ABSTRACT

STRUCTURAL MAPPING OF PAPER TOWELS: COMPARISON OF TWIN LASER PROFILOMETRY AND SYNCHROTRON X-RAY MICRO-COMPUTED TOMOGRAPHY

by Yan Huang

This investigation was to compare the structural properties, of commercial available single ply paper towels, as they vary in X-Y plane. The focus was to quantify the differences in selected regions of interest, as measured by two analytical methods, non-contact Twin Laser Profilometry (TLP) and synchrotron X-ray micro-computed tomography (XRμCT). The thickness and out-of-plane deformation (OPD) distributions were mapped using a TLP and XRμCT. The XRμCT samples were further used to calculate for the mass center surface. TLP enables web structures to be mapped to a micrometer resolution over 12.5mm regions. Synchrotron X-ray microtomography method provides sub-micrometer resolution of the complete volumetric structure (1.4mm×1.4 mm × thickness (μm)) for the imaged region. The features of individual fibers are revealed by XRμCT method, so that the true surface geometry may be quantified.
STRUCTURAL MAPPING OF PAPER TOWELS: COMPARISON OF TWIN LASER PROFILOMETRY AND SYNCHROTRON X-RAY MICRO-COMPUTED TOMOGRAPHY

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To my parents, Jianxin Huang and Linghua Ye, future wife, and children,

with love
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1. Introduction

Paper is one of the most important material inventions of human kind, first introduced around 2000 years ago. Nowadays, paper plays an important role in our daily activities as communication and information medium and home cleaning products among others. In recent decades, modern technologies and equipment, such as computers, the internet and scanners, have developed very rapidly so that a common misconception is that paper and paper products will be replaced, and the solid position once held might be in decline. In fact, as computer and internet use has grown wider and wider, the demand and consumption of paper products has also increased.

Like all other types of materials, paper has a characteristic structural hierarchy. End users are generally interested only in the end-use properties of this sheet material, such as strength, softness, absorption, water repellency and so on. They are not usually concerned with the structural hierarchy at all. However, as a manufacturer or researcher of paper products, we understand that the raw materials and the processes involved in manufacturing create a complex hierarchical structure we know as paper. It is this structure which has a major influence on the end-use properties.

By definition, structure is that which can be seen using a suitable measuring instrument [1]. The term structural hierarchy describes the existence of structure at a variety of scales. From lower scale to higher scale, paper can be identified by some basic constituents such as molecules, elementary fibrils, micro-fibrils, fibrils, cellulose, the fiber cell wall, individual fibers, flocs of fibers, induced patterns, composite layers and even the manner in which it stacks. Although this selection of constituents is very reasonable, it is not necessary or even desirable to consider the structure at all levels of detail when investigating the properties of paper [2]. In short, within the structural hierarchy of paper, the investigator must be continuously aware of the structural range that is most important to relate the input materials and processes and the output bulk properties that are being studied. This does not mean that
the nature of the structure outside of the scale of interest is not important. Rather, there exists a scale region where the characteristics of the structure account for most of the material properties which are of interest. Table 1-1 list some scale of the hierarchy structures of paper of interest.

In the papermaking processes, each individual process step can influence the properties of the resulting paper. Each step is also controlled by a set of process parameters. The choice of wood species, the pulping method, the forming section, the paper drying process, and the calendering conditions are all examples where the paper properties such as the formation, strength, optical properties, softness, and absorption of paper are influenced, shown in Figure 1-1 [2]. The schematic in figure 1-1 shows the relationship between the states of the structure, process parameters, and the corresponding properties. Each structure determines the

<table>
<thead>
<tr>
<th>Structure</th>
<th>Scale</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fiber length</td>
<td>1mm-6mm</td>
</tr>
<tr>
<td>Fiber width</td>
<td>20μm-60μm</td>
</tr>
<tr>
<td>TAD pattern</td>
<td>1mm-2mm; 0.1mm-0.5mm (spacing)</td>
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<td>Embossing pattern</td>
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</tr>
</tbody>
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Table 1-1 The hierarchical structure of paper of interest

![Diagram](image)

Figure 1-1 The Relationship of paper making process to product properties
properties of the material, and each process includes a set of process parameters. Fibers have their own properties such as length, width, coarseness, strength, and light absorption and scattering coefficient. The change from raw material to intermediate structure and then to final structure is realized by different processes. Final paper also has its own properties as described previously.

In this research, tissue paper, especially towel papers, are of interest. The tissue paper grade includes towel paper, napkin, facial tissue and toilet paper. The demand for tissue products is increasing all over the world and usage is expected to continue to increase in the future [3-6]. There is also an increased demand for better qualities of tissue papers. The advance in electronic technologies gives the paper industry and paper researchers an opportunity to develop new processes and products to meet this challenge. Pursuant to this, a deeper insight of paper structures and how they are influenced by the paper components and manufacturing processes are needed. Major efforts have been made during the last decades to understand the structure of paper and its relationship to the paper components, papermaking processes and the corresponding properties, such as mechanical properties, optical properties, and transport properties. But their efforts have not been sufficient and extensive so that a detailed understanding of the structure of paper and its relationship to the manufacturing processes and the corresponding end use properties is still incomplete.

Every day, millions of people reach for tissue products. Whether it is personal hygiene or cleaning towel products, consumers look for the same characteristics in: softness, absorption, and strength. Softness is a very important quality for bathroom tissue and facial tissue. However, an objective panel test of softness is rather difficult and confusing, so that tissue manufacturers generally have had to rely upon the subjective tests for evaluation. One of the methods is hand feel (HF) which is greatly depends on paper thickness (bulk) and flexibility [7]. Some others are based upon the measurement of surface compressibility; softness is thus regarded in a narrow sense to be almost identical to surface compressibility [8]. More recently, the terms bulk softness and surface softness have been used [7]. It is thought that flexibility (inverse of bending stiffness) is the main component in bulk softness, whereas
surface softness is related to surface compressibility. Flexibility and surface compressibility are closely related to the bulk and density distribution.

Absorption is a very important quality for kitchen towels and industrial wipes. The properties normally measured are absorption capacity and absorption rate [7]. In theory, absorption capacity reflects the amount of water the towel can absorb after infinite time, while absorption rate measures how quickly the product can absorb water. A high bulk will favor a rapid water uptake by presenting larger flow channels and minimizing the flow resistance. Practically, all of the water is transported in the interstices between fibers. Therefore, fiber orientation, pore geometry and crepe structure are some of the elements which will affect the absorption and all these elements are related to the thickness and density distribution of the tissue paper.

Tensile strength is an important property of kitchen towels and industrial wipes which are required to stand up to repetitive wiping. Strength is determined, in part, by relative fiber strength, bonding area, bonding strength, and formation. Usually, the higher the elastic modulus is, the higher the tensile strength. Increasing the paper density will also improve the elastic modulus of paper, and therefore the tensile strength. However, this is not a viable solution for tissue and towel products since the absorption and softness will be lost upon densification.

For tissue paper, softness, bulk, and tensile strength are hard to optimize. As tensile strength increases, softness and bulk decrease, and vice versa. Softness, absorption and tensile strength are the preferred properties by consumer. In order to produce tissue paper having better combination of softness, absorption and tensile strength, deep insights into the manufacturing processes and the structure properties, for example thickness, formation, deformation, and density, are needed. The focus of this investigation is on the scale of structure, both inherent and induced, imparted by different processes, and the relationship of that scale has to the corresponding processes.
2. Back Ground

In this chapter, the raw material of tissue papers and the conventional manufacturing processes will be described. The critical processes in determining the characteristics of the final towel paper will be discussed in details. The scale of structure induced by different processes indicates variation in mass distribution and morphology, and therefore the characterization of tissue papers and three-dimensional (3D) image acquisition devices used in the analysis of distribution of mass will be reviewed.

2.1 Tissue paper grade

Tissue and towel papers are made from either virgin pulp, recycled paper pulp, or a mixture of these two. If virgin pulp is used, it generally contains a mixture of hardwood fibers and softwood fibers. The hardwood fibers are used to improve softness and the softwood fibers are used to improve paper strength. Fibers are often bleached during the pulping process to make the color whiter to improve the appearance of the pulp [9].

Tissue papers are often embossed with patterns of shapes such as circles, ovals or diamonds, in order to enhance their absorption and softness, or to provide adhesion points in multi-ply products. To make them more appealing, tissue papers sometimes have intricate colored images, such as flowers, imprinted on them. Most of the tissue paper rolls are manufactured with two layers of thin paper called 2-ply, but some have only one, called single ply.

2.2 Tissue and towel manufacture

2.2.1 The forming section

In terms of the forming section, there are mainly three types of contemporary tissue machines: suction breast roll machines, twin-wire machines (“S-wrap” or “C-wrap”), and crescent former machines. In a review by Atkins [10], the configurations of these machines are provided, see figure 2-1. The suction breast roll machine, which was dominant during the
1960s and 1970s, is a modified Fourdrinier which has only a single forming wire [11]. The table length is short since it is possible to dewater in a short distance at high speed wire by the design of the lip of the headbox extended around the suction roll. This is similar to pressure forming used in boxboard production. The free water is forced from the dilute stock as the volume in the region of the lip contracts. The low grammage of these products allows this method of dewatering to be used.

Twin-wire machines developed in the 1970s are the most popular tissue machines in use today. In twin-wire machines, the stock is sprayed from the headbox in between the two converging wires. Most of the water is squeezed out of the sheet by the tension of the two wires as they wind around the forming roll, which may be solid or an open drilled suction roll. This type of machine provides more effective dewatering, as compared to the Fourdrinier type. Due to the path that the web takes, twin-wire machines include two categories called the “S-wrap” and “C-wrap” formers. In the “S-wrap” former, the outer wire is the conveying wire as it carries the sheet to a press felt after drainage zone. In the “C-wrap” former, the inner wire is the conveying wire. In the manufacturing process, the choice of an “S-wrap” or a “C-wrap” depends on several factors, such as contaminants in the stock and the design of the headbox. Details were discussed by Atkins [10].

