

# Demonstration of Strength Development in Initial Wet Paper Web using Field Emission-Scanning Electron Microscopy (FE-SEM)

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Various models exist that explain strength development in the wet web. Furthermore the scanning electron microscope (SEM) has been used in the paper industry to characterise cellulosic fibres and paper. The documentation of the initial wet web properties needs very specific requirements for sample preparation. An SEM image shows the sample's surface, so the wet sample's water film would cover all fine fibre structures. For this reason the samples must be dried prior to analysis. Freeze drying is a common method that is described to prepare samples for characterisation of single fibres before and after mechanical treatment. In this investigation the structure of the initial wet web was physically fixed by rapid freezing, followed by freeze drying. Afterwards, the samples were analyzed by Field Emission SEM (FE-SEM). The generated images support the hypothesis that fibrils partially extend themselves from the fibre and interact with adjacent fibres.

*Keywords: Dryness; Fibre collapse; Form fit; Hornification; Initial wet web strength*

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## INTRODUCTION

Initial wet web strength is one of the most important parameters to ensure effective paper machine runnability (Guldenberg *et al.* 2004; Sutman 2011; Ora and Maloney 2013). Generally, the designation “initial wet” spans a dryness level from approximately 10% during the sheet formation up to approximately 60% in the first dryer groups. Today, depending on the respective forming section construction and the utilised fibrous material, dryness levels of 18% up to a maximum of 25% at the end of the wire section are achieved (Strauß 2008). After the press section the dryness level is in the range of 40 to 55%; this also depends on press construction and the utilized fibre sources. Many different explanations and models, which describe initial wet web strength mechanisms, have been published.

Several papers mention capillary forces as the decisive factor for the initial wet web strength in the early dewatering stages of the paper web (Campbell 1933; Rance 1980; Page 1993; Persson *et al.* 2013). As the distance between two fibres becomes smaller and smaller while dewatering proceeds, the capillary forces between the fibres become greater – similar to the glass plate effect (Ek *et al.* 2009; Belle *et al.* 2014b). The fibre surface roughness significantly influences the capillary- and adhesion-forces during dewatering (Fuller and Tabor 1975; Page 1993; Kendall 2001; Alince *et al.* 2006; Feiler *et al.* 2007; van de Ven 2008; Huang *et al.* 2009); therefore, this fibre characteristic is important for the initial wet web strength development. Since recently it has become possible to measure a single

cellulose fibre's surface roughness, this can also be taken into account for the calculation of the above mentioned forces (Feiler *et al.* 2007; Heinemann *et al.* 2011). The capillary forces are explained using the two cylinder model (Wågberg and Annergren 1997; Ek *et al.* 2009). In this model the fibres are represented by two cylinders, which are orthogonally crossed with a water film in between. However, it can only be considered as a rough approximation, since fibres—especially in the wet state—are mobile, swollen, and deformable.

Electrostatic and van-der-Waals forces, as well as acid-base interactions, also have an effect on initial wet web strength (Brecht and Erfurt 1959; Kendall 2001; de Oliveira *et al.* 2008; Myllytie 2009; Lindqvist 2013; Belle *et al.* 2014a). Carboxylic- and sulphonic acid groups of fibres are very important for these models due to their impact on the surface charge of the fibre (Sjöström 1989).

Another model describes a partial dissolution of the cellulose fibre surface, so that the fibres partly penetrate each other during sheet forming (Casey 1960; McKenzie 1984; Pelton 2004; Pelton *et al.* 2000; Voyutskij 1963). This idea has been broadened, stating that the reducing end-groups of the cellulose chain start to dissolve and rise up from the rest of the fibre so that they are more easily available for linking (Clark 1978a). Through this effect, the fibres approach each other sufficiently closer during drying to form the required hydrogen bonds. This model emphasizes the high bonding ability of hemicelluloses, resulting from their large number of short molecules, which strongly interact during bonding. Wet sheet fibre bonding is explained *via* a gel-like fibre surface, which is responsible for fibre-fibre bonding during sheet dewatering and the corresponding fibre and fibril approach (Kibblewhite 1973). Research results from another group of scientist supports this theory (Wågberg and Annergren 1997).

Neuman published extensive work on cellulose surface force measurement (Neuman 1993), out of which one conclusion is the dangling tail model. This theory describes the swollen cellulose fibre surface in water as negatively charged cellulose tails, which tower above the fibre, are soft, and sensitive to applied stress. Neuman stated that this model could have a major implication for fibre-fibre bonding in respect to the inter-diffusion of cellulose chains between two fibre surfaces. In this model, another important connection between the fibres holding the paper web together is the frictional connection and/or form fit.

Alinec and coworkers described the initial wet web strength as adhesion among fibres (Alinec *et al.* 2006). This is defined as the friction force between fibres with tensile stress. Persson combines the partly dissolved surface model with his ideas of fibre's plastic flow during dewatering as a result of his trials (Persson *et al.* 2013).

According to the models above and the explained parameters, the interactions between two fibres are influenced mainly by the following characteristics:

- State of fibre swelling or hornification (Lindström 1980; Weise 1998; Laine *et al.* 2002; Linhart 2005),
- Fibre deformation, *e.g.* kinks, curls, and coarseness (Seth 1995; Odell 2001; Lindqvist 2013),
- Fibre flexibility and suppleness (Brecht and Langer 1953; Seth *et al.* 1984),
- Fibre surface tension (Lyne and Gallay 1954; Lyne and Gallay 1954a; Schwarz and Bechtel 2003; de Oliveira *et al.* 2008), and
- Fibre roughness (Alinec *et al.* 2006; van de Ven 2008; Huang *et al.* 2009).

All of these models and parameters can be summarized and addressed to different stages of strength development at certain dry contents. An early idea of these stages is explained by Brecht (1959), which was recently refined by Erhard as well as Tejado and van de Ven (both 2010) (Brecht and Erfurt 1959; Brecht and Erfurt 1959b; Erhard *et al.* 2010; Tejado and van de Ven 2010).

In the first stage, up to a dry content of about 25% +/- 5%, capillary forces are one part of the strength development with increasing dewatering (Kendall 2001). The morphology of the collapsed fibres enables their approach to each other and the generation of initial contact points. The fibre characteristic between the orthogonal and the longitudinal axis (*e.g.* coarseness) has a significant effect on the fibre collapse, also known as wet hornification (Paavilainen 1993a, b; Weise and Paulapuro 1996). A form fit and frictional connection is built up as a result of macroscopic and mechanical entanglement. At this stage, rigid and smooth fibres are most suitable to develop good capillary forces and an entanglement between fibres at higher distances (Belle *et al.* 2014b).

