Influence of beating and chemical additives on residual stresses in paper

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SUMMARY: Residual stresses are the stresses remaining in a material when all external forces are removed. Residual stresses in paper can influence the converting and end-use performance. There are well-established methods for determining residual stresses in paper, and some knowledge exists of how to control and tailor the residual stresses. However, there is an increasing demand to be able to tailor paper grades with respect to their mechanical properties. Pulp fibres are commonly beaten to improve the mechanical performance, but beating also increases the sheet density, de-watering resistance, and residual stresses of the paper produced.

This work examines whether beating and the addition of chemical additives, i.e., a single layer of poly(allylamine) or a multilayer of poly(allylamine) and poly(acrylic acid), exert different effects on the build-up of residual stresses in paper. Both beating the fibres and adding polyelectrolytes increased the in-plane strength, stiffness, and residual stresses of the paper sheets prepared. The fact that the residual stresses did not scale linearly with the stiffness of the prepared sheets suggests that both beating and polyelectrolyte addition made the fibre/fibre joints transfer load at a lower solids content, such that stresses were transferred between fibre layers in the sheet earlier in the drying process, thus increasing the residual stresses. The fact that the strength gain when building polyelectrolyte multilayers induced less residual stresses than when the strength was increased by beating indicates the possibilities for producing paper with high strength but less residual stress.

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Stress is typically the response of a material subjected to external load, but can exist irrespective of load provided the average force on every cross-section is zero. Residual stresses are the stresses that remain in a piece of material when all external forces are removed. In paper, residual stresses originate from the fact that paper dries inhomogeneously (Östlund et al. 2004b). For a paper dried such that vapour is allowed to escape from both sides of the paper (i.e., two-sided drying), the outer layers of the paper dry first (Bernada et al. 1998). As the outer layers dry and shrink, the interior of the paper will be compliant, due to its high moisture content, and no significant stress build-up will take place. However, towards the end of the drying, when the interior of the paper is drying and shrinking, the shrinkage will be opposed by the already dry and thus stiff surface layers. This will cause tensile stresses in the middle layers of the paper that are not allowed to shrink and compressive stresses in the outer layers that oppose the shrinkage. Methods for determining residual stresses in paper have been developed, and compressive stress in the outer layers and tensile stress in the middle layers are also found in paperboard after drying (Waterhouse et al. 1987; Östlund et al. 2005a; Östlund et al. 2005b), though not always in finished commercial paperboard (Östlund et al. 2005b).

Residual stresses have several important implications for paper materials. An uneven residual stress distribution leads to shape distortion, i.e., curl and twist. Curl is an important quality issue in the paper industry, especially in papers used in high-speed printing (Uesaka 1991), and so it is desirable to have good control of the stress development during paper manufacturing. Compressive stress at the surface, as usually found in commercial papers, is negative in the sense that, on exposure to water, the stress state is modified leading to dimensional changes that may be difficult to predict. In contrast, tensile stress at the surface could be harmful by facilitating fracture and thus reducing the strength of the material. The residual stress state can thus influence the likelihood of crack formation during the folding of paperboard. Residual stresses are also a source of error in material testing and in predicting the mechanical behaviour of materials. The desired stress state thus likely differs between paper grades. Therefore, to optimise a paper's performance during converting and end use, it would be beneficial to be able to control the residual stress state.

Some research has examined the possibility of controlling the residual stress state of paper. First, residual stresses are known to correlate well with the moisture gradients during drying, such that compressive stresses develop in the layers that dry first due to the shrinkage in the layers that dry later (Östlund et al. 2004b). Residual stresses in paper can thus be controlled by controlling the moisture profile during drying. Re-wetting is known to be able to reverse the residual stress state in some machine-made paperboards (Östlund et al. 2005b). Beating the pulp is known to induce additional shrinkage gradients in multi-ply paperboard and to increase the magnitude of the residual stresses (Östlund et al. 2004a). Beating the pulp used in the different plies of a paperboard to different degrees would therefore be one way to influence the residual stress profile.

