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# APPLICATIONS OF THICKNESS AND APPARENT DENSITY MAPPING BY LASER PROFILOMETRY

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## ABSTRACT

This paper describes the development of a method for mapping the apparent density of paper, nonwovens and other fibrous webs based on non-contact laser profilometry and  $\beta$ -transmission radiographic imaging. The method is applied in three complimentary studies that examine the in-plane non-uniformity of thickness, grammage and apparent density in printing papers. The first section focuses on the development and verification of the analytical method for mapping thickness. Through simultaneous scanning of the topography of both sides of the specimen by opposing range sensors, the surface contour of each side, the local thickness, and the out of plane deformation were measured. It was demonstrated that the laser based method provides results that closely approximate the intrinsic thickness that is independent of paper formation. The method was then used to examine the

#### Y.J. Sung, C.H. Ham, O. Kwon, H.L. Lee and D.S. Keller

structural differences in laboratory sheets pressed by soft and hard nip calenders. The well known difference in web densification mechanisms of the two was reaffirmed by mapping discrete changes in thickness and statistically comparing these with corresponding points on grammage maps. Densification was shown to be dependent on grammage for the hard calender, and independent of grammage for the soft calender. The final study used the thickness mapping method to monitor the hygroexpansivity of representative printing papers as equilibrium humidity was varied between 9% and 80%. Thickness maps were obtained for newsprint, SC-A (calendered and uncalendered), bulk offset and office copy. Differential thickness maps were used to compare the in-plane non-uniformity of hygroexpansion. The inplane hygroexpansion was characterized and corrected for using a recently developed algorithm known as Enhanced Digital Image Correlation (EDIC). Thickness change did not appear to correspond to grammage maps. The results suggest that a significant irreversible increase in thickness occurs for papers that are heavily calendered.

## INTRODUCTION

For thin fibrous web materials such as paper, paperboard, tissue and nonwovens, the structural density and thickness are important characteristics. Density is especially important since most of the key properties associated with mechanical strength, optical response and fluid transport are directly influenced by the distribution of fibers and pores within the structure [1, 2]. Non-uniformity of the apparent density within the principal plane of the web may result in production or coating problems. It may also cause quality defects such as poor optical formation (distribution of opacity), various print mottles (print density and gloss) or excessive out-of-plane deformation (cockle). The uniformity of apparent density is usually approximated by mapping the in-plane distribution of mass, or *paper formation*, using transmission radiographic methods [3–6]. Implicit in the use of paper formation is the assumption that either that thickness varies directly with formation or that thickness is relatively constant within the region of interest. Nevertheless, this approach has been widely applied in the characterization of mechanical [7], optical [8, 9], coating [10] and printing performance [11–13] with reasonably good success. Significant discrepancies arise when the web is subjected to densification processes that influence thickness and the uniformity thereof. Komppa and Ebeling [14] and Sara [15] demonstrated that calendering disrupts any correlation that may exist between mass and optical formation due to changes in the uniformity of local scattering coefficients caused by the non-uniform densification of the structure. In the absence of a reliable method to map the thickness to the same resolution currently attained by transmission radiography, a deficiency exists in our understanding of how the uniformity of structural density is affected by processes.

An almost universally accepted method for measuring thickness in industrial and scientific applications is by use of a hard platen caliper. This method brings two planer surfaces in contact with the web under a predefined normal pressure, e.g. TAPPI and SCAN standard caliper methods. The separation distance between the platens is taken as the thickness of the material and is well suited for foils and films. However, the unevenness of the surface and internal lumpiness of webs causes the planar approximation to depart from the "true" top and bottom surfaces. Fellers et. al [16] provided a review of the methods used to improve the accuracy of thickness measurements including soft platen caliper [17, 18] and mercury buoyancy [19]. While both methods enable the surface "probe" to conform to the structural irregularities of paper, neither provides the ability to map the non-uniformity of thickness. Each method of thickness measurement brought its own unique physical definition of the surface. To address this, Fellers et. al [16] used the intrinsic thickness as that which most closely defines the web surface. It essentially describes a linear relationship between thickness and grammage that passes through the origin.

Contacting probes were first used to measure local thickness to generate linear scans [20] and then used to map regions [21-23] to a spatial resolution as fine as 150 µm. Thickness maps were combined with grammage maps (formation) to map the local apparent density of papers. Schultz-Eklund *et al.* [23] used opposing spherical platens to study the relationship between thickness and grammage and the effect that calendering has on the relationship. They showed how the fine scale uniformity of density was influenced by this type of densification process. However, since the compressibility of the paper is non-uniform and is determined by a number of variables that contribute to the material structure, any contacting method such as the opposing spherical platens may be subject to artifacts unrelated to the true local thickness [18]. This complicates the interpretation of comparisons between different paper grades or those subject to different densification processes.

The introduction of laser range sensors with sub-micrometer level resolution enabled the development of non-contacting surface profilometers, useful in measuring the surface topography of paper and other fibrous webs [24–27]. The methods are based on either dynamic confocal ranging [24] or triangulation [26, 28]. While avoiding the artifacts presented by contacting methods, both optical methods have unique artifacts [26] that must be addressed in order to obtain an accurate representation of the paper surface.

The concept of non-contact profilometry was expanded for the measurement of local thickness by simultaneously scanning of both sides of the paper with the laser sensors [29–31]. Oba [31] used an instrument developed by Izumi and Yoshida [30] based on dynamic confocal sensors. They were able to map local thickness of a 100 mm  $\times$  100 mm region to an in-plane spatial resolution of 100  $\mu$ m and with micrometer precision in the Z-direction. The present investigation uses a method developed by Sung [29] that uses triangulation based sensors.

The purpose of this investigation was to develop a laser based method for mapping the local thickness of fibrous webs. A comprehensive examination of the operating characteristics and performance capabilities was conducted in the context of comparing commercial printing papers. Artifacts that could interfere with the accurate measurement and mapping of web thickness were systematically evaluated and addressed. The fundamental relationships between the three structural parameters, grammage, thickness and apparent density were examined using grammage and thickness maps with 100  $\mu m$  spatial resolution.

The specific objectives of the study were to apply the additional capabilities of the instrument to explore structural changes that occur with soft and hard nip calendering and compare results from the two densification methods. The increased thickness resolution and improved registration between grammage and thickness maps was utilized to compare subtle differences in the spatial uniformity of densification. A second objective was to investigate the uniformity of hygroexpansivity of printing papers exposed to various relative humidities from 9% to 84%. A comparison of the new method with conventional methods of thickness measurement was performed to provide appropriate context. The differences between thickness maps at different moisture contents should provide an accurate representation of the mean out-of-plane hygroexpansivity. It will also map the heterogeneity of Z-hygroexpansivity within the test region.

#### **DEVELOPMENT OF THE METHOD**

#### Principles of the mapping methods

The Twin Laser Profilometer (TLP) is based on the concept that surface topographic maps obtained from the two planar sides of a sample can be

spatially registered so that the distance between the surface maps at each point represent the thickness at that position [29]. While there is no general agreement on what constitutes the "surface" of fibrous webs or paper, the non-contacting optical sensors used in this study were shown to provide a close approximation of the outermost optically reflective surface. Specifically, the sensors detected 10  $\mu$ m diameter surface facets, or *spots*, that specularly reflected visible laser light at an incident angle of 35°. Surface irregularities contained within the 10  $\mu$ m region were integrated into a single value for that region. The semi-transparent nature of some papers was addressed by adjusting the sensitivity of the sensor to distinguish top surface reflectivity from that reflected from subsurface structure. In extreme cases of transparency, the back surface of the fibrous structure or film can be detected.