The second stage, between dry contents of ~25 % up to ~60%, is characterized by van-der-Waals forces of attraction and repulsion according to the DLVO theory (Derjaguin 1954; Derjaguin and Landau 1941; Pelton 1993; Wågberg and Annergren 1997; Israelachvili 2006). Contrary to the first stage, a flexible, visco-elastic and soft fibre surface is now needed for the formation of larger contact areas between the fibres (Nanko and Ohsawa 1989; Pelton 1993; Nilsson *et al.* 2000; Lindström *et al.* 2005). In this phase, the gelatinization between water and fibre is an important phenomenon for the formation of contact areas (Kibblewhite 1973; Voyutskij 1963; McKenzie 1984; Wågberg and Annergren 1997; Pelton *et al.* 2000; Pelton 2004). This property promotes the diffusion of polymer chains and polyelectrolytes from wood polysaccharides, especially from xylan (Casey 1960; Clark 1978b; McKenzie 1984; Pelton 1993), as well as a self-assembly between micro fibrils (Neuman 1993; Pönni *et al.* 2012). In this second stage, swellability of the fibres is an important parameter. A procedure for the determination of the swelling state is the measurement of the water retention value (Höpner *et al.* 1955; Zellcheming 1957; Thode *et al.* 1960). The swelling contributes to fibre flexibility, as a result receiving a considerably higher capacity to felt or interlock with each other due to higher suppleness (Brecht 1947; Lyne and Gallay 1954; Lyne and Gallay 1954a; Brecht and Erfurt 1959; Barzyk *et al.* 1997; Scallan 1983; Weise *et al.* 1998; Linhart 2005; Erhard *et al.* 2010).

There are, however, quite different models to be found in the literature explaining why paper holds together at low dryness levels, but until today, there was no possibility to show the behaviour of the fibre in the wet paper sheet. This work demonstrates the development of fibre entanglement during two different dewatering steps and one drying step using rapid freezing and freeze drying for sample preparation to conserve the structure of the pulp fibres in the wet and dry web.

## EXPERIMENTAL: MATERIAL AND METHODS

### Pulp Preparation

The procedure of pulp preparation is shown in Fig. 1. Never-dried northern bleached softwood kraft pulp (NBSK-ECF) from Zellstoff Stendal GmbH, Germany, was used. This pulp was diluted with deionized water, disintegrated and washed with deionized water until the filtrate conductivity was below 1  $\mu\text{S}/\text{cm}$ .

For Water Retention Value measurement (WRV), one part of the pulp was further dried at 20 °C and another part at 105 °C, according to Brecht and Erfurt (1959).

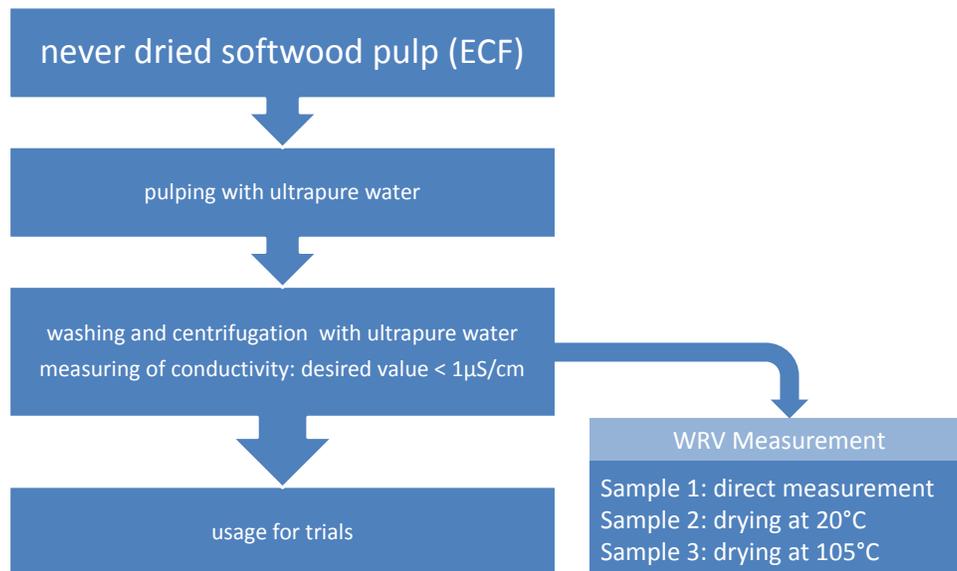


Fig 1. Procedure of pulp preparation

### Measurement of Water Retention Value (WRV)

The Water Retention Value was measured according to DIN ISO 23714.

### Sheet Preparation

Sheets were formed using a Retention and Drainage Analyzer (RDA) from Frank PTI (Lee *et al.* 2010; Ryu and Bong-Keun 2011). This special sheet forming device was chosen to enable proper and reliable sheet forming with only 1 liter of stock solution with a consistency of 0.3 %. The wire type used was according to DIN EN ISO 5269-2004. The vacuum settings are summarized in Table 1. This setup enables production-like sheet forming and avoids any washing effects, which usually occur with conventional sheet formers. The dryness after sheet forming was about 17 %.

Table 1. Vacuum Settings of Sheet Forming Device

Parameter	Value
Main Vacuum	250 mm Hg
Sub Vacuum	250 mm Hg
Suction time	5 s
Vacuum time	10 s

### Dry Content Adjustment

To adjust the different sample dryness, filter paper and a Rapid-Koethen couch roll were used to get a dryness of about 20%.

For preparing samples with 45% dry content, a laboratory roll press by Sumet-Messtechnik, Denklingen, Germany, with a load of 500 N was used. To obtain reproducible

solids content, the samples were pressed within a defined sandwich on a support plate (Fig. 1).

The completely oven dried sheets were prepared with a Rapid-Koethen drying unit from Labor Geräte Service, Mühlheim, Germany in accordance to DIN EN ISO 5269-2004.

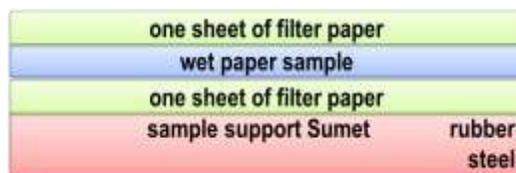


Fig. 2. Defined pressing sandwich and support plate

### Measurement of the Initial Wet Web Strength (IWWS)

The initial wet web strength measurement was conducted on the basis of the German standard “Prüfung der initialen Nassfestigkeit” (DIN 54514 2008).

