub micron to pixel resolution and is often used for microtomographic studies on porous materials (Jones et al. 2003; Willson 2005; W.B. Lindquist and Venkatarangan 1999; Banhart et al. 2001).

Magnetic resonance imaging (MRI) is another three dimensional image technique that is commonly used for imaging of biological tissue. Some work has also been done using MRI to image the pore structure of ceramic foams (Groß et al. 2008). Imaging metal foam using these techniques would be impossible due to magnetic interference.

There are many methods of obtaining two dimensional images of a porous material, of which optical microscopy and scanning electron microscopy are some of the most common method. To accurately describe the three dimensional structure of a porous material from two dimensional images, stereological sampling techniques must be implemented. Stereology is the study of how three dimensional structures can be quantitatively characterized using two or one dimensional probes with systematic sampling techniques, statistics and geometrical relationships. Several studies have used the serial sectioning techniques to characterize three dimensional morphological and topographical features of porous media (HJ Vogel 1997; H Vogel and Kretzschmar 1996; Harpreet Singh and Arun M Gokhale 2005).

1.7 Image Processing Techniques

Images of porous material must be processed before its information can be analyzed. First the concept of thresholding will be introduced, followed by a description into different structural characterization methods including statistical descriptors of the microstructure and morphological analysis of the microstructure.
1.7.1 Thresholding Techniques

Thresholding is a form of image segmentation, where the “regions of interest” are separated from the background, which a binary image based upon a calculated threshold pixel value. Pixels with values above the threshold value are associated with the regions of interest (pixel value= 1) and those below the threshold value are associated with the background (pixel value= 0). The thresholding technique is used in many image processing applications including document text recognition, noise removal, character extraction and object recognition (Sezgin and Sankur 2004; Otsu 1975; Hoover, Kouznetsova, and Goldbaum 2002). The various applications for thresholding have resulted in different segmentation methods including histogram shape, clustering, entropy, spatial, attribute based and local methods. In histogram shape based methods of thresholding, the image in separated into segments based upon statistical analysis of the pixels values within the image (see Figure 0.3). The convex-hull thresholding algorithm, which thresholds an image based on the concavity of the peaks as a method of thresholding an image. (Rosenfeld and LA TORRE 1983) For the clustering based methods of thresholding, the pixel values within an image are sorted into clusters using the statistical data analysis method known as cluster analysis. The Otsu method, one of the most commonly used thresholding techniques, uses cluster analysis to find the optimal threshold value by minimizing the weighted sum of the variance of the pixel values within the foreground and background clusters (Otsu 1975).
1.8 Structural Characterization Methods.

After an image of the metallic foam has been processed into a binary image using the thresholding algorithm, the distribution of solid and pore space must be described. There are several different methods of quantitatively characterizing structural features from
binary images of planar cross sections of metallic foams. Statistical methods and the geometry based methods of describing the structure are discussed in the below.

The statistical method of characterizing two phase systems such as porous material from binary images involves describing the spatial arrangement of a material in terms of probabilities. The geometrical methods involve analyzing the structure in using shapes and set theory-based operations. Both methods of these structural characterization methods require that stereological relationships be used to estimate the three dimensional properties from planar two dimensional cross sectional image.

1.8.1 Statistical Methods of describing the microstructure

The statistical method of characterizing a microstructure is based on a statistical correlation function which describes how the spatial arrangement of a phase of interest changes in space. N-point, surface, lineal path and pore size functions are some of the commonly used statistical correlation functions. Figure 0.4 illustrates how some of the statistically correlation functions are calculated. The n-point correlation function $(S_n(x_1, x_2, \ldots, x_n))$ is used in the determining the specific surface area (J Berryman and S Blair 1986; J Berryman 1987), electrical conductivity (Liang 2000b), heat transfer (Meier, Friess, and Steinfeld 2008; Yagi, Kunii, and Wakao 1960; Kunii and JM Smith 1960), residence time (Habisreuther, Djordjevic, and Zarzalis 2009) and fluid permeability (J Berryman and S Blair 1986) For a two phase material, this function describes the probability that all $n$ sampling points all lie within the same phase $i$. The surface correlation function $(F_{sv}(r))$, a special case of the n-point correlation function, describes the probability for $n$ sampling points where all sampling points lie on the surfaces between phase 1 and phase 2, have been used in the determination of fluid
permeability (Doi 1976; Rubinstein and S Torquato 2006). The lineal path function \( L(z) \) is also another statistical correlation function is related to the chord length function, which is often used in the estimation of heat transfer coefficients (Meier, Friess, and Steinfeld 2008), and cytology (Weibel, Kistler, and Scherle 1966; Karch et al. 2010). The pore size distribution function \( P(\delta) \) is typically used to characterize the pore space in porous media by calculating probability a radius, \( \delta \), will fit completely in the pore space. The function is more commonly seen as the cumulative pore distribution function, which is the complement of the pore size distribution function.

Figure 0.4. Schematic of the methods of measurement for different statistical correlation functions. Lineal path function \( L(z) \), 2 point probability function \( S_2(r) \), a surface-void correlation function \( F_{sv}(r) \) and a pore size density function \( P(\delta) \).
To obtain a statistical correlation function, several assumptions must be made. A statistical correlation function assumes that the microstructure is static at any given time and the realization $\omega$ of a two phase medium, which has a volume $V$, can be partitioned into two phases which for phase 1 and phase 2 such that:

$$V_1 \omega = \phi_1$$  \hspace{1cm} (2.1)  

$$V_2 \omega = \phi_2$$  \hspace{1cm} (2.2)  

Where $V_1 \omega$ corresponds to the volume of phase 1 and $V_2 \omega$ corresponds with the volume associated with phase 2. For a metallic foam, $V_1 \omega$ refers to the solid phase such that $\phi_1$ is the volume fraction of the solid phase and $\phi_2$ is the volume fraction of the pore space. Also $dV \omega$ is assumed to represent the surface between $V_1 \omega$ and $V_2 \omega$, which is useful in calculating the interfacial area between two phases.

To calculate the correlation function, an indicator function $T^i_{x: \omega}$ be used. This function calculates the probability at a particular instance for phase $i$ and point $x$ such that:

$$T^i_{x: \omega} = \begin{cases} 1 & \text{if } x \in V_i(\omega) \\ 0 & \text{otherwise} \end{cases}$$  \hspace{1cm} (2.3)  

Using this indicator function, the different statistical correlation functions can be calculated.
1.8.1.1 n-point probability functions

The n-point probability function is the most widely used correlation function. They were introduced to determine the effective transport properties of random media (S Torquato 2002) the n-point probability function \( S_n \) calculates the probability that \( n \) randomly selected sampling points all correspond with volume associated with phase \( i \) \((V_i, \omega)\). This function can be calculated using the equation shown in Eq. (1.4), as the product of the indicator function of \( n \) sampling points:

\[
S_n \left( x_1, x_2, \ldots, x_n \right) = \left( T_i x_1, T_i x_2, \ldots, T_i x_n \right)
\]

If the number of sampling points is 2 (\( n = 2 \)), then the n-point correlation function is called a 2-point correlation function. Typically, 2-point correlation functions are used to analyze images of real or model microstructures. To make the calculation of the 2-point correlation functions more manageable, simplifying assumptions are used. The first assumption is the structure is statistically homogenous, such that the n point probability function is translationally invariant (Karch et al. 2010; S Torquato 2002). Another assumption is that the structure is locally isotropic such that sampling is orientation independent. There the two point probability function simplifies from Eq. (2.4) into the equation below

\[
S_2 (x_1, x_2) = S_2 (r)
\]

Where \( r \) is the distance between sampling points \( x_1 \) and \( x_2 \). An example of the 2 point correlation function is shown in Figure 0.5.

Some of important properties of the 2 point correlation function (Eq. (2.5)) are shown in the equations below and can be seen in Figure 0.5.
Among the different statistical descriptors, the 2 point correlation function is one of the most commonly used spatial descriptors. Torquato, Berryman and Corson describe some of the properties of two point correlation functions and it is useful in describing the properties of porous media (S Torquato 2002; C Y Yeong and S. Torquato 1998; Corson 1974). Work has also been done to use Statistical descriptors like two point correlation functions and the lineal path functions to reconstruct heterogeneous structures(C Yeong and S Torquato 1998; Anon; Corson 1974; Meier, Friess, and Steinfeld 2008; Roberts 1997) and calculate the representative volume element(Zeman and Šejnoha 2007) used in the homogenization technique to describe a unit cell for a random microstructure.

For the 2-point correlation function of an isotropic randomly generated two phase material, the maximum value of the function is occurs at $r=0$ and should be equal to the porosity (see Eq. (2.6) and Figure 0.5). The function should decay exponentially to an asymptotic value equal of the square of the porosity (see Eq (2.7)). For a structure with long range order, the 2- point correlation function behaves similar to that of a randomly generated microstructure; except for some fluctuations occur in the probability as $r$ increases, which mean that there are certain distances between a single phase that are more probable. This increase in probability at certain distances is an indicator of

\[ S_{2} \ 0 = \phi \]  \hspace{1cm} (2.6)

\[ \lim_{r \to \infty} S_{2} \ r = \phi^{2} \]  \hspace{1cm} (2.7)

\[ \frac{d}{dr} S_{2}(r) \bigg|_{r=0} = -\frac{s}{\pi} \]  \hspace{1cm} (2.8)
structural periodicity. The effective surface area of the microstructure can be obtained by taking the derivative of the function at \( r=0 \).[61]

![Graph showing two point correlation functions](image)

Figure 0.5. Two point correlation functions of two simulated microstructures, one with impenetrable spheres and another with penetrable spheres. Quantities that can be obtained from the two point correlation function are porosity \( S_2(r=0) \), specific surface area \( (dS_2(r=0) = -s/\pi) \) and an estimate of the particles size (minimum of \( S_2(r) \)) (James G Berryman 1985)

### 1.8.2 Geometry Based Method of describing the microstructure

A geometric description of the pore structure involves how the pores deviate from simple geometrical shapes and forms. To analyze structures using shapes and forms, mathematical morphology techniques are used. The basic concepts of morphological image processing will be discussed in this section, followed by a description of how these
algorithms are used to analyze the structure. Several image processing algorithms that use morphological operations to obtain features of the pore structure are also discussed.

1.8.2.1 Morphological Image Processing Algorithms

Morphological image processing algorithms are often times used to detect edges, isolate structural features or extract topological properties from an image. Morphological image processing algorithms use set theory to analyze a structure based upon a structuring element. The structuring element is a matrix made of a matrix of zeros and ones; the ones within the structuring element define the neighborhood or region of interest; whereas the zeros values define the background. A structuring element can have any arbitrary shape and size; however the structuring element is typically smaller than the image. The origin of the structuring element can be located anywhere within the structuring element matrix. The structuring element acts as a probe into the image by moving the origin of the structuring element to each position in the image in turn, and the points within the translated structuring element are compared with the pixel values from the underlying image. The outcome of the comparison between the structuring element and the image pixel values create a set of pixels position with assigned values, which is determined by the morphological operation used and the type of image being processed (Maragos, Schafer, and Butt 1996).

Typically an image that has been processed using a morphological image processing algorithm results in a binary image; however there are certain cases where a binary input image results in a grayscale image such as a distance mapping algorithm, where the input image is a binary image, but the output image is a grayscale image with pixel values corresponding with topographic distance from an edge (Serra 1994).
Of the different morphological operations, erosion and dilation are the most commonly used operations. All of the morphological operations use translation of a structuring element to probe an image. The concept of translation for a set A by a distance x can be mathematically expressed by expressed by Eq. (2.9), where $A_x$ is the translated set. For processing an image, the erosion operation and the dilation operation are morphological operations that are commonly used. The erosion operation is defined as the set of all pixel locations in the image plane where translated structuring image set $B_x$ is contained in the input image A (see Eq. (2.10)). The erosion operation typically has a “shrinking effect” whereas the dilation operation typically expands a region of interest. The dilation operation uses a reflection of the structuring element to “dilate” an image and can be described as the complement to the erosion of the reflected structuring element ($\tilde{B}$) and the complement of the input image ($A^c$) (Eq. (2.11)).

$$A_x = a + x : a \in A$$  \hspace{1cm} (2.9)

$$A \oplus B = (A^c \ominus \tilde{B})^c$$  \hspace{1cm} (2.10)

$$A \ominus B = x : B_x \subseteq A$$  \hspace{1cm} (3.11)

By combining the morphological operations of erosion and dilation other morphological operations such as opening and close can be created to analyze images. The morphological opening operation can be described as the dilation of an eroded set. This operation removes objects in the foreground (white space) smaller than the structuring element. The morphological opening operation is translationally invariant, monotonically increasing and idempotent. The morphological closing operation can be described as an erosion of a dilated set which removes holes from a region of interest.
The morphological closing operation has similar properties; it is translational invariant, idempotent and can be expressed monotonically. However, unlike the morphological opening operation, it is extensive, which means its algorithm is dependent on the input image and the structure element.

1.8.2.2 **Medial Axis Transformation**

The medial axis transformation is a morphological image processing algorithm that creates medial lines of symmetry using dilation. Blum introduced the concept of the medial axis function (MAF), which he described as the “locus of the corners” of a wave propagating through time and also as “locus of points equidistant from the pattern and in this sense represents a line of symmetry of the pattern”. (Blum 1967) Also referred to as the skeletonization algorithm, it has been used to simplify complex geometrical shapes into single pixel lines of symmetry (Liang 2000a; W.B. Lindquist and Venkatarangan 1999; Willson 2005; Glantz and Hilpert 2007). Figure 0.6 depicts the “skeleton” created by the medial axis transformation (MAT) algorithm.

Due to the geometry-sensitive branching of the skeleton, a “cleaning” algorithm can be performed to obtain useful information regarding the topological features of the pore space and thinning algorithms can be used to remove end points (Liang 2000a), thus allowing for the pore space and be partitioned based upon adjacency (Liang, Ioannidis, and Chatzis 2000).

There are three methods for creating a skeleton: using a distance map, Voronoi diagram or from an iterative erosion technique. Different methods of creating skeletons are used to preserve desired aspects of the original image. Some methods of
skeletonization preserve the topography of the structure, allowing for the transport properties of the structure to be modeled more accurately, whereas other methods preserve the geometric aspects of the image by creating an invariant skeleton which is unaffected by operations such as translation, rotation and scale.