Figure 1 shows a photograph of the Twin Laser Profilometer in the arrangement used to determine sample thickness. The laser range sensors are mounted on positioning stages that move the sensors along the axis perpendicular to the sample surface. The displacement of the positioning stages, summed with the range values acquired from the laser sensors, is subtracted from a constant reference distance to determine the thickness of the specimen at that location. The instrument constant is determined before each session using foil standards of known thickness.

Figure 2 illustrates the strategy for mapping the various structural parameters for each sample [29]. The top and bottom surface topographies are determined simultaneously. The separation of the surfaces, or local thickness, is calculated from the sensor positions. An imaginary center-plane that bisects the bulk phase is determined at each position. Since the two surfaces may have significantly different roughness, the center position calculated at each spatial point will contain components of each surface roughness plus the out-of-plane deformation. Filtering of the center plane surface with image analysis smoothing techniques provides a good approximation of the *out-of-plane deformation* of the sample. Any cockling or curling of the sample will be apparent in this surface. The thickness map, independent of out-ofplane deformation, was calculated from the difference between the top and bottom surfaces.

#### A new approach for mapping surface roughness

Conventional methods for determining surface roughness have involved probing only one surface of the specimen. In as such, the analysis relies on one of two assumptions. Either it is assumed that 1) the opposing side of the sample is ideally in contact with the basal reference plane, or 2) there is no out-of-plane deformation and any variation in thickness (lumpiness) is

Y.J. Sung, C.H. Ham, O. Kwon, H.L. Lee and D.S. Keller



Figure 1 Photographs of the TLP instrument.



**Figure 2** Example of thickness measurement by using twin laser profilometer instrument. Sample is Newsprint Scanning interval (X–Y); 25 μm.

distributed uniformly between the measured topographies of the top and bottom surfaces. The roughness is therefore the variation in the distance from the basal reference plane to the sample surface, i.e., variation in the profile. By this approach, it is impossible to distinguish between out-of-plane deformation and lumpiness. Furthermore, one cannot isolate these features from the true roughness of the surfaces. The schematic diagram of a new approach is shown in Figure 3. By measuring the three-dimensional position of the opposing surfaces, the true surface roughness may be determined. The filtered center plane is used as the reference plane to determine the deviation, i.e. roughness, of each surface independently. The surface roughness and lumpiness are partitioned from the out-of-plane deformation. By varying the filtering kernel (zone size) used to smooth the center plane, the scale of surface roughness may be analyzed in greater detail to determine the size of surface features.



Figure 3 The schematic diagram of roughness measurement by the TLP instrument method (left) and the conventional method (right). Arrows indicate the length measurement used to determine roughness. The TLP instrument measures from an imaginary center plane, filtered to remove the effects of roughness. The conventional method measures the length between the top surface and a reference plane.

Instrument design

The Twin Laser Profilometer (TLP) instrument consists of three main parts; the sample frame, the opposing laser sensing systems and the main (X–Y) positioning units [29]. Figure 1 illustrates the configuration of the instrument. The sample frame is rigidly affixed to the base and holds the test specimen vertically. The two laser sensor units are held in opposition along the same (Z) axis, normal to the sample surface, so that each can independently measure the distance to one side of the sample surface at precisely the same inplane position. The main positioning unit moves the laser sensing system vertically and horizontally, i.e. within the (X–Y) plane of the test specimen. A square region of up to 130 mm can be mapped to 3-dimensional resolution of 1  $\mu$ m.

The laser sensing system consists of optical range sensors affixed to high precision (Z) positioning stages. This arrangement is uniquely different from earlier approaches [30, 31] in that the sensors use a triangulation algorithm to detect range. The sensors can be independently repositioned to track the surfaces of samples with excessive lumpiness or out-of-plane deformation, such as cockle, curl or buckling, that exceeds the operating range of the sensors. The sample may be tested in various states of restraint including free hanging.

A control computer applies a mapping sequence for main stage positioning and acquires the range data sent from the sensors. The Z-positioning stages are repositioned using a feedback loop from the range data. The stage positioning and range data were coordinated to maximize sampling rate and mitigate noise from stage vibration and other sources. Sampling rate ranged from 0.2 to 1 Hz and was limited by the surface roughness and the planarity of the sample. For excessively rough surfaces more Z-stage adjustments are needed to reliably track the surface. The range data obtained from the sensors was then converted into thickness maps that were then used to calculate bulk density maps from corresponding transmission radiographic images. The instrument was isolated from the ambient environment to control temperature and humidity and to mitigate noise and vibration.

### Optical range sensors

Two DRS 300 Digital Laser Sensors (Cyber Optics Corp., Minneapolis, MN), were used to measure the range between a fixed spatial position and the sample surface. Figure 4 illustrates the optical triangulation method that was used to measure the distance to a digital resolution of 0.2  $\mu$ m in a 300  $\mu$ m working range. A 670 nm laser beam was focused onto the surface at an incident angle,  $\theta = 35^{\circ}$  producing a 10  $\mu$ m spot on the surface. A CCD detector positioned along the path of specular reflectance detects the position and intensity of the spot. A distribution of intensities as a function of position is sensed on the CCD array. The nature of the peak shape provides insight into the transparency and surface gloss of the specimen under examination [32]. A threshold intensity value is set so that light attributed to diffuse reflectivity or subsurface penetration in semi-transparent materials is excluded. The mean value of the peak that lies above the threshold indicates the average distance to the surface for the region contained within the beam spot. This effectively filters surface roughness for features less than the spot





size. While the sensors are capable of distinguishing individual softwood tracheids [26], they are unable to resolve detailed surface features of coated papers. Similar sensors have been used for measuring surface profiles and mapping surface topography [33–36]. The sensors were calibrated by the manufacturer using NIST-traceable standards. The operating accuracy was determined in this study to be 1 $\mu$ m using standard foils.

Range sensors based on triangulation of a laser beam are subject to an artifact known as the *shadow effect* [33]. The reflected beam is interrupted by extreme depressions in or protrusions from the sample surface [26, 37, 38]. There are actually two types of shadowing effects that may occur on an excessively rough surface. In one case the incident or reflected beam is blocked from detection by the sensor. In the other case the incident beam may be specularly reflected away from the sensor so that again, no spot is detected. These shadowing artifacts may be substantial for sample surfaces that have abrupt changes in surface profile where the extrema that greatly exceed the spot diameter, such as fine wire mesh. This artifact may interfere with the measurement of materials such as linerboards or paper toweling where pore size and diameter may greatly exceed the beam spot diameter. For such samples, beam blockage will cause many points to go undetected yielding surface maps that are incomplete to a greater or lesser extent depending on the sample roughness. The surface topographies of the printing papers examined in this study are smooth enough so that this artifact will have little or no effect on results. A subtle amount of beveling may occur for the most abrupt surface features which will provide a slight loss in spatial resolution.

#### Positioning stages

By mounting each laser range sensor on a high precision, linear positioning stage, the standoff distance between the sensors and the sample surface was controlled. The sensor positioning stages (Z-axis) were ATS 100-050-20P stages (Aerotech Corp., Pittsburgh, PA). These provided Z-axis motion with a range of 50 mm and a resolution of 0.5  $\mu$ m. The range sensing systems was thereby extended beyond the 300  $\mu$ m limits of the optical sensors. This enables the system to map heavily cockled or curled samples with out-of-plane deformation up to ±20 mm. This arrangement also enabled the system to overcome a beam displacement artifact characteristic of the triangulation ranging method that will be discussed in the section that follows.

