# 6 Microscopic study of sheet densification by wet pressing

## 6.1 Introduction

Fibre bonded area and sheet density are strongly affected by wet pressing. A better understanding of the mechanisms of the changes of paper structure in wet pressing is of critical importance for the understanding of the fibre bonding and sheet densification. This information is also important for network modelling. In a sense wet pressing will bring fibres closer together and thus promote bonding. Wet pressing will also compress the fibres in the network and thus increase the degree of fibre collapse. However, how much these changes contribute to the sheet densification is still not clear.

Paulapuro (Paulapuro 2001) recently established that the observed effects of wet pressing on paper properties are related to changes of sheet density, the z-direction density distribution created by the pressing and surface evenness (topography). Szikla and Paulapuro (Szikla 1986; Szikla 1989) found that increasing fibre bonding is the dominant mechanism in the densifying effect of wet pressing (Szikla 1989). Gorres et al (Gorres 1993) believed that fibre collapse and deflection of free fibre segments are the predominant mechanisms at low pressing pressure, while at high pressure all spaces within the sheet which can be filled by such deflections have been filled, and therefore completion of fibre collapse and perhaps other effects then predominate. From these studies, it appears that the mechanisms in the densification effect of wet pressing on paper structure are still not completely understood.

Previous studies of fibre cross-sections measured the transverse dimensions of individual pulp fibres deposited on glass slides or embedded individually in resin (Kibblewhite 1991; Gorres 1993; Jang 1998). Gorres et al pressed very thin fibre networks (less than 0.1g/m<sup>2</sup>) on glass slides and used a stylus profilometer to evaluate the transverse dimensions of fibres in the networks (Gorres 1993). However, no quantitative studies have been reported on the behaviour of fibre cross-sections in "true" paper in wet pressing, primarily because before the work of this thesis there was no technique to measure fibre cross-sectional dimensions directly in a sheet.

The collapse degree of fibres has been characterized in different ways, such as by the fibre aspect ratio (Kibblewhite 1991) and the fibre collapse index, which is defined either as the reduction of fibre thickness (Gorres 1993) or the reduction of fibre lumen area (Jang 1995), compared to an un-collapsed fibre. To calculate the fibre collapse index, the original fibre shape must be assumed to be either circular or rectangular in shape (Gorres 1993; Jang 1995). The collapse index cannot be measured directly and it only describes the shape of the fibre lumen area, completely neglecting the shape of fibre wall area.

One factor that has not been considered in studies of pressing is the twisting of the fibres so that their cross-sectional major axis rotates to be closer to parallel to the paper plane. The orientation of the fibre with respect to the paper plane could strongly affect the structure of the paper. Decreasing the number of twists in fibres will increase sheet strength (Mohlin, Dahlbom et al. 1996).

In this chapter, a fibre shape factor and a twist angle of the fibre cross-section are defined. These parameters are measured directly in handsheets using the cross-section preparation and analysis technique describe in Chapter 4. The results are used to quantify the changes of fibres in the handsheets in wet pressing. The mechanisms behind densification effects of wet pressing on paper structure are discussed.

# 6.2 Theory

#### 6.2.1 Degree of fibre collapse in paper

For the purpose of quantifying the degree of collapse of fibres in sheets we defined the new term of shape factor,  $f_m$ , as the ratio of the fibre wall area,  $A_f$ , to the area of the smallest rectangular bounding box,  $A_s$ , that can completely enclose the irregular shape of the fibre. (Refer to subsection 4.4.5). In this section, we will examine the relationship between the shape factor and the degree of collapse of the fibre. To do this we begin by assuming that the fibre cross section before wet pressing is circular with diameter, D, and wall thickness, t, and that the fibre wall area,  $A_f$ , is constant with

wet pressing. We assume that if the thickness decreases by a distance  $\delta_D$ , then the width will increase by the same amount (see Figure 6-1). Accordingly,  $f_m$  is:

$$f_m = \frac{A_f}{A_s} = \frac{A_f}{(D - 2\delta_D)(D + 2\delta_D)} = \frac{\pi t (D - t)}{D^2 - 4\delta_D^2}$$
 6.1

Equation 6.1 can also be written as:

$$f_m = \frac{\pi^2 (t/P)(1-t/P)}{1-(\delta_D/r)^2}$$
6.2

where P is the fibre perimeter, and r is the fibre radius.