From a binary image, a distance map is generated from a binary image, and where the ridges of the distance map create the skeleton. (Kimmel, Shaked, and Kiryati 1995) This method of skeletonization preserves the geometrical aspects of the structure, but does not preserve the topography of the structure. From a Voronoi diagram generated from an infinite number of generating points, the skeleton of the input image can be created (Brandt and Algazi 1992). Creating the skeleton using a Voronoi diagram preserves both the geometry and topography of the input image, but is computationally expensive and therefore is rarely used.

Using iterative thinning algorithms results in a skeleton where topographical information is preserved, but geometric information is not. The skeleton that is produced often contains many disconnected regions, and must be used with geometric descriptors of the input image to quantitatively characterize its original properties. The iterative thinning algorithm is a computationally inexpensive and fast algorithm, therefore making it appealing to use.
1.8.2.3 Watershed Algorithm

Watersheds, commonly referred to as dividing lines divide an image into segments based upon the geometry of the region of interest. Typically, the watershed algorithm is used to segment objects that are partially overlapping. One of the most common examples of this can be seen in Figure 0.7 where the watershed algorithm has been used to separate the touching spheres and or overlapping spheres into individual spheres. A watershed or dividing line separates a region of interest into catchment basins (segments) based upon the location of the minima (located in the center of the catchment basin).
Before the computation of a watershed can be described, the critical concepts used must be defined in greater detail. Among these concepts is the concept of a minimum \((M)\). For a watershed, a minimum of an image can be defined as a “connected iso-intensive area where the gray level is strictly darker than its neighboring pixels” (Luc Vincent and Soille 1991). Another concept used is catchment basins\((C(M))\) which are associated with a minimum \(M\). A catchment basin can be described as a set of pixels, \(p\), such that a drop of water located at \(p\) it would flow toward the minimum \(M\). In morphological terms, the catchment basins of an image \(I\) can be described as the influence zones of a minimum. Equation (2.12) describes the algorithmic definition of a catchment basin based upon the immersion method of computing the catchment basins.

For a grayscale image, the pixels within catchment basin \((C_h M)\) associated with a minimum \(M\), the grayscale value (or elevation) of the all the points within the catchment basin must have a value greater than the value \(h\) where \(h\) is the value corresponding to the catchment basin.

\[
C_h M = \{ p \in (C M), I(p) \leq h \}
\] (2.12)
To compute a watershed on a binary image, a marking function must be applied. The marking function selects the regions to be segmenting and creates a gradient used for the segmentation process. Typically, some form of a distance or elevation map (see section 1.8.2.2) is used as the marking function for the watershed algorithm (Luc Vincent and Soille 1991). However, using a distance map alone as a marking function tends to over-segment the image, where the objects of interests are lost within a mass of segments.

To prevent this over-segmentation, several different methods of isolating regions prior to applying the watershed algorithm including gradient controlled and marker controlled segmentation (Gonzalez, Woods, and Eddins 2004; L Vincent and Beucher 1989) have been developed. The gradient controlled method of watershed segmentation uses the changes in pixel values of the marking function as a method to select regions of interest and select minima to use as seeds for the watershed algorithm. For the marker controlled watershed segmentation, the user selects regions of interests to be segmented and also selects where the minima within the regions of interest are located and with the structuring element (shape) used to analyze the image.

1.9 Methods to quantify characteristics of particles and pores.

Typically, pore-size distributions are calculated by generating the statistical descriptor called the pore size probability density function, which describe the radius of the circle that can be drawn within an area of the pore space (S Torquato 2002). For open celled foams, partitioning the pore space and treating the partitioned pores as particles allows for the characterization techniques developed for quantitatively describing particle size distributions to be used to describe pore space. Also for the materials characterization in this work, partitioning the pore space allows for the direct comparison
of the particle size distribution of the sacrificial template polymer beads to the resulting
distribution of pores in the metal foams.

1.9.1 Particle descriptors

In this section, some of the methods used to quantify particle that can be used to
quantify the particles are described. Particle size distribution, shape descriptors and
effective diameters are some of the most useful characteristics in describe particles.
These characteristics will also be used to describe the pores within a sample.

1.9.1.1 Particle Size

Particle size is defined as the diameter of an equivalent sphere with the same
volume (H Merkus 2009). The equivalent diameter be used is used to measure particle
size from images. The equivalent diameter ( \( D_{equiv} \)) can be calculated using one of two
methods: using Eq.(2.13) where \( A \) is the cross sectional area of the particle, or using the
ASTM expression for calculating the equivalent circle diameter ( \( D_{equiv}^{ASTM} \), see Eq.(2.14)).
The Defined as the distance between two parallel tangents on the opposite sides of an
image of a randomly oriented particle (H Merkus 2009), the feret diameter is another
method that can be used as a measure of particle size (Walton 1948).

\[
D_{equiv} = \frac{4A_{pore}}{\pi}
\]  
(2.13)

\[
D_{equiv}^{ASTM} = \left( \frac{4A_{pore}}{\pi} \right)^{\frac{1}{2}}
\]  
(2.14)
1.9.1.2 **Shape Descriptors**

In addition to describing its size, describing the shape is useful in predicting the performance of particles within a system. From an image analysis standpoint, the quantitative shape descriptors are a method sorting and removing any objects that deviate from the expected shape of a particle. Some of the most commonly use shape descriptors are compactness (sometimes referred to as circularity (Mikli et al. 2001) or form factor (Tissues and Distribution 2010)), eccentricity, aspect ratio (sometimes referred to as ellipticity) and solidity.

Compactness \((f_{\text{circ}})\) can be mathematically described as the ratio of the area of the particle divided by the square of the perimeter of the particle times the \(4\pi\) (see Eq.2.15)(Acharya and Ray 2005). It is commonly used to determine the roughness of a particle due to the perimeter value being in the denominator.

\[
f_{\text{circ}} = \frac{4\pi A_{\text{pore}}}{P_{\text{pore}}^2}
\]  

(2.15)

The shape descriptor compactness is a dimensionless value that describes how the shape of an object deviates from a circle, a value of 1 indicates that the projected area of the particle resembles a circle. If compactness is 0, this indicates that the projected image of the particle resembles a line.

Eccentricity is the ratio of the particle’s minor and major axis and ranges between 0 and 1, with 0 indicating that the particle resembles a circle and a value of 1 indicates that the particle resembles a line. Eccentricity, \(E\), is mathematically expressed in Eq. (2.16)
\[ E = \frac{\mu_{2.0} + \mu_{0.2} - \sqrt{(\mu_{2.0} - \mu_{0.2})^2 + 4\mu_{1.3}^2}}{\mu_{2.0} + \mu_{0.2} + \sqrt{(\mu_{2.0} - \mu_{0.2})^2 + 4\mu_{1.3}^2}} \]  

(2.16)

where \( \mu_{p,q} = \sum \sum (x - \bar{x})^p (y - \bar{y})^q \) is the central moment of the shape and \((\bar{x}, \bar{y})\) is the centroid of the shape. Although compactness and eccentricity are both dimensionless numbers used to measure how a particle deviates from a circle or a line, the two shape descriptors quantify two different characteristics regarding the shape of a particle. The compactness describes how the shape of the particle deviates from a circle, whereas the eccentricity describes the elongation of the particle in terms of the major and minor axis.

Another important shape descriptor that is commonly used to characterize particles is the aspect ratio, \( AR \) (see Eq. (2.17)), which can be defined as the ratio of the major diameter(\( D_{\text{max}} \)) to the minor diameter(\( D_{\text{min}} \)), where the major axis is defined as the longest straight line that can be drawn between two points of the projected image of a particle and the minor diameter is length that span the particle projection normal between the two lines.

\[ AR = \frac{D_{\text{max}}}{D_{\text{min}}} \]  

(2.17)

1.9.2 Methods to represent the pore size distribution using image analysis

Recent studies have used watershed algorithms to segment the pore space of cellular materials (Brun et al. 2008). By using the watershed method of segmenting the pore space into individual pores, the characteristics of the individual pores can be easily quantified, and partitioning the data allows for simpler calculations.
Given a number of particles (or pores) taken from a sample, there will always be variability in the size and shape of the particles, therefore the particle sizes will exhibit some sort of distribution. Methods of representing the particle size distribution that involves calculating number of occurrences within a size range then applying a weighting factor (H Merkus 2009; HG Merkus 2008). This method of representing the particle size distribution can allow for particular features of the particle distribution to be better represented. The mean weighted diameter is often used to characterize the particle size distribution for a weighted particle size distribution. By calculating weighted mean diameters, the performance of the particle in a system can be better understood. The Sauter mean diameter, for example which is a surface area weighted mean diameter, is commonly used as a measure of the total surface area for a given volume (HG Merkus 2008; Tissues and Distribution 2010; Anon).

The calculations of the weighted particle size distributions are based upon statistical moments of a distribution (Anon). The Sauter mean diameter can be mathematically expressed as:

\[
< D_{3,2} >= \frac{\sum n_i D_i^3}{\sum n_i D_i^2}
\]

(2.18)

where \( D_{3,2} \) is the Sauter mean diameter and \( n_i \) is the normalized number of occurrences within size range \( i \) and \( D_i \) represents the mean diameter within size range \( i \). In statistical terms the Sauter mean diameter can be defined as the skewness of the distribution divided by its variance. The volume weighted mean diameter is where the diameter of each pore is weighted by its volume. This method favors larger particles to small particles, meaning that a few occurrences of particles with a large volume can
increase the volume weighted mean diameter. Expressed in equation (2.19), it can be described as the kurtosis of the distribution (fourth central moment of the distribution) divided by the skewness of the distribution (third central moment of the distribution) (H Merkus 2009).

\[
< D_{1.3} >= \left( \frac{\sum n_i D_i^4}{\sum n_i D_i^3} \right)
\]

(2.19)

1.10 Basic concepts of fluid permeability

Using the quantitative characterized structural features of a porous material, the fluid permeability of the material can then be estimated using modeling techniques and mathematical expressions. Permeability, for example, is a property that is unique to porous materials with a interconnected flow paths such as open celled metal foams or packed beds of sand. It can be a useful property in predicting the operating parameters used for evaporative heat transfer in open celled metal foams used as heat exchangers, vapor chambers and heat pipes.

Permeability, which can be described as the rate at which a viscous fluid flows through a porous medium is described by Darcy’s law where the flow rate \( (q) \) is equal to the permeability \( (K) \) times the flux \( (J) \)

\[ q = KJ = -K grad \phi \]  

(2.20)

Many different methods have been developed to predict the permeability of materials. A few that are commonly used to estimate permeability will be described. Empirical methods to estimate the permeability based upon the work of Kozeny and Carman is discussed in section 1.10.1 followed by a discussion of the Brinkman Stokes
effective medium methods in section 1.10.2. Network modeling techniques used to estimate the permeability will be discussed in section 1.10.3.

### 1.10.1 Kozeny Carman Empirical method

The Kozeny Carman model is a widely used empirical model used to calculate the flow through porous media (James G Berryman and Stephen C Blair 1987; Yu 2002; Meier, Friess, and Steinfeld 2008; Martins et al. 2007; S Torquato and Pham 2004; James G Berryman and Stephen C Blair 1986; Mauran, Rigaud, and Coudevylle 2001). The Kozeny Carman model uses smooth walled straight capillary tubes to model the fluid pathways of the porous medium. The general expression of the Kozeny Carman equation to calculate the fluid permeability ($k_{\text{kc}}$) can be expressed as

$$k_{\text{kc}} = \frac{\phi^2}{F s^2}$$

(2.21)

Where $\phi$ is the porosity, $F$ is a form factor and $s$ is the specific surface area (James G Berryman and Stephen C Blair 1987) and the permeability can be expressed in terms of m$^2$. The permeability value calculated from the Kozeny Carman expression does not fit experimental data well when the porous media exhibits one or more of the following characteristics: high porosity (greater than 60%), non spherical particles, consolidated porous medium, non-normal particle size distributions (Mauran, Rigaud, and Coudevylle 2001). Modified forms of the Kozeny Carman equation have been made to better fit experimental data of consolidated porous media and materials with high porosity and anisotropic consolidated media, by using a specific area term instead of a particle diameter (Mauran, Rigaud, and Coudevylle 2001). The modified Kozeny Carman equation has been used to calculate the fluid permeability for porous media used for high
heat flux applications (Raffray and Pulsifer 2003) and titanium foams (R Singh et al. 2009). One of the modified forms of the Kozeny Carmon equation that is used to express the effective fluid permeability of a consolidated porous media with a high porosity is Eq. (2.22).

\[ k = \frac{\phi^3}{Fs^2} \]  

(2.22)

1.10.2 Stokes -Brinkman Methods

Another method used to estimate permeability of a material involves describing single phase flow through porous media using the Stokes- Brinkman equations. Using this method, flow through the pores can be treated in terms of stokes flow. Theses equations can be expressed in terms of a permeability tensor related to the Darcy permeability (K), the pressure (p), the velocity field (u) and an effective viscosity (\(\mu\)) where f corresponds with the fluid moving within the material.

\[ \mu K^{-1} \Delta \vec{u} + \nabla p - \tilde{\mu} \Delta \vec{u} = 0, \quad \nabla \ast \vec{u} = f \]  

(2.23)

In the fluid domain, where free flow occurs, the permeability tensor goes to infinity and the effective viscosity becomes equal to the fluid viscosity such and Eq (2.23) simplifies to the Stokes equation.

\[ -\mu \nabla \ast \nabla \vec{u} + \nabla \vec{u} + \nabla p = 0, \quad \nabla \ast \vec{u} = f \]  

(2.24)

In the porous domain, the effective viscosity equals zero so that the Stokes-Brinkman equation simplifies back to the Darcy- Stokes equations [80].

\[ \mu K^{-1} \vec{u}_D + \nabla p_D = 0, \quad \nabla \ast \vec{u}_D = f \]  

(2.25)
1.10.3 Network Model Methods

The basic concept of a network model is the defining of elementary cells to represent the local structure and describing the flow through these cells to estimate the permeability of a porous material (Martins et al. 2007). Calculating the fluid permeability using network model methods allows for the fluid flow paths within the porous material to be tracked.