The sensing systems, consisting of the range sensors affixed to the sensor position stages, were moved in unison by the main (X–Y) positioning stages. This allowed the sensors to sample all positions within a 130 mm square region to a precision of ~1  $\mu$ m. The main positioning stages were Aerotech

ATS 206 series with a maximum travel of 140 mm. The stage movement was programmed to dampen vibration that could be detected by the sensors.

#### Registration of the laser sensing systems

Two strategies were used to register the opposing laser systems so that the two surfaces of the sample could be measured in precise opposition. The original configuration of the instrument enabled the in-plane alignment of the spots to within 10  $\mu$ m in the X and Y directions. Alignment was calibrated before each experiment using the detection of a vertical and horizontal edge of a 57  $\mu$ m thick tungsten foil. If the residual differences between the positions of the lasers were significant, the images were digitally translated to improve registration. The present configuration of the instrument uses micrometers to adjust beam registration to  $\pm 1 \mu$ m.

An artifact of triangulation range sensors is that the position of the spot on the surface will change with respect to the center axis of the sensor as a function of the distance from the sensor to the surface (Figure 4). The result is an irregular spacing of points as the sensor is moved to collects data at regularly spaced intervals as demonstrated by Elgay [26]. When the two sensors are equidistant from the surface and the centerlines of the sensors are aligned, the spots are in opposition, Figure 4(a). If the surface is closer to one sensor or the other, c.f. Figure 4(b), the detector will sense the difference in distance,  $\delta Z$ . However, the spot will also shift from the centerline so that the opposing spots are no longer aligned by a displacement of  $\delta X$ . For the DRS-300 sensors  $\delta X = \delta Z \tan\theta = 0.7\delta Z$ . To overcome this artifact, the sensor positioning stages move the laser sensors to the same standoff distance,  $\pm 20$ µm, before each distance value is recorded thereby reducing potential lateral displacement to the dimensions of the beam spot diameter.

#### Environmental control

The instrument was enclosed in a clear acrylic chamber so that humidity and temperature could be closely controlled. All experiments were conducted under TAPPI standard condition (23° C and 50 % RH) unless specified otherwise. The enclosure also served to isolate the test specimen from extraneous air movement generated from air currents and sounds in the room. Sung [29] demonstrated that without the enclosure the ambient sound levels and air currents caused drum like vibration of the sample that exceeded 3  $\mu$ m depending on the stiffness of the material. Single point variability was reduced to < 1  $\mu$ m by enclosing the instrument and situating the instrument on a vibration isolation table to eliminate contact vibration.

## EXPERIMENTAL

## Materials

Commercial samples were obtained from a variety of sources. Table 1 gives a description of the samples tested in this study, and provides grammage values. Laboratory paper samples were formed by using a British handsheet mold according to TAPPI Method T205. Standard research pulps obtained from the National Institute of Standards & Technology (NIST) were used to prepare handsheets. The softwood pulp [39] had a nominal coarseness of 17.6 mg/100m and a mean fiber length (length weighted) of 2.85 mm. The hardwood pulp [39] had a nominal coarseness of 9.5 mg/100m and a mean fiber length of 0.65 mm. Both were beaten using a laboratory valley beater [40] to CSF freeness of 521 ml for the hardwood and 634 ml for the soft wood. All handsheets considered in this report consisted of 60 % softwood and 40 % hardwood.

Experiments were conducted under TAPPI standard conditions of 23  $\pm 2^{\circ}$ C and 50% RH, unless stated otherwise. Samples were preconditioned at the standard or modified conditions for at least 24 h before tests were performed. Samples were marked with a rectangular hole or with three pinholes in order to register thickness and grammage maps for subsequent mapping of density.

Sample ID	Description	Grammage (g/m <sup>2</sup> )	Ash Content (wt%)	
Fine Coated	C2S and Calendered	149		
Copy-A	201b Recycled Office Copy	76.9	9.4	
Copy-B	24lb Office Copy (Superior Quality)	105		
Envelope	Envelope paper	104		
SC-A	Supercalendered A	51.2	25.7	
SCA-UnCal	Uncalendered stock for SC-A	51.2	25.7	
Newsprint	Newsprint (Superior Quality)	48.8	1.2	
Thin-Pad	Thin bleached packing paper	42	0	
Tracing	Tracing Paper, Medium Wt.	44	0	
Handsheet	Handsheet 40% BHWK/60%BSWK	80	0	
HB Offset	High bulk offset paper	116.7	8.9	

 Table 1
 Identification of the Commercial Samples

## Paper formation imaging

The local grammage distribution (paper formation) of the samples was determined using a storage phosphor  $\beta$ -radiographic imaging system. Exposures were made using a <sup>14</sup>C PMMA source and a storage phosphor imaging screen. After a one hour, the screens were processed using a PhosphorImager (Molecular Dynamics, Sunnyvale, CA, USA). Exposed screens were scanned and digitized at a 50 µm pixel size. The captured data array was converted into a grammage map using a calibration constant determined using Mylar of various thickness as a reference. This system has a linear response to  $\beta$  ray exposure and grammage sensitivity of ~0.1 g/m<sup>2</sup> as detailed by Keller and Pawlak [6, 41].

## Mercury displacement

Mercury (Hg) buoyancy measurements were conducted using a modified approach introduced by Wasser [16, 19]. The volume of a paper specimen of known length and width was determined by measuring the weight change that occurs as it is immersed in Hg. In this study, the specimens were oriented parallel the Hg surface so that when immersed to 30 mm, a uniform head pressure of 4 kPa was applied to the paper-Hg interface.

## Apparent density mapping

The thickness maps were measured at a resolution of 25  $\mu$ m spacing and then dilated to 100  $\mu$ m elements by local averaging. This enabled a pointwise registration of the formation images and thickness maps by division into apparent density. The image of local grammage was fitted visually together with the image of local thickness using registration marks. After visually fitting, a final adjustment was performed based on the two-dimensional correlation coefficient between two images.

## STRUCTURAL MAPPING OF VARIOUS PAPER GRADES

## Comparison with caliper methods

In order to examine the performance of the Twin Laser Profilometer (TLP) instrument for the analysis of commercial paper samples, a variety of representative paper grades were tested, as identified in Table 1. The results from the TLP were considered in the context of existing thickness measurement methods by comparing results with two standard caliper methods; TAPPI T-411 hard platen micrometer method [42] and a modification of TAPPI

#### Y.J. Sung, C.H. Ham, O. Kwon, H.L. Lee and D.S. Keller

T-551 soft platen method [17]. Both apply a 50 kPa normal load over a 200 mm<sup>2</sup> circular region with a diameter of 16 mm. The modified soft platen used two types of elastiomeric latex, Latex-L10 and Latex-L25, (Small Parts Inc., Miami, FL.) Latex L10 has a lower compressive modulus. The TLP structural thickness,  $Z_{TLP}$ , was determined by averaging local thickness values determined at 0.1 mm spacing within a 20 mm × 10 mm region for a total of 20,000 points. The same region was tested using the hard and soft calipers by averaging 10 measurements. Error bars in the figure represent the standard deviation of the data. Figure 5 shows the results from the four methods for the commercial samples.

Fellers and coworkers [16] described the limitations of the hard platen methods for determining the true or *intrinsic thickness* of paper. Essentially, the caliper methods integrate regions that are sufficiently large so that any variation in thickness or roughness will contribute to the measured value. Deviation from the intrinsic thickness occurs when the hard platen surface makes contact with the thickest areas while the indentations or thinner areas remain undetected. The result is an increase in measured thickness that is a function of structure non-uniformity, referred to by Fellers *et al.* as the *apparent thickness*. The soft platens address this situ-



Figure 5 Thickness of the Commercial Paper Samples measured with four different methods.

ation to some extent by conforming to the irregularity of thickness. As evident in Figure 5, thickness values for the soft caliper are generally lower than those for the hard caliper. Exceptions occurred for the super-calendered samples that are highly uniform in thickness, i.e. SCA and SuperCal Coated.