Figure 2-1 Configurations of tissue forming section. Top left: suction breast roll former; top right: “S-wrap” former; bottom left: “C-wrap” former; bottom right: Crescent former.[10]
The crescent former, which is found in wide use throughout the tissue industry after 1980s, looks like an inverted “C-wrap” with the headbox on the top. The arrangement of this former, in which the stock is sprayed in between a wire and a felt, results in higher runnability and lower cost since there is no need to take off the sheet from the wire and convey it onto another press felt.

2.2.2 Drying: Conventional wet pressing (CWP) and the Yankee dryer

In the tissue paper manufacturing processes, the dryer section is of great importance. In conventional tissue machines, the lightweight paper from the forming and press sections is pressed against and dried on a single large diameter cylinder called a Yankee dryer. The surface of the cylinder is continuously sprayed with a solution of chemicals containing adhesive compounds and often one or more release agents. Figure 2-2 shows the schematic of a Yankee dryer. The water is pressed from the wet paper to the Yankee machine to be evaporated. No backing felt is used on a Yankee drier and so the strength of the wet web is crucial for runnability. The heat transfer rate is very high because the tension of the wet web holds it tightly against the highly polished Yankee surface. The paper is usually creped as it is scraped off the dryer by a special creping doctor blade. The effect that creping has on tensile strength, web discontinuity and thickness depends on several factors, including the raw materials used in the stock, the strength of the adhesive used on the Yankee cylinder, the web moisture, the duration of surface contact, and the doctor blade geometry[9]. During creping process, the speed at which the sheet moves away from the doctor blade is slower.

![Figure 2-2 The schematic of tissue web being dried and creped on a Yankee dryer.](image-url)
than the rotational speed of the Yankee dryer. The action that takes place at the doctor blade causes the flat web adhered to the Yankee cylinder to buckle and form cross-direction ridges which are recognized as creping [12]. In general, the degree of creping increases with a higher angle of contact between the doctor blade and Yankee cylinder [13]. Depending on the dryness of the web during the creping process, creping can be divided into three categories: wet crepe, semi-dry crepe, and dry crepe [12]. Single and two-ply napkins and industrial wipes are made by semi-dry creping; facial tissues and toilet tissues are the major uses of dry crepe tissues. Paper towel is the principle focus of this investigation and wet creping is usually used for this product. After creping, the densified web becomes softer and bulkier. Figure 2-3 depicts the structure of a group of fibers on a Yankee dryer before creping and after creping. The transition from a dense state to a bulky creped state, characterized by kinks or deformations in individual fibers, is illustrated.

![Figure 2-3 Schematic representations of fiber changes as a web on a Yankee dryer is creped.](image)

### 2.2.3 Drying: Through-air-drying (TAD)

Compared to the conventional wet pressing drying method, the through-air-drying (TAD) technology removes water by pulling high temperature air through the sheet. This process significantly reduces pressing load, helps maintain the thickness of the sheet and produces a higher soft and more textured web.

In the through-air-drying process, the moist paper web from a forming and press sections is transferred from one fabric to a second slower-moving fabric under carefully controlled conditions. It is then that the moist web is placed onto an advanced three-dimensional
through-drying fabric. Hot air then passes through the web to dry it in a morphology corresponding to that of the supporting fabric [12], see figure 2-4 The hot air passing through the web provides high heat transfer rates and effective drying without significant compression of the web.

Unlike the Yankee drying of CWP, through-air drying does not densify the web and bulk is maintained at the microscopic level. The texture of the fabric imparts high bulk at macroscopic scale. The morphology of the web after through-air-drying is shown in Figure 2-5.

Figure 2-4 Tissue web being dried on a through-air-dryer

Figure 2-5 Schematic of morphology of fiber changes as a web passed a through air dryer.
2.2.4 Embossing

Most of the tissue papers are embossed to produce a greater softness. Paper can sometimes be embossed for decorative reasons. There are mainly three types of embossing: traditional embossing, nested embossing, and foot-to-foot embossing [11].

The traditional embossing method was virtually the only embossing method used up until early 1980s and embosses all tissue plies simultaneously. The two embossing rolls can be made of steel, or one might be steel and the other rubber. This type of embossing method is mainly used to emboss bathroom tissue. The nested embossing method introduced in the early 1980s embosses each ply separately, after which the plies are aligned and bonded together. Alignment is achieved by matching the narrow peak of one ply to the broad valleys of the other. Compare to the traditional method, this method results in bulk which is almost doubled. This type of embossing method is mainly used to emboss kitchen towels. Foot-to-foot embossing is similar to nested embossing. Here again, each ply is embossed separately and the plies are then aligned and bonded together. The difference of foot-to-foot embossing to the nested embossing is that the alignment is done by matching the peak of one ply to the peak of the other ply. This further improves the bulk to a greater level and enhances the tissue’s rate of absorption. Figure 2-6 shows the schematic of tissues after different embossing processes.

![Figure 2-6 A: alignment of 2 ply of traditional embossing; B: alignment of 2 ply of nested embossing; C: alignment of 2 ply of foot-to-foot embossing.](image-url)
2.3 Scale ranges that are important

Figure 2-7 Void surface volume created by features in tissue and towel papers [14].

In towel manufacturing, the raw fibers are subjected to processes leading to a structure which is just the interaction and distribution of the fibers in a three-dimensional space. The structure, as shown in figure 2-7, includes induced features and inherent features as defined by Keller [14]. The induced features, from the processes, consist of creping patterns, fabric marks, TAD patterns and embossing patterns. The inherent features include the stochastic distribution of fibers that results in paper formation, variation of thickness (lumpiness), surface roughness and unresolved out-of-plane deformation. These various features have their scale ranges, some of which are shown in figure 2-8.

Figure 2-8 scale of towel paper: a: molecular; b: fiber width; c: free fiber length and bonding diameters; d: TAD pattern spacing; e: TAD pattern; f: Embossing pattern and fiber bonding; g: conventional wet pressing pattern; h: Embossing space. i: fiber length; j: formation.
As previously noted, the investigation must be continuously be aware of the structural range that is most important to relate the input materials and processes and the output bulk properties that are being studied. Apart from using the inter-fiber bonds and millimeter length network to provide strength, tissue and towel papers apply pressed regions, which are densified, in organized patterns to form a reinforcing network to meet the strength requirement. The bulk of the paper is increased by the un-pressed regions which have fewer bonds. Furthermore, creping and through-air-drying improve the bulk of the entire web to a higher level. Therefore, the critical consideration of the web properties of tissue and towel papers occurs at a scale range between $10^{-3}$ mm and 10 mm, although the internal bonding and fiber properties remain important factors in the web properties. The distribution of fibers in a sample area, covering one or several duplicate regions of that scale range, will be characterized and quantified by formation, thickness, and density.

### 2.4 Characterization of towel structure

In the past, paper has been modeled as a two-dimensional (2D) web composed of a random distribution of fibers. Dodson built a 2D network geometry model to describe the structural statistics of web materials in details [15]. Norman [16] used “paper formation”, defined as the in-plane variation of local grammage, to characterize web structure by measuring the mass distribution in the plane of the web. The scale of formation is actually considered to range from 1mm to 100mm, and therefore including the patterns induced by the forming wire and other supporting fabrics (press and dryer). However, due to web thickness and severe out-of-plane deformation induced by CWP, creping, TAD, or embossing, towel papers are more appropriately recognized as three-dimensional structure because of the fibers displaced in the Z-direction which results in high bulk. Therefore, two-dimensional paper formation falls far short of describing the structural properties of towel paper. Thickness and Z-directional distribution play a much more important role for tissue structure than that for communication paper structure. In describing towel papers using three-directional geometry, thickness variation in the Z-direction and the distribution of density should be considered.
The material density, which is the formation reduced by thickness, provides a complete view of the mass distribution and therefore will provide a deeper understanding of the relationship between the manufacturing processes and the structure.

2.4.1 Measurement of local grammage

The principle method of measuring grammage of paper is gravimetrically by dividing the weight of a region by its in plane area. A more robust method to reflect the distribution of grammage within a region is referred to as formation. Several energy sources including visible light [17], electron beam [18, 19], soft X-ray [20] and β-ray [21, 22] have been used to determine the formation of paper by recording the local transmission within a spatial region. Absorption is related to the mass in the region. Among these, β-radiographic imaging and light transmission imaging are the most commonly used techniques. A detailed comparison and review of these formation measurement methods are given by Tomimasu [19].

Compare to light transmission, β-radiographic imaging provides higher resolution and therefore could measure the distribution of mass with a higher precision. Light transmission imaging is based on the material’s ability to absorb and scatter the incident beam, whereas β-radiographic imaging depends almost entirely on the absorption of the radiation in the material. Both techniques are therefore greatly influenced by the mass of the material and its distribution which can be affected by the manufacturing and finishing processes of the products [19, 23]. The principle of the measurement of the attenuation of β-radiation is the direct relationship between the attenuation of incident-electrons and the mass in that region. Equation 1 reflects the transmission of the β-electrons through a web [19]:

\[ T_\beta = e^{-\mu w} \]

Where, e= mathematical constant
μ= the absorption coefficient of material
w= grammage, g/m²
\( T_\beta = \) the transmission value
The formation data from β-radiography could be recorded using either X-ray film or a storage phosphor screen as the detector. If X-ray film is used as, generation of formation is based on the interaction of the transmitted β-radiation source and the sensitive silver halide in the X-ray film. Once exposed, the film is developed, fixed and dried, and a “latent image” is formed. This image is then digitized using an optical scanner to generate grayscale maps. The grayscale maps may then be calibrated to represent the grammage at each sub-region (pixel) by using a standard step wedge made from Mylar of various thickness (known grammage). By using a storage phosphor system, the processing time and experimental variables are reduced, while maintaining a high spatial resolution [22]. After a storage phosphor screen is exposure to β-radiation, a latent image is created which could be digitized by the scanning system to create a formation map.

2.4.2 Measurement of local paper thickness

Thickness is defined as the perpendicular distance between the two principal surfaces of paper under specific conditions [24]. For paper towels, thickness and density are closely related to some desired properties such as softness and absorption. Most papers are thin with respect to the in-plane dimensions; so the density uniformity is more depended on the variation of thickness. Therefore, an accurate calculation of the thickness, which grammage is divided by to obtain density, is of great importance.