Beating is commonly used to improve both the strength and stiffness of the finished paper in both tension and compression, but beating pulp fibres also has several drawbacks. The density of the manufactured paper increases with beating, because beating makes the fibres more flexible such that they can pack closer together during the consolidation of the paper (Samuelsson 1964). Density is a key property, since it has a large influence on bending stiffness, which in turn has considerable impact on the performance of many paper grades. For example, high bending stiffness reduces the tendency for boxes to buckle and fail under compressive load, prevents printing-grade papers from folding under their own load when read from, and increases the runnability of sack paper during converting. Therefore, to maximise bending stiffness it is desirable to have as high tensile stiffness as possible while maintaining low density. Secondly, beating increases the swelling and water-retaining ability of the fibres thus rendering the dewatering of the paper web more difficult, influencing the rate at which paper can be produced on dryer-limited paper machines. Thirdly, the energy required for beating is costly.

The fact that common paper-making unit operations such as beating and wet pressing are always associated with increased density and, in the case of beating, with increased de-watering resistance, means there usually are few degrees of freedom when optimising the mechanical performance of a paper grade. Since the competition on the paper market and the product performance demands are constantly increasing, there is a pressure to improve and tailor paper qualities with respect to strength, toughness, stiffness, and residual stresses without impairing other important properties such as density and de-watering resistance. Chemical additives offer one way to improve certain properties without degrading others. For example, the build-up of polyelectrolyte multilayers on pulp fibres can be used to increase tensile strength with less densification than if chemical fibres are PFI beaten (Wågberg et al. 2002) or if the energy input in refining mechanical pulp fibres is increased (Lundström 2009). In addition, increasing the surface charge of chemical pulp fibres by the irreversible adsorption of carboxymethyl cellulose to the fibre surfaces has been demonstrated to increase in-plane tensile strength while producing less densification than does PFI beating (Laine et al. 2003).

This study investigated whether a single or multilayer addition of polyelectrolyte could be used to alter the residual stresses in paper in a way different from beating, and thus increase the degrees of freedom for controlling the residual stresses in paper. Since the build-up of residual stresses is closely linked to how fibres shrink and interact during drying, the study also sought clues as to whether beating and polyelectrolyte addition have different effects on how fibres shrink and interact during drying.

Materials and Methods

Fibres

The fibres used were unbleached never-dried laboratory pulped softwood kraft fibres. Spruce wood chips were lab cooked in steel autoclaves using an effective alkali of 200 kg/ton and a cooking temperature of 160°C, resulting in an H-factor of 1381. The resulting pulp had a yield of 49.7%, a kappa number of 34, and a degree of polymerisation of 1014 ml/g as determined by viscosity measurements. The pulp was subsequently washed at both high and low pH in order to remove most of the remaining adsorbed metal ions and dissolved and colloidal material, as described previously by Gimåker et al. (2007). Since polyelectrolyte addition influences fines retention, it was necessary to remove the fines from the fibres so the fines concentration would not vary between sheets and to avoid fines gradients through the thickness of the sheets. This was done using a Britt Dynamic Drainage Jar (PRM Inc., Seattle, WA) according to the Tappi T 261 cm-94 standard, before adding polyelectrolytes to the fibres or after beating the fibres.

Chemicals

Poly(allylamine hydrochloride) (PAH) with an average molecular mass of 15 kDa and poly(acrylic acid) (PAA) with an average molecular mass of 8 kDa were purchased from Sigma-Aldrich, Sweden. The sodium bicarbonate ($NaHCO_3$) used to make buffer solution for the polyelectrolyte adsorption was of analytical grade.

Adsorption of polyelectrolytes

The build-up of multilayers consisting of PAH and PAA on cellulose fibres is a very well-documented and robust method (Wågberg et al. 2002; Eriksson et al. 2005). However, it is crucial that pH be controlled during the adsorption of PAH and PAA, since both are weak polyelectrolytes, i.e., their charge density depends on the present pH. Consequently, all adsorptions were conducted in a 10^{-2} M $NaHCO_3$ buffer to maintain a constant pH of approximately 8.3 throughout the adsorptions. It has previously been demonstrated that a background electrolyte concentration of 10^{-2} M NaCl results in the adsorption of 15 kDa PAH to the exterior of the fibres (Gimåker, Wågberg 2009), ensuring that the multilayers will build up on the fibre surfaces.