Following Jang (Jang 2001), the collapse index, CI, is defined as  $CI = (\delta_D / r)^2$ . For a thin walled fibre,  $t \ll P$ , and  $(1 - t / P) \approx 1$ , and Equation 6.2 becomes:

$$f_m = \frac{\pi^2(t/P)}{1 - CI}$$
 6.3

Obviously, uncollapsed fibres and fully collapsed fibres will have the minimum and the maximum values of  $f_m$  respectively. These maximum and minimum values of  $f_m$  can be used to create an approximate scale to separate fibres in a sheet into three collapse-states, viz. uncollapsed fibres, partially collapsed fibres and fully collapsed fibres (Figure 6-2). The validity of using the boundary values of  $f_m$  in describing the collapse-state of a fibre will be verified in this study.



Figure 6-1 Model fibre cross-section



Figure 6-2 Different collapse states of fibre: uncollapsed fibres (A, B), partially collapsed fibres(C, D), and fully collapsed fibres (E, F).

#### 6.2.2 Reduction in paper thickness by fibre twist and fibre collapse

Assuming the paper has layered structure, fibres in each layer are twisted and collapsed in wet pressing, reducing the thickness of each layer. The total reduction in the paper thickness is the sum of the thickness reduction of each layer. Figure 6-3 shows schematically the *i*<sup>th</sup> fibre in a layer of a sheet before and after it is pressed. Before the fibre is pressed, the fibre has width,  $D_{wi}$ , thickness,  $D_{hi}$ , and twist angle,  $\beta_i$ . After it is pressed, the width, thickness and twist angle are  $D'_{wi}$ ,  $D'_{hi}$  and  $\beta'_i$  respectively. If the paper has *m* independent layers and each layer has *n* fibres, the total reductions in paper thickness from the fibre twist,  $\overline{\Delta T}_{twist}$ , and from fibre collapse,  $\overline{\Delta T}_{collapse}$ , are given by Equations 6.4 and 6.5 respectively:

$$\overline{\Delta T}_{twist} = \frac{m}{n} \sum_{i=1}^{n} \left[ D_{wi} \sin \beta_i - D'_{wi} \sin \beta'_i \right]$$
6.4

$$\overline{\Delta T}_{collapse} = \frac{m}{n} \sum_{i=1}^{n} \left[ \frac{D_{hi}}{\cos \beta_i} - \frac{D'_{hi}}{\cos \beta'_i} \right]$$
6.5



Figure 6-3 Schematic showing the reduction in paper thickness contributed by fibre twist and fibre collapse in any layer of a sheet

## 6.3 Sample used in this study

The low kappa pulp was used in this study (refer to Appendix A). This is the pulp labelled L<sub>0</sub> in subsection 5.2.3. Five sets of  $60g/m^2$  square handsheets were made on the Moving Belt Sheet Former. Each set of handsheets was pressed statically at one of 5 pressures, viz 100kPa, 200kPa, 500kPa, 2000kPa and 4000kPa (for details refer to subsection 5.2.1). Cross-sections of handsheets pressed at 100kPa, 500kPa and 4000kPa, which are denoted as L<sub>0</sub>P<sub>1</sub> L<sub>0</sub>P<sub>3</sub>, and L<sub>0</sub>P<sub>5</sub> respectively, were examined in a confocal microscope. The method for sample preparation for the confocal microscope has been given in subsection 4.3.2. The methods for measuring the shape factor,  $f_m$ , and the twist angle,  $\beta$ , have been given in Subsection 4.4.1.5.