For calendered products, such as printing and copy papers, it is much easier to measure the thickness accurately due to the relative uniformity of smoothness and thickness. Tissue and towel papers, on the other hand, are creped or embossed during manufacturing process, so they have more surface height variation and web discontinuity. Tissue paper also has low grammage, and is highly compressible. Due to the roughness, web discontinuity, compressibility and the difficulties in defining the true outer boundary of tissue and towel grades, it is very difficult to measure thickness accurately. To measure the thickness, the surface or boundary definition of the sample is adopted from the experimental method of measurement, although the thickness value obtained under different experimental definitions
will result in different values. Fellers [25] made a comparison of the various thickness measurement methods, including TAPPI standard method [26], the piling thickness [27] and the effective thickness by mercury displacement, soft rubber platens and the opposing spherical platen profilometry methods. All these methods will add some pressure on, and will contact with, the sample surface. This will deform the surface contours to a greater or lesser extent.

Recently, high resolution non-contact thickness measurement methods were done by Yoshida [28] and Sung [29, 30]. In the non-contact thickness tester described by Yoshida, two different auto-focusing opposing laser sensors were mounted on to sense the top and bottom side of the paper samples placed in between two sensors. The sample thickness was calculated by the range value obtained from both lasers. Due to the limited z-direction measuring range value of both lasers, it was hard for this instrument to measure large out-of-plane deformations, such as cockles, embossing and indentation. Sung [29, 30] described a high resolution Twin Laser Profilometer (TLP) to measure the thickness and surface topography of paper samples. This instrument provided a wider range of surface topography, so that surface roughness (10^{-2} mm) and cockle (1mm) could be measured at the same time. More description about the TLP and principle will be given in the following chapter.

**2.4.3 Measurement of local density map**

The density of paper has been regarded as one of the most fundamental paper properties, because it strongly influences the end-use properties of paper. The density of paper could be obtained by dividing the grammage by the thickness of the paper of the same zone. Depending on the papers, the variability in thickness may be high (as for tissue and towel papers), so that thickness is much more important for determining the uniformity of density.

Oba and Sampson [15] used opposing laser non-contact thickness tester developed by Izumi and Yoshida [28] to measure the local thickness and local density variation map, in order to
investigate the relationship between the variance and the coefficient of variance of local thickness, local grammage, and the local apparent density for various samples. However, the local thickness value obtained by this method had limitations in spatial resolution. Sung and Branca were more successful at mapping thickness, and thus density, for printing paper [29, 30] and tissues and woven materials [14, 31]. Sung calculated the local apparent density distribution of laboratory samples with thickness variation caused by hard nip and soft nip calendaring, by dividing the grammage distribution, obtained from β-radiographic imaging, and thickness distribution, obtained from TLP. Branca, using the same method as Sung to obtain the grammage and thickness values, compared the mean density of the embossed or creped regions with that of the non-embossed or non-creped regions of certain tissue paper, crepe paper and nonwovens. They found that the embossing process increases the mean density by reducing the thickness of the web, while the creping process decreases the mean density by increasing the thickness of the web. The method used by both Sung and Branca will be used in this investigation to evaluate the thickness and mean density distributions of regions of interest in commercial paper towel samples.

2.5 The analysis of paper structure in three dimensions

Detailed characterization of formation, bulk and density is important as they are all characteristics of manufacture parameters and affecting specific end use properties of paper. Characteristics including free fiber distributions, fiber bonding, patterns (induced by creping, air drying and embossing), and formation are all important features of tissue and towel papers. These features range from $10^{-3}$ mm to 100 mm. Therefore, an adequate representation of the paper structure in this range is essential. In recent years, a major research topic is the analysis of 3D paper structure using a variety of analytical methods. Chinga [32] reviewed the importance of structure on printing paper, and provided a list of common 3D image acquisition methods. Some of these are provided in a partial list in Table 2-1. In this table, the large difference of the resolution of the devices as well as the respective advantages and disadvantages are mentioned. A suitable scale of resolution of some of the more common methods are defined in Table 2-2 [32].
The thickness and bulk structure of paper can be characterized based on several microscopy techniques listed in Table 2-1. In order to yield the information on how the mass in the paper web is distributed in two dimensions, Szikla [33] and Rättö [34] examined the Z-direction

Table 2-1 some reported 3D image acquisition devices used in the analysis of paper structure. [32]

<table>
<thead>
<tr>
<th>Technique</th>
<th>Approximate Resolution(μm)</th>
<th>Assessed Structure</th>
<th>Advantage</th>
<th>Disadvantage</th>
</tr>
</thead>
<tbody>
<tr>
<td>Light microscopy (LM)</td>
<td>0.2</td>
<td>Surface and bulk</td>
<td>Printing ink visualization</td>
<td>Time consuming</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Automatic 3D reconstruction.</td>
<td>Preparation may involve microtoming</td>
</tr>
<tr>
<td>Automated serial sectioning</td>
<td>1.0</td>
<td>Surface and bulk</td>
<td>Lack of accessibility to pores.</td>
<td></td>
</tr>
<tr>
<td>Confocal laser scanning microscopy (CLSM)</td>
<td>0.2-0.7</td>
<td>Surface and bulk</td>
<td>Decreasing intensity and resolution in the z-direction.</td>
<td></td>
</tr>
<tr>
<td>Scanning Electron Microscopy (SEM)</td>
<td>0.001-0.02</td>
<td>Surface and bulk</td>
<td>High quality and sub-micrometer resolution</td>
<td>Time consuming</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Limited sample size</td>
<td></td>
</tr>
<tr>
<td>Optical Coherence Tomography (OCT)</td>
<td>15</td>
<td>Surface and bulk</td>
<td>Non-destructive</td>
<td>Low resolution</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>Large sample size</td>
<td></td>
</tr>
<tr>
<td>X-ray micro-computed tomography (XRμCT)</td>
<td>0.7-1.0</td>
<td>Surface and bulk</td>
<td>Same resolution in the x, y, and z direction</td>
<td>Relatively low resolution. Low contrast between different components.</td>
</tr>
</tbody>
</table>

Table 2-2 classification of some image acquisition devices and their effective resolution

<table>
<thead>
<tr>
<th>Resolution (r)</th>
<th>Definition</th>
<th>Image acquisition device</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.0 nm≤ r &lt; 100nm</td>
<td>Nano</td>
<td>SEM</td>
</tr>
<tr>
<td>0.1 μm ≤ r &lt;1.0 μm</td>
<td>Sub-micrometer</td>
<td>LM, CLSM, XRμCT</td>
</tr>
<tr>
<td>1.0 μm ≤ r &lt;100μm</td>
<td>Micrometer</td>
<td>XRμCT, OCT</td>
</tr>
<tr>
<td>0.1 mm ≤ r &lt;1.0 mm</td>
<td>Sub-macro</td>
<td></td>
</tr>
<tr>
<td>r ≥ 1.0 mm</td>
<td>macro</td>
<td></td>
</tr>
</tbody>
</table>
density distribution in the paper web from images of paper cross-sections obtained using a light microscope. Later, Hasuike [35] obtained a good representation of 3D structure of paper by combining several such two-dimensional (2D) images into a 3D image rendering processing program using a digital microscope. This work was tedious, time-consuming, and destructive. Also, only small region (0.2mm x 0.2mm) were studied. Recently, a novel technique called automated serial sectioning was developed by Wiltsche [36]. This technique allows serial section of hundreds of slices to be assembled into a 3D representation of the material under examination, in this case. This instrument combined a motorized rotary microtome and a digital optical microscopy. The instrument is capable of digitizing paper samples with a size of more than 1 cm² to a 3D resolution of less than 1 μm. However, this technique cannot access the porosity of paper because of the difficulty in distinguishing the walls of some fibers, where bonds occur and the pores in the fibers network. A second generation of this automated serial sectioning technique equipped additionally with fluorescent microscopy is currently under construction [36].

Mangin [37] and Auran [38] applied the confocal laser scanning microscope (CLSM), a non-destructive method which is capable to achieve 3D resolution in the sub-micron range to measure the 3D surface structure of paper by assembling a stack of the two-dimensional images from successive focal planes. He [39] used CLSM to analysis the transverse morphology of individual fibers. Xu [40] used this technique to analyze the distribution of fibers in paper thickness direction. However, the interior structure of the paper is difficult to measure because the laser beam is refracted by the surface layer of fibers and thus it has highly limited depth of focus.

Scanning electron microscopy (SEM) cross-section analysis [41-46] has been widely used for quantification of paper bulk structure, such as analysis of pore structures in paper sheet, analysis of individual fiber properties and reconstruction of the fiber network. SEM has high resolution, but the application of SEM to 3D analysis is usually quite time consuming and therefore has met with limited application.

Alarousu [47] used optical coherence tomography (OCT), a non destructive image acquisition
device, to analyze paper surface and bulk structure. The low resolution (15μm) is the main limitation of OCT, although large sample size can be analyzed.

All of the methods discussed above are either destructive (cutting), time consuming, structure modification due to the introduction of resin or lack of robust characterization of the interior structure of paper samples. Therefore, a nondestructive and efficient 3D imaging system for paper is needed. The present work is concerned with the use of synchrotron X-ray micro-computed tomography (with resolution of sub-micrometer) and with a sample region about 1mm³ for the purpose of structural analysis.

2.6 Synchrotron X-ray tomography and image processing

X-ray tomography is a newer method used to analyze paper and before 2000 was mostly used for medial and biological material analysis. X-ray is a form of electromagnetic radiation. X-ray tomography is the process of determining the internal structure of an object without sectioning it by physical cutting. Rather, sectioning is performed through the use of penetrating X-rays. Tomography can be applied to any solid material for which the energetic beam can penetrate and detect differences in density. When an X-ray beam passes through an object that is partially opaque to the radiation, various areas in the object will attenuate the beam in different ways. Usually the denser spots in the object will attenuate the X-ray beam more than less dense spots. Therefore a map of the X-ray projection is really just a map of the density distribution of the object from a given viewing angle. X-ray tomography combined with digital image processing can generate a 3D representation of the interior structure of an object from numerous two-dimensional X-ray images taken around a single axis of rotation.