For the addition of a single layer of PAH, the fibres were suspended in a 10^{-2} M $NaHCO_3$ solution at a concentration of 5 g/l. Dissolved PAH was added to the pulp suspension at a dosage of 10 mg/g fibre and allowed to adsorb for 15 min, after which the fibres were transferred to the sheet preparation equipment. To prepare a multilayer of PAH and PAA, PAH was first adsorbed to the fibres as described above, after which the fibres were washed with twice the volume of sodium bicarbonate solution as used during the adsorption. The fibres were then re-suspended in 10^{-2} M $NaHCO_3$ solution, again at a concentration of 5 g/l. Dissolved PAA was added at a dosage of 10 mg/g fibre and allowed to adsorb for 15 min. The fibres were then washed and a third PAH layer was adsorbed using the exact same methodology as used for the first layer. After a third layer had been adsorbed, the fibres were transferred to the sheet preparation equipment. The pH was closely monitored during all adsorption steps and found to remain between 8.0 and 8.5.

Sheet preparation

Before sheet preparation, polyelectrolytes were added to the fibres as described above or the fibres were PFI beaten according to the ISO 5264-2:2002 standard for 0, 500, 5000, or 7000 revolutions. Sheets with a grammage as close as possible to 160 g/m^2 were prepared using a Rapid-Köthen sheet preparation equipment (Paper Testing Instruments, Pettenbach, Austria) following the ISO 5269-2:1998 standard. Sheets were dried under restrained conditions at 93°C and at a pressure of 95 kPa below atmospheric pressure for 20 min. The time needed for dewatering in the forming section of the Rapid-Köthen equipment was also measured for the various furnishes in order to quantify the effects of beating and chemical addition on the dewatering resistance.

Paper testing

Tensile testing in one in-plane direction of the paper was conducted according to the SCAN P:67 standard for tensile testing laboratory-made sheets. As the sheet making was symmetrical, the papers were assumed to be isotropic in the plane of the papers. The thickness of the prepared sheets was measured as structural thickness (Schultz-Eklund et al. 1992) and was used to calculate sheet density.

Determination of residual stress state

The through-thickness distribution of residual stresses in the plane of the paper was determined by a previously described layer removal method (Östlund et al. 2005). Thin layers were removed from the paper specimen by surface grinding, which changes both the stress distribution and the bending stiffness of the substrate, resulting in a change of curvature of the substrate. The surface grinding was performed on narrow strips (8 mm) of the paper so that the strips would be able to curl in both in-plane directions, which is known as a geometrically linear behaviour. After each grinding increment, which reduced the average strip thickness by 4–20 μm, the curvature in the strip direction was calculated by least-squares fitting a circle to the positional co-ordinates of the centre line of the strip measured by triangulation with a laser displacement sensor. Thickness was also measured after each grinding increment using the same system as above. The measured data provide the curvature versus grinding depth trend. By grinding paper specimens to the middle from alternate sides, and measuring the curvature versus grinding depth trend as described, the stress distribution in the original specimen could be calculated. Because the sheet preparation was symmetrical, the stress state was assumed to be equibiaxial. Full details of this method and extensive discussion of its merits are available in Östlund et al. (2005a).

Results and Discussion

The effect of beating and the addition of poly(allylamine) (PAH) as a single layer or as a multilayer with poly(acrylic acid) (PAA) was studied as described above. The effect on the tensile strength index of the prepared laboratory sheets is presented in *Fig 1*. The addition of polyelectrolytes increased the strength of the sheets considerably, in fact more than could be achieved by beating, without any significant densification. The fact that the tensile strength index increased so significantly without densification demonstrates the possibility of producing paper possessing both high strength and bulk. The fact that the strength increased without densification also indicates that the addition of the current additives caused no significant flexibilising or collapse of the fibres during drying, since this would have resulted in densification (Samuelsson 1964; Page 1985). Previous BET measurements and fibre contact zone analysis of sheets treated with PAH/PAA multilayers (Eriksson et al. 2006) suggest that the main effect of the additives was to increase the number of fibre/fibre joints, the fibre/fibre joint strength, and the molecular contact area in each joint. The results also indicate that fibre flexibilisation is not necessary to obtain high paper strength, but that the surface properties of the fibres largely determine the strength of the fibre/fibre joint and the resulting paper. Such results and ideas have been previously described by, for example, Wågberg et al. (2002), Laine et al. (2003), and Torgnysdotter and Wågberg (2004).