There are two components that make up a pore network: the *pore bodies* and the *pore throats*. The pore network can be divided into chambers (pore bodies), which represent the large voids in the porous media, and channels (pore throats), which are the smaller voids that connect two chambers. (S Torquato 2002) Networks models can be established based upon a materials measured topological and geometric feature of the real material being studied. Therefore information regarding the connectivity and shape of the individual pore bodies and pore throats is necessary

To set up a simple network model, pore bodies and pore throats are assigned shapes and sizes based upon material characteristics or simplifying assumptions. These components are then arranged into a network with a lattice and a characteristic unit cell (S Torquato 2002; Armatas 2006). A network can also be formed from the components based upon topographical information obtained from image analysis (Liang 2000a). After the configuration of the network has been resolved, boundary conditions are applied and the flow through the network is modeled using as equivalent electric circuit where pore throats acts as resistors and pore bodies act as nodes (Martins et al. 2007; Liang 2000a). It is assumed that: the flow is isothermal, incompressible and steady state and the gravitational effects are negligible. Therefore the pressure drop associated with fluid flow
through two pore bodies ($D_j$ and $D$) with a pore throat ($\theta$) connecting the two pore bodies can be written as

$$\Delta p_j = R_j q_j$$  \hspace{1cm} (2.26)

Where $R_j$ is the hydrodynamic resistance of pore throat $j$ and $q_j$ is the flow rate through pore throat $j$. In Figure 0.8, Kirchhoff’s laws and resistive network analysis are used to obtain a series of equations used to determine the pore network’s overall hydraulic conductance. The relationship between the hydraulic conductance ($K$) and the permeability ($k$) is shown in equation (2.8) expressed in terms of the dynamic viscosity of the fluid ($\mu$) and the density of the fluid ($\rho$)

$$K = \frac{k \rho}{\mu}$$  \hspace{1cm} (2.27)

Figure 0.8. Electrical analogy used for the network modeling of fluid permeability. A) Electrical analog of network model for a network with periodic and normal channels. B) Analogy for pore level flow within an network (Martins et al. 2007)
MATERIALS AND METHODS

In this chapter the development of image processing algorithms used to characterize the different structural features relevant to the calculation of the fluid permeability for a porous structure will be discussed. Then, a description of how the procedures used to characterize particle sizes from a distribution were developed. Several different studies will be presented. These studies document the validation of the image analysis based method of quantifying particle size distribution of different sized poly(methyl methacrylate) (PMMA) beads, image analysis based methods for estimating the fluid permeability and digitally constructed microstructures. The results from these studies will be presented and discussed in following chapter.

1.11 Image Analysis for quantitative characterization of open celled metal foams

In this section, the development of image processing algorithms used to quantify the porosity, surface area per unit volume and tortousity of open celled metal foams will be discussed. First the development of the thresholding algorithms will be discussed followed by how a description of how the structural parameters are obtained from the image processing algorithms. Then marker controlled watersheding algorithm developed for the partitioning of individual cells within the foam will be described and the method developed to calculate weighted particle diameter will be discussed.

1.11.1 Development of a thresholding algorithm

To obtain information from an grayscale image, the image must be segmented such that the object or region of interest must be separated from the background using one of the thresholding algorithms described in Section 1.7.1. In this work, the Otsu
method of image segmentation is used. After an image has been thresholded, a morphological opening and closing operation is performed on the binary image using a square 3 x 3 pixel structuring element to remove objects that are smaller than the structuring element within the regions of interest. The morphological closing operation is done to remove holes within the background that are smaller than the structuring element. Then opening and closing operations are performed on the complement of the resulting image, thus removing noise from the background as well as the foreground.

After thresholding, the resulting binary image can be processed to quantitatively characterize porosity, surface area per unit volume and the tortousity. Since porosity of the porous material can be described as the volume fraction of pores \( V_{roi} \), the stereological relationship between the volume fraction and the area fraction per image \( A_{FOV} \) can be used to calculate porosity (Russ 2002). A binary image can be represented as a logical array where values for the pore space are 0 and solid space is 1. The area fraction of pores in an image can then be calculated counting the number of zeroes within the logical array and dividing the number of zeroes by the total number of elements within the array.

\[
V_{roi} = \frac{A_{FOV}}{3.1}
\]  

(3.1)

To calculate the surface area per unit volume of a porous material, the stereological concepts can be used on images of a planar cross sections of the structure. An edge detection algorithm can be used to detect the solid-pore interface from binary images of the planar cross section of the porous material (where 0 represents pore space and 1 represents solid). The edge detection algorithm results in a binary image with a pixel-thin outline of the solid–pore interface (see Figure 0.1). From this resulting binary
image, the planar surface area per unit area can be calculated by counting the number of 1s within the binary image.

![Binary Image before edge detection and Result of the edge detection algorithm](image)

**Figure 0.1 Result of the Edge Detection Algorithm.** Left: Binary Image before edge detection. Right: Result of the edge detection algorithm.

To eliminate error associated with edge effects, a mask is created from the pixels at the edges of the binary image, and two of the four edges are randomly selected. The edges that fall on those randomly selected edges are calculated into the planar surface area.

After a series of images corresponding to a sample has been processed using this modified edge detection algorithm, the surface area per unit volume ($s_{true}$) is calculated using the stereological relationship between the planar surface area and the true surface area per unit volume developed by Smith & Guttmann (Smith and Guttman 1954):

$$s_{planar} = \frac{\pi}{4} s_{true}$$  \hspace{1cm} (3.2)

### 1.11.2 Tortousity

Tortousity can be defined as the average pore length divided by the nominal length of the main fluid flow path (i.e. the thickness of the sample), and this
topographical feature will be used as the form factor in Kozeny Carman equation (Eq (2.3)) used to estimate the fluid permeability of a open celled metal foam. To calculate tortuosity a skeleton of the pore space must be created (Willson 2005). As discussed in Section 1.8.2.2 of chapter 2, pore space can be partitioned into individual pores based upon the changes in geometry through the use of a medial axis transformation algorithm (Willson 2005; Solymar and Fabricius 1999; Liang 2000b). The nodes of the skeleton can be labeled and identified using a neighborhood look up table operation. An indexed list of image skeleton nodes and branches creates an incidence matrix $R$ of size $nv \times ne$, where $nv$ and $ne$ represent the number of nodes and branches in an image skeleton, respectively. Matrix value $R(i,j)$ is equal to 1 if node $i$ is connected to branch $j$ (Diestel 2005). Skeleton branches on the image boundary are generally excluded.

Next, the adjacency matrix $A$, size $nv \times nv$, is determined by Eq (3.3), where $I$ is the identity matrix of dimension $nv$ (Diestel 2005)

$$A = R^T R - 2I$$ (3.3)

Matrix value $A(i,j)$ is equal to 1 if node $i$ is connected to node $j$ and 0 for all other cases. Matrix $G$ is a modified adjacency matrix, formed by replacing every non-zero element of $A$ with the tortuosity the length of the skeleton branch connecting node $i$ to node $j$. Taking the modified adjacency matrix $G$, the shortest fluid flow path ($L_{flow}$) can be calculated between two arbitrary nodes a graph traversal algorithm (Diestel 2005).

For the calculation of tortuousity, tortuousity ($\tau$) is equal to the shortest fluid flow path between two arbitrary nodes divided by the nominal distance between the two arbitrary nodes ($L_{nom}$) (see Figure 0.2)
To determine tortuousity from a skeleton of binary image is first obtained by using medial axis transformation algorithm within the MATLAB image processing toolbox. Then, the spurs and isolated pixels within the skeleton are removed and analysis of the skeleton branches and nodes begins. For each image, the incidence matrix, $R$, is obtained for the skeleton as well as and the adjacency matrix $A$ and the modified adjacency matrix $G$.

From the modified adjacency matrix $G$, random starting and ending nodes are picked. A shortest path algorithm computes the shortest length ($L_{flow}$) between to two nodes using the skeleton. The nominal distance($L_{nom}$) is calculated for the two nodes using the pixel locate of the nodes. The tortuousity is calculated using Eq (3.4), this procedure is performed 1000 times for each image.

$$\tau = \frac{L_{flow}}{L_{nom}} \quad (3.4)$$

Figure 0.2. Pore Skeleton with nodes shaded in red and a representative fluid path shown in green. Randomly selected starting node $C_1$, randomly selected end node $C_2$. 

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1.11.3 Development of a pore space portioning algorithm

To partition the pore space, the watershed algorithm (introduced in section 1.8.2.3 of chapter 2) is used to segment the pore space based on shape. Watershed algorithms require using the Euclidean distance map (EDM) of a binary image as a marking function. However, the complex morphology of open cell metal foams revealed through image analysis often leads to over-segmentation of the images. Therefore, a marker-based method watershed algorithm was developed to mitigate this effect.

To create a marker-controlled algorithms, first the external limit of the region of interest must be selected. The region of interest was isolated by creating a mask from the EDM, and the external limit is set using the criteria that any point on the EDM with a pixel value less than 3 is removed, followed by morphological opening and closing operation. After the external limit of the watershed algorithm is found, seeds (minima for the watershed algorithm) are created by using an extended-maxima transformation where regional maxima are selected and expanded based upon the H-maxima transformation. The H-maxima transformation suppresses all the maxima in the image with values lesser than a selected value on the EDM (see Figure 0.3.b). The mask generated from the marking function overlaid on the EDM of the binary image is shown in Figure 0.3c. This mask is used as the marking function for processing using MATLAB’s watershed algorithm. This produces a logical matrix, which can be visualized in Figure 0.3.d, where lines (watersheds) divide the pore space into pores based upon morphology.
After a binary image has been portioned using the marker controlled watershed algorithm, the individual pores can be indentified and sorted based upon their quantitative characteristics (such as area or perimeter) or shape descriptors (eccentricity or circularity). An indexing algorithm is thus performed on the watershed binary image, where the pixel locations corresponding to each individual pore are associated with a number label. This indexing is performed so that individual pores may be accessed at other instances during image processing.

An edge removal edge algorithm was developed to indentify and remove the pores lying on the boundary of the image. Before the pores can be sorted, those that lie on
the edge of an image must be removed. The resulting removal algorithm creates a mask with locations of the pixels at the edges, and then searches the pixel locations of each pore removing the index number of those pores whose pixels are located on the edges of the field of view. The resulting list of numbers corresponds to individual pores within a watersheded binary image.

1.1.4 Representation of particle size distributions from laser diffraction based particle size analysis

Laser diffraction based particle size analysis of the PMMA beads used in the processing of the open cell copper foam was performed to determine if the image analysis based methods of calculating particle distributions are valid. The data obtained from a laser diffraction based particle size analyzer are presented the percent absorbance associated with channels that correspond to the size ranges of particles. The percent absorbance is equivalent to the amount of particles which have a diameter within the size range specified by the channel. The particle size analyzer used in this work produces both tabular data and a graphical representation of the weighted particle size distribution and calculation of different weighted mean diameter as described below.

The volume weighted mean diameter (MV) describes the particles in terms of the volume distribution and is the summation of the percent volume change between size channels(V) and the size of the center of the channel (d) divided by summation of the volume percent (V)( see Eq.(3.5)). The MV is weighted by a change in volume of the larger particles within the distribution, and it is therefore it is a commonly used to express the average particle size based upon the central tendency (Plantz).
\[ MV = \frac{\sum V_i d_i}{\sum V_i} \]  

(3.5)

The number weighted mean diameter (MN) describes the mean particle size as equation (3.6). The number weight mean diameter (MN) is weighted toward the smaller particles within the distribution and is associated with the average particle size obtained from counting individual particles within a distribution. This method of calculating the average particle diameter favors the smaller particles, resulting in a smaller value for particle diameter than MV.

\[ MN = \frac{\sum \frac{V_i}{d_i^2}}{\sum \frac{V_i}{d_i^3}} \]  

(3.6)

The area distribution mean particle diameter (MA), is also an average particle size value calculated from the volume distribution data, and this is associated with the surface area of the particles. It can be expressed in equation(3.7). The standard deviation (SD) obtained the particle size analyzer is a measure of the width of the distribution, not the variability within the measurement. The standard deviation (SD) for a particle size measurement can be expressed as half of the width of the normal distribution (84% volume -16% volume)(see Eq.(3.8)).

\[ MA = \frac{\sum V_i}{\sum \frac{V_i}{d_i}} \]  

(3.7)

\[ SD = \frac{(84\% - 16\%)}{2} \]  

(3.8)
1.12 Statistical Descriptors of Microstructures

To generate two point correlation functions for a series of images, the technique described by Yeong and Torquato was used, where sampling occurs along the two orthogonal directions of a binary image (along the rows and columns) (C l Y Yeong and S. Torquato 1998). To implement this sampling algorithm for a series of binary images in the Matlab environment, a box smaller than the size binary image must be placed inside the image, centered at its centroid. The box will act as were one endpoint of sampling line must be located as shown in Figure 0.4.

![Figure 0.4 Schematic of the sampling window used to calculate the two point correlation function using the method described by Yeong and Torquato.](image)

To generate the point (corresponding to a distance $r$) of the two point correlation function, a sampling line of length $r$ must be created with the coordinates of the sampling line being equal to $(x, y)$ and $(x, y+r)$. The sampling line of length $r$, will act as a two point probe into the image, in the same way as a structuring element is used in the morphological algorithm. If the two endpoints of the sampling line both lie within the pore space, the instance will be recorded as a success. The sampling line is then translated one pixel length down or across dependent of the orientation of the image. This
procedure is iterated until one of the endpoint encounters an edge. When this happens, the sampling begins in the next row or column (dependent on sampling). This process is repeated for all rows (or columns) and the value obtained from the two point correlation function for length $r$ can be calculated by the dividing the number of successes by the total number of translations. This process is repeated for all lengths $r$ up to $2/3$ the width of the images, thus allowing for the two point correlation function to be generated for lengths range from one pixel up to two thirds the width of the image.

For a set of images that correspond to a particular sample, the two point correlation function is generated for each individual image, then the average values for each value of $r$ is calculated to estimate the two point correlation function corresponding to the spatial arrangement of the pore space.
1.13 Particle size analysis of PMMA beads using image analysis

Characterizing the particle size distribution of the PMMA beads is important because they act as the templates for the pores of the final structure and the distribution of the PMMA beads affects the rheological behavior of the precursor material during the processing. Obtaining particle size distributions from laser diffraction-based particle size analyzer is the standard method of quantitatively characterizing particle size distribution. However because most laser diffraction-based particle size analyzers are expensive and require a considerable amount of maintenance to obtain reliable results, another method of quantitatively characterizing the particle size distribution may be desirable. Thus image analysis techniques were developed to quantitatively characterize the particle size distribution of the PMMA beads and these results were compared against those from a laser diffraction based particle size analyzer (Microtrac X100).