The two-dimensional images are recorded by a detector that represents the spatial maps of the linear attenuations, μ, of the sample that fit the Lambert-Beer law:

\[ I_n = I_0 e^{(-\mu L)} \]

where \( I_0 \) is the incident radiation of the X-ray beam, \( I_n \) is the transmitted radiation, \( \mu \) is the attenuation coefficient and \( L \) is the path length through the sample. The image is simply the
radon transform of attenuation [48]. The data recorded from multiple radial angles are collected and input into a tomographic reconstruction algorithm in order to reconstruct an approximation of sample volume of the interior structure of the object. The algorithm is a form of Fourier-like integral called an inverse radon transform [49]. Usually, the more rotational angles used to generate projection images, the more precise is the three-dimensional reconstruction. However, no matter how many angles are used to rotate the object, there is always a difference between the original object and the reconstruction volume. Differences are generally introduced into the volumetric data in the form of noise. Therefore, to obtain a better reconstruction, a noise reduction procedure must be applied. There are several types of noise inside the images, including Gaussian noise, exponential noise, impulse (salt-and-pepper) noise among others [50]. Noise can arise from a variety of factors, such as the low quality of the sensing device, the environment disturbances during the image acquisition process, and light levels and sensor temperature of the CCD camera. With regard to the transmitted signal, noise reduction may be achieved by applying mathematical filters to the signal to reduce or enhance certain aspects of that signal. These filters include mean filter, median filter, adaptive filter, bandpass filter, notch filters and so on [50]. After the noise is filtered out, the signal to noise ratio is increased. Also, the segmentation between regions of different density will be more clearly defined, and there will be a closer approximation between the 3D representation and the original object.

Compared to conventional X-ray, synchrotron X-ray reveals invaluable information for those fields of research that depends on a wide energy range and high resolution. In 1988, a group of European countries joined together to sponsor and create the synchrotron facility in Grenoble, France, called the European Synchrotron Radiation Facility (ESRF). There are about 50 synchrotrons around the world that are being used by an ever growing number of scientists. Among those, ESRF is one of the largest and most powerful synchrotrons in the world. An aerial view of the site of ESRF is shown in figure 2-9.
Three-dimensional reconstructions based on XRμCT (Computed Tomography) have been applied for characterizing paper at the sub-micrometer scale in the last decade. Samuelsen et al. [52] paved the way for the XRμCT three dimensional characterization of paper structure. They used synchrotron X-ray in phase contrast mode to show details of voids and individual cellulosic fibers by a series of cross-sectional slices of handsheet samples. Details of phase contrast were discussed in that article. A relatively low image quality that resulted from the reconstruction procedure was a limitation of this first attempt. The obtained images require extensive processing to reduce noise and improve contrast. The image process, including image reconstruction, noise and artifacts filtering, edge detection, and the binarisation of the pores, fibers and lumens, was detailed by Antonie et al. [53] who used phase contrast synchrotron X-ray source to study the 3D structure of different types of papers including handsheets, newsprint, and super-calendered magazine paper. Rolland du Roscoat et al. [54, 55] applied synchrotron X-ray micro-computed tomography in absorption mode to investigate the microstructural properties of paper and made use of a segmentation method to separate the different components of paper to determine the porosity and specific surface areas of the network structure. Analysis of the relationships between structural properties and the manufacturing processes was also conducted using synchrotron X-ray micro-computed
tomography. Although phase contrast synchrotron X-ray micro-computed tomography is better in distinguishing the border and interfaces between different materials and has a high resolution, the absorption mode is the most straightforward method of providing the necessary contrast for imaging [52, 53]. Goel et al. [56] used X-ray micro-computed tomography to analyze the impact of papermaking variables on the structure and transport properties of paper samples. They showed that among all the paper making variables involved in their study, calendering plays a dominant role in altering the sheet structure, yielding denser paper with smaller pores and higher resistance to transport. Holmstad et al. [57, 58] investigated the applicability of high resolution phase contrast synchrotron radiation to obtained reasonable measures of 3D characteristics while preserving the topology. This revealed detailed information of the 3D structure and transport properties. They also made a comparison of XRμCT and high resolution synchrotron X-ray tomography and pointed out that synchrotron X-ray has higher resolution, better image quality, and therefore is better suited for fundamental research. Most recently, Janne and Keranen [59] used X-ray tomography to study the paper properties with paper under different drying conditions.

2.7 Measurement of z-direction density distribution

Due to the rough surface of the paper and the complexity of fibers distribution in 3D network, the measurement of the density distribution in the Z-direction of the paper presents difficulty. Although there were not many articles in investigation of Z-directional density distribution of paper, several approaches have been tried.

Szikla and Paulapuro [33] used an optical microscope to image the fiber distribution in the cross section (with a thickness of 2μm or 4μm) of the handsheets and divide the cross section into several layers from top to bottom. The density of each layer was calculated based on the average density and the proportion of the fiber wall area in each layer to the total fiber wall area in the sample. The limitations of this measurement are cutting the sections with a microtome equipped with a glass knife and the difficulty in distinguishing of the fibers wall and void space confined by the image processing. Rättö [34] used a digital microscope to
image the cross section (with a thickness of 1.5μm) of the calendered and uncalendered uncoated paper and divide the section into a number of horizontal layers by the contour lines drawn at the top and bottom surface of the sample. The contour lines were all parallel and equidistant. The width of each layer is approximately 1μm. The percentage of fiber area in each layer was evaluated using a binary map with the assist of a designed Matlab code. The local density of each layer of the samples was calculated as the product of the fiber area ratio and the density of cellulose. The limitations are that the fibers will swell after the staining process and the five top and bottom layers, which had most influence on the surface properties of paper, were disregarded for these layers had a high density. Holmstad [60] used SEM to image the cross machine direction (CD) cut of commercially available SC-grad paper samples and apply the rolling ball technique from Chinga [61] to determine the paper surface. The density was also calculated based on the fact that each layer has a local thickness proportional to the local thickness of the paper. However, the rolling ball diameter will affect the surface definition and this method was just suited for smooth calendered paper. Correia and Roy [62] investigated the density profile of commercially paperboards through the Z-direction using an X-ray densitometer. The algorithm is based on the relationship of X-ray attenuation and density of the material. However, the resolution is low and this technique is then not available for lower thickness papers.
3. Problem statement and objective:

Statement of problem:
Paper towels formed by different processes result in patterned deformation and redistribution of fibers within the plane of the web. Depending on the processes used to induce creped or embossed structures, the web may exhibit variations in the thickness and density that are characteristic of the processes which are used. The measurement of thickness of paper towels presents a difficulty since the material is subject to compression when contact methods are used. The material also has a discontinuous surface that interferes with some of the more conventional non-contact optical methods, such as micro-focusing profilometry [63] and white light interference methods[64]. Subjected to the limitations of conventional instruments, there is an inadequate understanding about the structural properties of tissues and towels. In this investigation, two techniques, Twin Laser Profilometry and synchrotron XRMCT, are used to characterize the structure of paper towels (a subject of the tissue grade of paper). There is also an insufficient confidence in the agreement of the results generated by these two analytical techniques based in part on the different scale of detection. Also, the hypotheses that embossing will cause localized thinning of the web by pressing and that through air drying (TAD) samples have lower density than CWP sample as a result of pulling air through the wet web will be tested.

Project Objectives:
Overall, the objective of this investigation is to study the structural properties of paper towels. Specifically, the thickness and out-of-plane deformation maps of the samples, especially of the induced patterns in the samples, will be measured and compared for Twin Laser Profilometry and synchrotron X-ray micro-computed tomography methods. Secondly, the Z-directional density distribution of towel samples will be studied by synchrotron X-ray micro-computed tomography method, and attempts to quantify this distribution will be made. Clarification of important limitations of the non-contact optical method (TLP) as it is applied to low density fibrous structure, such as paper towels and tissue, will be sought.
4. Experimental Methods

This section will include the identification of the paper towel samples tested, part of the photography of the samples, and two thickness and density measurement techniques, which are Twin Laser Profilometry (TLP) and synchrotron X-ray micro-computed tomography (XRμCT).

4.1 Materials

Commercially available paper towels were examined in this study. Thickness mapping and out-of-plane deformation were measured and compared for single ply samples. Some samples were measured based on both single ply and 2-ply. Table 4-1 identifies the samples and provides the values for grammage and flat platen thickness measured by TAPPI standard.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Identification</th>
<th>Method</th>
<th>Grammage (g/m²)</th>
<th>TAPPI STD Caliper (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>GP Mardi Gras Top</td>
<td>CWP</td>
<td>21.9</td>
<td>90</td>
</tr>
<tr>
<td>A2</td>
<td>~ Bottom CWP</td>
<td></td>
<td>21.6</td>
<td>90</td>
</tr>
<tr>
<td>A3</td>
<td>GP Sparkle Top</td>
<td>CWP</td>
<td>22.5</td>
<td>95</td>
</tr>
<tr>
<td>A4</td>
<td>Bottom CWP</td>
<td></td>
<td>23.0</td>
<td>125</td>
</tr>
<tr>
<td>A5</td>
<td>Marcal Top CWP</td>
<td></td>
<td>24.7</td>
<td>100</td>
</tr>
<tr>
<td>A6</td>
<td>~ Bottom CWP</td>
<td></td>
<td>24</td>
<td>95</td>
</tr>
<tr>
<td>A7</td>
<td>Kruger White Cloud Top CWP</td>
<td></td>
<td>21.9</td>
<td>98</td>
</tr>
<tr>
<td>A8</td>
<td>~ Bottom CWP</td>
<td></td>
<td>21.9</td>
<td>100</td>
</tr>
<tr>
<td>B1</td>
<td>GP Brawny Top</td>
<td>TAD-3</td>
<td>29.6</td>
<td>235</td>
</tr>
<tr>
<td>B2</td>
<td>~ Bottom TAD-3</td>
<td></td>
<td>29.3</td>
<td>215</td>
</tr>
<tr>
<td>B3</td>
<td>PG Bounty White Top</td>
<td>TAD-1</td>
<td>20.3</td>
<td>140</td>
</tr>
<tr>
<td>B4</td>
<td>~ Bottom TAD-1</td>
<td></td>
<td>21.0</td>
<td>145</td>
</tr>
<tr>
<td>B5</td>
<td>PG Bounty Basic 1-ply Top TAD</td>
<td>39.2</td>
<td>196</td>
<td></td>
</tr>
<tr>
<td>B6</td>
<td>KC Scott Top</td>
<td>TAD</td>
<td>38.8</td>
<td>300</td>
</tr>
<tr>
<td>B7</td>
<td>PG Bounty Extra Top</td>
<td>TAD</td>
<td>28.8</td>
<td>155</td>
</tr>
<tr>
<td>B8</td>
<td>~ Bottom TAD</td>
<td></td>
<td>28.6</td>
<td>155</td>
</tr>
<tr>
<td>B9</td>
<td>GP 19682 eTAD</td>
<td>~</td>
<td>~</td>
<td>~</td>
</tr>
</tbody>
</table>
methods T410 [65] and T411 [26], respectively. For TLP analysis, samples were prepared by cutting into 70mm square regions in order to fit into the instrument’s sample frame. A centimeter square region near the center of each sample was scanned.