For the measurement, a vertical tensile testing machine from Zwick was used. Immediately before each measurement, the test strip was weighed to determine the actual dryness of the paper for the evaluation. A special clamp with a grooved roll was designed to ensure that the strip is not wedged in directly above or below the free clamping length. The clamping force was distributed via a 90° redirection on the grooved roll, ensuring that no water was pressed in the span. The tensile strain measurement was performed with a speed of 1.5 mm/s until the strip ruptured (Schwarz and Bechtel 2003).

The results of the IWWS measurements were used to calculate the value of  $F_{index}$  (Eq. 1):

$$F_{index} = \frac{\frac{F_{max}[N]}{\text{test strip width [mm]} \times 10^3}}{\text{basis weight of test strip} [\frac{g}{m^2}]} \left[ \frac{Nm}{g} \right] \quad (1)$$

### Design of Experiments

To obtain a valid procedure for the laboratory trials, a full factorial design of experiment was developed with the software model 10.1 from Umetrics (Belle *et al.* 2014a; Eriksson *et al.* 2008).

### Sample Preparation for Electron Microscopy Measurements

Some scientists have mentioned in their work that even the critical point drying method (CPD) can lead to artifacts by drying (De Silveira *et al.* 1995; Daniel and Duchesne 1998; Duchesne and Daniel 1999). To minimize the artefacts by preparation, the samples were physically fixed by rapid freezing in a propane/pentane mixture of 3:1 at -80 °C to -100 °C (in accordance to (Fritz *et al.* 2007)). This mixture remains fluid at this temperature; also, the lack of the Leidenfrost effect (Curzon 1978) facilitates excellent cooling rates. The samples were retained in the fluid for at least 30 s and manually broken in a frozen state to get breaking edges for later observations. After temporary storage in liquid nitrogen, the samples were freeze-dried overnight.

Surface samples were mounted on stubs with carbon conductive tabs and edge samples were fixed with carbon conductive glue (Plano GmbH, Wetzlar, Germany). After coating with gold (Biorad SC510 SEM Coating Systems) the samples were examined in a

FEI field emission scanning electron microscope Quanta FEG 250 from FEI Munich, Germany.

## RESULTS

The results are divided in three parts: Firstly, the paper surface is shown to illustrate differences during paper processing including dry paper surfaces produced with unrefined pulp. Secondly, pictures of the samples' edges are shown to elaborate some more details of the paper structure in the transverse direction. Finally, the fibre entanglement formation is shown in several pictures.

Overall more than 400 images were evaluated for this investigation.

### Water Retention Value

Figure 3 shows the development of the water retention value depending on the hornification and dewatering resistance of the pulp used. The pulp used for producing the paper sheets for the SEM images was unrefined pulp at a refining level as measured by water resistance of SR 12. It can clearly be seen that at this SR, the hornification was rather low. The values of the unrefined pulp reflected only the fibre collapse. The effect of hornification during drying was larger with refined fibres. At SR 30 refining level, this pulp contained fibres and fibrils and confirmed the findings of earlier research (Szwarcstajn and Przybysz 1977).

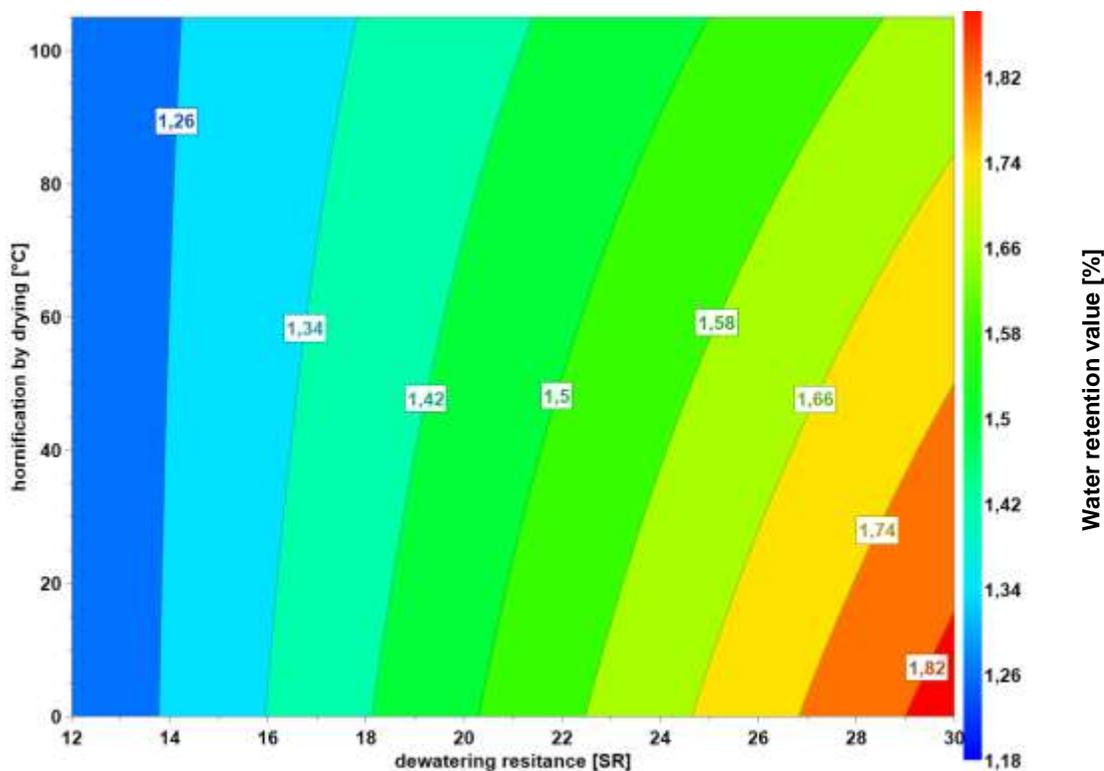
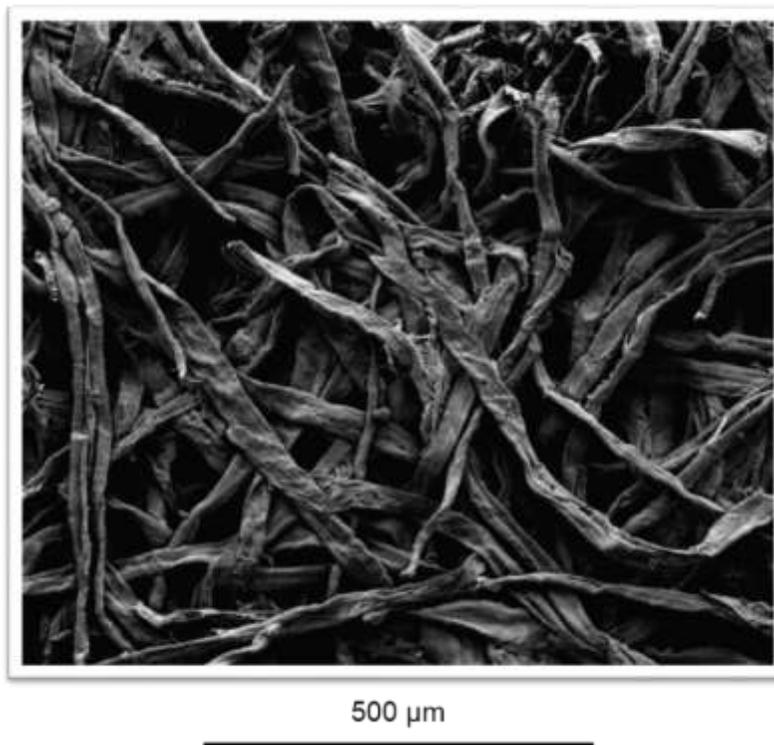