Samples of two different size distributions of PMMA beads used in the precursor material for the open cell copper foam were analyzed using both image analysis and laser diffraction-based particle size analysis. The coarse PMMA sample was obtained from Scientific Polymer Products Inc (Ontario, NY) and the fine PMMA sample was obtained from Altuglas (Philadelphia, PA). For the laser diffraction based particle size analyzer, the Microtrac x100 has a measurement range of 0.12 to 700 microns. 12 runs per sample of each of the different sized PMMA beads were performed. For each run, PMMA beads were suspended in deionized water and sonicated. The suspended PMMA beads were then placed into a variable speed recirculator using a pipette until the optimal sample loading of 0.10 transmittance. Within the Microtrac X100 software, the volume distribution-based particle size distribution would be calculated using the assumption that
the sample had a spherical shape and an index of refraction of 1.6. Each run consisted of three particle size distributions taken for 90 seconds of sampling.

For the image analysis method of obtaining the particle size distribution, optical micrographs of samples of two different batches of PMMA beads were taken using a Leica M165C with a light source projected from below the PMMA beads such that an outline of the PMMA sphere could be clearly identified. (see Figure 0.5) The optical micrographs were then processed using the thresholding method described in section 1.11.1 of chapter 3.

Figure 0.5 Optical Micrograph of a sample of PMMA beads

The marker controlled watershed algorithm, described in section 1.11.3, was performed on binary images of the optical micrographs to partition the pore space into individual pores. Since the watershed algorithm is useful only for segmenting a small cluster (2 or 3) of PMMA beads, a compactness term (Eq.(2.15)) is used to filter out only the projections of individual PMMA beads from clusters of PMMA beads. Particles with a compactness value less than .75 (i.e. Clusters of PMMA beads) were excluded from the
data set. To obtain information regarding individual particle sizes, two different metrics were used; the feret diameter also known as the maximum caliper, and the equivalent diameter. Both of these metrics were discussed in section 1.9.1.1.

To calculate the equivalent diameter \( D_{\text{equiv}} \) of the particles using Eq. Error! Reference source not found., the area and perimeter of the individual pores were measured using the REGIONPROPS function within matlab. The area weighted pore size distribution was obtained using the equivalent diameter of the pore as a measure of the pore size. Eq.(3.9) was used to calculate the area weighted effective diameter(\( D^\text{eff} \)) where \( A_{\text{pore}} \) is equal to the area of the particle and \( A_{\text{total}} \) correspond to the total area of all the particle in an image .

\[
D^\text{eff} = D_{\text{equiv}} \frac{A_{\text{pore}}}{A_{\text{total}}}
\]  

(3.9)

The area weighted pore size distribution was calculated by dividing the pores into classes. Each class has size range of 4.5 microns (due to the resolution of the images), starting from 0 to the maximum value of the equivalent diameters observed within a sample. The probability density of a bin, \( x \), with \( n \) number of pores can be calculated by summing all of weighted effective diameter \( D^\text{eff} \) within bin \( x \) (see Eq.3.10).

\[
\text{PDF } x = \sum_{i=1}^{n} D_i^\text{eff}
\]

(3.10)

After all of the probability densities have been calculated for all bins, the PDF for a particle distribution can be plotted against the size range associated with each bin such
that a graphical representation of the particle size distribution can be obtained. The cumulative probability of the particle can be calculated from the sum of all of the probability densities corresponding to the size ranges lower than \(x\). (see Eq. (3.11)). From the cumulative probability function, the area weighted mean particle diameter can be calculated by finding the size where the probability is equal to 0.5.

\[
CDF(x) = \sum_{i=1}^{x} PDF(i)
\]  

(3.11)

1.14 Procedure for quantitative characterization of sintered stainless steel filters

To validate the method developed to estimate the permeability using the modified KC equation (Eq. (2.3)) and quantitative characterization using image analysis, a open celled metal foam with known permeability and porosity values, obtained using ISO testing methods was characterized using the techniques described in section 1.11.

In this study, three commercially available sintered stainless steel foams (GKN Sinter Metals, Ohio) with manufacturer average pore sizes, permeability (obtained using ISO 4022) and manufacturer provided effective pore diameters (see Table 0.1). These were used to validate the image analysis based methods to quantitatively characterize the structure of the porous material and estimate the fluid permeability using a modified version of the Kozeny Carman equation.
Table 0.1 Table of Manufacturer Provided Data for sintered stainless steel foams

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Porosity</th>
<th>Pore Diameter Min(µm)</th>
<th>Pore Diameter Max(µm)</th>
<th>Effective Pore Diameter</th>
<th>Permeability Constant α</th>
</tr>
</thead>
<tbody>
<tr>
<td>S3</td>
<td>31%</td>
<td>4</td>
<td>11</td>
<td>6</td>
<td>0.6</td>
</tr>
<tr>
<td>S10</td>
<td>43%</td>
<td>12</td>
<td>25</td>
<td>17</td>
<td>3.8</td>
</tr>
<tr>
<td>S20</td>
<td>43%</td>
<td>12</td>
<td>37</td>
<td>20</td>
<td>7.2</td>
</tr>
</tbody>
</table>

1.14.1 Sample Preparation of Stainless Steel Filters

To prepare samples for imaging, samples were vacuum infiltrated and mounted in a low viscosity epoxy (Buehler Epoheat) and then sectioned. After sectioning, the vacuum infiltrated samples were ground flat and polished using standard metallographic techniques. Indentions were placed using a Vickers pyramid diamond indenter with a load of 1kg at 12 different locations on the polished surface of each sample, shown in Figure 0.7, consistent with the montage serial sectioning technique described by Gokhale (A. Tewari, A Gokhale, and German 1999).

Figure 0.6 Several views of the stainless steel filter
Digital images of the sample were acquired using backscatter scanning electron microscopy (BSEM) at a magnification of 110× (resolution 2.27 pixels/µm) using the Hitachi S-4700 scanning electron microscope. BSEM micrographs were obtained across the length of the sample as shown in Figure 0.7. After all images across a section were imaged, a thin layer was removed from the sample using metallographic grinding/polishing techniques; the amount removed was approximately 3-5 µm as determined from changes in the width of the hardness indentations. BSEM image acquisition was then repeated, using the micro hardness indents as a method of registering and locating the position of a particular image within the sample. The layer removal and image acquisition cycle were repeated resulting in a total of five sets of images per sample, as described in Figure 0.8.

Figure 0.7. Schematic of the BSEM micrographs per slice with location of indents marked with crosses
1.14.2 Image processing and image analysis

Conversion of the digital images from the original format (8-bit grayscale) to a binary image was performed using the thresholding algorithms described in section 1.11.1. Small pores and solid features less than 1.9 microns (10 pixels) were removed during the image processing to remove noise from the binary image. Porosity (\( \phi \)), surface area per unit area (\( s \)) and tortousity (\( \tau \)) were calculated for each binary image using the quantitative characterization techniques described in section 1.11. Eq.(2.22) was used to calculate the permeability for each binary image.

1.15 Effect of processing parameters on the structure of open celled copper foams

To better understand how processing affects the permeability and the structural characteristics of an open celled copper foam, open celled copper foams were made varying the batch composition and the heat treatment temperature. These samples were then quantitatively characterized using the method described in section 1.11.
1.15.1 Volume percentage of copper

The effect of changing the batch composition of the precursor slurry used to make an open celled copper foam on the permeability was studied by estimating the permeability of samples made with different percentages of copper powder used in the batch composition of the precursor slurry. Due to processing limitations associated with changes in rheological behavior of the slurry, only two volume percentages of copper were tested: 17.06% vol. copper and 25.05% vol. copper. The slurries were spun at 1000 rpm for 15 seconds onto a copper substrate on a LAURELL WS-650-23 spin coater. Immediately after the spinning step, the sample of the 25.06% vol. copper had an average thickness of 295 microns. Both samples were processed in the same manner. The samples were heat treated using a heat schedule of 3°C/min to 800°C with hydrogen flowing at a rate of 0.10 liters per minute for both the heating and cooling stages of the samples.

The samples were processed in a method similar to the method described in section 1.14.1 and shown in Figure 0.10, where the copper foam samples were cross-sectioned, vacuum infiltrated with a low viscosity epoxy, ground and polished, microindented and then imaged. However, to the geometry of the sample, a greater magnification was used to acquire BSEM images of the open celled copper foams. The image processing procedure performed on the imaged microstructure of the open celled copper foam is the same procedure described in section 1.14.2 where the porosity, surface area per unit volume and tortuosity are calculated from binary images. The permeability of the samples was calculated using Eq. (2.22). The pore space in the binary images was partitioned using the marker controlled watershed algorithm. And the area weighted particle size distributions were obtained for the samples with the two different
compositions, and the effective mean particle diameter was obtained for each of the samples.

### 1.15.2 Heat Treatment

To study the effect of heat treatment on the structural parameters of an open celled copper foam, the heating schedule was modified such that a set of copper foam samples were heated to 700°C and 800°C. Each set of copper foam samples was created from the same precursor batch which had 17.06% vol. copper powder and 82.94% coarse PMMA beads. The samples were spun at 1000rpm for 15 seconds. The average thickness immediately after spinning of the pre-heat treatment samples was 286.4 microns. The sample were heated at a rate of 3°C/min either to 700°C or 800°C with hydrogen flowing at a rate of 0.10 liters per minute during the heating and cooling of the sample.

These open celled copper foam samples were processed in the same method as the open celled copper foams were processed in section 1.15.1.

### 1.16 Digitally constructed microstructures

From the particle size distributions obtained from the image analysis of PMMA beads, a digital construction of the open celled copper foam can be generated based upon the packing of the PMMA beads. In this section, the procedure involving in generating packed beds of spheres using the particle size distribution of the PMMA beads is described followed by the generation of pore neck. After the pore necks geometry is discussed, the procedure involved in the spherical contraction algorithm is be described.
1.16.1 Generation of packed beds

Digital microstructures were generated using a particle packing algorithm developed by Kulpe (Kulpe 2010). This algorithm was being developed as a tool to predict the resulting structure of a open celled metal foam based upon the particle packing of the precursor sacrificial template material: the PMMA spheres.

The particle packing algorithm begins with the generation of a seed configuration, where three touching spheres are placed within an specified domain with dimensions x, y and z. For these digitally constructed microstructures, the radii of the touching sphere are obtained by randomly selecting from an array of PMMA particle sized measured values obtained from the image analysis. They are the same beads used in the processing of open celled copper metal foams. After the seed configuration is generated, a new sphere is placed to form a tetrahedron using the packing configuration where the center to center distances are used as the slide lengths of the packing tetrahedron in place of using sum of the length of the radii (see Figure 0.9). This packing configuration uses the center to center distances instead of the length of radii was used due to the computational speed at which the microstructures could be generated. After the initial tetrahedron is formed, three of the four spheres are randomly selected; another particle is placed based on the formation of the packing tetrahedron described. This process is repeated until a target volume fraction of spheres within sampling domain is achieved.
Figure 0.9 Two different packing methods. Left: Tetrahedron formed using radius of the spheres. Right: Tetrahedron formed from center to center distances.

1.16.2 Pore Neck Geometry

Once the target volume fraction of spheres is achieved, inter-particle necks for the packed spheres are generated. This is associated physical phenomena of extraction of the PMMA beads; because the copper particles used in the processing of the copper foam, necks form where two PMMA touch due to the fact that the copper particle cannot fit into the interstitial space where the two PMMA beads touch. The necks are formed by placing a revolved circular arc at each inter-sphere connection point and Eq. (3.12) was developed to describe how the geometry of the neck is calculated, where $\rho$ is the neck radius and $\kappa$ is the proportional constant and $r_i$ and $r_j$ are the radii of the two spheres from which the pore neck is formed. The proportionally constant, $\kappa$, ranges from 0 to 1. When $\kappa = 0$, no pore necks are generated, and when $\kappa=1$ the pore neck generated resembles a straight between the two radii $r_i$ and $r_j$.

$$\rho = \kappa \left( \frac{r_i}{r_2} \right)^2 r_i + \left( \frac{r_i}{r_j} \right)^2 r_j \right)$$ (3.12)
1.16.2.1 Study of Pore geometry

To study the how the $\kappa$ value changes the digital constructed microstructure, a packed bead of spherical beads with the radii of the spheres randomly selected from the particle size distribution of the coarse PMMA beads is created. The packed bed of spheres has 75.6% volume fraction of the sampling domain consisting of spheres. A series of binary images were generated from the digitally constructed microstructure with different $\kappa$ values used to calculate the pore necks using the “sphere_vol” script described in the work of Kulpe (Kulpe 2010b). The different $\kappa$ values used to generated the pore necks were used to determine how the pore space changes as a result of the changing the neck geometry. In the resulting binary images, the spheres and necks are represented as 0 and space that does not correspond with either sphere or neck is presented as 1, thus implying that the spheres and necks represent the pore space whereas the areas represented by 1s correspond with the sold phase. This representation of the packed sphere more closely resembles the final structure of the open celled copper foams because during the heat treatment of the copper foams. The PMMA beads act as sacrificial template and thermally decompose thus leaving pores that have characteristics similar to the original beads.

Quantitative characterization of the microstructure of the four different digital constructed microstructures was performed using the same method similar described from the previous studies, where the porosity, surface area per unit volume and tortuousity are calculated for all of the images. The binary images are partitioned so that quantitative characterization of the pores can be also performed. In addition to these methods of

55
quantitative characterization, the two point correlation functions were generated for all of
the digitally constructed microstructures.
1.16.3 Spherical Contraction

After a sphere packing is achieved, a spherical contraction algorithm is used to simulate the contraction of the solid phase that occur during heat treatment. The method by which the algorithm computes the contraction of the solid phase is based upon the work of Kulpe (Kulpe, Lin, and Nadler 2010) and the procedure is outlined in Figure 0.10. In the spherical contraction algorithm, pore necks are generated for the initial sphere packing using a given \( \kappa \) value. The porosity of the domain is calculated and if the porosity of the domain \( (\phi_{\text{pore}}) \) is less than the desired porosity \( (\phi_{\text{derived}}) \), then the radius of each sphere within the domain is multiplied by a factor \( \gamma \). The pore neck geometries are then recalculated based upon the value obtained from multiplying the radius by \( \gamma \) and the \( \kappa \) value used to calculate the initial pore necks. Then the porosity is recalculated for the domain and compared against the desired porosity. If the porosity of the domain is less than the desired porosity then the process is repeated until the porosity of the domain is equal to or greater than the desired porosity. The corresponding process diagram is shown in Figure 0.10.