#### 6.4 **Results and discussion**

#### 6.4.1 Changes in paper cross-section

Figure 6-4 shows typical cross-sectional images of the three samples  $L_0P_1$ ,  $L_0P_3$  and  $L_0P_5$ . The sample thickness and the void space are reduced with increasing pressing pressure. In sample  $L_0P_1$ , it can be seen that most fibres are either uncollapsed or partially collapsed, but there are also some fully collapsed fibres. In sample  $L_0P_3$ , the number of uncollapsed fibres is dramatically reduced, and around half the fibres are fully collapsed. In sample  $L_0P_5$ , most of the fibres have been fully collapsed, but a few uncollapsed fibres still can be seen.

Figure 6-4 also shows that fibres in the sheet have been rearranged in wet pressing. Some of the fibres are pressed into gaps or void space. The type of movement is called gap closure in this project.

In summary three types of movement of fibres in wet pressing have been observed in this study including collapse of fibre, twist of the fibre and gap closure. These three types of movements, which control the change in paper thickness, have been quantified in this study and the results are discussed in the following paragraphs.



\* The size bar is 10µm

Figure 6-4 Images of cross-sections of sample  $L_0P_1$  (A),  $L_0P_3$  (B) and  $L_0P_5$  (C)Table 6-1 summarizes the fibre dimensions and the sheet properties measured in this work. The fibre wall area was not changed by wet pressing and the mean value of  $f_m$  increases with increasing wet pressing pressure. The twist angle falls from 9° to 6.8° with increasing wet pressing.

Sample	Wet press pressure (kPa)	Shape factor, $f_m$ (%)	Average twist angle	<sup>*</sup> Fibre wall area (μm <sup>2</sup> )	Out-of- plane angle (degree)	Number of fibres measured	Sheet density (kg/m <sup>3</sup> )	Paper thickness (µm)	Tensile index (kNm/kg)
$L_0P_1$	100	$0.548 \pm 0.014^{**}$	9.0±1.1	206±9**	5.13±0.61	247	286	239	34.45
/	200	/	/	/	/	/	375	172	36.98
$L_0P_3$	500	0.589±0.013	8.2±1.0	198±9	4.49±0.48	241	522	125	45.98
7	2000	/	/	/	/	/	590	109	48.04
$L_0P_5$	4000	0.603±0.011	6.8±0.8	205±9	4.64±0.52	247	596	107	49.95

 Table 6-1 Handsheet properties and mean fibre properties

\*Fibre wall area and fibre axes have been corrected by its angle to the surface of the paper cross-section.

\*\*  $\pm$  is 95% confidence interval.

Figure 6-5 shows the twist angle distribution of fibres in the three samples  $L_0P_1$ ,  $L_0P_3$ and  $L_0P_5$ . As the pressing pressure is increased, the number of fibres with twist angle less than 10° increases, while the number of fibres with twist angle more than 10° decreases. It can be seen that 67% of the fibres in sample  $L_0P_1$  have a twist angle less than 10° and this percentage is increased by wet pressing to 71% for sample  $P_3$  and to 78% for sample  $L_0P_5$ . This reduction in fibre twist will reduce the amount of space taken by the fibre in the paper structure, therefore reducing the void space and increasing the density of the paper. The reduction in fibre twist could also increase the potential bonding surface area of the fibres, especially for collapsed fibres.



Figure 6-5 Frequency distribution of twist angle of fibre cross-section of sample  $L_0P_1$ ,  $L_0P_3$  and  $L_0P_5$ 



Figure 6-6 Frequency distribution of the fibre shape factor of sample  $L_0P_1$ ,  $L_0P_3$ and  $L_0P_5$ 

The frequency distributions of  $f_m$  are shown in Figure 6-6. For sample  $L_0P_1$ ,  $f_m$  has an almost symmetrical distribution. When the pressing pressure is increased, the distribution of  $f_m$  skews to a higher  $f_m$  range and becomes narrower. Since the three samples were made from the same pulp, fibres in the three samples should have the same wall thickness and perimeter. In that case, according to Equation 6.3, the value of  $f_m$  should be mainly affected by the degree of collapse of the fibres. The trends in  $f_m$ are therefore most likely indicative of fibre collapse.