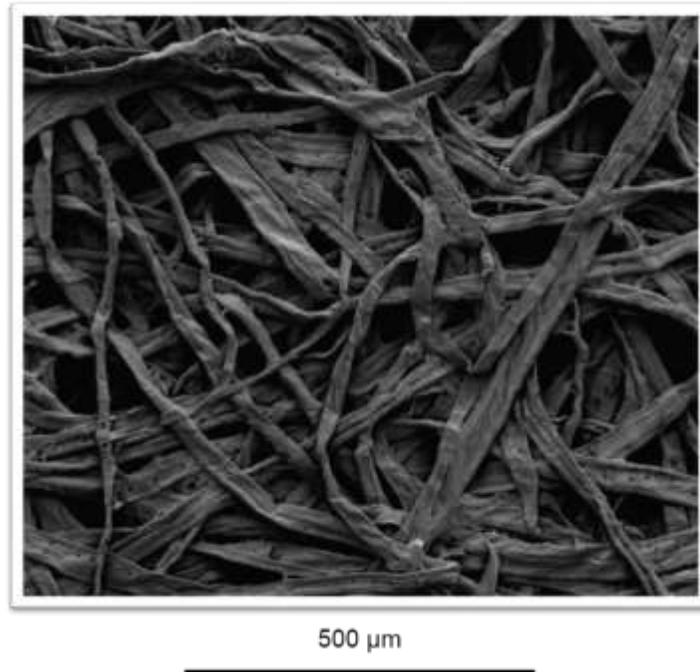
Fig. 3. Development of water retention value vs. dewatering resistance and hornification by drying

### Paper Surface

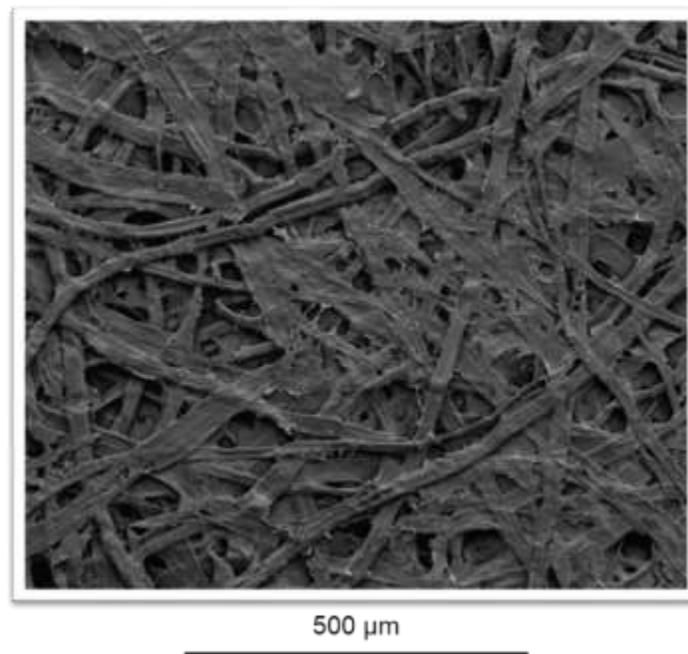
Figures 4 to 6 show the paper surface at different dryness levels at the same magnification. In Fig. 4, a paper surface freeze-dried at a solids content of 20% is shown. An open structure with spaces between the fibres is visible. Due to the huge amount of water in the sheet, the fibres had a slightly oval shape and a swollen outline (better visible in transverse fracture, Figs. 7-9). Figure 5 shows the paper freeze-dried at a dryness of 45%. The sheet structure was already more dense and the fibres flatter. This can be explained as a manifestation of wet hornification (Weise 1998; Paulapuro 2001; Fernandes Diniz *et al.* 2004). Figure 6 shows the paper surface after heat drying. The once oven-dried fibres had become completely flattened and collapsed (Scallan 1974), making the sheet as dense as possible for the unrefined pulp. Some areas were covered with film-like cellulose structures (Mou *et al.* 2013).