Incident light photography of CWP paper towel samples (A1, A8, B1, 70mm x 70mm, respectively) are shown in figure 4-1 and that of TAD samples (B6, B8, B7,) are in figure 4-2. The photography in the right column includes 10 mm square region sketched by a red box. The thickness and out-of-plane deformation figure of that region were scanned by TLP. Incident light photographs of the rest of the samples in this study are shown in Appendix 1. The patterns from the conventional wet pressing (CWP), or through-air-drying and/or embossing processes are evident in these figure.
Figure 4-1 Light photographs of CWP towel samples A1, A8 and A7.
Figure 4-2 Light photographs of TAD towel samples B6, B8 and B7.
4.2 Thickness mapping

Thickness mapping was performed using the Twin Laser Profilometry (TLP) method. A detailed description of the principle and operation of TLP instrument was provided in an earlier work by Sung [29, 30]. As shown in Figure 4-3, the instrument is composed of four main parts: digital laser sensors (Z), laser sensor positioning stages (Z), the main positioning stages (XY), and the sample frame. The laser sensor positioning stages (Z) move the left and right laser sensors along the Z-axis that is perpendicular to the principal plane of the paper sample which is held vertically between the sensors. The main positioning stages (XY) move both Z stages/sensors assembling simultaneously in the X and Y direction. This allows raster scanning within the principal plane of the sample. The sample frame holds samples vertically to reduce the out-of-plane effect caused by gravitation.

Each laser sensors emit a 670nm laser beam to form a 7~15μm diameter spot on the sample surface. The sensors are only sensitive to distances of up to 300μm range and provide a distance resolution of 1μm. The laser beam is specularly reflected from the sample surface and detected by the CCD detector after passing through two lenses. The received laser beam is detected as intensity distribution and digitized so that a smooth test surface, such as glass, appears as a normal distribution. This distribution is processed by centroid algorithm to

Figure 4-3 Schematic of TLP instrument
obtain the center which is used to calculate the average range value (distance) from the sensor to the sample surface. If the sample surface is irregular, such as the case with paper, the distribution detected in the CCD detector may be irregular multimodal and discontinuous. In such case, the centroid is still calculated and an average range is recorded. This provides a source of artifact or error when detecting very rough surfaces, such as tissues and towels. For surface irregularities that exceed the 300μm range limit of the laser sensors, the Z-positioning stages move the laser sensors towards or away from the sample so that the sensor can detect the surface. The combination of the Z-positioning stage displacement and the range measured by the laser sensor provides a relative spatial position of the surface to a resolution of 1μm.

If the laser spot encounters a large deformation or a void on the sample surface, so that the surface is not detected, the laser sensor enters a seek mode to search for the surface. For irregular surfaces, this slows the scanning processes. If the surface at that spot cannot be found, the instrument moves to the next X-Y position and considers this a “dead spot”. This point is excluded from the working data matrix. A tungsten foil calibration standard (57μm) is used to calibrate the three dimensional spatial position of the instrument. Thickness is calibrated against the known thickness of the foil. A small square hole is etched into the center of tungsten foil. The horizontal and vertical edges of the square are used to align the laser sensor in the X and Y directions. This ensures that the spots are aligned in precise opposition for analysis. While each sensor is moved independently along the Z-direction, they are moved simultaneously in the X-Y plane by the main positioning stages (X-Y). The sample frame can hold four samples. The exposed sample area is 50×50mm and the main positioning stage can move up to 130mm in each direction in the X-Y plane with a resolution of 1μm.

During the experiment period, paper samples were mounted to the sample frame and were fixed at the top edge by tap. Samples were marked with a felt tip pen to mark the starting location of each scan with an ink spot. For all samples, machine direction (MD) was aligned in the vertical direction and recorded on the sample. The instrument was set to map a 12.5 mm square region with an X and Y step of 25μm. The scan time varied from 4 to 6 days of continuous scanning time, but if a surface discontinuity was encountered, then it took a
longer time to finish scanning, depending how discontinuous the paper sample was. After each experiment, eight text files containing numerical data were stored. An information file contained the instrument calibration constant and the scanning parameters. Four important files consist of integers for the output of the left and right laser sensor, and for the positions of the left and right Z-positioning stages. Three other files were stored, two contained the calculated range values and the last contains a calculated thickness. These last three should be neglected since their data is erroneous and the first five were used to calculate the thickness value in Matlab. The five important text files for each sample were loaded into Matlab workspace (.mat files). Calibrated thickness and out-of-plane deformation were calculated in the Matlab command window and stored in .mat files in workspace (where Sp constant could be read from the information .txt and all the other values are from the other four files). It is from these calculated files that graphical representations of thicknesses and surfaces were produced.

4.3 Synchrotron X-ray micro-computed tomography (XRμCT)

4.3.1 High resolution synchrotron X-ray micro-computed tomography imaging

At ESRF, imaging was conducted at the ID19 beam line which is dedicated to high resolution X-ray imaging. This is a long beam line (145m) which provides a highly parallel source of X-ray that produces highly monochromatic and narrowly focused (<100nm) analyzing beam

![Figure 4-4 Schematic of the XRμCT experimental set-up [54].](image)
(10-35 keV). The schematic of the experiment setup is shown in figure 4-4[54]. One factor that limits the resolution and total volume tested was the time it took to rotate the sample on an axis normal to the beam, and to move the sample in steps along that axis to obtain multiple slices from which the 3D-position of material is deconvoluted. The more steps, due to resolution or to sample volume, the more time is required for testing. The second limiting factor is the amount of data that must be stored and processed (deconvolution processing time) which is confined by the computational resources that are available. The thousands of images captured by the detector in the ID19 beam line lab were deconvoluted into a 3D data arrays that contain intensity information at each point that indicates the existence of material, void space or the interface between the two (an edge).

For the samples tested in this study, the base resolution is 0.7μm, and the 3D data array is 2048×2048×1024; or with voxel dimensions that are ideally 0.7μm×0.7μm×0.7μm. The diameter of all of these cylindrical samples is 1.4mm. The sample was positioned so that the principal plane (X-Y or MD-CD) occupied the larger two dimensions and the shorter dimension measured position along the Z-axis. The rotation along the Z axis produced a circle of usable data confined by the in-plane dimensions. Stepping along the Z-axis and combining circles produces a cylindrically shaped region within the 3D array that contained tomographic information.

4.3.2 Tomographic file and image processing

The data sets as received from ESRF required further processing in order to distinguish between solid and void space. This required an appropriate regiment of denoising and segmentation. There are no generally accepted methods for treating tomographic data sets since the nature of the image depends on the experimental conditions used to obtain the data and the absorption response of the material being scanned. Thus, a unique protocol was developed to process the images obtained in this study.

For each sample, the deconvoluted data was transferred from ESRF to Miami University as four, 1GB data files in the *.raw format. This format is simply the linearization of individual
Z-directional slices, with dimensions of X and Y (2048). The four files were recombined by concatenation to form a 4GB file that was resaved in the *.raw format. This format is recognized by ImageJ, Avizo and Matlab as long as the array dimensions are provided. The two maps shown in figure 4-5 and figure 4-6 illustrate the volumetric structure of two typical CWP samples A1, A8 and two TAD samples B6 and B8, using Avizo software (VSG). Volumetric structure for other samples is given in Appendix II.

The images contained a speckled pattern of random noise, sweeping rings and central disk artifacts that resulted from sample rotation, and a grey level intensity difference between void/solid and interface. Images also contained the backing paper, metallic support platform, and in some case a considerable amount of void space above the sample that could be cropped from the file. For all of the samples, steps were taken to reduce the presences of each of these defects. ImageJ, a public domain software application provided by NIH (http://rsbweb.nih.gov/ij/) was used to address these defects.

Concatenated raw files were loaded into ImageJ using the dimensions provided in the attached *.info files. A despeckle (median) filter was applied twice to reduce the salt and pepper random noise apparent when viewing individual X-Z slices from the array. The upper and lower thresholds of the gray level distribution were adjusted to create a uniform field for the voids space, while not seriously affecting the location of the interface (edge) of the fibers. Slices were removed from the top and bottom of the array and so that data file size was reduced to contain all of the sample volume, while reducing file size. Finally, as series of rotations, reslices and crops were applied to the data arrays in order to eliminate the backing paper and as much of the non-sample material (e.g. Post-it adhesive) as possible. Once loaded in ImageJ, determination of the lowest X-Y slices where first contact with the backing paper was observed. Then, the angle needed to align the tip axis along the Y-axis was determined. This permitted the volume to be resliced along this axis. Inspection of the resulting X-Z profiles revealed the rotation required to align the desired sample volume with the X-axis. Reslicing once again, this time on the Z-axis produced a sample volume where bottom slices were removed to the point where only the desired sample volume remained. Often additional,
Figure 4-5 Three dimensional volumetric structure of two typical CWP samples A1 and A8
Figure 4-6 Three dimensional volumetric structure of two typical TAD samples B6 and B8
top slices were also removed. Samples that have undergone the rotate and reslice procedure to produce “cleaned” volumes contained the letters “RnR”. These were considered to be the optimized data sets that contained the most useful sample volumes for further analysis.