As discussed in subsection 6.2.1, the maximum and the minimum values of  $f_m$  can be used to assign the fibres as collapsed, partially collapsed or uncollapsed. The boundary values of  $f_m$  used to do this were set somewhat arbitrarily. Fibres with  $f_m$  less than 0.50 were treated as uncollapsed fibres, fibres with  $f_m$  greater than 0.60 were treated as fully collapsed fibres and fibres with  $f_m$  in between 0.50 and 0.60 were treated as partially collapsed fibres. Using this classification 35% of fibres in the slightly pressed sample P<sub>1</sub> were uncollapsed and 33% were fully collapsed (Figure 6-6). After the sample was pressed at 500kPa, the percentage of uncollapsed fibres dropped to 24% and the percentage of fully collapsed fibres rose to 49%. After the sample was heavily pressed, the percentage of fully collapsed fibres increased to 54% and the percentage of uncollapsed fibres fell to 13%. The percentage of partially collapsed fibres stayed relatively stable at around 30% in all the three samples. These quantitative results are consistent with the observations shown in Figure 6-4, but quite different from those reported by Gorres and his coworkers (Gorres 1993) who found fibres of a softwood kraft pulp can be totally collapsed at a pressing pressure of 2240kPa. This indicates that the collapse behaviour of fibres on glass slides and in paper is different.

There are two major factors that may cause the observed non-uniform collapse of fibres. Firstly, fibre collapsibility will vary between fibres and at different points along fibres. Secondly, and perhaps more importantly, the non-uniform structure of paper causes an uneven pressure transfer in the network that results in the non-uniform collapse of fibre.

# 6.4.2 Relationship between apparent density and the changes in fibre cross-section

The apparent density of the handsheets increased from 286 kg/m<sup>3</sup> to 522 kg/m<sup>3</sup> as the pressure increased from 100 to 500 kPa. However, pressing at 4,000 kPa increased the sheet density only slightly, to 596 kg/m<sup>3</sup>, compared to pressing at 500kPa (Table 6-1). Gorres et al (Gorres 1993) found a similar relationship between density of paper and the wet pressing pressure. They failed to explain the continuing increase in density at high pressure where they believed that fibres are totally collapsed. One reason is that they used the measured degree of collapse of fibres pressed on glass slides to represent the degree of collapse of fibres in paper, which as discussed before is a different situation to collapse in the pressing of paper. Another reason is that they did not take account of the contribution of fibre twist to the density of paper.



Figure 6-7 Frequency distribution of out-of-plane angle of sample  $L_0P_1$ ,  $L_0P_3$  and  $L_0P_5$ 

Gorres and Luner developed a model for the apparent sheet density (Gorres and Luner 1992). The model assumed that increasing fibre deflection with increasing wet pressing pressure is a major mechanism in the densification of the paper. However, no evidence was given to prove this relationship. In this study, the out-of-plane deflection angles of fibre segments in samples  $L_0P_1$ ,  $L_0P_3$  and  $L_0P_5$  were measured. If the sample is not arranged exactly vertically, an error will arise for the measured value of the out-of-plane A correction, similar to the correction for the twist angle deflection angle. measurements, has been done for each individual fibre to avoid this error. The results (Table 6-1) show that the average out-of-plane deflection angle of fibre segments in the three samples ranges from 4.49° to 5.13°, and no regular increase with wet pressing pressure can be observed. The distribution, shown in Figure 6-7, of out-of-plane defection angles, also shows no regular trends with pressing level. If the out-of-plane angle of the fibre segment stays constant, the deflecting distance of the fibre segment will be reduced as the pressing pressure is increased. This is because wet pressing will reduce the distance between fibre-fibre contacts.