**Fig. 4.** Paper surface freeze-dried at 20% dryness



**Fig. 5.** Paper surface freeze-dried at 45% dryness

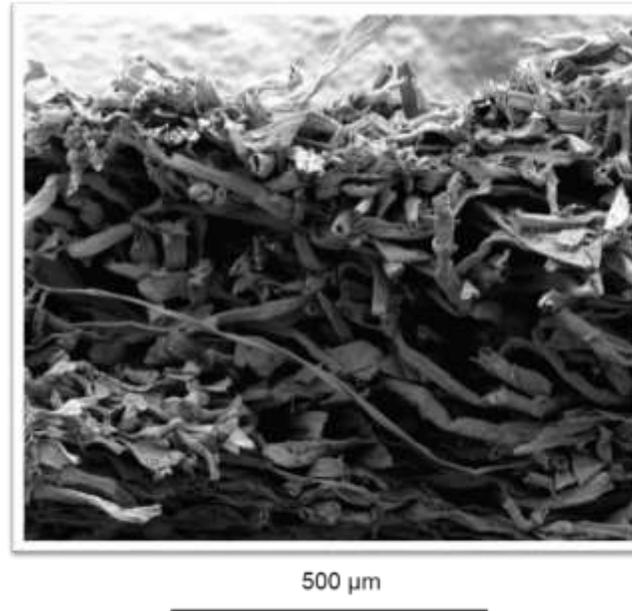


**Fig. 6.** Paper surfaces heat dried to 95% dryness

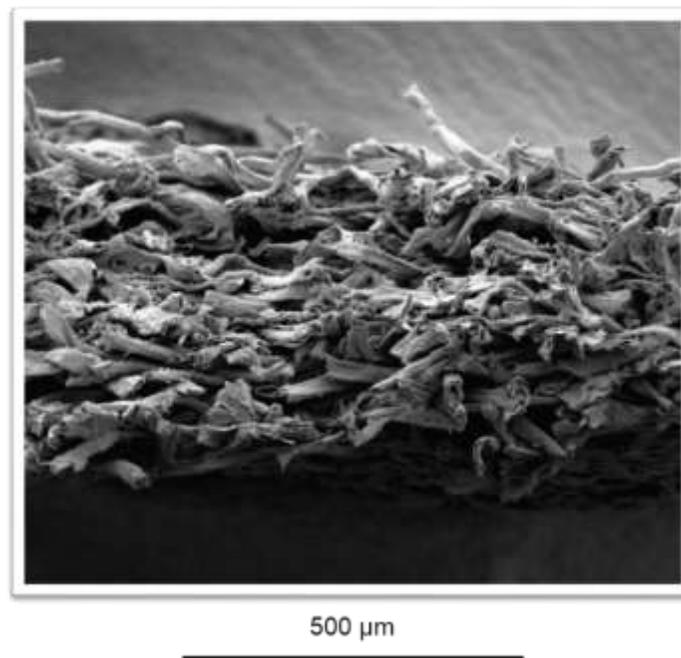
### Paper in Z-direction

Figures 7 to 9 show the papers' z-direction at different dryness levels at a constant level of magnification. The paper thickness reduction due to sheet dewatering can easily be seen. Considering the 20% dryness as 100% thickness, the sample at 45% dryness has only 70% thickness. At 95% dryness, only around 40% thickness is left due to the drying

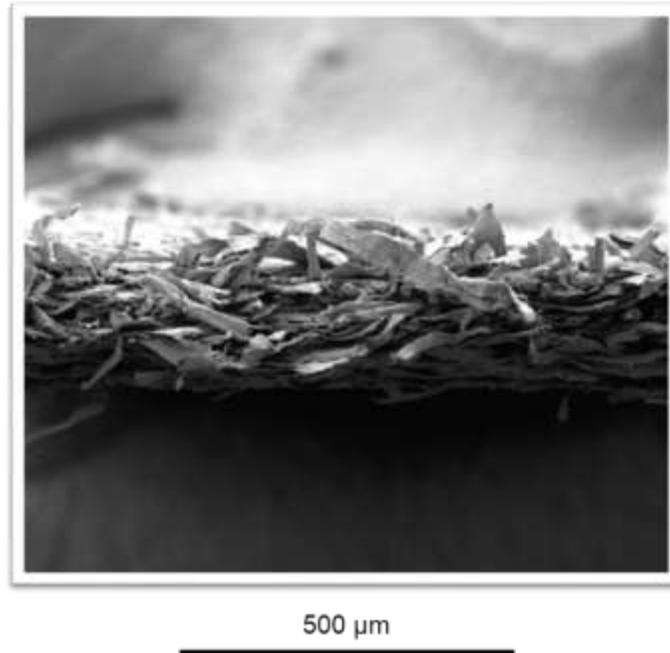
step. The consequences of the drying process are more fibre contact points. The initial wet web strength index of these papers show an increase from 0.30 Nm/g at 20% solids content to 0.88 Nm/g at 45% solids content (Fig. 10) and finally to 60.4 Nm/g at 95% solids content.



**Fig. 7.** Z-direction of paper freeze-dried at 20% dryness

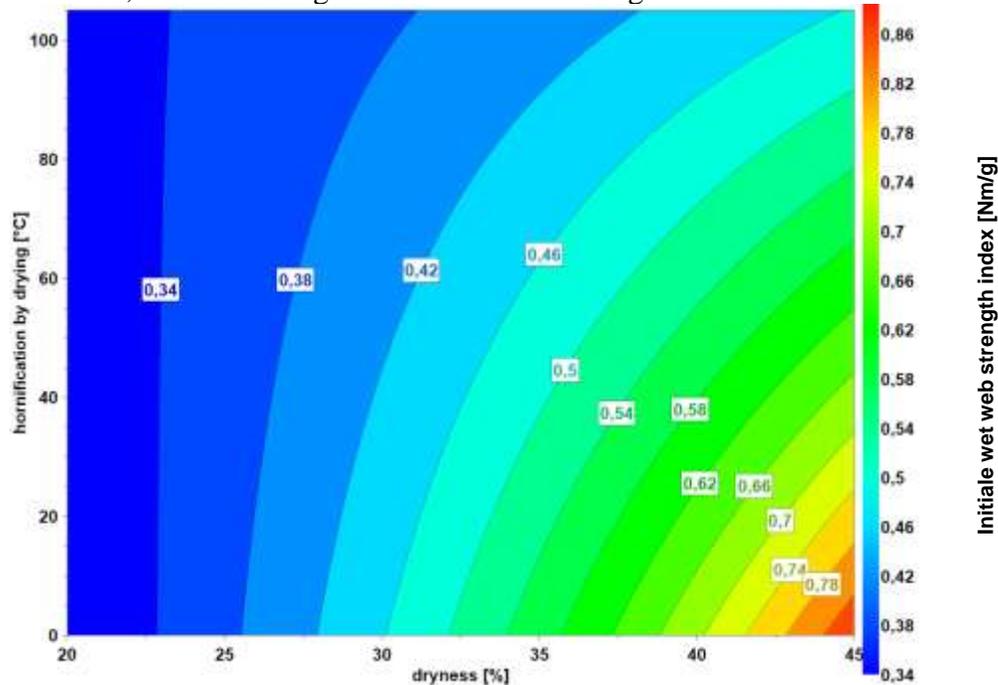


**Fig. 8.** Z-direction of paper freeze-dried at 45% dryness



**Fig. 9.** Z-direction of paper heat dried to 95% dryness

In the context of the images, Fig. 10 shows the initial wet web strength development depending on dryness and hornification. The figure reveals that the hornification of the pulp at a dryness of 20% had no impact on the strength index. The more the paper sheet was dewatered, the more a negative effect on the strength index can be seen.