4.3.3 Surface definition and generation of thickness and OPD maps

After the image processing of all of the slices, Matlab was used for all subsequent analysis. The thickness and OPD maps and other statistical analyses are generated based on these 2D slices. The algorithms used to generate the thickness and OPD maps are shown in figure 4-7. The figure also shows one X-Z slice out of the 2048 that composes the total volume. The surface boundaries are defined as the first occurrence of distinguishable material, i.e. fibers or fines, proceeding from the top or bottom along the Z axis. The thickness value, \( H_{x,y} \), is equal to the vertical difference between the top boundary and bottom boundary of the sample. The out-of-plane deformation, \( C_{x,y} \), is equal to a half of the sum between the top bounding and bottom bounding surfaces, defined as the imaginary center surface. The surface definition and algorithm of generating thickness and OPD maps for the Twin Laser Profilometry (TLP) was the same as that and detailed by Sung[29, 30]. The only difference is the scale of resolution.

![Figure 4-7 Schematic of algorithm calculating thickness and OPD map and one slice out of 2048](image-url)
To obtain the thickness and out-of-plane deformation map for the entire sample, all of the 2048 slices are processed using this algorithm. Thickness values and center plane height values are a function of the location in the X-Y plane. Thereafter, it is possible to generate thickness and OPD maps for this 1.4mm×1.4 mm region that are similar to those obtained from the TLP method. In order to improve the usefulness of these maps to study features much larger than individual fibers, a median smoothing filter was applied using 50μm window.

4.3.4 Threshold value defining

In order to define an agreeable threshold value to calculate the thickness and out-of-plane deformation map, several threshold values were tried, and the graph of the median thickness value to threshold value was plotted. Figure 4-8 and figure 4-9 shows the relationship of median thickness value to threshold value. In figure 4-8, the median thickness value was not significantly affected by the threshold value. Therefore, we could choose a threshold value between 30 and 40. This is because these images are denoised to have a high quality. Another case, shown in figure 4-9, is that the change of threshold value would affect the median thickness value a lot due to the noise within the tomography images. For example, the median thickness value obtained at the threshold of 40 is quite different from that of 25. Therefore, we selected a threshold value large enough to eliminate the noise while retaining the fiber information in the result.
4.4 Statistical analysis

The matrix containing the thickness and out-of-plane deformation information was reshaped into a vector in Matlab, and the vector was copied and pasted into the M.S. Excel in order to generate a histogram graph. The thickness values of the same paper towel sample obtained by different methods, XRμCT and TLP, were plotted in the same frequency vs. thickness graph to compare their relationship. The same was performed for the out-of-plane deformation values. The following is the procedures for plotting the histogram graph of the thickness and out-of-plane deformation values in Excel.

(1) A histogram dialog was opened in the Data/Data Analysis. The thickness data was selected; the bin was set to a step value of 1, and the frequency data was output to a column.
(2) For the vector values from X-ray method, subtract the frequency number of bin “0” by the number of zeros surrounding the cylinder.

(3) The frequency data was then normalized using the following equation:

\[ F = \frac{F_i}{F_1 + F_2 + \cdots + F_i + \cdots F_n} \times 100 \]

Where, \( F_i \) is the frequency number in each category, 1 to n.

(4) The histogram was then drawn using the bin as the X-axis and normalized frequency calculated using thickness values or out-of-plane deformation values from TLP method and XR\( \mu \)CT methods as the Y-axis, to compare the relationship.
5. Result and Discussion

The thickness and the out-of-plane deformation maps of the conventional wet pressing (CWP) and through air drying (TAD) samples are illustrated and compared to provide further insight into the structural properties of towel samples. Two analytical methods, Twin Laser Profilometer (TLP) and synchrotron X-ray micro-computed tomography (XRμCT) were used to obtain the thickness and OPD maps. An agreement of these two techniques, with respect to generate thickness and OPD maps, is present by comparing and quantifying their result.

5.1 Conventional wet creping (CWP) samples

5.1.1 Twin Laser Profilometry (TLP)

The three maps shown in figure 5-1 illustrate the results obtained from profilometry measurement using the TLP instrument for a typical CWP paper towel sample (A1). The size of the sampled region is 10 mm×10 mm. The top image is the thickness map and the out-of-plane deformation map is shown at the bottom left. The thickness and out-of-plane deformation vary as a function of the position in the X-Y plane. As seen from the thickness map, most of the spots inside the embossed region have thickness values less than 30μm, which, assuming a collapsed fiber thickness of ~8μm, corresponds to a coverage of 4 fibers or less. There appears to be a thicker region, at X=1.8 mm and Y=9 mm, which is about 70μm thick. This is likely a higher grammage flocculated region, although this was not confirmed by formation mapping. Close inspection of both the thickness and out-of-plane deformation maps shows horizontal ridges, which are created by the creping process. Note that the cross machine direction (CD) is oriented parallel to the X-axis. This creping pattern is also clearly visible in the light photograph in figure 4-1, where shadows are cast by the incident light. The areas within the circular embossed feature are generally thinner. However, significant thickening occurs at the periphery. Thickness values at the edge are ~150μm or more, cf. X=4.5 mm and Y=2.3 mm. These thicker regions may result from the mechanical disruption of the web during the embossing process where the stresses are dissipated to the surrounding
Figure 5-1 Structural maps obtained from TLP for CWP sample A1. Thickness map (top) and out-of-plane deformation map (bottom left) are plotted at a resolution of 25μm; and binary map used to partition the embossed and non-embossed region (bottom right).
Region via the 3mm long fibers. Part of the excessive thickness may result from an artifact of the profilometer and the method it uses to detect the sample surface.

From the OPD map, one can see that the deformation value is about 150μm to 200μm. In order to compare the mean thickness values of the embossed region with that of non-embossed region that surrounds, a mask was created using the out-of-plane (OPD) map, cf. the bottom right map in figure 5-1. The mask was used to partition the embossed and non-embossed regions so that the respective thickness values for each region could be determined. Histograms of the thickness distributions for the two regions are shown in figure 5-2 for comparison. The median of the embossed region is 15μm and that of the non-embossed region is 20μm. Clearly, the embossing process for this sample has caused thinning in addition to planar displacement. The magnitude of this thinning has been quantified and the uniformity of the thickness may also be compared. The variance of the thickness values of the embossed region appears smaller than the non-embossed region, suggesting the hard and smooth surface used in the embossing process makes the surface more uniform.

![A1](image)

Figure 5-2 Frequency distribution of thickness values for CWP sample A1. Dash line: pressed region with a median of 15μm; solid line: non-pressed region with a median of 20μm.
5.1.2 Synchrotron X-ray micro-computed tomography (XRµCT)

Confined by the size (dia.≈1.4mm), the imaged region was not large enough to include examples of all of the features present in many of the towel samples tested. Some tomographs illustrate certain features better than others, depending on where the image was obtained from. The two maps in figure 5-3 show the results obtained from XRµCT imaging of a typical CWP paper towel sample (A1). In these two maps, one could clearly see the embossed feature, as a semicircular in the upper half. The deformation value is about 200μm, which is the same as that obtained using TLP. From the thickness map, the embossed region has thickness values which are generally less than 30μm. This is equal to what was observed using the TLP method. Close inspection of the OPD, 3D volumetric tomograph and a single slice shows parallel undulations, which are created by the creping process. These are identified in figure 5-4. The machine direction (MD) or cross machine direction (CD) was not marked when the samples were tested in the synchrotron. Creping ridges are actually oriented in the CD, and so for papers alignment of the MD/CD to X/Y, the sample should be rotated about 90° clockwise or counterclockwise. The areas within the semicircular embossed feature are generally thinner. However, there is a thicker region at the periphery with a thickness value of 150μm or more, cf. X=0.35mm and Y=1.10mm. This thicker region is clearly the result of the artifact of the algorithm used to calculate the sample thickness from tomographic volumes, detailed in section 5.4.
Figure 5-3 Structural maps obtained from XRµCT for CWP sample A1. Thickness map (top) and out-of-plane deformation map (bottom) are plotted at a resolution of 0.7µm.
Figure 5-4 Top: OPD of A1 with white oval sketching the creping patterns; middle: the three dimensional volume; bottom: a slice out of 2048 (illustrating creping feature)
5.1.3 Comparing TLP and XRμCT maps

The mapped region of the paper towel samples for TLP instrument is 10mm × 10mm which allows all of the induced features to be including in a single test. Therefore, there is a high likelihood that the region (with a diameter of 1.4mm) analyzed using the XRμCT will compare well with a region in the TLP map. And so the thickness and out-of-plane deformation maps obtained by both TLP and XRμCT methods should be comparable to each other if a proper spatial alignment was achieved. Since the dimensions of XRμCT sample are small, finding a comparable region could be difficult, depending on the clarity of the features. There are several XRμCT samples that have distinguishable regions that allow a connection to be made. Comparison of both thickness map and out-of-plane map were conducted and statistical analyses were performed.

5.1.3.1 Thickness map correlations

The thickness maps obtained from both methods were firstly matched. The sample compared here is A1. The thickness values of the matched spot were statistically compared. Figure 5-5 shows the thickness maps of TLP sample and XRμCT sample and the image after the map from XRμCT was fitted into that of TLP. The XRμCT image was rotated 90° counterclockwise to align the ridges (creping feature).

Once the spot in TLP sample that matches with XRμCT sample was found, a binary map was used to extract the thickness values of the spot with the assist of Matlab. The thickness frequency distributions were plotted on the same axis, as shown in figure 5-6. The frequency distribution also includes the boundaries of the thickness distributions which were obtained by plotting all the 8 important and valuable matched spots. From the thickness probability distributions we can see that both data have similar distribution with comparable median value of 25μm for a single matched spot of TLP sample and 27μm for XRμCT sample.
Figure 5-5 Top: thickness image from TLP with dimension of 10mm×10mm; middle: thickness image from XRμCT with dimension of 1.4x1.4mm; both samples have the same color bar range from 0 to 150μm; bottom: image after the image from the XRμCT was fitted into that of TLP.
Figure 5-6 Thickness probability distributions of both samples with median value of 25μm for a single matched region in TLP sample and 27μm for XRμCT sample. The top and bottom dash lines are the boundaries of the thickness distribution of the eight vital matched regions in TLP sample.

5.1.3.2 OPD map correlations

In the second step, correlation was done by matching the out-of-deformation map from TLP method to that from XRμCT method. Figure 5-7 shows the out-of-plane deformation images from TLP sample and that from XRμCT sample and the image after the map from the XRμCT sample were fitted into that of TLP sample.