The sheets used in the present study were prepared from pulp from which the fines had been removed. Increasing the fines content generally increases tensile strength and strain-to-break (Sandgreen, Wahren 1960; Htun, De Ruvo 1978). However, increasing the fines content simultaneously increases sheet density so, if the sheets in the present study had been produced from the original pulp containing the fines, they would probably have had a slightly higher tensile strength but also a higher density. Sandgreen and Wahren (1960) found that the relationship between tensile index and sheet density was not changed by fines removal. Thus, with respect to mechanical properties, the absolute values would have differed if fines were present, but the relative relationship between the sheets would likely have been unaffected. The effects of beating and polyelectrolyte addition on the tensile stiffness index of the prepared sheets are shown in *Fig 2*; as can be seen, both additive addition and beating increased the stiffness of the prepared sheets slightly but significantly. In the case of the additives, the stiffness increase would be primarily due to a change in the fibre network structure resulting from improved joint development during consolidation (Seth, Page 1981). Furthermore, as seen in *Fig 2*, beating increased the stiffness index more than did polyelectrolyte addition. Like the effect of added polyelectrolytes, the effect of beating on stiffness was likely partly due to a change in fibre network structure resulting from improved joint formation during consolidation. Low-consistency refining such as PFI beating is also known to exert a straightening effect on the fibres (Peakes 1967; Page 1985; Mohlin, Alfredsson 1990), which in turn have a stiffening effect on the resulting paper sheets (Page, Seth 1980; Joutsimo et al. 2005). Thus, the reduction of fibre curl and fibre deformation explains the higher stiffness achieved by beating than by adding polyelectrolytes.

By measuring the dewatering time in the Rapid-Köthen sheet forming equipment, the effect of beating and polyelectrolyte addition on dewatering resistance was also quantified. Beating increased the dewatering time from 20 sec for the reference to 28 sec for the pulp beaten for 5000 revolutions. Polyelectrolyte addition, however, had no significant effect on dewatering resistance (20 sec dewatering time with the addition of PAH and 19 sec dewatering time with the addition of PAH/PAA/PAH multilayers). That multilayers do not influence the dewatering resistance of unbeaten pulp was also found by Brännvall et al. (2007), who demonstrated that the dewatering resistance of unbeaten pulp measured as the Schopper-Riegler value did not change with the addition of multi-layers of cationic and anionic starch. Brännvall et al. (2007) also found that, for beaten pulp, dewatering resistance actually decreased with the addition of starch multilayers.

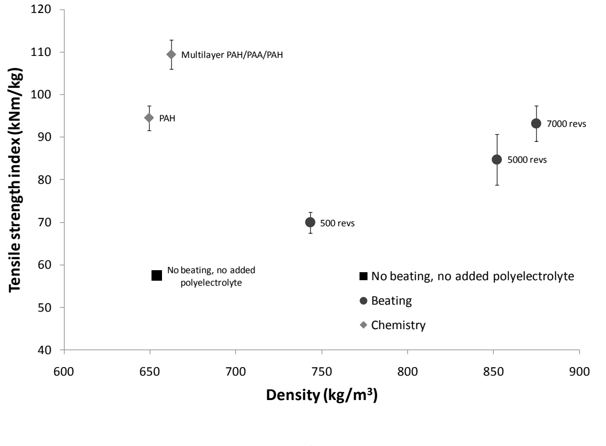

Fig 1. Tensile strength index for the prepared sheets as a function of the apparent sheet density.

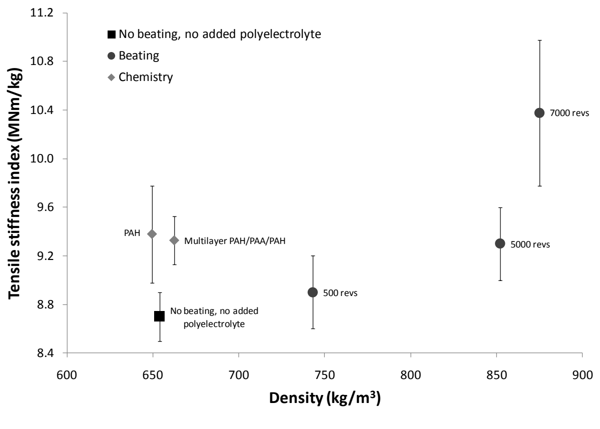

Fig 2. Tensile stiffness index of the prepared sheets as a function of the apparent sheet density.