![Logic Diagram describing the steps used in the spherical contraction algorithm](image)

Figure 0.10 Logic Diagram describing the steps used in the spherical contraction algorithm (Kulpe, Lin, and Nadler 2010)
RESULTS AND DISCUSSION

In this chapter, laser diffraction based particle size distributions are compared against particle size distributions obtained by using the feret diameter and equivalent diameter used as a measure of particle size to determine if image analysis can be used as a method of characterizing particle size distribution. The results of a study documenting the validity of using the Kozeny Carman equation and image analysis to estimate the fluid permeability of a structure will be presented. A study exploring the effect of heat treatment and precursor composition on the permeability and structure of a open celled copper foam to determine how the optimize the performance of the copper foam as a thermal wick. Then characterization of digitally constructed materials used to simulate the pore structure of an open celled copper foam are presented and the digitally constructed materials are compared with real microstructures. Conclusions drawn from the data analysis are presented in the next chapter.

1.17 Particle size analysis using image analysis

The particle size distributions of two sizes, types PMMA spheres are obtained using the feret diameter as a measure of particle size and the equivalent diameter as a measure of particle size as described in section 1.13. The results from image analysis are compared against data obtained from laser diffraction based particle size analysis.

1.17.1 Image Analysis based Particle Size Distribution

From the image processing of the optical micrographs of the fine PMMA samples, the area weighted particle size distribution was calculated using the equivalent
diameter as method of quantifying particle size described in section 1.13. The resulting particle size distributions can be seen in Figure 0.1.

Figure 0.1. Particle Size Distribution of the Fine PMMA beads obtained using equivalent diameter and feret diameter as a measure of particle size. a) Particle Size Distribution of PMMA beads using the Equivalent Diameter as a measure of particle size. B) Particle Size Distribution of the PMMA beads using the Feret Diameter as a measure of particle size. c) Cumulative distribution function of the particle size distribution using the two different methods of describing the particle sizes.
The distribution of fine PMMA beads had an average feret diameter of 15.06 μm with a standard deviation of 5.23 μm and an average equivalent diameter of 12.86 μm with a standard deviation of 4.42 μm (see Figure 0.1 and Table 0.1).

Table 0.1 Mean Particle Sizes of the different sized samples of PMMA beads

<table>
<thead>
<tr>
<th>PMMA Sample Size</th>
<th>Average Feret Diameter (μm)</th>
<th>Average Equivalent Diameter (μm)</th>
<th>Average Major Axis (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fine</td>
<td>15.06(±5.23)</td>
<td>12.86(±4.43)</td>
<td>14.34(±5.06)</td>
</tr>
<tr>
<td>Coarse</td>
<td>93.31(32.86)</td>
<td>77.37(±26.12)</td>
<td>86.91(±31.25)</td>
</tr>
</tbody>
</table>

To compare the different methods of quantifying the distribution of the fine PMMA particles, a cumulative distribution function (CDF) for each particle size distribution was created using Eq. (3.11). The calculated CDFs are shown in Figure 0.1c. The calculated CDF using the feret diameter as a measure of particle size is similar in shape to the CDF of using the equivalent diameter as a measure of particle size. However, the feret diameter based CDF results in a larger overall particle size distribution. These results are due the methods by which particle size is quantified, the equivalent diameter uses the area of the particle to calculate an equivalent circle whereas the feret diameter calculates the maximum distance that spans the projected particle.

The distribution of coarse PMMA beads using the feret diameter as a measure of particle size (see Figure 0.2a) had an average particle diameter of 93.31 μm with a standard deviation of 32.86 μm (see Table 0.1), and the distribution of coarse PMMA
beads using the equivalent diameter as a measure of particle size (Figure 0.2b) results in an average particle diameter of 77.37 μm with a standard deviation of 26.13 μm (see Table 0.1). The shape of the particle size distribution using the equivalent diameter as a measure of particle size (Figure 0.2a) is very similar in shape to the particle size distribution obtained using the feret diameter as shown in Figure 0.2b.

To compare the different methods of quantifying the particle size distribution of the coarse PMMA beads, the procedure used to calculate the CDFs for the fine PMMA beads was also performed on the coarse PMMA bead sample. The resulting particle size distributions can be seen in Figure 0.2. When comparing the shape of the cumulative distribution functions of the two methods of measuring particle size, there are subtle differences in the particle size distributions at particle sizes less than 50 μm. From the CDFs of the coarse PMMA beads, the feret diameter as a measure of particle size results in a larger measurement of the particle size.
Figure 0.2. Particle Size Distribution of the Coarse PMMA beads using the equivalent diameter and feret diameter as a measure of particle size. a) Particle Size distribution of the Coarse PMMA beads using the Equivalent Diameter as a measure of particle size. b) Particle Size distribution of the Coarse PMMA beads using the feret diameter as a measure of particle size. C) Cumulative Probability Distribution of the two different methods of representing the particle size distribution.
1.17.2 Particle Size Analyzer Data

Samples of the fine PMMA beads were run on a laser diffraction based particle size analyzer (Microtrac X100) using the procedure described in section 1.13. The average of volume weighted particle size distribution obtained from 12 runs on the particle size analyzer for the fine PMMA beads are plotted in Figure 0.3a and Table 0.2 summarizes the mean diameters of the different weighting methods used for on the particle size distribution obtained from the particle size analyzer.

The MV for the fine PMMA sample was calculated to be 18.90 microns, whereas the MN of the fine PMMA sample was calculated to be 11.86 microns and the MA of the PMMA sample was calculated to be 17.41 (see Table 0.2). From the volume weighted particle size distribution of the fine PMMA sample (Figure 0.2a), the fine PMMA particle size distribution had two distinct peaks: one in the 6-7 micron range and another within the 20-25 micron range. Although the volume associated with the 6-7 μm range peak varied from measurement to measurement, its location did not change. The peak at the 20-25 μm range behaved in a similar manner. Using the peak deconvolution algorithm in the Microtrac X100 software program, it was found that 41.25% of the volume of the fine PMMA bead sample is inside the peak with a particle diameter centered at 6.41 μm and the other 58.75% of the volume of the fine PMMA beads sample is inside the peak centered at 17.84 μm (see Figure 0.2).
Table 0.2. Summary of the data obtained from the particle size analyzer

<table>
<thead>
<tr>
<th></th>
<th>MV(μm)</th>
<th>MN(μm)</th>
<th>MA(μm)</th>
<th>Error(μm)</th>
<th>Particle Diameter(μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fine</td>
<td>18.90</td>
<td>11.86</td>
<td>17.41</td>
<td>5.97</td>
<td>17.84(58.75%) 6.41(41.25%)</td>
</tr>
<tr>
<td>Coarse</td>
<td>99.58</td>
<td>83.59</td>
<td>93.83</td>
<td>21.21</td>
<td>91.26(100%)</td>
</tr>
</tbody>
</table>

Figure 0.3. Particle Size Distribution of the two different samples of PMMA beads obtained from laser diffraction based particle size analyzer. Left: Fine PMMA beads Right: Coarse PMMA beads

The average particle size distribution result obtained from 12 measurements on the particle size analyzer for the coarse PMMA beads are plotted in Figure 0.3b. The calculated values for MV and MN for the coarse PMMA sample is 99.58 microns and 93.59 microns respectively. The calculated MA for the coarse PMMA sample is 93.83 microns. The coarse PMMA sample exhibits normal distribution with single peak centered at 91.26 μm (see Figure 0.3).

For the processing methods used to produce open celled copper foams, the volume weighted mean diameter (MV) will be used to describe the particle size.
distribution of particles within the system because the MV based method describes the central tendencies of the particle size distribution, such that the effect of the big particles is weighted more than that of the small particles.

1.17.3 Comparison of Particle Size Distributions

Comparing the volume weighted particle size distributions obtained from the particle size analyzer with particle size distributions obtained from image analysis of the PMMA bead samples requires that the data obtained from the image analysis also be weighted by a weighting factor. The particle size distribution obtained from image analysis is weighted with the projected area of the PMMA beads using Eq (3.9) and the probability density function associated with a size range is calculated from Eq.(3.10). The area weighted mean diameter obtained from image analysis of the particle is compared against the volume weighted mean particle diameters obtained from the particle size analyzer (see Figure 0.3 and Table 0.2).

For the fine PMMA beads particle size distribution, using the mean of the unweighted particle size distribution results in value of 15.06 microns. These values of the effective diameter obtained from overestimates the MN obtained from particle size analyzer by 26.95% and underestimates the MV obtained from particle size analyzer by 20.30%, and underestimates the MA obtained from the particle size analyzer by 13.49% (see Table 0.2). Using the equivalent diameter to calculate area weighted mean diameter as the effective diameter results in the calculated value for the effective diameter being 16.31 microns. This value calculated for the effective diameter overestimates the MN obtained from the particle size analyzer by 37.56%, and underestimates the MV obtained
particle size analyzer by 13.65% and underestimates the MA particle size analyzer by 6.26% which means that the effective particle calculate using the area weighting method estimates the area-weighted particle diameter obtained from particle size analysis most accurately. This result is to be expected because the both methods of calculating the mean particle diameter rely on a weight factor that is based upon the projected area of a particle.

Table 0.3. Comparison of the Mean Particle Diameters calculated from particle size analyzer and image analysis

<table>
<thead>
<tr>
<th></th>
<th>Mean particle diameter (µm)</th>
<th>Area weighted particle diameter(µm)</th>
<th>MN(µm)</th>
<th>MV(µm)</th>
<th>MA(µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fine PMMA</td>
<td>15.06</td>
<td>16.31</td>
<td>11.86</td>
<td>18.90</td>
<td>17.41</td>
</tr>
<tr>
<td>Coarse PMMA</td>
<td>93.31</td>
<td>99.58</td>
<td>83.59</td>
<td>99.58</td>
<td>93.83</td>
</tr>
</tbody>
</table>

For the coarse PMMA bead sample, using the mean of the unweighted particle size distribution results in a value of 93.31 microns for the effective diameter. This calculated value for the effective diameter results in a 11.63% overestimate of the MN and a 6.30% underestimate of the of the MV and a 0.55% underestimate of the MA (see Table 4.3). Using the equivalent diameter to calculate the area weighted mean diameter as effective diameter results in a value of 99.97 microns for the effective diameter. This calculated value for the effective diameter overestimates the MV by 0.004%, and overestimates the MN by 19.95% and overestimates the MA by 6.55%. The results indicate that the area weighted method of calculating the mean particle diameter accurately describes the volume weighted mean particle diameter obtained from the
particle size analyzer. These results are not consistent with the results obtained from the fine PMMA beads, the discrepancy between the two methods of measurement could be a result of how the distributions differ. However, the area weighted method of calculating the mean particle diameter does provide an estimate of the MA (6.55% error).

The cumulative distribution function of the area weighted particle size distribution and the volume weighted particle size distribution obtained from the particle size analyzer are plotted in Figure 0.4. From these graphs in, using the area weighted method of calculating the particle size distribution provides a good fit for the volume weighted particle size distribution of the coarse PMMA beads. However the area weighted method of calculating the pore size distribution for the fine PMMA beads does not provide a good fit to the volume weighted particle size distribution obtained from the particle size analyzer. The reason for this discrepancy can be seen from by comparing the particle size distributions obtained from the particle size analyzer (see Figure 0.4); the particle size distribution of the coarse PMMA beads contains only one distinct peak, where as the particle size distribution of the fine PMMA beads shows two distinct peaks.
Figure 0.4 Cumulative Probability Distribution Function of the volume weighted particle size distribution obtained from the particle size analyzer (blue) and using the area weighted equivalent diameter (red) for a) coarse PMMA beads b) fine PMMA beads

1.18 Validation of Image Analysis Techniques: Stainless Steel Study

The results obtained from the image analysis of three stainless steel samples are compared against the known fluid permeability values to validate the techniques that are used to calculate fluid permeability for other types of porous materials in terms of determining structural parameters and fluid flow parameters of a open celled sintered metal foam. The procedure used in this study is described in greater detail in Section 1.14. The permeability of these structures was calculated using a modified Kozeny Carman equation (see Eq (2.22)) and these observations and their interpretations are presented below.

1.18.1 Quantitative Structural Characterization

For the quantitative structural characterization of the sintered metal filters, 3 samples of stainless steel metal filters were characterized using image analysis. The three samples consisted of a same with 3 micron feature size (S3), a 10 micron feature size (S10) and a 20 micron feature size(S20). The porosity, surface area per unit volume and tortousity was calculated for each sample and the values obtained were used to calculate
the permeability of each sample. A pore level analysis was also performed on each sample, where the effective pore diameter and pore size distribution of each sample was calculated.

1.18.1.1 Structural Parameters and Permeability Calculations

Figure 0.5 shows binary images of the planar cross sections of the three stainless steel samples used in this study. Table 0.4 summarizes the results of the quantitative characterization of porosity, surface area per unit volume and tortousity needed to calculate the permeability.

The 3 micron pore size sample (S3) had the least amount of porosity (20.27%) and the greatest surface area per unit volume (0.08 µm²/µm³), yet the calculated permeability of the 3 micron pore size sample was the smallest (0.63 × 10⁻¹² m²), indicating that the 3 micron pore size sample was the least permeable of all three samples. These results correspond well with the manufacture provided data (see Table 0.1) with the calculated value of fluid permeability overestimating the manufacture provided value of fluid permeability by 5.16 % (see Table 4.4).

The 10 micron pore size sample (S10) had a porosity of 36.64%, a 16.37% increase from the porosity of S3 micron pore size sample. For S10, there was a 18.05% reduction in surface area relative to S3 and the calculated value for the fluid permeability of the S10 sample was 3.80 × 10⁻¹² m², a 0.11% overestimate of the manufacturer provided permeability (see Table 4.4).

The 20 micron pore size sample (S20) exhibited a 2.51% decrease in porosity relative to S10 and exhibited the smallest surface area per unit volume (0.05 µm²/µm³) of all samples. S20 exhibited 40.98% less surface area then S3, yet S20 micron pore size
sample exhibited the largest fluid permeability value (7.56) of the commercial metal foams. The calculated value of fluid permeability was 4.97% greater than the manufacturer provided value (see Table 4.4).