Once the spot in TLP sample that matches the XRμCT sample was found, a binary map was used to extract the out-of-plane deformation values of the spot using calculations in Matlab. The frequency distributions were plotted on the same axis, as shown in figure 5-8. From the out-of-plane deformation probability distributions one can see that both data have similar distribution. The deformation value obtained by both is about 180μm to 200μm. It means that the deformation values obtained from both methods match each other.
Figure 5-7 Top: OPD map from TLP sample with dimension of 4.99x12.5mm; middle: OPD map from XRμCT sample with dimension of 1.4x1.4 mm; bottom: image after the OPD map from XRμCT sample was fitted into that of TLP sample.
5.2 Through air drying (TAD) samples

5.2.1 Twin laser profilometry

Now, the structure properties of through-air-dry (TAD) sample (B6) will be considered. The size of the mapped region is 12.5mm×12.5mm. Figure 5-9 illustrates the thickness (top) and out-of-plane deformation maps (bottom left). Two distinct features, which are the TAD screen and creping patterns, are clearly observed. There are regions where the wet web is supported during the TAD process, and where a backing roll compresses this region of the web. Thinning has clearly occurred as evident in the thickness map, and the web centerline is displaced as seen in the OPD map. Once again, the thicker region along the border of the vertical ridges results from the abrupt discontinuity of the paper web. In the thickness map, closer inspection reveals a creping pattern parallel to the horizontal axis. This pattern is clearly shown in the out-of-plane deformation map and photograph figure 4-1. This deep creping results from the wet creping process which causes bulking of the web that are evident in the thickness map.
Figure 5-9 Structural maps for TAD sample B6. Thickness map (left), out-of-plane deformation map (right) are plotted at a resolution of 25 μm.
The structural map of a second TAD sample (B8) is shown in figure 5-10. The patterns induced by through-air-drying (TAD) process are clearly shown in both the thickness and out-of-plane deformation maps. The patterns that are seen were caused by the TAD cylinder. Inspection of the thickness map shows considerable thinning of the structure along the margins between the patterned TAD features, which is caused by compression of the web between the bars of the cylinder and a backing roll used. Comparison of the thickness distribution of the two regions is made using an OPD mask to partition the pressed and non-pressed region, cf. figure 5-10 bottom right. A histogram is plotted to demonstrate the difference in the thickness distributions of these two regions, cf. figure 5-11. The median values of the pressed and non-pressed region are 68μm and 118μm, respectively, which is far

Figure 5-10 Structural maps for TAD sample B8. Thickness map (left), out-of-plane deformation map (right) are plotted at a resolution of 25μm.
Figure 5.2.2: Frequency distributions of thickness values for TAD sample B8. Dash line: pressed region with a median of 68μm; solid line: non-pressed region with a median of 118μm.

This means the non-pressed region is bulked to a very high level due to the air pulled through to dry the wet web. The bulkiness of this region contributes to the absorptivity and softness of this towel paper. On the other hand, the increased inter fiber bonding found in the pressed region forms a continuous meshed structure, cf. figure 2-4. This contributes to the tensile strength of the whole paper web.

5.2.2 XRμCT

The structural properties of through air dying (TAD) samples (B6) from XRμCT will now be considered. Figure 5-12 illustrates the thickness (top) map, OPD (center) map, the 983rd slice (first bottom) viewing from the cross machine direction and the 1515th slice (second bottom) from the machine direction. The TAD pattern (out-of-plane deformation) is clearly seen both in the OPD map and the slice in the MD. This is the region where the wet web is supported during the TAD process, and where a backing roll compresses this region of the web to make thinning. This is similar to what was observed in the TLP method. Bulking and thinning are obvious in both the thickness map and the slice in the MD. The slices obtained by cutting
from the cross machine direction (bottom) were also inspected in order to see whether the creping process could be detected. Due to the small size and location of the sample in the paper towel, the creping pattern could not be observed in the both the maps and slices.
Figure 5-12 Structural maps of TAD sample B6 from XRμCT. From top to bottom, they are thickness map, OPD map, MD slice and CD slice, respectively.
5.2.3 Comparing TLP and XRµCT maps

For this sample (B9), the comparison of TLP and XRµCT is conducted in the same manner as that in section 5.1.3.

5.2.3.1 Thickness map correlations

Figure 5-13 shows the thickness maps of TLP sample and XRµCT sample and the image after the map from XRµCT was fitted into that of TLP. Once the spot in TLP sample that matches with XRµCT sample was found, a binary map was used to extract the thickness values of the spot. The thickness frequency distributions were plotted on the same axis, as shown in figure 5-14. From the thickness probability distributions one can see that both data have similar distribution with comparable median value of 49µm for TLP sample and 52µm for XRµCT sample. It means the results from both methods are in agreement with each other.
Figure 5-13 Top: thickness image from TLP with dimension of 4.99x12.5 mm; middle: thickness image from XRμCT with dimension of 1.4x1.4mm; both samples have the same color bar range from 0 to 200μm; bottom: image after the image from the XRμCT was fitted into that of TLP.
Figure 5-14 Thickness probability distribution of TAD sample (B9) with median value of 49 for TLP sample and 52 for X-ray sample

5.2.3.2 OPD map correlations

Figure 5-15 showed the out-of-plane deformation images from TLP sample and that from XRμCT sample and the image after the map from the XRμCT sample were fitted into that of TLP sample.

Once the spot in TLP sample that matches with XRμCT sample was determined, a binary map was used to extract the out-of-plane deformation values of the spot with the assist of Matlab. The frequency distributions were plotted on the same axis, as shown in figure 5-16.

Free fiber protruding at about 450μm-500μm was detected from both methods. It is not an artifact of the experiment, but a discontinuous characteristic of the sample. From the out-of-plane deformation probability distributions one can see that both data have similar distribution. This means that the deformation values obtained from both methods are in agreement with each other.
Figure 5-15 Top: OPD map from TLP sample with dimension of 4.99x12.5mm; middle: OPD map from XRμCT sample with dimension of 1.4x1.4 mm; bottom: image after the OPD map from XRμCT sample was fitted into that of TLP sample.
5.3 Comparison of CWP and TAD paper towels

5.3.1 Thickness distribution

Figure 5-17 shows the thickness maps for four paper towels, two formed by CWP (A1, A8) and two by TAD (B6, B8). The color scale for each sample were adjusted to the same range, 0-450μm, so that the thickness can be compared. It is evidently that the two TAD samples are significantly thicker than the two CWP samples. Among these four samples, B6 is the thickest. It is well known that that TAD was introduced to maintain high bulk (thickness) in order to improve softness and absorptivity. One can not only observe the large scale structural network that is used to achieve strength (see the mesh network in B8) while maintaining regions of high bulk, but quantification of the patterns of densification for both the CWP and TAD can now enable the mechanical behavior to be modeled. For these materials, the mapping of density would require imaging of regions using a suitable formation measurement method, such as transmission X-radiography. However, this was not performed in this investigation. Figure 5-18 shows a comparison of the thickness distribution of pressed and non-pressed regions for CWP (A1) and TAD (B8) samples. This figure demonstrates the
Figure 5-17 Comparison of the thickness value of CWP and TAD samples after adjust the color bar to be the same range, 0-450μm. Top left: A1 with grammage of 21.9 g/m²; top right: B6 with grammage of 38.8; bottom left: A8 with grammage of 21.9; bottom right: B8 with grammage of 28.6g/m².
Figure 5-18 comparison of the thickness distribution of CWP sample (A1) and TAD sample (B8). The median of embossed and non-embossed regions of A1 are 15μm and 20μm, respectively; the median of pressed and non-pressed region of B8 are 68μm and 118μm, respectively.

The utility of the thickness distributions for quantifying the differences in uniformity of thickness between samples, and in regions within each.

The thickness map of sample A8 shows the relative uniformity of thickness distribution and little information of embossing. However, the embossing patterns are evident in its OPD map, as shown in figure 5-19. This implies that the embossing process for this sample, unlike for the proceeding sample A1, causes displacement of the web with a minimal amount of thinning. A regional mask is once again made using the OPD map and histograms quantify differences in the thickness distributions. As seen from figure 5-20, the distributions of embossed and non-embossed regions superimpose each other. This means that the thickness values of the embossed features remain essentially unchanged from the original web. Both regions have close median thickness values, which are statistically equivalent (26μm). Therefore, the embossing process for this sample only causes out-of-plane deformation, but no thinning.
Figure 5-19 Structural maps for CWP sample A8. Thickness map (top) and out-of-plane deformation map (bottom left) are plotted at a resolution of 25μm, and binary map used to partition the pressed and non-pressed region (bottom right)
**Figure 5-20** Frequency distributions of thickness values for CWP sample A8. Dash line: pressed region; solid line: non-pressed region. The median of pressed and non-pressed regions are both 26µm, respectively.

### 5.3.2 Void regions

For the TAD process that disrupts the web by pulling air through the paper web during drying, one might assume that the TAD samples will have a higher percentage of void spaces or pinholes, as compared to CWP samples which are dried on a slightly curved smooth surface. For TAD, the structure is stretched and opened as air passes through. Figure 5-21 illustrates the spatial composition of void space for the mapped region that have thickness less than 1µm, or where thickness was not detected. Table 5-1 summaries the relative void area along with the grammage for each sample. Clearly, the CWP samples have more areas that appear as holes or voids as compared to the TAD samples. There are two possible reasons that may account for this. First, the TAD samples generally have higher grammage, which reduces the likelihood of the formation of pinholes due to the greater fiber coverage. Secondly, the CWP samples appear to have more void spaces associated with embossed features, which may result from fracturing of the structuring (minor tensile breakage) that could have occurred during the embossing step. Embossing takes place when the web is in a dried state, and are therefore slightly fractured. The TAD, on the other hand is deformed when the web is wet,
and fibers are free to move away from each other when displaced. The observation of the

Figure 5-21 Comparison of the void percentage of CWP and TAD samples after adjust the color bar to be the same range, 0-450μm.