The *structural thickness* data obtained from the TLP instrument tend to be lower than the caliper methods in all cases, cf. Figure 5. This follows the explanation given by Fellers and coworkers [16] who described a similar situation for the thickness values determined using the STFI opposing spherical platen method [SCAN-P88:012001]. Although this is a contacting method, the spherical platen contact diameter is < 4 mm and the normal load is low, ~0.1N, so that the instrument senses the non-uniformity of thickness at a finer scale than the caliper methods. Thus a more accurate representation of the sample thickness is determined; one that more closely approximates the *intrinsic thickness*. The standard deviation of the TLP thickness values,  $\sigma_s$ , shown as the error bars in Figure 5, provides an indication of the variability of the thickness measured in the tested region. The graph shows that the samples with greater variability in thickness exhibit the greatest differences between the TLP and the hard platen thickness results. The relationship between variation of thickness as quantified by the standard deviation of thickness and the differential between the hard platen caliper and TLP thickness values is shown in Figure 6. The relationship suggests that the two methods correspond well for smooth samples with  $\sigma_s < 5 \ \mu m$  such as SC-A and SuperCal Coated. The difference becomes greater with increasing thickness variation up to  $\sigma_s = 10 \ \mu\text{m}$ . Above a thickness variation of 10  $\mu\text{m}$ , the thickness difference increases without further increase in thickness variation. This may result from the scale of spatial non-uniformities, although this was not pursued further in this investigation.

#### Relationship between TLP mean thickness and grammage

The relationship between the *integrated mean thickness* measured by the TLP, and the more ideal *intrinsic thickness* was explored in more depth by conducting controlled experiments using laboratory handsheets. Many investigators found that for a paper, a linear relationship existed between mean grammage,  $\overline{W}$ , and mean (structural) thickness,  $\overline{Z}$ , and that there was no significant difference in the bulk density. This was especially true for laboratory paper samples [16, 43–45].

Thus, the basic relationship is given by

$$\overline{Z} = a\overline{W} + \beta$$
 where:  $a,\beta$  are empirical constant. (1)



**Figure 6** The relationship between thickness variation and the difference between TAPPI Std. Caliper thickness and mean thickness measured by the TLP instrument.

and the mean bulk density,  $\overline{\rho}$ , of the paper is given by

$$\overline{\rho} = \left(\frac{\overline{W}}{\overline{Z}}\right) = \left(\frac{\overline{W}}{a\overline{W} + \beta}\right) \cong \frac{1}{a} (if \beta \cong 0)$$
(2)

Therefore, the mean bulk density theoretically becomes constant and is defined by Fellers *et al.* [16] as an *intrinsic density*, for  $\beta \rightarrow 0$  (*intrinsic thickness*). The constants *a* and  $\beta$  may be significantly affected by the properties of the furnish, the papermaking processes, and the thickness measurement methods. For commercial papers, an accurate determination of the relationship between thickness and grammage and the resultant bulk density is difficult. The indirect method based on the standard caliper thickness and the *intrinsic density* once the offset  $\beta$  is determined.

Figure 7 shows the average thickness values of laboratory handsheets, measured by the methods described above and plotted as a function of sample grammage. A linear relationship between grammage and thickness was evident for all methods. The caliper methods provided higher values of thickness as compared to the TLP instrument with an incremental difference for all samples tested. By applying the empirical relationship between thickness and grammage in Equation (1), the slopes,  $a = 1/\overline{\rho}$ , were found to be quite similar. This is expected since the handsheets were prepared from the same furnish and using the same pressing and drying conditions. They should therefore have similar bulk density,  $\overline{\rho}$ . However, the offset in thickness,  $\beta$ , differed significantly between the methods. The hard platen has the greatest offset of 21 µm, the soft platens, 14 µm, and the TLP essentially passes



Figure 7 Average thickness measured by four different methods as a function of the sample grammage. The  $30 \times 30$  mm area of each handsheet sample was tested 10 times with the caliper methods. The region was scanned at 100µm interval by the TLP instrument.

#### Y.J. Sung, C.H. Ham, O. Kwon, H.L. Lee and D.S. Keller

through the origin. This clearly demonstrates the inadequacies of the *apparent thickness* as measured by the TAPPI hard platen caliper method and the tendencies to overestimate thickness, especially for rough samples such as the handsheets tested in this study. The soft platen method was shown to alleviate the overestimation by conforming to surface irregularities. The *structural thickness* measured by the TLP non-contact method,  $Z_{TLP}$ , provides results that are consistent with the ideal *intrinsic thickness*. Therefore, bulk densities determined using TLP thickness method should closely approximate the *intrinsic density* of the materials. Figure 8 shows how the structural densities determined from the TLP thickness values are effectively constant with grammage as is the *intrinsic density* by definition [16]. The platen methods vary with grammage and differ depending on the ability of the platens to conform to surface irregularities.



Figure 8 Bulk density as a function of grammage for the data plotted in Figure 7.

#### Effect of sample reflectivity and transparency on measured thickness

Based on the principals of operation of the optical triangulation sensor as discussed above, the reflectivity of the surface will affect the shape and intensity of the spot as detected by the CCD in the sensor. This may have serious implications for the ability of the detector to resolve the correct distance to the top surface. The optical properties, including opacity (transparency), surface roughness, and gloss may all influence the results [26].

#### Reflected beam characteristics

For an ideally transparent material such as Mylar film, the sensor's incident beam will be reflected off of the top and bottom surfaces where a change in the refractive index occurs. The result is that two beam spots may be detected by the sensor. If the sample is thin enough, both spots may fall within the 300 µm range of the CCD detector and be recorded. The thickness of such material is obtained by measuring the distance between the detected spots taking into consideration the refractive index of the material as demonstrated in an earlier investigation [26]. Even from samples that are translucent, such as some paper and paperboards, the penetration of the incident laser beam may occur to a greater or lesser extent depending on scattering and absorption coefficients of the material. Figure 9 shows typical sensor output obtained from a tracing paper with ISO opacity of 36.2 [29]. The plot shows the characteristic primary peak that originates from reflectance off of the top surface of the sample. A second peak that occurs to the right of the first is generated by reflectance that occurs within the fibrous structure. The correct distance to the surface, represented by the peak with greater intensity can be captured by adjusting the threshold limit of the detector (horizontal line) to a level high enough to exclude any internally reflected light. For papers with



Figure 9 Intensity distribution of beam captured by the sensor for Tracing Paper with 36 ISO opacity. The threshold limit may be set above the second peak so that the correct surface range may be calculated.

low scattering coefficient such as the handsheet and the tracing paper, the exact location of the top surface was detected by adjusting the threshold limit.

#### Sputter coating experiments

The effect of beam penetration on surface mapping was examined in more detail in experiments conducted using sputter coating surface treatment to block beam penetration. The samples for this experiment were coated with the Gold-Palladium (Au-Pd) using a cold sputter coater (Desk 2, Denton Vacuum Co., Moorestown, NY). This sputter coater provide a uniform 100 Å coating layer to all externally exposed surfaces of the samples. The coating application was small enough to have no significant effect on the measured thickness of the samples. The effects of the Au-Pd sputter coating on the optical response of the tracing paper may be seen in Figure 10. Before coating there was some penetration and scattering of the laser light within the structure as indicated by the presence of the peak shoulders. The Au-Pd coating eliminated the laser light scattering and penetration and reduced peak width substantially.