Table 4.4 Summary of the calculated structural parameters of the three different stainless steel samples.

<table>
<thead>
<tr>
<th></th>
<th>S3</th>
<th>S10</th>
<th>S20</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porosity</td>
<td>20.27%</td>
<td>36.64%</td>
<td>34.13%</td>
</tr>
<tr>
<td>Surface Area ($\mu m^2/\mu m^3$)</td>
<td>0.08</td>
<td>0.07</td>
<td>0.05</td>
</tr>
<tr>
<td>$K_{calculated}$ ($10^{-12}$ m$^2$)</td>
<td>0.63</td>
<td>3.80</td>
<td>7.56</td>
</tr>
<tr>
<td>$K_{manufacturer}$ ($10^{-12}$ m$^2$)</td>
<td>0.60</td>
<td>3.80</td>
<td>7.20</td>
</tr>
<tr>
<td>Error</td>
<td>-5.16%</td>
<td>-0.11%</td>
<td>-4.97%</td>
</tr>
</tbody>
</table>
Figure 0.5. SEM images of the surface and the planar cross section of the three stainless steel filters, S3, S10 and S20.
1.18.1.2 Pore Space Calculations

A set of binary images for each of the three samples were processed using a marker controlled watershed algorithm, where the pore space was partitioned into individual pores based on the morphology of the pore space as described in section 1.14.2. The area weighted pore size distributions were calculated for each set of images using Eqs.(3.9) and (3.10). Figure 0.6 illustrates the area weighted pore size distribution of for the stainless steel specimen S10, with the cumulative distribution of the pores (dotted line) plotted over the PDF. The effective diameter, which can be quantified by the mean area weighted particle diameter, using the equivalent diameter as a measure of particle size and Eq. (3.9), is analogous with the hydrodynamic concept of an equivalent diameter (J Bear 1988). Thus the calculated value for the effective diameter of S10 occurs at 16.64 microns with a standard deviation of 7.83 microns and calculated value for the effective diameter of S3 occurs at a diameter of 6.56 microns, and the pore size distribution has a standard deviation of 4.09 microns (see Table 0.5). The area weighted pore size distribution for S20 has a calculated value for the effective diameter occurring at 24.08 microns and the standard deviation of the distribution was 10.93 microns (see Table 4.5). These results indicate that the feature size of the three different stainless steel specimens have significantly different feature sizes, which means that the surface area per unit volume and tortuosity values for these specimens should be inversely related to the feature size. Thus the S20 specimen should have the smallest surface area to volume ratio and the S3 specimen should have the greatest surface area to volume ratio.
Table 0.5. Effective pore diameter values for the three stainless steel specimens

<table>
<thead>
<tr>
<th>Sample</th>
<th>S3</th>
<th>S10</th>
<th>S20</th>
</tr>
</thead>
<tbody>
<tr>
<td>Calculated effective pore diameter (µm)</td>
<td>6.56</td>
<td>16.64</td>
<td>24.08</td>
</tr>
<tr>
<td>Manufacturer provided effective pore diameter (µm)</td>
<td>6.00</td>
<td>17.00</td>
<td>20.00</td>
</tr>
</tbody>
</table>

Comparing the calculated values of the effective diameter to the manufacture provided data on the effective diameter as shown in Table 4.5, the calculated value of effective pore diameter for S3 over estimates the manufacture provided value by 9.33%. For the S10 sample, the calculated value for the effective pore diameter underestimates the manufacture provided data by 2.11% and the calculated effective pore diameter of the S20 sample overestimates this effective pore diameter by 20.4%. The mean effective diameter, calculated from the area weighted distribution, can be a method used to quantify the hydrodynamic equivalent diameter. However, future development of the portioning algorithm is needed to reduce the error associated with partitioning the pore function by refining the external limit used on the marking function of the marker-controlled watershed function (see section 1.11.3). Also if there was an expected pore morphology, a structuring element with characteristics resembling the expected pore morphology could be used to partition the pore space more accurately. To determine the structuring element to be used, the shape of the pore template should be used. If the shape of the pore template is not known, the user must define a shape based upon visual inspection of a binary image of the structure.

The cumulative distribution of the area weighted pore size distribution for the three different samples are plotted in Figure 0.7. Comparing the cumulative distribution
functions of the S3 to the S10 and S20 samples, the shape of the cumulative distribution function for the S3 sample approaches 100% at the lowest values, indicating that the pore size distribution for the S3 sample is narrower relative to the other particle size distributions. In a likewise manner, the cumulative distribution function for the S10 sample approaches 100% at values lower than the S20 sample did, indicating that the pore size distribution of the pores in the S10 samples are smaller than the pores found in the S20 sample. The pores in the S3 sample are much smaller than the pores of the S10 or S20 sample with approximately 80% of the pores in the S3 sample having an equivalent diameter smaller than 5 microns. For the S10 sample, approximately 80% of the pores in the S10 sample have a pore size smaller than 20 microns and for the S20 sample, approximately 80% of the pores in the S20 sample have a pore size smaller than 25 microns. These results are consistent with the expected results and provide a graphical representation that the pore size distributions of the three different stainless steel samples are distinctly different.
Figure 0.6 Area weighted particle size distribution of the pores in the S10 sample with the cumulative distribution function plotted on the secondary axis.

Figure 0.7 Cumulative Distribution Function for the three stainless steel samples S3 (yellow), S10 (blue), S20 (red).
1.19 Effect of Processing Parameters on the Pore Structure of Copper Foam

In this section, the results of quantitative characterization of the porosity, surface area per unit volume and tortuosity of copper foams with different batch compositions and heat treatment temperatures are discussed to determine the effect of processing parameters on the structure and permeability of the copper foams. The results for the pore level analysis of the copper foams and its implications will also be discussed.

1.19.1 Processing Parameters: Volume percent of Copper

The effect of changing the measured solids volume fraction of precursor on the porosity, surface area per unit volume, tortuosity, effective pore size, and permeability of a open celled copper foam was examined. Figure 0.6 shows an example of a cross section of the copper foam sample. In the pre heat treatment processing, two different batch compositions were used; one with 25.05% volume of copper and 74.95% volume of coarse-sized PMMA beads, the other with 17.06% volume of copper and 82.94% volume of coarse-sized PMMA beads. The processing of the two samples was described in section 1.15.1. Table 0.6 summaries the results from the quantitative characterization of the open celled metal foams with different batch compositions.
From Table 0.6, there is a reduction in the porosity resulting from increase in the volume of copper by 11.27%. These results are expected because volume of copper powder available for sintering in the 25.05% vol. copper sample is greater than that of the 17.06% vol. copper sample. The 17.06% vol. copper sample exhibited a 25.57% reduction in pore space from the pre-heat treatment composition (82.94%) where as the 25.05% vol. copper sample exhibited a 28.85% reduction in pore space compared to the pre-heat treatment composition (74.95%). This reduction in porosity could be a result of an increase in number of the copper particle to particle contact in the sample with the greater volume of copper. These contact points are as sites where the onset of sintering
occurs, which would result in a more rapid densification of the structure for a given temperature.

Regarding the surface area per unit volume, the 17.06% volume copper sample exhibits 18458.76 m²/m³ more surface area than the 25.05% volume cooper sample. The greater surface area seen for the 17.06% vol. copper sample indicates either less densification of the copper particles in the 17.06% vol. copper sample or that pores in the 17.06% sample are more evenly dispersed throughout the sample. The tortuosity value for the sample with the 17.06% vol. copper (5.70) is greater than the tortuosity value for the sample with 25.05% vol. copper (2.69) (see Table 4.6). These results may indicate that the samples with 17.06% vol. copper tend to have longer flow paths due to the greater tortuosity and greater surface area per unit volume. The result also indicates that the copper particles are more likely to be evenly distributed throughout the sample rather than agglomerating. This could be investigated by obtaining the two point correlation function or the pore size distribution function using image analysis.

The 25.05% vol. copper sample has a greater calculated value for the fluid permeability than the 17.06% vol. copper sample. These results do not correspond with what was expected, the greater porosity and increase tortuosity should indicate that the 17.06% vol. sample would be more permeable than the 25.05% vol. copper sample due to the increase number of possible flow paths (see Table 4.6). Calculations of copper samples made with different volume percentages of copper indicate that the difference between the calculated tortuosity values contributed to the deviation from what is expected. The 17.06% vol. copper foam sample has a calculated tortuosity value (5.7) that is 2.12 times the calculated value obtained for the 25.05% vol. sample. This
significant difference in the calculated tortuosity could be due to the tortuosity algorithm handles the small pores associated with the less sintering densification seen in the 17.06% vol. copper foam sample but not seen in the 25.05% vol. copper foam sample (see Figure 4.9). The small pores seen in 17.06% vol. sample would create skeleton that would be much more tortuous than the skeleton of the sample without the small pores present (see Figure 0.9). Further preprocessing such as an “fill holes” operation, of the image would be necessary to remove the small pores seen in the 17.06% vol. copper foam sample. These small pores can be computationally ignored for the permeability calculations because they do not significantly contribute much to the fluid permeability after the sample is saturated with the fluid.

Another reason for the discrepancy between the expected and actual permeability is due to some of the assumptions in the Kozeny Carman equations; one must assume that simple connectedness properties for the porous structure are described using an average tortuosity value. It only takes into account the average length of the pathway the fluid takes, but not the total number of possible pathways through which a fluid would flow. For these open celled sintered copper foam sample, Eq.(2.22) is therefore not sufficient for the estimation of the fluid permeability. Either another method such as the Stokes-Brinkman methods (see section 1.10.2) must be used to estimate the fluid permeability or the Kozeny Carman equation needs to be modified to account for the number of the possible pathways. Modifying the Kozeny Carman equation to account for the number of possible pathways would be a more feasible approach, because the information need for the calculations can be obtained by modifying the tortuosity
algorithm to calculate the number of possible pathways from one randomly selected node
to another randomly selected node.

Table 0.6. Summary of the Structural Parameters obtained from Quantitative
Characterization of the structure using image analysis.

<table>
<thead>
<tr>
<th></th>
<th>17.06%</th>
<th>25.05%</th>
</tr>
</thead>
<tbody>
<tr>
<td>Copper</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Porosity</td>
<td>57.37%</td>
<td>46.10%</td>
</tr>
<tr>
<td>Surface Area (m²/m³)</td>
<td>60284.4</td>
<td>41825.6</td>
</tr>
<tr>
<td>Tortuosity</td>
<td>5.7</td>
<td>2.69</td>
</tr>
<tr>
<td>Permeability (m²)</td>
<td>25.74</td>
<td>27.69</td>
</tr>
<tr>
<td>Effective Pore Diameter (µm)</td>
<td>40.70</td>
<td>45.56</td>
</tr>
</tbody>
</table>

Figure 0.9 Processed binary cross sectional image of the pore structure (right) and
skeleton of the pore space (left) of a 17.06% vol. copper foam sample and the 25.05% vol. sample.
To more completely characterize the effect of changes in the batch composition, area weighted pore size distributions were calculated for the two open celled sintered copper foam with different copper volume percentages of 17.06% and 25.05%. The area averaged pore size distributions for both compositions are shown in Figure 0.10. From the cumulative probability functions of the two samples, the calculated value for the effective pore diameter of the 17.06% vol. copper foam sample is 40.704 microns, whereas the calculated value of the effective pore diameter for the 25.05% vol. copper foam sample is 45.557 microns (see Table 4.6). These results are consistent with the expectation that as the volume fraction of the copper increases, the pores will become increasing isolated due to the copper particle agglomeration. Reducing the volume fraction of copper in turn reduces the ratio of copper to templating material, so distribution of the two constituents should be more evenly distributed throughout the structure. In addition as the template polymer spheres thermally decompose during heat treatment, the remaining pores are more evenly dispersed. This effect of pore distribution could also be a result of the increase in the number of inter-bead contact points. During the heat treatment process the sacrificial templates thermally decompose at temperatures lower than the onset of significant copper sintering, so the pore space shrinks due to the way the copper phase contracts.

The results obtained from the quantitative image analysis of the structural and fluid flow parameters also correspond with the data obtained from the portioning of pore space and the quantification of the effective diameter described in section 1.15.1; the smaller value for the effective pore diameter of the 17.06% vol. copper sample than the
25.05% vol. sample indicates that the tortuosity of the 17.06% vol. sample should be greater than the tortuosity value of the 25.05% sample. These results indicate that the calculation of an effective pore diameter may be useful in predicting differences on the surface area per unit area, tortuosity and permeability of an open celled copper foam. Studies have used the effective diameter as a measure of pore size (ARYA et al.; Armatas 2006; Liang 2000a) and the pore size can play an important role in estimating heat transfer in open celled copper foams (W Lu, Zhao, and Tassou 2006; Virto, Carbonell, and Castilla 2009; PETRASCH et al. 2008).

From the cumulative pore size distribution of the two copper foam samples shown in Figure 0.10(c, the pore size distribution of the 17.06% vol. copper sample is much smaller than that of the 25.05% vol. copper sample; 80% of the pores for the 17.06% vol. copper sample are below 40 microns while as for the 25.05% copper sample, 80% of the pores have an equivalent diameter of less than 50 microns. This indicates that the pores within the 25.05% vol. copper sample tend to be larger than the pores of the 17.06% vol. copper sample.

From the probability density functions shown in Figure 0.10(a and b, the distribution of the pore sizes within each size range is very different. The pores with the 17.06% vol. Copper foam samples tend to occur within the 26-34 micron size range with approximately 40% of the pores occurring within 26-34 micron size range. For the 17.06% vol. copper foam sample, the frequency of pores having an equivalent diameter in the 44-60 micron range decreases from the frequency of pores having an equivalent diameter in the 26-34 micron range or the 40-44 micron range of 15% or greater to a frequency of less than 10%. A different frequency distribution shape is seen for the pores.
of the 25.05% vol. copper sample. The frequency of these pores having a equivalent
diameter within the different size ranges is approximately 10% for the sizes classes
ranging from 26-54 microns, with a small peak occurring at 30-34 microns and another
peak occurring at 50-54 microns, and a small trough occurs at 46-50 microns( see Figure
4.10c).