The effect of beating and polyelectrolyte addition on the residual stress state was determined according to the above-described procedure and the results are shown in *Fig 3*. The residual stresses were small in the reference paper, which agrees with previous results for paperboard made from unbeaten pulp (Östlund et al. 2004a). Still, the stress was definitely compressive at the surface of the reference paper, as in the stronger papers. This is typical of paper after drying, as indeed of many plastics after cooling. The residual stresses in the stronger papers, whether resulting from pulp beating or chemical additives, were significant though not of the magnitude found in strong commercial paperboards. The magnitude of the residual stresses increased with the number of both beating revolutions and adsorbed polyelectrolyte layers. The dotted parts of some curves in *Fig 3b* were calculated by extrapolating the measured trend of curvature versus grinding depth to the middle of the specimen, for completeness of the stress profiles.

Since the residual stress profiles of sheets containing added polyelectrolytes (*Fig 3a*) appeared to have less steep stress gradients than the profiles of sheets made of beaten fibres (*Fig 3b*), the profile shape was analysed in more detail. In doing so, it is important to bear in mind that the sheet thickness decreased with beating. Therefore, to determine whether the more curved appearance resulted solely from the decreased sheet thickness or whether there were additional causes, the in-plane stress was plotted against the normalised thickness, as shown in *Fig 4*. It can be seen that the slope of the stress gradient in the outer part of the sheets tended to increase with increasing surface stress. However, if two samples with similar surface stresses, i.e. the PAH sample and the 500 beater revolutions sample, are compared, no significant difference in slope of the gradient can be seen. The multilayer sample and the 5000 beater revolutions sample also have gradients with similar slopes even though the 5000 beater revolutions sample had a somewhat higher absolute residual stress. It can therefore be concluded that there was no significant difference in slope of the stress gradients between papers made of beaten fibres and papers with added polyelectrolyte, and that the apparently steeper stress gradients in sheets made of beaten pulp in *Fig 3b* resulted solely from the decrease in sheet thickness as a result of beating.

In *Fig 3* and *Fig 4*, however, it can be seen that, for sheets containing added polyelectrolytes, the in-plane stress did not monotonically increase towards a maximum stress in the centre of the sheets. Instead, there was a maximum in stress some 60 micrometers from the middle. The stress dip in the middle could, for example, correspond to a low-density region in the middle of these papers. The fact that this stress dip was not seen in the papers made of beaten fibres could suggest that most of the thickness decrease, i.e., consolidation, achieved by beating (versus without beating) occurs mainly in the middle of such papers. This would correspond to a situation where the middle of papers, made from beaten fibres, carry more load than in the papers containing additives.

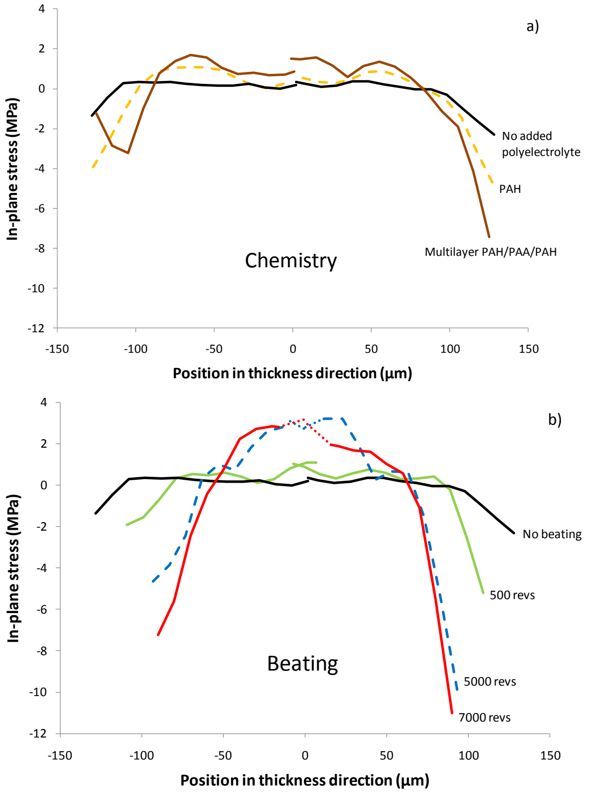

Fig 3. Distribution of in-plane residual stress in the thickness direction for a) papers made of fibres with various levels of polyelectrolyte addition and b) papers made of fibres subject to various degrees of beating.