A closer examination of the effects of beam penetration was conducted using the bleached kraft handsheet sample that exhibited a relatively large change after sputter coating. The TLP instrument was used to map the thickness of and area 10 mm  $\times$  20 mm at 50µm intervals. The thickness maps of the handsheet sample obtained before and after coating are shown in Figure 11. The left half region was protected from the coating process by loosely covering the region with a film "shield". The shielded region (left) was subjected to all of the preparation steps including evaporation, without undergoing the actual sputtering deposition. Comparison of the thickness results from the shielded side, before and after coating, indicates the effect of sample



Figure 10 Intensity distribution of beam captured by the sensor for Tracing Paper with and without Au-Pd sputter coating. The uncoated top and bottom are on the left. The coated top and bottom are on the right.



Figure 11 The effects of coating process condition and Au-Pd coating on the paper thickness. The exact same area of no filler handsheet was scanned with 50 μm interval before and after Gold-Palladium Coating.

preparation on the measured thickness map. The difference between the structural thickness of the shielded side (left) before and after coating was  $\sim 1 \mu m$ . This demonstrated that the preparation process caused only a small change in thickness to occur, at the reproducibility limit of the instrument.

By coating the surface, all beam penetration was blocked. The difference between a region before and after coating is shown in Figure 11. The observed change of the measured structural thickness that occurs with sputter coating was about 4  $\mu$ m. This exceeds the reproducibility of the instrument, and should be considered for papers that are more translucent.

All of the commercial samples were tested in similar Au-Pd sputter coating experiments. For each, a 10 mm square sample area was scanned with 50  $\mu$ m intervals before and after sputter coating. The use of an alignment bracket enabled the same region to be scanned by the TLP before and after coating. It was therefore possible to evaluate the effect of surface coating, on a point by point basis. The results are shown in Table 2. For each sample, the structural thickness values after coating were found to be slightly higher than those measured before coating. This suggests that there was only a small amount of

#### Y.J. Sung, C.H. Ham, O. Kwon, H.L. Lee and D.S. Keller

Sample	Before Coating (µm)	After Coating (µm)	Difference (µm)
Coated Fine	113 ±2	115 ±2	1.7
Copy-A	86 ±8	88 ±8	2.6
Copy-B	109 ±6	111 ±5	1.9
Envelope	136 ±9	137 ±8	0.8
SC-A	44 ±4	44 ±4	0.2
SCA-UnCal	66 ±9	$68 \pm 10$	2.2
Newsprint	61 ±6	63 ±6	2.5
Thin-Pad	34 ±8	37 ±8	2.4
Tracing	26 ±8	30 ±6	3.6
Handsheet	$124 \pm 10$	$128 \pm 10$	3.9

**Table 2** Thickness of commercial paper samples before and after Au-Pd sputter coating. Since the nearly same area of each sample was compared, the direct point wise comparison rather than statistical approach was conducted in this experiment.

penetration and scattering of the laser beam. However, the observed difference between average thickness values was small for most of the commercial paper samples. Also, the variation of thickness represented by the standard deviation of each thickness data did not appear to change by surface coating. This indicated that reduction in thickness due to laser light penetration occurred over the entire scanned surface, essentially to the same extent.

Sung [29] conducted a more detailed investigation of the relationship between the results shown in Table 2, and optical properties including opacity, scattering coefficients and gloss. He found no consistent relationships or trends relating these optical properties and the change in reflectivity imparted by the Au-Pd coating treatment on the TLP thickness measurement.

# EFFECTS OF CALENDERING PROCESS ON THE IN-PLANE DISTRIBUTION OF STRUCTURAL THICKNESS AND DENSITY

The changes in structure that paper undergoes when it is calendered can be significantly different depending conditions such as hard or soft nip, temperature, moisture, pressure and dwell time. Rather than conduct an in depth investigation of the calendering process, this study sought to demonstrate the utility of non-contact mapping of thickness and density for improved quantification of the differences between the densification processes. A comprehensive comparison of hard and soft calendering and the effect of various conditions have on product properties has been performed by Steffner *et al.* [46] for woodfree papers and Crotogino *et al.* for groundwood. [47]. Schultz-Eklund *et al.* [23] conducted a cursory examination of the differences between the thickness vs. grammage distributions for hard and soft nip calender using the opposing spherical platen method to acquire local thickness. The work in this section elaborates on work previously reported by Sung [29, 48] of how a similar study was conducted using the TLP instrument.

#### Experimental

#### Calendering

Calendering of laboratory handsheets was performed using pilot scale calenders. A hard nip calender stack (Borg-Warner Inc., Denver, CO) and soft nip calender (B. F. Perkins & Son Inc., Holyoke, MS) were used. In order to directly compare the effects of the two calendering processes, the same nip dwell time, 16 ms, and nip pressure, 10 MPa, were applied to the samples. The other operating constraints are given in Table 3. Calculation of nip pressure was based on the assumption that the load was evenly distributed on each calender roll.

The paper formation and structural thickness maps were determined for each handsheet sample before and after calendering. Since significant thickness recovery could occur after calendering, and it may take as long as 24 h for thickness to relax to a steady state [49], samples were conditioned in a TAPPI standard environment for 24 h before post-calendering testing was performed.

Operating Parameters	Soft Nip	Hard Nip
Calender Speed (m/min)	30	9
Ingoing Nip Width (mm)	8.4	2.4
Nip Length (mm)	1816	391
Linear Load (kN/m)	83.6	24

Table 3	Operating	parameters	for	soft	nip	and	hard	nip	calendering	•
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#### Results

#### Effects of hard nip calendering

The grammage and local thickness maps for the sample subjected to hard calendering are shown in the first column of Figure 12. The local thickness map of the sample before calendering exhibits a pattern which is quite similar to that of the local grammage map. This indicates the existence of a spatial correlation between grammage and thickness, i.e. greater grammage corresponds to greater thickness [29]. Although the pattern of local thickness variation can be observed in the local thickness map after hard nip calendering, the intensity of the pattern is significantly diminished. This is expected since, by design, the hard nip calendering substantially reduces the variation of the local thickness regions.

This is more clearly illustrated in a correlation plot of the thickness vs. grammage for each point in the sampled region shown in Figure 13. Before calendering, the thickness and grammage suggest a bivariate Gaussian distribution which is consistent with the linearity of the relationship, The slope and intercept of the principal axis are given in Table 4. The significance of this relationship has been proposed by by Dodson and coworkers [50, 51] especially for characterization of the porous structure. The compression of the thicker regions during calendering decreases the slope of the relationship so that thickness becomes increasingly independent of grammage. The structural density of the handsheet was initially independent of grammage with a correlation coefficient of  $R^2 = 0.005$ . Hard platen calendering increased the correlation to  $R^2 = 0.62$  and resulted in an approximation of the bivariate Gaussian distribution, cf. Figure 13. The density maps are provided in Figure 14. The overall increase in density with calendering is apparent, although any change in correlation with grammage is difficult to recognize since both the before and after images show features that are consistent with the grammage map, cf. Figure 12. The map of local densification, calculated by a point wise difference between the before and after maps, also shows a pattern of features similar to the local thickness map, cf. Figure 12B. This agrees with the observation that the thickest regions, e.g. floc or lumps, are preferentially compressed and densified by the hard nip calender. Therefore, after the hard nip calendering, the variation of local density could roughly be predicted by the original variation of local grammage.

Figure 12 (opposite) The grammage maps and local thickness distribution from before and after calendering are shown. The left column shows results using the Hard

Nip Calender. The right column shows the results for the Soft Nip Calender.



Figure 13 Plots of the thickness vs. grammage and local density vs. grammage relationships for the Hard Nip Calender (left) and Soft Nip Calender (right).