The 17.06% vol. copper sample has a narrower distribution of pores with a
distinct peak at the size range of 30-34 microns( see Figure 4.10a), where as the
distribution of pores within the 25.05% vol. copper sample has a more uniform
distribution of pores( Figure 4.10b). Studies have shown that shape and range of pore
size distributions are can play a role in the diffusive transport properties of a porous
material. (S Torquato and Avellaneda 1991; VOGEL 1997; Armatas 2006) , with
Armatas stating that the most effective pore network( for diffusive properties) should
have a well connected pore network consisting of one pore size (Armatas 2006).
Therefore, the transport properties of the 17.06% vol. copper sample should have more
diffusive pore network with pores having a larger coordination number than the 25.05%
vol. copper sample due to the narrower, more uniform particle size distribution.
Figure 0.10 (a) Probability Density Function for the equivalent diameter of the pores of a copper foam sample with 17.06% volume of copper. (b) Probability Density Function for the equivalent diameters of the pores of a copper sample with 25.05% volume of copper. (c) Cumulative Density Function of the equivalent diameters of the pore of the copper samples with different volume percentages of copper.
1.19.2 Processing Parameters: Heat treatment

The effect of heat treatment on the structural parameters is discussed in this section following the procedure described in section 1.15.2. Two copper foam samples of 25.05% vol. copper were prepared where one of the copper samples was heated to 700 °C at 3°C/min, while the other was heated to 800 °C at 3°C/min. Figure 0.11 shows the processed binary images of a cross section of these copper foam samples processed at two different heat treatment temperatures.

![Binary Images of the cross section of two copper foam samples](image)

Figure 0.11 Binary Images of the cross section of two copper foam samples with the same composition heat treated at different temperatures with a summary of the structural parameters associated with the different heat treatments

Table 4.7 summarized the changes in the structural parameters due to heat treatment. When the temperature of the heat treatment is changed from 700 °C to 800 °C, the porosity is reduced by 8.44% (from 69.45% to 61.01%). Similarly, the surface area is also reduced 4.16% from 59266.2 m²/m³ to 56803.5 m²/m³. When comparing 700°C to 800°C respectively. This reduction in both the surface area and the porosity could be due to densification associated with the later stages of sintering (Kang 2005), which would be more prevalent for the 800°C sample due to the longer heating schedule.
Table 0.7. Summary of the Quantitative Characterization of Copper foams using image analysis

<table>
<thead>
<tr>
<th>Heat Treatment</th>
<th>700 C</th>
<th>800 C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bead Size</td>
<td>Coarse</td>
<td>Coarse</td>
</tr>
<tr>
<td>Porosity</td>
<td>69.45%</td>
<td>61.01%</td>
</tr>
<tr>
<td>Surface Area</td>
<td>59266.2</td>
<td>56803.5</td>
</tr>
<tr>
<td>Tortousity</td>
<td>4.93</td>
<td>4.83</td>
</tr>
<tr>
<td>Permeability</td>
<td>22.77</td>
<td>24.71</td>
</tr>
<tr>
<td>Effective Pore Diameter</td>
<td>39.72</td>
<td>52.42</td>
</tr>
</tbody>
</table>

From the calculated tortousity values show in Table 4.7, increasing the heat treatment temperatures causes a decrease in the tortousity, mainly could be from the reduction in the surface area attributed to the densification that occurs at a higher heat treatment temperature. This reduction in surface area results in a smoother surface of the pore walls (see Figure 0.12), and due to the sensitivity of the morphological image processing algorithm’s to changes of geometry, the differences in the morphology between the two structures can be detected. This reduction in the tortousity cannot be suggested as a trend, because as the heat treatment temperature increases, more sintering will occur causing the structure to change, possibly causing pores to become isolated.

Typically the different stages of sintering have distinct pore morphologies (JOHNSON 1970; Shi 1999; Kang 2005), however with the presence of a sacrificial templates (coarse PMMA beads) which are 40 times bigger than the powders used for sintering (~100 micron PMMA beads and ~2.5 microns) and uncompacted particles, the kinetics associated with the sintering process may not accurately predict the sintering behavior. Therefore using the traditional models of describing changes in morphology
due to sintering is not sufficient to predict structural changes in the open cell copper foams

Figure 0.12. Process Binary Image of the pore walls of open celled copper foams processed at different heat treatment temperatures Left: 700ºC Right: 800ºC

From the area weighted pore size distributions shown in Figure 0.17, the effect of heat treatment on the pore size distribution can be seen. The area weighted pore size distribution of the pores of the 700ºC heat treated sample has a relatively uniform pore distribution compared to that of the 800ºC sample. The 800ºC heat treatment sample also exhibits a large occurrence of pores in larger size ranges (greater than 80 microns), and an increase in the probability at the size range 54-58 microns. The cumulative pore size distribution (shown in Figure 0.13) also provides a graphical representation of the differences in the pore size distribution. From the shape of the cumulative pore size distribution functions for the 700ºC and 800ºC heat treated copper samples (see Figure 0.12), the pores of the 800ºC should be larger than the pores of the 700ºC samples. From the cumulative pore size distribution function, the calculated effective diameter for the 700ºC heat treated copper sample is 39.72 microns and the calculated effective pore diameter for the 800ºC heat treated sample is 52.42 microns (see Table 0.7). From these calculated results, the tortousity of the 700ºC heat treated sample should be greater than
the tortousity of the 800°C, which is consistent the results obtained from the quantitative characterization of the porous structural using image analysis (see section 1.19.2).
Figure 0.13 Pore Size Distributions of the equivalent diameter of the copper foam samples with heat treatments of 700°C and 800°C.
1.20 Simulated Sphere Packing

Figure 0.14 shows a cross sectional view of the digitally constructed microstructures where the procedure for the construction and processing of the digital microstructures are described in section 1.16. Microstructures were generated from the same initial packing configuration, obtained from using the coarse PMMA bead distribution and target porosity of 76%, with the geometry of the necks calculated using \( \kappa \) values of \( \kappa = 1/25, \kappa = 1/30, \kappa = 1/35 \) and \( \kappa = 1/5000 \).

Table 0.8 summarizes the porosity, surface area per unit volume, tortousity and permeability calculated from quantitative characterization of binary images from those digitally constructed microstructure. As the \( \kappa \) values decrease, the surface area consistently increases. When the \( \kappa \) value is decreased from 1/25 to 1/30, the surface area is increased 4.24% relative to the surface area for the microstructure with pore generated with a \( \kappa \) value of 1/3. Similarly, when the \( \kappa \) value was decreased from 1/30 to 1/35, the surface area per unit volume increased 3.29%. When the \( \kappa \) values are decreased for 1/35 to 1/5000, the surface area increases 11.65%. These results were expected; decreasing the \( \kappa \) value reduces the appearance of the pore necks (see \( \kappa = 5000 \) in Figure 0.14).

From the area weighted pore size distribution, calculated using Eq. (3.9) and Eq(3.10), of the different microstructures digitally constructed pore structures with different \( \kappa \) values are shown in Figure 0.15.: 72.47 microns for \( \kappa = 1/25 \), 72.375 microns for the \( \kappa = 1/30 \), 72.50 microns for \( \kappa = 1/35 \), 73.26 microns from \( \kappa = 1/5000 \)( see Table 0.8). The pore size distributions corresponding to the calculations for the effective pore diameter are shown in Figure 0.15. Comparing the four pore size distributions, all are very similar in shape, with subtle differences. The cumulative pore distribution, shown in
Figure 0.16, is used with the pore size distributions shown in Figure 0.15 to effectively compare how the pore distributions differ. For the constructed microstructure with κ value of 1/25, there is a peak in the number of pores ranging from 72-76 microns in diameter and also at 84-88 microns in diameter. This trend is reflected in Figure 0.16 at 65-80 microns, where a drastic change in the slope of the cumulative distribution function can be see for κ=1/25. This change in the slope is also seen in κ=1/35 and κ=1/5000 cumulative distribution functions, however the changes are less drastic.

Table 0.8 Summary of the quantitative characterization of the digital generated microstructures using image analysis of generated cross sections

<table>
<thead>
<tr>
<th></th>
<th>κ=1/25</th>
<th>κ=1/30</th>
<th>κ=1/35</th>
<th>κ=1/5000</th>
</tr>
</thead>
<tbody>
<tr>
<td>Porosity</td>
<td>80.54%</td>
<td>79.60%</td>
<td>79.08%</td>
<td>76.58%</td>
</tr>
<tr>
<td>Surface Area (m²/m³)</td>
<td>18551.6</td>
<td>19372.3</td>
<td>20031.5</td>
<td>22673.8</td>
</tr>
<tr>
<td>Tortuosity</td>
<td>2.63</td>
<td>2.64</td>
<td>3.53</td>
<td>4.36</td>
</tr>
<tr>
<td>Permeability (m²)</td>
<td>697.4</td>
<td>596.01</td>
<td>509.54</td>
<td>260.85</td>
</tr>
<tr>
<td>Effective Pore Diameter (µm)</td>
<td>72.47</td>
<td>72.375</td>
<td>72.50</td>
<td>73.26</td>
</tr>
</tbody>
</table>
Figure 0.14 Binary Images of the cross sections of digitally constructed foam structures with different $\kappa$ values used to calculate the geometry of the necks and summary of the structural parameters calculated for the digitally constructed copper foams.
Figure 0.15. Probability density functions of the partitioned pore space of the digital reconstructed pore structures shown in Figure 4.14. (a) Pore size distribution for $\kappa$ value of 1/25. (b) Pore size distribution for $\kappa$ value of 1/30. (c) Pore size distribution for $\kappa$ value of 1/35 and (d) Pore size distribution for $\kappa$ value of 1/5000.

Figure 0.16. Cumulative Probability Distribution Functions for the digitally constructed microstructures with different $\kappa$ values used for the generation of necks.
Figure 0.15 shows the pore size distributions of the simulated microstructures for \( k \) values of \( 1/25, 1/30, 1/35, 1/5000 \). The two point correlation functions for the simulated sphere function are shown in Figure 0.17. The two point correlation functions were generated using the procedure described in section 1.12. The boundary conditions Eqs. (2.6) and Eqs. (2.7), were used as method of troubleshooting of the two point correlation function algorithm developed. The two point correlation function can be used to describe the spatial distribution of the pores, and Figure 0.17 displays the two point correlation functions for the microstructures constructed with \( \kappa \) values of \( 1/25, 1/30, 1/35, 1/5000 \). The shape of the two point correlation function for all of these different microstructures exhibit similar behavior, with an increase in probability at line of length \( r \) placed randomly anywhere in the structure lands with both endpoints within the pore phase of the image (y axis of the graph) is greater than 55μm. This increase in the probability indicates that there is some level of order associated with the imaged structure, because the two point correlation of a series of binary images of randomly generated microstructures should look like the that of a series of binary images generated from randomly overlapping spheres shown in Figure 0.5 where the randomly overlapping spheres which show an exponential decay to an asymptotic value. However, this curve exhibits a shape similar to the two point correlation function generated from the binary images of a packed bed of uniformly sized beads shown in Figure 0.5, where there are the changes in probability associated with periodicity appear as oscillations in the curve.
1.20.1 Spherical Contraction Algorithm

After the spherical contraction algorithm was performed on the set of packed spheres, necks were formed between the contracted spheres with a $\kappa$ value of 0.0301, which was found to minimize the effective pore diameter. A three dimensional model of then digitally generated porous material was created (see Figure 0.18). A Voronoi diagram based skeletonization algorithm was performed on the three dimensional model to quantify geometric and topographical information regarding the pore structure and provide a visual representation (see Figure 0.18.a). From the analysis of the pore space using the skeletonization algorithm, the pore bodes and pore channels within the pore...
structure can be identified and fluid flow pathways within the structure can be quantified and visualized (see Figure 0.18.b).

Figure 0.18. A) Three dimensional visualization of the digitally constructed microstructure with contraction. b) Visualization of the skeleton of the pore space of the digitally constructed microstructure.

The two point correlation function for the digitally constructed microstructure with spherical contraction was generated and compared against that of a microstructure of a real open celled copper foam processed with the coarse PMMA beads (see Figure 4.19). The two point correlation functions of the two functions behave differently at shorter length, but exhibit similar long range order behavior. The two point correlation function for the real metal foam decays at a much more rapid rate at shorter distances, indicating a greater surface area for the real metal foam than for the digitally reconstructed microstructure. These results correspond to the quantitative characterization of the two samples: the real reticulated foam had an average surface area per unit area 2.06 times greater than the average surface area per unit area of the digital
constructed microstructure with contraction. These results indicate that algorithms used to construct digital microstructures can simulate the long range order of the real open celled copper foams. The smoother surfaces seen for the simulated microstructures could represent the morphology of the pore structure after the surface has been wetted by the fluid.

Figure 0.19. Two point correlation function of a copper foam heat treated at 800°C and a digitally constructed metal foam with processed with necks generated using a κ value of 0.0301 and the spherical contraction algorithm.
CONCLUSIONS AND FUTURE WORK

1.21 Results

1.21.1 Particle size distribution

Using the feret diameter as a measure of the particle size resulted in a larger overall particle size distribution for both PMMA samples compared to using the equivalent diameter. The particle size distributions obtained from using those two different metrics as a measure of particle size were compared against results for the PMMA samples using a laser diffraction based particle size analyzer. Since the data obtained from the particle size analyzer was expressed in terms of a volume weighted particle size distribution, an area weighted method was used to compare the image analysis particle size data against the particle size analyzer data. The results indicated that using the area weighted method of calculating the average particle size from image analysis provided a valid alternative to using the area weighted particle diameter provided by the particle size analyzer.

1.21.2 Quantitative Characterization of Metallic Foams

1.21.2.1 Stainless Steel Study

Using image analysis for the quantitative characterization of the pore structure of the stainless steel sample provided the values for the structural parameters needed to calculate the fluid permeability using the modified version of the Kozeny Carman (Eq(2.22)). The permeability values calculated for the stainless steel samples indicated that the modified Kozeny Carman equation is a valid method of estimating the fluid permeability of the structure of the stainless steel samples. Also the methods developed
for calculating the average particle diameter using image analysis can be used to estimate
the average pore diameter with reasonable accuracy (±10%) using a pore portioning
algorithm. To improve the accuracy of the calculation of the average pore size,
knowledge of the pore morphology can be used to refine the pore portioning algorithm.