To quantify, using Equations 6.4 and 6.5, the contributions of fibre twist, fibre collapse and gap closure to the measured reduction in paper thickness we assumed that the samples have 10 layers and the number of layer stays constant as the pressing pressure is changed. These data were used to calculate the  $\overline{\Delta T}_{twist}$  and  $\overline{\Delta T}_{collapse}$  between samples L<sub>0</sub>P<sub>3</sub> and L<sub>0</sub>P<sub>1</sub> and between samples L<sub>0</sub>P<sub>5</sub> and L<sub>0</sub>P<sub>3</sub>, and the results are given in Table 6-2. The measured total thickness reduction between samples L<sub>0</sub>P<sub>3</sub> and L<sub>0</sub>P<sub>1</sub> is 114µm, of which the contributions from the fibre twist and fibre collapse only accounts for 16%. We believe that the rest of the thickness reduction is caused by closing the gap between the layers. The total reduction in paper thickness between samples L<sub>0</sub>P<sub>5</sub> and L<sub>0</sub>P<sub>3</sub> is 18µm which is mostly contributed by fibre twist and fibre collapse. These findings clearly show that fibre twist, fibre collapse and gap closure occur simultaneously at low pressing pressures and the gap closure is the predominant mechanism in paper structure densification at low pressures. When the pressing pressure was increased from 500 to 4000kPa, the density increased only slightly. This density increase was mainly due to the additional twist and collapse of the fibres at the high pressure.

Table 6-2 Reduction in paper thickness

	$L_0P_3 - L_0P_1$	$L_0P_5 - L_0P_3$
$\overline{\Delta T}_{twist}$ (µm)	7	3
$\overline{\Delta T}_{\textit{collapse}}$ (µm)	11	10
** $\overline{\Delta T}_{gapclosure}$ (µm)	96	5
*** $\overline{\Delta T}_{measured}$ (µm)	114	18

\*  $L_0P_3$ -  $L_0P_1$  and  $L_0P_5$ -  $L_0P_3$  represents thickness reduction between those samples.

\*\*  $\overline{\Delta T}_{gapclosure} = \overline{\Delta T}_{measured} - \overline{\Delta T}_{twist} - \overline{\Delta T}_{collaspe}$ . \*\*\*  $\overline{\Delta T}_{measured}$  is the measured total reduction in paper thickness.

## 6.5 Conclusions

A fibre shape factor and twist angle of fibre cross-section in paper have been defined for the purpose of quantifying the changes in the transverse dimensions of fibres in paper in wet pressing. It was found that fibre twist, fibre collapse and gap closure are the major types of movement of fibres in paper in wet pressing. In particular, fibre twist has been found and quantified for the first time. The results show that the number of fibres with twist angles greater than  $10^{\circ}$  is reduced by wet pressing and that the average twist angle decreases as the pressing pressure was increased. The fibre shape factor of fibres in the lightly pressed handsheets showed an almost symmetrical distribution, which indicates that the degree of collapse of fibres in the handsheets is symmetrically distributed. The distribution was narrowed and skewed to the high value range when the handsheets were pressed at 500kPa or at 4000kPa. These results show that fibres in the handsheets cannot be totally collapsed by wet pressing even when the very high pressing pressure (4000kPa) is used. The experimental data also suggest that out of plane fibre deflection angle is independent of wet pressing pressure.

Fibre twist, fibre collapse and gap closure occur simultaneously at low pressing pressures, and the gap closure is the predominant mechanism in paper structure densification at low pressing pressures (less than 500kPa). Increasing pressure beyond 500kPa only increases the apparent density slightly and the density increase is mainly contributed by the additional twist and collapse of the fibres at the higher pressing pressure.