Figure 14 (opposite) The local apparent density maps before and after calendering and the net local densification. The results for the Hard Nip Calender (left) and the Soft Nip Calender (right) are shown.



0 kg/m<sup>3</sup> 90 105kg/m<sup>3</sup>

#### Y.J. Sung, C.H. Ham, O. Kwon, H.L. Lee and D.S. Keller

	Hard	l Nip	Soft Nip		
	Before	After	Before	After	
Slope	0.947	0.279	0.953	0.747	
Intercept	6.5	60.3	11.4	21.1	
Correlation Coefficient	0.554	0.244	0.468	0.563	

Table 4	Statistical	parameters for	the plots	in Figure 13
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## Effects of soft nip calendering

The local thickness maps for the sample before and after soft calendering are shown in the right column of Figure 12. The before calendered thickness pattern corresponds well with the formation image as was observed for the hard calendered "before" sample. The apparent direct linear relationship is evident in Figure 13C. A distinct thickness pattern remains after soft nip calendering that appears similar, albeit thinner, to the "before" calendered thickness image. The effect of soft calendering is clearly represented in the thickness vs. grammage correlation plot. Figure 13C shows that the calendered sample continues to approximate a bivariate Gaussian distribution with a similar correlation coefficient and slope as given in Table 4. The local density maps for the uncalendered and soft calendered samples show little correlation with grammage, cf. Figure 14 and Figure 12, respectively.

#### Comparison between hard nip calendering and soft nip calendering

With soft calendering, the reduction in thickness is distributed uniformly across the high and low thickness regions. The result is a paper that has a more uniform distribution of apparent density as compared to hard calendering, cf. Figure 14. This is quantitatively represented by comparing graphs B and D in Figure 13 for both calendered samples. The effect of hard nip and soft nip calendering processes on the local paper structural properties were summarized in Table 5. Since the two calendering processes were applied to similar handsheet paper samples using the same nip pressure and nip dwell time, the average differences in thickness and density do not differ significantly. However, the hard nip calendering gave slightly higher reduction in overall thickness and consequently increased densification. Inspection of small scale, local value of thickness and density reveals that the hard nip calendering shows larger differences between before and after processing.

Mean Structural	Structural Hard Nip				Soft Nip				
Parameter	Be	fore	After	Difference	e Before		After	Difference	
Grammage (g/m <sup>2</sup> ) Thickness (µm) Density	95.1 99 961	2 (5.0) (7) (37)	- 87 (3) 1067 (46)	- 12 (4) 136 (53)	95.3 103 929	8 (4.8) (7) (42)	- 93 (5) 1030 (42)	- 10 (3) 101 (33)	

**Table 5** The changes in the mean structural properties on calendering. The differ-ence in thickness and density were determined by point wise comparison of each localdata. The table includes mean values with standard deviations in parentheses.

This indicates that the more selective densification has occurred by the hard nip calendering process, already discussed above.

The uniformity of local paper structure, inferred by the coefficient of variation, CV, showed the difference between the two calendering processes. The variation of local thickness was greatly decreased by the hard nip calendering, which resulted in the non uniform local densification and a small increase in the CV of local apparent density. However, the soft nip calendering showed the decrease in the CV of local apparent density, which came from rather uniform local densification and the increase in the average of apparent density.

#### Summary

The systematic methods for evaluating the basic structural parameters for paper, i.e. grammage, thickness, apparent density and surface roughness were presented. Specifically, the combining of the local thickness map obtained using the twin laser profilometer (TLP) instrument with the local grammage map by a storage phosphor  $\beta$ -radiography provided the local apparent density map with 200  $\mu$ m pixel size. Therefore, it is possible to evaluate the local variation of the structural parameters by using this method in order to characterize changes at the scale of fiber flocs.

The calendering process significantly affected the paper structure and the relationship between local thickness and local grammage. The hard nip calendering resulted in the selective reduction in local thickness especially in areas that were thicker. This acted to slightly increase the variation in local density after hard nip calendering. The local density had a linear relationship with the local grammage which appears to approximate a bivariate Gaussian distribution. Grammage and local thickness appeared independent of one another. In the case of the soft calendering, the reduction in local thickness

took place uniformly over the sample area, which led to less variation in local density. The relationship between the local thickness and the local grammage remained the same before and after the soft nip calendering.

## HYGROEXPANSIVITY OF COMMERCIAL PRINTING PAPERS

Paper is a hygroscopic material that will respond to exposure to liquid or vapor-phase water. This may result in large-scale paper sheet distortions such as curl, waviness and cockle [52, 53] and small-scale paper distortion such as fiber rising [54, 55] that contribute to the paper surface roughening. In pigmented coating and offset printing, water is introduced to the paper surface. This results in a pronounced roughening of the paper surface that may adversely affect the uniformity of print quality and coating color distribution [55–60]. The sorption of water that may result in dimensional changes is referred to as hygroexpansivity [53]. This may cause several end use problems, such as misregistration in multicolor printing or stack lean of form papers. It is also recognized as a problem in inkjet printing. Mao and coworkers [61] used optical scanning interferometry to examine the rewetting of commercial printing paper, with water or inkjet ink. Steffner *et al.* investigated the effects of rewetting of wood free calendered samples, and compared the Z-hygroexpansion using a hard and soft nip [62].

Several investigators have dealt with the measurements of two- and threedimensional strains of paper and paper products under varying moisture conditions. Salmén and Fellers [59] considered hygroexpansion as volume expansion, and reported that for sheets dried under restraint, an irreversible expansion occurred resulting in an increase in the thickness. Uesaka *et al.* [60] determined the in-plane hygroexpansivity of paper under controlled cyclic humidity conditions. They reported that for handsheets dried under restraint, the relationship between hygroexpansion and moisture content was approximately linear and reversible in the lower moisture range, but nonlinear and irreversible after exposure to a relative humidity greater than 65%. Many studies have investigated the principal mechanisms causing paper roughening [55, 63, 64]. They found that irreversible fiber swelling, fiber debonding, release of internal stresses, and non-uniform water distribution were the principal causes.

#### Experimental

#### Materials

The five commercial papers used in this study are identified as Newsprint, SCA, SCA Uncalendered, Copy A and HB Offset as described in Table 1. Eight square specimens, with sides of 70 mm, were cut for each paper sample. One specimen was used for formation and thickness testing. The remaining specimens were used to monitor the moisture content of the sample during the experiment.

#### Methods

Grammage maps for each sample were determined using transmission radiography. Samples were affixed at the top to the TLP sample frame and restrained from out-of-plane deformation along the sides and bottom. Since the sample frame holds only four samples, the SCA-Uncalendered was tested separately from the other four.

Humidity was controlled in the environmental chamber of the TLP instrument using saturated salt baths. Samples were equilibrated for at least one day before thickness testing was begun. The remaining specimens for each sample were held proximate to the imaged sample in the test chamber and were later used for the determination of moisture contents. Air was slowly recirculated only during the equilibration time prior to testing. Initially the samples were conditioned at 51% RH and  $23^{\circ}\pm 2^{\circ}$ C. The thickness map for each sample was then acquired. Once all maps were obtained, the salt solution was exchanged, and the samples were permitted to equilibrate for approximately two days at the new relative humidity. The humidity was cycled up from RH of 51% (Ca(NO<sub>3</sub>)<sub>2</sub>.4H<sub>2</sub>O), 74% (NaClO<sub>3</sub>) and 84% (KBr), then reduced to 51%, 29% (NaCl) and 9% (H<sub>3</sub>PO<sub>4</sub>.<sup>1</sup>/<sub>2</sub>H<sub>2</sub>O).