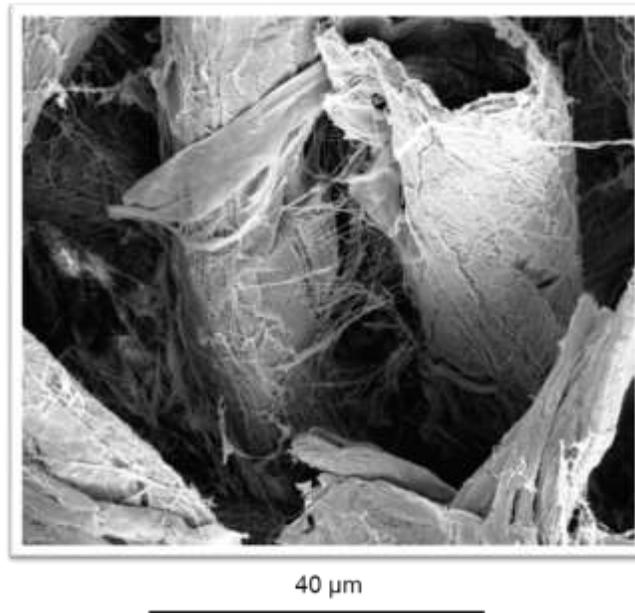
**Fig. 10.** Development of initial wet web strength index

### Strength Development

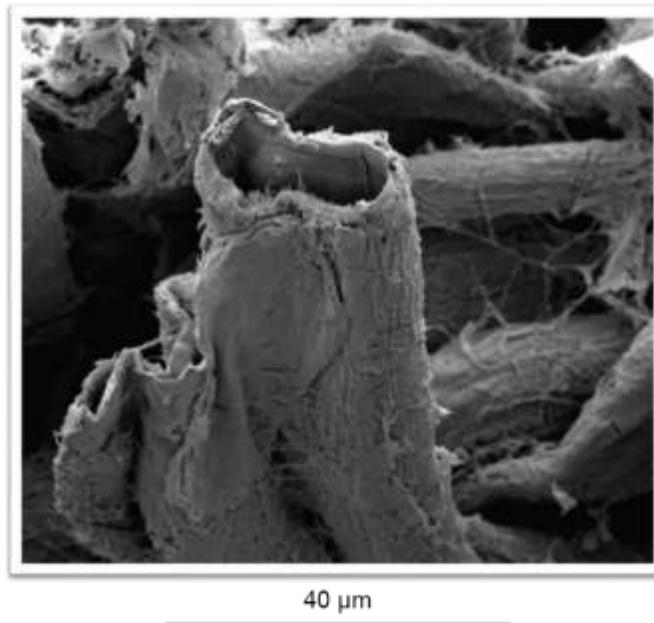
As mentioned above, strength development can be explained by different phenomena. In this work, fibre collapse and hornification were analyzed, as well as the water-gel structure with the approach of fibrils.

#### *Fibre collapse, wet and dry hornification*

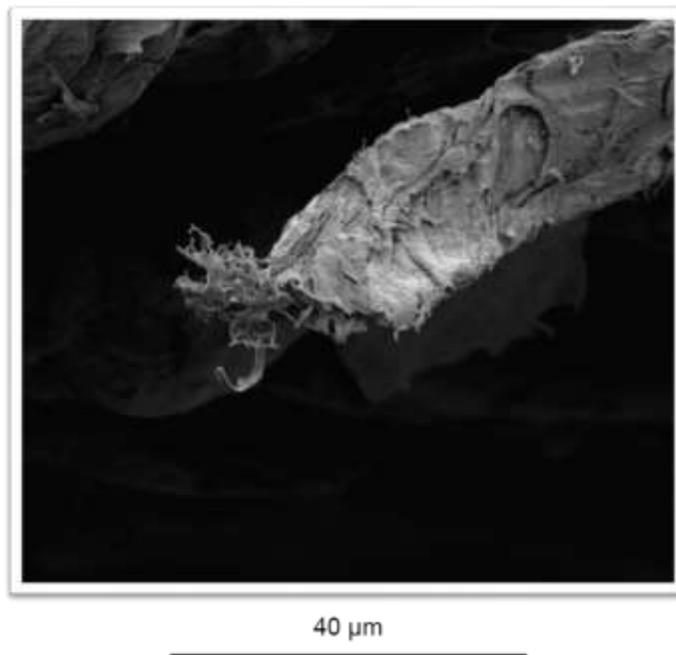
Figures 11 to 13 show the fibre collapse during dewatering and drying.



**Fig. 11.** Process of fibre collapse due to wet and dry hornification for paper freeze-dried at 20% dryness



**Fig. 12.** Process of fibre collapse due to wet and dry hornification for paper freeze-dried at 45% dryness



**Fig. 13.** Process of fibre collapse due to wet and dry hornification for paper heat dried to 95% dryness

In Fig. 11, a broken fibre with an open lumen can be seen at the top of the image. Some outward-extending fibrils between two fibres are also visible in the middle of the picture. In Fig. 12, freeze-dried at 45% dryness, the fibre is already flattened to some extent. On the right side of this fibre, in the background, some outward extension of fibrils can be observed. In Fig. 13, the sample heat dried to a solids content of 95%, the fibre lumen is completely collapsed and the fibre is flattened with no outwardly extending fibrils (Reeves 1991; Wågberg 2009).

#### *Fibre surface and fibrils*

In Fig. 14 on the left, the fibre surface freeze-dried at a dryness level of 20% is shown. Although it is an unrefined pulp sample, some fibrils on the surface can be observed. At a dryness level of 20% the fibres show a slightly round shape, indicating their swollen state.

The horizontal fibre shows a partially peeled-off S1 wall that is already joined with the vertical fibre. In this joint area there are some other small fibrils visible. It is imaginable that here a gel-like structure is formed by water and fibrils (Wågberg and Annergren 1997). The angular surface structure of the S1 layer cannot yet be observed on these fibres at this degree of magnification (Young 1986).