1.2.1.2.2 Copper Foam

The calculated permeability values for the copper foams processed with different
batch composition and heat treatment temperatures did not follow the expected trends. It
was expected that the larger loading of copper should make the structure less permeable
because of the increased amount of copper within the structure, however using the
Kozeny Carman equation to estimate the fluid permeability resulted in the 25.05% vol.
copper foam sample having a greater value of fluid permeability than the 17.06% vol.
copper sample. Regarding the effect of heat treatment on the structural characteristics of
an open celled foam, it is expected that the structure with the lower heat treatment
temperature should have the more permeable structure. However from the calculations of
the fluid permeability using Eq.(2.22), the calculated fluid permeability for the 800°C
sample is greater than the calculated fluid permeability for the 700°C sample. These
unexpected results can due to many different factors including the larger tortuosity values
and greater surface area per unit volume for the 17.06% vol. copper sample compared to
the 25.05% vol. copper and similar behavior for the 700°C sample compared to the 800°C
sample.

Regarding the pore level analysis, the pore size distribution of the 17.06% vol.
copper foam was smaller than the pore size distribution of the 25.05% vol. copper foam.
In a likewise manner the effective diameter of the 800ºC heat treatment temperature sample was 31.97% larger than effective diameter of the 700ºC. These results provide insight as to why the calculated permeability values were not consistent with the expected results. Also using the effective pore diameter to compare one structure to another can provide an estimate of material properties such as relative permeability and tortuosity which can be useful in estimating the effective heat transfer and diffusivity of a copper foam.

**1.22 Future work**

Further work on modifying the Kozeny Carman equation used to calculate the fluid permeability of the open celled copper foams to better predict the fluid permeability values by including a term that describes the average connectivity of the pore throats and the number density of pores.

Also other statistical descriptors such as the pore size distribution and the lineal path function should be obtained for both the real and simulated structures so that structural differences can be indentified easily and several studies have used the two point correlation function as a tool to create stochastic reconstruction of porous media (Liang 2000a; Roberts 1997; C 1 Y Yeong and S. Torquato 1998; Bentz and Martys 1994; S. Torquato 2002; Habisreuther, Djordjevic, and Zarzalis 2009). Therefore the development of an algorithm to determine the lineal path function for a microstructure would allow the reconstruction of a copper foam using binary images of the structure. Two point correlation functions can also be used to find a statistically equivalent periodic unit cell (SEPUC), which could be used with the unified treatment of microstructures such that the large scale simulations on realistic microstructure can be
produce with much less computational time (Zeman and Šejnoha 2007). Therefore methods of determining the SEPUC of a copper foam should be developed.

The algorithms associated with the digital construction of microstructure will be developed and refined. Ideally different particle distributions will be used to create different sacrificial template packing scenarios and provide a powerful tool for predicting the resulting pore structure for a given different particle size distributions. Also in addition to the further development of the digitally constructed microstructures, methods to analysis of the three dimensional network of the digital constructed microstructure should be developed, particularly the quantification of tortuosity in three dimensions.
APPENDIX A: MATLAB CODE

modwatershed.m

purpose:
Partition the pore space based on the selected min and max values.

Notes:
1. Structuring element is a 5 by 5 square
2. Uses the user defined function poreview
3. Can be modified to visualize the steps

function [ n vp va new]=modwatershed(G)
% modified version of the WATERSHED cell semgementation processing
% using the work from http://blogs.mathworks.com/steve/2006/06/02/cell-segmentation/ and the
% imoverlay algorithm for purely for visualization
% INPUT: G – grayscale image of the microstructure
% OUTPUT: n: number of pores
% vp: perimeters of the individual pores
% new: watershed image
% va: areas of the individual pores

%% Create a grayscale DISTANCE MAP
I=bwdist(G)./255;
%% turn distance map into a binary image
I_eq=im2uint8(I);
bw=im2bw(I,.02);
%%
bw1 = bwperim(G);
bw2 = imfill(bw,'holes');
bw3 = imopen(bw2, ones(5,5));
bw4 = bwareaopen(bw3, 10);
bw4_perim = bwperim(bw4);
%%
mask_em = imextendedmax(I_eq, 5);
mask_em = imclose(mask_em, ones(2,2));
mask_em = bwareaopen(mask_em, 40);
%%
I_eq_c = imcomplement(I_eq);
I_mod = imimposemin(I_eq_c, ~bw4 | mask_em);
L = watershed(I_mod);
new=~L|~bw;
[n vp va]=poreview(new, 10);

poreview.m

Purpose:
index and count the portioned pores and provide

Notes:
1. must define the number pixels to be filter out (fit),
2. Code can be modified so that partitioning of pores can be visualized
3. Outputs the number of pores (Nvc), perimeter (per) and area associated with each pore (can be modified so that more outputs such as the feret diameter can be exported as well)

function [nVC, per, va] = poreview(new, fit)
% Function to create an image that clearly displays watershed pores and
% the associated labels.
% INPUTS:
% new: watershed binary image
% OUTPUTS:
% VC: CENTROIDS OF PORE SPACES
% VEQ: EQUIVALENT DIAMETERS OF PORE SPACES
% Based upon nodeview by Jason Kulpe
%% Create size and matrix for labeling
size = size(new);
[vertex_lab, nv] = bwlabel(~new, 8);

% get pore info
% Get centroid properties
vp = regionprops(vertex_lab, 'PixelIdxList', 'Area', 'Centroid', 'EquivDiameter', 'Perimeter');
vc = cat(1, vp.Centroid);
va = cat(1, vp.Area);
per = cat(1, vp.Perimeter);

%% Filter out pores smaller than fit
ind = (find(Va > fit)); % FIND ONLY THE PORES WITH AN AREA GREATER THAN FIT
for i = 1:length(ind)
    VC{i} = vc(ind(i),:); % create new centroid list for images
    va(i,:) = Va(ind(i),:);
    per(i,:) = per(ind(i),:);
    pore{i} = pix{ind(i)}; %]
end
%% Remove boundary effect
[A B] = size(new);
bound = zeros(A, B);
bound(1,:) = 1;
bound(:,1) = 1;
bound(A-1,:) = 1;
bound(:,B) = 1;
ind = find(bound == 1);
var3 = rmvedg(pore, ind);
va = va(var3);
per = per(var3);
for jj = 1:length(var3)
    nVC(jj,:) = VC(var3(jj,:))
end
NV = length(nVC);
**tort_image_skeleton.m**

*purpose:* calculates the tortuosity factor of an image

*Notes:*
1. This algorithm takes a while to run
2. This code creates a skeleton, adjacency matrix and biograph
3. The code outputs all the calculations for tortuosity and also the mean tortuosity

```matlab
function [tau_mean tau]=tort_image_skeleton(M)
% [tau_mean tau]=tort_image_skeletonb(M)
% Take the binary image M and form the skeleton. Compose the graph of the
% skeleton and trace it to find the tortuosity, defined as the longest path
% between two points divided by their nominal distance.
% The algorithm works by taking 1000 random starting nodes and finds the
% furthest distance in that part of the skeleton, subject to a few
% conditions. Tau_mean is the average of the values tau

% INPUTS:
%   M: binary image
% OUTPUTS:
%   tau_mean: mean tortuosity value
%   tau: tortuosity values
% modified from the code by J Kuple

%% BASE IMAGE ANALYSIS
% Create skeleton and clean parts
S=bwmorph(~M,'skel',inf);
S1=bwmorph(S,'spur',5); % Remove skeleton spur pieces smaller than 5 pixels
S1=bwmorph(S1,'clean',1);
% Apply look up table to remove nodes of skeleton
lut1=makelut('x(2,2)&(sum(x(:))>3)',3); % A pixel node is ON and has more than 2 ON neighbors
Y=applylut(S1,lut1); % Image with only nodes remaining
ind=find(Y); % Find linear image indices of each node
N=S1;
N(ind)=0; % Turn off where the nodes are on
N=bwmorph(N,'clean',1);
[branch_lab num_branch]=bwlabel(N,8);
W=xor(N,S); % IMAGE OF vertex of skeleton
[vertex_lab nv]=bwlabel(W,8);
[edge_lab ne]=bwlabel(N,8);
v_prop=regionprops(vertex_lab,'pixellist','area','centroid');
e_prop=regionprops(edge_lab,'pixellist','area','centroid');

% Reform e_prop
for ii=1:ne
    edgept{ii}=e_prop(ii).PixelList; % list of points for each edge
    ecentroid(ii,:)=e_prop(ii).Centroid; % list of centroid location for each edge, for visualization later
end
% Reform v_prop
for ii=1:nv
    vpt{ii}=v_prop(ii).PixelList; % list of points for each vertex
    vcentroid(ii,:)=v_prop(ii).Centroid; % centroid location for each vertex, for visualization later
end

```
%% FORM INCIDENCE MATRIX %
%% The incidence matrix, R, is number_vertices x number_edges matrix.
%% R(i,j) is 1 if vertex i is connected to edge j and 0 otherwise.
%% Initialize for speed
R=zeros(nv,ne);
%% Search through each vertex point, checking criteria of each edge at each
%% vertex point
for ii=1:nv
    pts=vpt{ii}; % pts is a npt x 2 matrix
    for jj=1:size(pts,1) % Search through each point in the vertex(ii)
        atpt=pts(jj,:); % current point of vertex ii
        % Now search through all edges and see if an edge is adjacent to
        % the vertex point atpt (8 connectivity)
        for kk=1:ne
            e=edgept{kk}; % current edge point list
            % At each vertex (ii), point (jj), check distance this point
            % and all edge points
            edge_distance=sqrt((atpt(1)-e(:,1)).^2+(atpt(2)-e(:,2)).^2);
            % edge_distance is a ne x 1 vector
            % Conditional statement for connectivity criteria.
            % 8-connectivity, the maximum separation distance is sqrt(2)
            check=edge_distance<=sqrt(2);
            % If an edge(kk) has at least one non-zero element,
            % then edge(kk) is connected. So make R(ii,kk)=1 to show this.
            % If not, R(ii,kk)=0, if not already 1 from previous iterations
            % of points
            if R(ii,kk)==0 % R(ii,kk)=0
                if sum(double(check))>0
                    R(ii,kk)=1;
                end
            end
        end
    end
end

%% INCIDENCE MATRIX SPECIAL CASE
H=R*R';
%% The diagonal elements are non-zero...fix this
for i=1:nv
    H(i,i)=0;
end

%% Metric for matrix G
for i=1:ne
    G_metric(i)=e_prop(i).Area;
end

%% Form G matrix with metric values for common edges
G=zeros(nv);
for i=1:nv
    for j=1:nv
        if H(i,j)
            % Identify common edge
            common_edge=intersect(find(R(i,:)),find(R(j,:)));
            % etc...
        end
    end
end

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% There can be more than 1 common edge, if this is the case the
% metric is the maximum
if numel(common_edge) > 1
    G(i,j) = min(G_metric(common_edge)); % minimum of metric
else
    G(i,j) = G_metric(common_edge);
end
end
end

%% ANALYZE GRAPH FOR TORTUOSITY
% 1. pick random node to start and find maximum value
% 2. remove infinite's from shortest path matrix
% 3. if max length less than 10 pixels or distance (in X) is less than 10
%    pixels, ignore and iterate
% 4. find the distance and calculate tortuosity at each iteration
% 5. tortuosity should be greater than 1

% Create graph
bio = biograph(sparse(G));
num = 5;
index = 1;
tau = zeros(1,num);
while index <= num
    con = randi(nv);
    a = shortestpath(bio, con); % a is the distance of every node from node con
    aaa = isinf(a); % find infinite values to be removed
    w = max(a(~aaa)); % location of the shortest path for algorithm
    ind1 = find(w == a);
    ind = ind1(1);
    % compute length ONLY in X direction
    len = abs(vcentroid(con,1) - vcentroid(ind,1))
    % make checks
    ck1 = w > 5;
    ck2 = len > 10;
    ck3 = w > len;
    % if passes all 3 checks, compute tau and increment counter, else
    % repeat
    if ck1 && ck2 && ck3
        tau(index) = w / len;
        index = index + 1;
    end
end

% Outputs
tau_mean = mean(tau);

---

**Image_filter.m**

**Purpose:** creating a binary image from an input image.

function M = image_filter(varargin)
% M = image_filter(O, map, siz)
% M=image_filter(loc,file,siz)
% M=image_filter(siz)
% Converts the RGB image O to binary and does the necessary processing.
% OR
% Reads the gray-scale image pointed to by strings loc and file, converts
% to binary and does the necessary processing and filtering to find the
% image M.
% If there is only one argument than a user window appears asking for the
% file name.
% INPUTS:
%       O: RGB image
%       map: colormap of RGB image, from imread
%       loc: string of the directory of the file
%       file: string of the filename of the file, including extension
%       siz: value describing how to scale the output image, used with
%            imresize
%
% OUTPUTS:
%       M: processed binary image
%
% Jason Kulpe
% 11/13/09

in1=varargin{1};

% Parse input arguments
if ~ischar(in1) && nargin > 1 % assume input in1 is a matrix
    in1=varargin{1}; % image
    in2=varargin{2}; % map
    % Convert image to B&W
    Gray=ind2gray(in1,in2);
    thresh=graythresh(Gray); % uses Otsu method of thresholding
    BW=im2bw(in1,thresh);
    siz=varargin{3};
elseif ischar(in1) && nargin > 1
    loc=varargin{1}; % location
    file=varargin{2}; % directory
    [O map]=imread(strcat(loc,file)); % read pixel data of image into G
    % Convert image to B&W
    Gray=ind2gray(O,map);
    thresh=graythresh(Gray); % uses Otsu method of thresholding
    BW=im2bw(O,thresh);
    siz=varargin{3};
elseif nargin==1
    [si li]=uigetfile("*.tif");
    siz=varargin{1};
    if ischar(si)
        [O map]=imread(strcat(li,si));
        % Convert image to B&W
        if numel(map)==0
            Gray=O;
        else
            Gray=ind2gray(O,map);
        end
        thresh=graythresh(Gray); % uses Otsu method of thresholding
        BW=im2bw(O,thresh);
end
else
    error('Wrong input arguements....');
end

%% Morphological operations to clean and filter images

% Remove small pieces
n1=10;
n2=10;
C=bwareaopen(BW,n1); % Remove pieces smaller than n1
D=bwareaopen(~C,n2); % Remove pieces smaller than n2
D=~D;

% Combination of C and D
% H=D;
% Morphological operations
s1=strel('disk',2);
Mr=imopen(D,s1);
% Resize image
M=imresize(Mr,siz);
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