Thickness maps were obtained using the TLP instrument by raster scanning a square region with 12.5 mm sides located in the center of the 50 mm  $\times$  50 mm exposed area. Samples were scanned at a 25 µm interval so that thickness was measured at 250,000 locations within the region. The scanning time for each sample was approximately four to five days at each humidity level. At the end of each thickness test, a hanging test specimen removed for the gravimetric determination of sample moisture content. The higher resolution thickness maps at 25 µm spacing were dilated to 100 µm elements by local averaging of the maps. This allowed a point wise registration of the formation images and thickness maps to generate apparent density maps. Coarse alignment of the images was performed visually by using the registration hole. Final alignment was adjusted based on the two-dimensional correlation coefficient between two images.

## Analysis

The thickness maps were aligned using the square holes as references. Inspection of the maps that were generated by taking the difference between equilibrium states, e.g. 51% and 84% RH, suggested small scale misalignment of the maps. This originated from in-plane hygroexpansivity of the samples that is expected to occur with the change in humidity. Figure 15 shows the effect that in-plane displacement has on the difference map. The thickness maps for 51% RH and 84% RH are shown with the uncorrected difference map across the top of that figure. A pattern of small light features with dark features of similar shape immediately adjacent may be seen across the top and bottom of the image. While these could be mistaken as non-uniformities in the z-hygroexpansion, they actually result from localized in-plane strain. In order to accurately map changes in thickness, it was necessary to account for this artifact by quantifying and then correcting for in-plane displacements.



Figure 15 Method for determining local density maps (top row). The correction algorithm for in-plane hygroexpansivity is shown in the second row. The second map is analyzed using EDIC to obtain an in-plane displacement field. A corrected region is determined and the difference map is calculated.

#### In-plane displacement characterization and correction

A method known as the Enhanced Digital Image Correlation (EDIC) technique developed by Kwon [65] was used to quantify the local in-plane displacement that occurred between the humidity intervals of 51% to 84%RH and 84% to 51%RH. The EDIC technique is an improvement of the Digital Image Correlation (DIC) technique [66–71] that utilizes a low frequency tracking (LFT) algorithm, fast normalized cross-correlation (FCC) algorithm and the concept of phase correlation. This provided increased tolerance of intensity changes and low contrast in the data array.

The in-plane displacement maps were determined at several sampling resolutions. The ideal resolution was found to be 10000 elements, since features could be tracked with minimal error and the between point interpolation was short. A 0.75 mm margin was required to perform the technique. Figure 16 illustrates the in-plane displacement fields for the uncalendered SC-A and SC-A papers for the two humidity intervals. Arrows indicate the direction and relative intensity of displacement.

The spatial correction for in-plane displacement was conducted based on the displacement fields determined by the EDIC technique. An estimated displacement map for all points in a region of interest in the reference image was obtained by the application of the cubic interpolation algorithm to the displacement field. To minimize the estimation error by the interpolation algorithm, the number of tracking points was maximized. The thickness values in the deformed image were located by the estimated displacement map to generate a corrected thickness map from the deformed image. An example of the correction algorithm used to obtain thickness difference maps from properly registered thickness maps is shown in Figure 15. The displacement fields were determined with the EDIC technique by using the initial state as the reference image. A slightly smaller, corrected thickness map was determined. The region of interest was subtracted from the corresponding coordinates in the initial map to generate the thickness difference map. The properly registered difference map may be compared with the unregistered map in Figure 15. The presence of misregistration artifacts has been greatly reduced, although not entirely eliminated. It is also interesting to note that the registered thickness difference map is relatively uniform, suggesting a low correlation between hygroexpansivity and thickness or grammage distribution. This will be discussed in greater detail below.



**Figure 16** In-plane displacement fields for the SC-A Uncalendered (left) and SC-A (right). The top row represents the transition from 51% to 84% RH. The second row represents the transition from 84% to 51% RH.

#### **Results and discussion**

Figure 17 shows the structural maps, including grammage, thickness and density for the five samples tested. The grammage maps show differences in formation that are expected for each of the grades. This includes regular patterns from wire marks as seen in the copy and newsprint samples. The thickness maps have certain large scale features that correspond to similar features in the grammage maps.

Although the density distributions of the samples were not uniform, only the SCA sample showed a close correlation between features in the density and grammage maps. This logically follows as the supercalender compresses the sheet, the highest grammage regions will be more densified. The extent to which this relationship is developed may be quantified using the distributions



Figure 17 Spatial maps of grammage, thickness, and apparent density of the samples equilibrated at 51% RH.

13th Fundamental Research Symposium, Cambridge, September 2005

of thickness and grammage plots, as was demonstrated in the previous section for hard vs. soft nip calendering. This plot is provided in Figure 18 (top row) for SC-A samples at TAPPI standard conditions. The uncalendered sample shows little correlation, with a correlation coefficient of  $R^2$ =0.393.



Figure 18 Thickness plotted as a function of grammage for the SCA papers. All points within the region of interest are plotted.

With supercalendering, the thickness decreases and essentially becomes independent of grammage.

Thickness maps were collected at the different humidity levels. The structural thickness,  $\bar{Z}_{TLP}$ , and standard deviation,  $\sigma_s$ , were determined for each map. The effects of in-plane hygroexpansion did not affect  $\bar{Z}_{TLP}$  values, although  $\sigma_s$  values were slightly reduced once the maps were properly registered. Structural thickness values from the TLP are compared with values obtained from the TAPPI hard platen caliper and from mercury buoyancy tests in Figures 19–22 for the five samples.

Figure 19 shows the results for the uncalendered SC-A and the SC-A product. In this figure, the error bars on TLP points represent  $\sigma_s$  values. They are useful in indicating the distribution of thickness about the mean value for each sample. The error bars associated with caliper points represent the repeatability of sampling according to TAPPI T-411. The TLP measures a significant expansion as humidity is increased from 51% to 74% and from



Figure 19 Measured thickness of SC-A Uncalendered and SC-A product using the TLP instrument, mercury buoyancy and hard platen caliper, plotted as a function of moisture content.



Figure 20 TLP mean roughness and Sheffield smoothness plotted as a function of sample moisture content. Samples include SC-A uncalendered, SC-A, Newsprint, Office Copy and High Bulk Offset.



Figure 21 Measured thickness of Newsprint using the TLP instrument, mercury buoyancy and hard platen caliper, plotted as a function of moisture content.



Figure 22 Measured thickness of Office Copy paper using the TLP instrument, mercury buoyancy and hard platen caliper, plotted as a function of moisture content.

74% to 84%. The uncalendered sample appears to expand with increasing humidity and contract reversibly as humidity is decreased. The results from mercury buoyancy for this sample are incrementally greater by 5–7  $\mu$ m and also show an increase in thickness with moisture content. The caliper data are significantly greater than the TLP values. However, the difference can be attributed to non-uniformity of the thickness,  $\sigma_s$ , consistent with the relationship shown in Figure 6.

The TLP, caliper and mercury buoyancy results were in close agreement for the SC-A sample at the initial 51% RH as shown in Figure 19. With increased humidity, the SC-A paper showed a lower hygroscopicity as compared to the uncalendered sample; although the hygroexpansivity it exhibited was significantly larger. The mercury buoyancy method detected a similar hygroexpansion of the sample. The caliper showed no increase in thickness with moisture. This may have resulted from the normal force applied to the structure during the test acting in opposition to any expansion that occurs. In addition to a hysteresis response in the humidity cycle, an irreversible expansion of about 10  $\mu$ m was observed using the TLP instrument. The values for thickness, as well as the irreversible expansion of the structure agree well with the results of Forseth and Helle [72] who also studied the Z-hygroexpansion of SC-A. In their study they used scanning electron microscopy and image analysis of sectioned samples exposed to various conditions of hydration. They observed that an SC-A paper, with initial thickness of 43  $\mu$ m, swelled irreversible to 51  $\mu$ m after cycling to 95%RH for 8hr and back to 51%RH. They demonstrated that the irreversible hygroexpansion resulted from fibers recovering their original shape, based on analysis of lumen cross sectional area in section images.