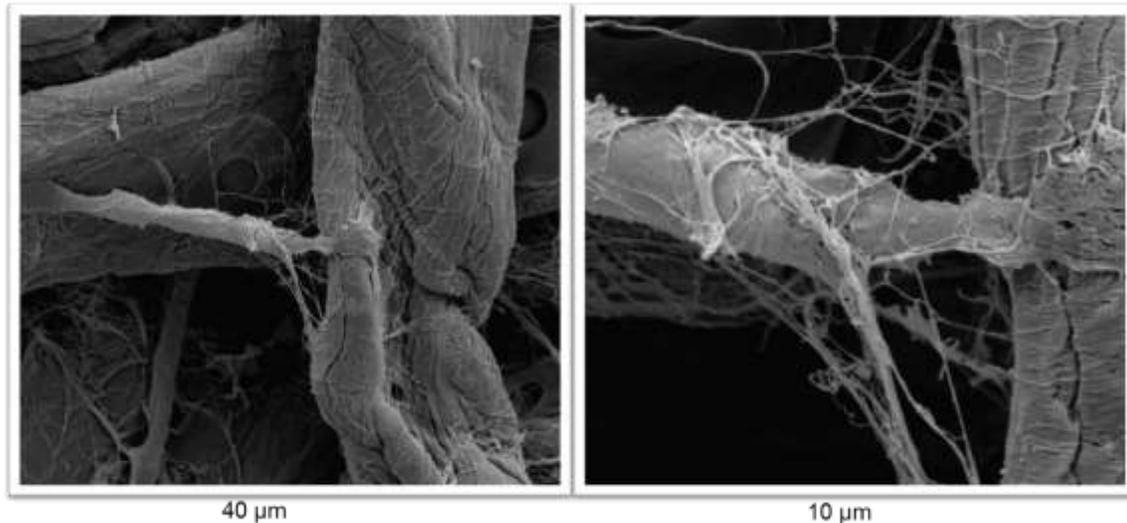
The image on the right side has been taken with higher magnification. It can be seen that many fibrils are looming out of the S1 layer to the next fibre. It looks like dangling tails, which are approaching the surrounding fibres by clamping together and adhering to each other (Neuman 1993; Yan and Li 2013).

Figures 15 to 17 show different fibre intersections. In Fig. 15 (freeze-dried at 20% dryness), a part of an S1 layer connects two fibres. Interacting macro fibrils are visible in this S1 layer. In Fig. 16 (the sample freeze-dried at a dryness of 45%), some fibrils are joined in a self-assembly manner at the intersection area of two fibres. Figure 17 shows a

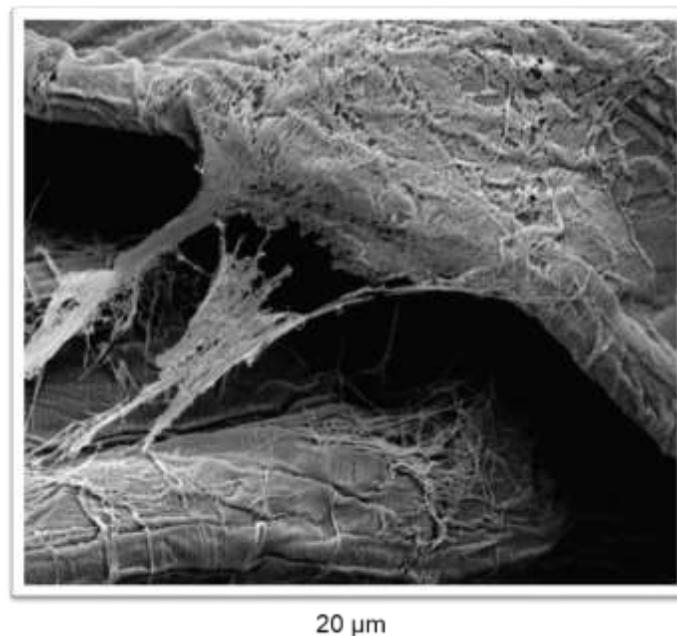
bonding area between two fibres in the paper heat-dried to a dryness of 95%. The bonding area looks like a kind of canvas.

Due to its uniform structure, this fibre bonding area could have been formed by dewatering and drying of cellulose gel (Laivins and Scallan 1993; Maloney *et al.* 1998). At this canvas-like structure, no macro fibrils are visible any more. This kind of bonding was observed in nearly all images of dried papers as already shown by various scientists (Nanko and Ohsawa 1989; Klein 2011; Mou *et al.* 2013).

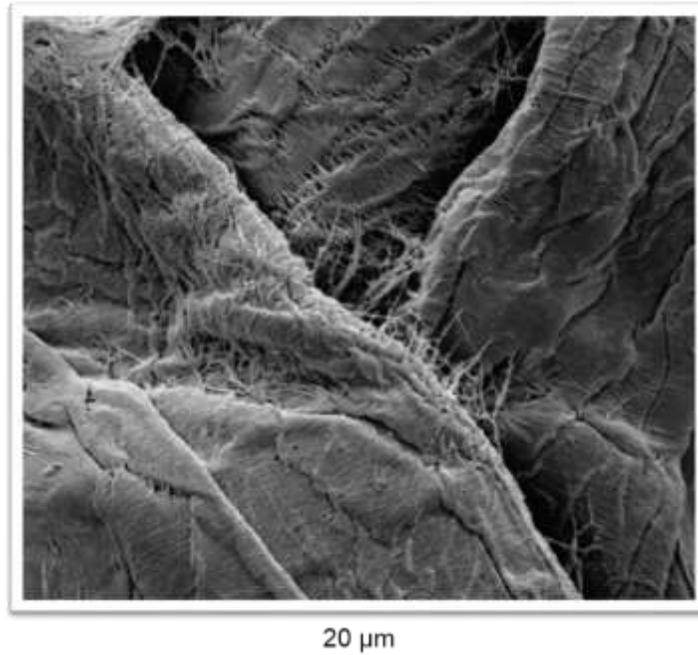
In Fig.17, a rupture in the mentioned bonding area can be seen. The cause for this defect could be explained by the shrinkage of the fibre structure during drying.