<table>
<thead>
<tr>
<th></th>
<th>CWP</th>
<th>Percentage void</th>
<th>Grammage (g/m²)</th>
<th>TAD</th>
<th>Percentage void</th>
<th>Grammage (g/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>17.1%</td>
<td>21.9</td>
<td>B6</td>
<td>2.9%</td>
<td>38.8</td>
<td></td>
</tr>
<tr>
<td>A8</td>
<td>12%</td>
<td>21.9</td>
<td>B8</td>
<td>6.8%</td>
<td>28.6</td>
<td></td>
</tr>
</tbody>
</table>
void space may also find use in the future for modeling of air permeation or for characterizing of filter media.

### 5.3.3 Reduction in web thickness

The relative reductions of the thickness for the different samples are listed in Table 5-2. Percentages are calculated based on the ratio of pressed to non-pressed regions for a given sample. TAD samples generally have a higher thinning ratio as compared to CWP samples. Knowing the extent of densification by the different process variables can be an important advantage for future investigations that explore the effect of hygroexpansibility and its uniformity between regions. It may also be an important factor for understanding the influence that local density has on performance properties, such as softness or absorptivity.

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Identification</th>
<th>Method</th>
<th>Thickness of un-pressed region (μm)</th>
<th>Thickness of pressed region (μm)</th>
<th>Percentage (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A1</td>
<td>GP Mardi Gras</td>
<td>top CWP</td>
<td>25.8</td>
<td>21.1</td>
<td>18.2%</td>
</tr>
<tr>
<td>A2</td>
<td>~</td>
<td>bottom CWP</td>
<td>28.8</td>
<td>27.2</td>
<td>5.5%</td>
</tr>
<tr>
<td>A3</td>
<td>GP Sparkle Top</td>
<td>CWP</td>
<td>24.3</td>
<td>24.2</td>
<td>0.2%</td>
</tr>
<tr>
<td>A4</td>
<td>~</td>
<td>Bottom CWP</td>
<td>24.3</td>
<td>22.4</td>
<td>7.9%</td>
</tr>
<tr>
<td>A5</td>
<td>Marcal Top</td>
<td>CWP</td>
<td>48.4</td>
<td>48</td>
<td>0.9%</td>
</tr>
<tr>
<td>A6</td>
<td>~</td>
<td>Bottom CWP</td>
<td>45.5</td>
<td>45.5</td>
<td>0%</td>
</tr>
<tr>
<td>A7</td>
<td>Kruger White Cloud Top</td>
<td>CWP</td>
<td>42.8</td>
<td>30.5</td>
<td>28.8%</td>
</tr>
<tr>
<td>A8</td>
<td>~</td>
<td>Bottom CWP</td>
<td>29.8</td>
<td>29.3</td>
<td>1.6%</td>
</tr>
<tr>
<td>B1</td>
<td>GP Brawny top</td>
<td>TAD-3</td>
<td>88.8</td>
<td>57.4</td>
<td>35.4%</td>
</tr>
<tr>
<td>B3</td>
<td>PG Bounty White Top</td>
<td>TAD-1</td>
<td>76.6</td>
<td>66.0</td>
<td>13.8%</td>
</tr>
<tr>
<td>B4</td>
<td>~</td>
<td>bottom TAD-1</td>
<td>79.7</td>
<td>62.2</td>
<td>22%</td>
</tr>
<tr>
<td>B7</td>
<td>PG Bounty Extra Top</td>
<td>TAD</td>
<td>143.9</td>
<td>77.6</td>
<td>46.1%</td>
</tr>
<tr>
<td>B8</td>
<td>~</td>
<td>bottom TAD</td>
<td>128.5</td>
<td>87.6</td>
<td>31.8%</td>
</tr>
<tr>
<td>B9</td>
<td>GP 19682 1-ply eTAD</td>
<td></td>
<td>69.3</td>
<td>68.0</td>
<td>1.9%</td>
</tr>
</tbody>
</table>
5.4 Density distribution

In section 5.1.2, it was determined that the thicker region at the periphery of the embossed pattern in the thickness map from XRμCT is clearly the result of the artifact of the algorithm used to calculate the sample thickness from tomographic volumes. This is because the algorithm measures the thickness based on the Z-direction which is parallel to the general principle plane of the sample, but not orthogonal to the local principal plane of the material represented by the center surface. This presents problems when the web is oriented at steeper angles such as the transition zone between the pressed and non-pressed regions, as shown in figure 5-22. This method will give a much larger thickness value as it calculates the distance from the top and bottom boundary, than it would if it calculated thickness through surface normal.

In order to calculate the thickness and local distribution of mass along surface normal, cf. figure 5-23, a more representative surface will be considered. Because the samples investigated in this study had huge out-of-plane deformation, shown discontinuity in the principle plane, and exhibited folding back on itself as a result of creping or other disruptions, a strategy was adopted to determine the center surface of the samples. This center
Figure 5-23 measurement of thickness and local distribution of mass in surface normal surface was further filtered and used to determine local orientation of the sample plane to calculate the surface normal, $Z'$. Therefore, the thickness and local distribution of mass (fiber) will be determined along $Z'$ for each point in the sample space.

This center surface was calculated along $Z$ direction by using the 2048 X-Z slice images (See Appendix II for the center surface for all of the samples). Figure 5-24 shows the algorithm of

$$M_{x,y} = \text{Median}(Z_{x,y})$$

Figure 5-24 the algorithm of calculating the center surface of a slice out of 2048 using median function.
calculating the center surface of one slice. The surface determined by this method will overcome some of the obvious defects, such as surface curvature, but it was demonstrated that this method does not properly address folds in the plane and the abrupt discontinuities due to low fiber coverage, free fibers and holes. Figure 5-25 illustrates the center surface maps for the samples A5, which clearly reveals the egg shaped induced feature. Close inspection of the images reveals a local discontinuity in the calculated center surface that results from irregularities in the fibrous mass and surface irregularity of the plane. Due to the existence of these artifacts, the data contained within is inadequate for continued analysis since the surface normal vectors calculated from this surface would not be consistent between local regions. A 2D median filter in Matlab was applied to smooth this surface. Figure 5-26 shows the result when this filter is applied to the center surface. Although most of the surface discontinuity was eliminated, the algorithm has introduced surface tiling and step functions which might interfere with the calculation of surface normal. More importantly, the “Median40” does not eliminate the major flaws that occur with folding and surface discontinuity that some of the samples exhibit.

Essential, the proposed approach for determining the local distribution of mass and thickness by using the surface normal vectors, and regional space calculated from these normal vectors, depends on the specular smoothness of the center surface. Having a diffusing surface, filled with numerous facets and irregularities, will produce uncertain results. Over filtering the surface will either eliminate interesting features, or mask sub-section of the features, such as corners or indentations within, where density difference are of interest. It was therefore decided not to apply the local distribution algorithms to the center surface maps that were generated based on the Z-median with spatial median filters applied.
Figure 5-25 Center surface of the XRS-μTomographs B9
Figure 5-26 Center surface of the XRS-μTomographs With a 2D median filter/40μm (B9).
6. Conclusion and Future Work

1) TLP and synchrotron X-ray micro-computed tomography
Twin Laser Profilometry (TLP) enables web structures to be mapped to a micrometer resolution over 12.5 mm×12.5 mm regions of interest. The thickness distribution of the pressed and non-pressed regions of towel samples was partitioned and quantified. While some artifacts, such as discontinuities, free fibers and beam diversion, conceal the true nature of the surface to some extent, the method remains valuable in providing meso-scale structural dimensions over a relatively large scanning region, with minimal instrumental effort or cost.

Conversely, synchrotron X-ray micro-computed tomography method is capable of providing sub-micrometer resolution of the complete volumetric structure for the imaged region. The features of individual fibers are revealed by this method, so that the true surface geometry may be quantified.

Although there is a large difference of the scale of detection, results from the two analytical techniques show there is an enough agreement of these two methods in term of measuring the structural properties of towel samples including the thickness and out-of-plane deformation maps.

2) Conventional wet pressing (CWP) and through air drying (TAD)
Creping and through air drying both cause bulking, but it is evidently that the TAD towel samples in this investigation are significantly thicker than the CWP samples. The CWP samples appear to have more areas that contain pinholes or voids than TAD samples, while TAD samples generally have higher thinning percentage as compared to CWP samples. In addition, embossing process could sometimes only cause out-of-plane deformation, but no thinning.
Future work:
1), Measuring a formation map using a accurate technique, and dividing the formation map by the thickness map to obtain the density map.

2), It may be useful to partition into more than two sections, therefore the contributions of each pattern could be tested.

3), The observation of the void space may also find use for modeling of air permeation or for characterizing of filter media.

4), Testing the structural maps under different humidity and various stretch degrees, and investigate the effects of hygroexpansibility and stretch on the pressed and non-pressed region.

5), Calculating the thickness and density distribution along surface normal.
7. Reference


[64] Jahanmir, J. and J. C. Wyant, "Comparison of Surface Roughness Measured with an Optical Profiler and

Appendix I: Incident light photographs

Figure I-1: top: A7 Brawny top; middle: B2 Brawny bottom; bottom: B5 Bounty basic
Figure I-2: Top: B3 Bounty white top; middle: B4 Bounty white bottom; bottom: A4 Sparkle bottom.
Figure I-3: Top: A5 Marcal top; middle: A2 Mardi gras back; bottom.
Appendix II: XRµCTomographs

Left: volumetric structure; right: center surface; bottom slices.

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<th>Figure II-1</th>
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Figure II-3: A3        GP Sparkle (bottom)

Figure II-4: A10        GP Sparkle (top)
Figure II-5: A2  GP Mardi Gras (bottom)

Figure II-6: A1  GP Mardi Gras (top)
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<th>Figure II-7: B4</th>
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Figure II-9: B8    PG Bounty Extra (bottom)

Figure II-10: B7    PG Bounty Extra (top)
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Appendix III: Thickness and out-of-plane deformation map from TLP and XRμCT

Figure III-1:  A2  Mardi gras bottom
Figure III-2: A4  sparkle bottom

Figure III-3: A5  Marcal top
Figure III-4: A7  Kruger white cloud top

Figure III-5:  B2  Brawny bottom
Figure III - Bounty ex top
Appendix IV: Thickness comparison of CWP and TAD samples

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Appendix V: void percentage

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