The standard deviation of the thickness increased 20% as humidity was increased from 51% to 84%RH. This is illustrated more clearly in Figure 20 where small changes in the topographical roughness and the Sheffield air leak smoothness can be seen for the SC-A, SC-A uncalendered, and other samples. Figure 18 shows a more comprehensive representation of the distribution of the thickness and grammage of the two SC-A samples, and the nature of the changes they undergo with increased humidity. The uncalendered



Figure 23 Measured thickness of High Bulk Offset using the TLP instrument, mercury buoyancy and hard platen caliper, plotted as a function of moisture content.

SC-A sample has a low correlation between thickness and grammage. It is not surprising that, other than a change in the center of the distribution reflecting the change in mean thickness, no other changes in the distribution are evident.

The SC-A also undergoes a change in the mean thickness. However, the fact that the distribution did not change more significantly with exposure to humidity by becoming more dispersed about the principal axis was unexpected. It was hypothesized that the high grammage/high density regions would swell preferentially, returning the calendered sample to a state that approached the original non-uniformity in thickness. Some roughening occurred although the thickness change was distributed rather uniformly within the sample.

Figure 21–Figure 23 show the measured thickness as a function of moisture content for the newsprint, copy and high bulk offset samples, respectively. The three show some hysteresis, in the humidity cycle. Only the newsprint showed evidence of significant irreversibility. In all cases, mercury buoyancy gave structural thickness results that were slightly higher than the TLP values and showed a similar increase with moisture. Figure 24 shows the



Figure 24 Thickness plotted as a function of grammage for the Newsprint, Office Copy and High Bulk Offset papers.

13th Fundamental Research Symposium, Cambridge, September 2005

thickness vs. grammage distributions for the three samples. The copy and newsprint have lower slopes than the high bulk offset. This results from the lower density of the high bulk offset, and less rigorous calendering.

The comparison of the TLP roughness and Sheffield smoothness in Figure 20 shows that the ranking of the samples is consistent between the two systems, except for the reversal of SC-A uncalendered and copy which have close to the same values. The roughness values determined from the TLP were calculated as the root mean square roughness,  $R_a$ . The roughening of the surface after hydration can be seen using both methods. The TLP has limited sensitivity of roughness due to the spacing selected for mapping the surface, i.e. 25 µm. However, the roughening of the calendered samples is evident. As the Sheffield smoothness tester measures air passage under a ring held in contact with the paper under load, the rougher samples seemed to increase smoothness slightly with increased humidity. This may be due to increased deformability of the surface features which enables the air passages to be closed off.

#### Summary

The focus of this study was to characterize the fine scale (~100  $\mu$ m) nonuniformity of thickness of newsprint, SCA (calendered and uncalendered), office copy, and bulk offset samples and the changes they exhibit with variation of equilibrium conditioning humidity. Thickness and roughness results obtained from the twin laser profilometer were compared with conventional TAPPI caliper and Sheffield air leak smoothness results, respectively. Differential thickness maps were used to compare the in-plane non-uniformity of hygroexpansion. Correction for in-plane hygroexpansion was applied in order to improve the point to point registration of the thickness maps for a more accurate comparison.

The results suggest that a significant irreversible increase in thickness occurs when calendered paper moisture content is increased. The TLP detected an irreversible swelling of the SCA calendered sample and to a lesser extent the newsprint sample when exposed to increased relative humidity. The uncalendered sample showed lower hygroexpansivity than the SC-A product. The caliper detected only small changes in thickness with sample moisture content. This may be attributed to the contacting nature of the caliper and changes in the sample compressibility with moisture content. While local thickness showed spatial correlation with paper formation and to a limited extent the local apparent density, the maps of the Z-directional hygroexpansivity showed little correlation with thickness, formation or apparent density.

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## **Transcription of Discussion**

# APPLICATIONS OF THICKNESS AND APPARENT DENSITY MAPPING BY LASER PROFILOMETRY

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Hannu Paulapuro Helsinki University of Technology

Nice work, Steven. Have you tried to correlate the profilometer data or the local density data to print unevenness?

#### Steven Keller

Yes, that is an area which we are actively working on right now, coupling with the group over at Rochester Institute of Technology. We will focus on those distinct features that I pointed out in the talk. While we as paper scientists always like to look through the paper and see the features or defects and how wonderful they look visually comparing one paper with the next, we really need to quantify it. Visual inspection, even of thickness or apparent density maps, should only be used to identify where a specific defect is occurring and then try to look at multiple regions and compare printing defects to see if you can recreate a particular defect.

#### Discussion

### William Sampson University of Manchester

Steve, you showed that there was no real correlation between local density and local grammage for handsheets before you calendered them. This is what we have seen before, but I have not yet seen the data for machine-made papers, where you press at higher wet-pressing pressures. Do you see, before calendering, distributions of local density which are correlated with local grammage?

#### Steven Keller

I would expect on a paper machine that you absolutely would, because you have the possibility of preferential compression or compaction where the rigidity of the roll or the fabrics is forcing down on the structure. We know this because we see the imprint of the fabric into the structure itself. So, you know that there is a likelihood of preferential densification and, because you are pushing on only one side, I would anticipate the distribution would show some correlation with grammage.

The next question would be "at what scale"? It may be that, at the finer scales, you see no correlation. So, this is taking all sizes into consideration, or at least all sizes within the specimen range.

## Wolfgang Bauer Graz University of Technology

With the digital image correlation you use you get z-directional information for the displacement when moisturizing the paper, but at the same time you are also going to have some displacement in the x- and y-direction due to hygroexpansivity. So which one do you use – just one or both?

#### Steven Keller

The first image used is the thickness image before deformation and the second image is the thickness image after. The features on those two images are used for the digital image correlation, so the thickness will be used to essentially find marker features and the movement of these features. So when you calculate the displacement of each piece, then what you are doing is moving those back to coincide with the place they originally were, using the low frequency filter, which eliminates the finest scales where the displacement would adversely impact the accuracy of results directly. So you are having displacement, but the filter is eliminating those small features first, you then are using the larger features in order to track local displacement. If you do not perform that low frequency filtration, then you end up with a much more scattered and random noise, which I believe is attributed to a combination between the in-plane change and also possibly the thickness value itself.

## Tetsu Uesaka Mid Sweden University

I am interested in your ZD hygroexpansion particularly the variation within the plane. What is the typical length scale of the variation of ZD hygroexpansion?

### Steven Keller

It is very much dependant on the paper sample you are looking at and also in the humidity differential range that you are looking at as well. As you are increasing or decreasing moisture content you see differences, but the average value varied substantially. For each differential there is a hygroexpansion curve as well and a correction that goes along with that.

## Stephen l'Anson

So, do you imagine there ever being an instrument available to others, perhaps not for paper mills, but for other research laboratories doing this?

## Steven Keller

I can think of some great ways of improving on this, given the money available at some of the major institutes sitting in front of me right now.

## Stephen I'Anson

So you would be interested in getting involved with producing it?

## Steven Keller

No, it would be fun for a while but there are ways of doing it faster. I do believe that truly looking at the structure and taking into consideration the intrinsic values is critical for developing relationships between mechanical or optical properties and apparent density.