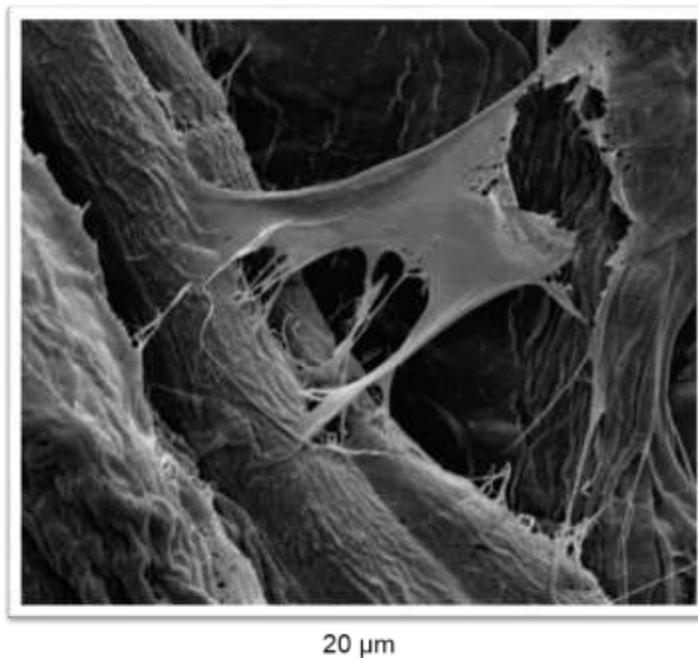
**Fig. 14.** Fibrils between fibres forming inter fibre bonds (unrefined pulp) freeze-dried at 20% dryness



**Fig. 15.** Development of fibre-fibre bonding in paper freeze-dried at 20% dryness



**Fig. 16.** Development of fibre-fibre bonding in paper freeze-dried at 45% dryness



**Fig. 17.** Development of fibre-fibre bonding in paper heat dried to 95% dryness

## DISCUSSION

At low dryness levels up to ~25%, one might expect that there is a lot of space between fibres because of the huge amount of water in the paper sheet. But due to fibre swelling, meaning inclusion of water in the fibre wall and in the lumen, as well as gel formation in striking distance of the fibres, they do approach each other already in several spots (Scallan 1983; Scallan and Tigerström 1992; Wågberg and Annergren 1997).

To build up sufficient fibre-fibre contact points, the moment of fibre collapse is important (Erhard *et al.* 2010). At this point the fibres get flattened and the initial approach of the fibres to each other is starting. As shown in Figs. 11 to 13, the fibre collapse starts somewhere between 20% and 45% solids content for the type of fibres used in the present work.

For fibre-fibre interactions, a small space between the fibres is necessary. The required distances for the formation of hydrogen-bridge-bonds, as described in the literature, are between 0.2 nm and 0.35 nm (Desiraju and Steiner 1999; Pelton *et al.* 2000; Gardner *et al.* 2008; Linhart 2005). This hydrogen-bridge-bond distance is much smaller than the surface roughness of the fibre, which is between 10 nm and 10,000 nm (Page 1993; Hubbe 2006; Pelton 2004; Heinemann *et al.* 2011). The fibre roughness might be one of the limiting factors for these interactions (Hubbe 2006). McKenzie stated that it is very unlikely for hydrogen-bridge-bonds to be formed directly after pressing, since the water layer at the fibre surface acts as a spacer in addition to the high fibre surface roughness (McKenzie 1984).

The pictures presented in this paper show, however, that even with a certain roughness and at low dryness levels, there exists a huge variety of contact possibilities between fibres. These contact points are maybe not directly on the fibre surfaces, but on fibrils and the partially peeled-off S1-layer. The water layer at the fibre surface does not seem to act as a spacer between the fibres. Moreover, water and fibrils at the fibre surface are forming a gel-like structure that enables the fibrils to extend outwards from the fibre like a dangling tail (Neuman 1993). These tails entangle with other tails like a hook and loop fastening system.

It is also probable that van-der-Waals forces occur between fibres if they approach each other close enough, meaning closer than 1 nm (Wågberg and Annergren 1997; Eriksson 2006; Hubbe 2006). In theory, van-der-Waals forces are unlikely to occur in initial wet paper, mainly due to the adsorbed water in the web, which increases the distance between the fibres. But the pictures, especially those in Fig. 15 to 17, show that the distance between the fibres is bridged by fibrils and other small fibre components, so that even van-der-Waals forces and hydrogen-bridge-bonds might act after pressing. These forces start to act even slightly above 25% dryness (Belle *et al.* 2014a,b).

In addition, the water retention values in Fig. 3 and the strength index in Fig. 10 show the negative effect of the hornification of the fibers even at dryness above 25%. Hornification means less flexible fibres and fibrils (Szwarcstajn and Przybysz 1977; Weise 1998) and a poorly formed fibre-water gel (Scallan and Tigerström 1992; Wågberg and Annergren 1997) that enables the fibrils to stretch out of the fibre surface to bridge the gaps between the fibres. This means a contradiction for paper machine runnability: In principle higher dryness is the best for paper strength. To achieve a higher solids content during paper production, a lower water retention value is needed. A lower water retention value is, however, counterproductive for water-fibre gel formation and flexible fibres and fibrils that bridge the distances between the fibres.

## CONCLUSIONS

Many researchers have developed and published models on the topic of paper strength development (Brecht and Erfurt 1961; Casey 1960; Clark 1978a; Hirn *et al.* 2013; Kibblewhite 1973; McKenzie 1984; Neuman 1993; Pelton 1993; Linhart 2005; Alince *et al.* 2006; van de Ven 2008; Kulachenko *et al.* 2009; Tejado and van de Ven 2010; Persson *et al.* 2013). Most of these models deal with the idea of the partial solubility of micro fibrils and cellulose chains on the fibres' surfaces.

1. The details shown in the presented pictures demonstrate the process of strength development during dewatering. With this sample preparation method, it is possible to implement FE-SEM imaging in order to illustrate the behavior of the fibre surface's fibrils and fine structures during their approach in water whilst pressing and drying.
2. The presented pictures, especially in Figs. 11, 12, 14, 15, and 16, demonstrate a high nonuniformity of the fibre surfaces even with unrefined pulp. Particularly the peeled-off S1 wall and fibrils on the fibre surface are shown in Figs. 14, 15, and 16. On this basis, it is questionable if the fibre surface roughness measurements can be used for calculating the initial wet web strength as mentioned in some papers (Page 1993; Alince *et al.* 2006).
3. The space between the fibres at 20% dryness is about 10 to 20  $\mu\text{m}$  (*e.g.* Fig. 11 and 15) and at dryness of 45% it is only about 3 to 10  $\mu\text{m}$  (*e.g.* Fig. 16). The fibrils are capable of bridging these distances if they are able to stretch out from the fibre surface in a well formed fibre-water gel (Scallan and Tigerström 1992; Wågberg and Annergren 1997).
4. It can be stated that the pictures show macro fibrils extending outwards from the fibre and forming contacts to the next fibres or fibrils *via* self-assembly. At these initial contact points, the van-der-Waals forces could act, and this seems to be the cause of strength in the wet paper web. Due to the extending fibrils, the required conditions for hydrogen-bridge-bond formation could be given.
5. The method used for sample preparation of initial wet paper webs in this work will be useful for future work in order to get deeper insights into paper strength development during manufacturing.

## ACKNOWLEDGMENTS

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