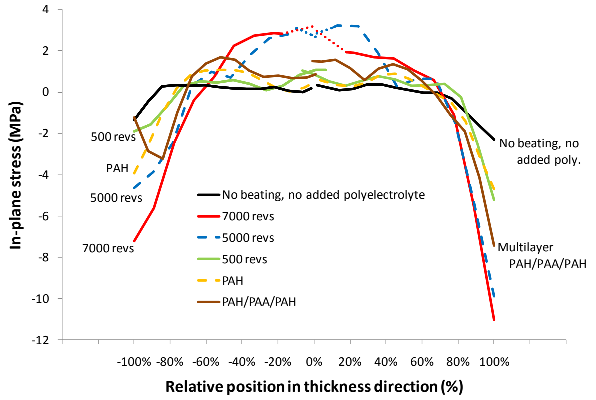

Fig 4. In-plane residual stress as a function of relative position in the thickness direction. The slope of the stress gradients increased with absolute surface stress, but, if compared at equal surface stress, there was no significant difference in gradient steepness between papers made of beaten fibres and papers containing added polyelectrolyte.

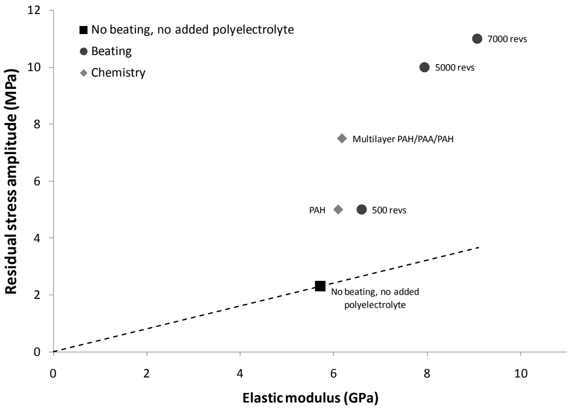

Fig 5. Residual stress amplitude in the prepared sheets plotted as a function of their stiffness. The dashed line shows where the residual stress amplitude would have been if the residual stresses had scaled linearly with sheet stiffness.

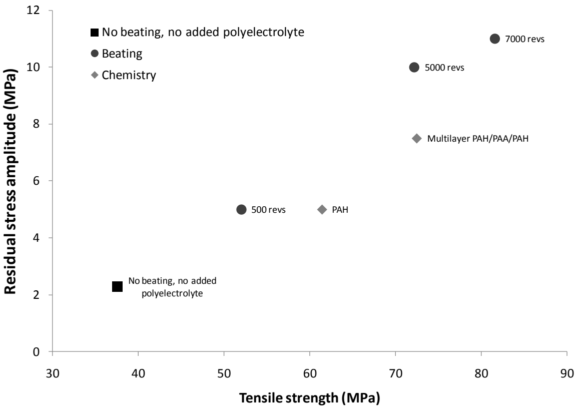

Fig 6. The residual stress amplitude as a function of tensile strength. Beating produced higher residual stress for a given tensile strength than did polyelectrolyte addition.

Fig 5 shows the residual stress amplitude, i.e., the absolute value of the surface stress at the side where it was the highest, as a function of the sheet modulus. In the method of residual stress determination, the stiffness is simply a scaling factor applied to the measured deformation and geometry data. It is thus natural for the influence of beating and chemical additives on stiffness to be manifested also in the residual stress amplitude. However, as seen in *Fig 5*, the residual stress amplitude did not scale linearly with the stiffness in the prepared sheets, as the data points would then have fallen on the dashed line in *Fig 5*.

The fact that the residual stress amplitude did not scale linearly with the stiffness means that there must be some additional mechanism apart from increased stiffness underlying the increase in residual stresses with beating and polyelectrolyte addition. Increased beating is known to increase shrinkage during drying (Brecht et al. 1956), whereas the build-up of PAH/PAA multilayers on fibres has been demonstrated not to influence shrinkage during free drying (Larsson, Wågberg 2008). The fact that beating increases dewatering resistance and shrinkage during drying implies that there will be greater moisture and shrinkage gradients and thus greater residual stresses in sheets made of beaten fibres. The finding of Ivarsson (1954), that sheets start to shrink at higher moisture contents the more beaten their constituent fibres, also suggests that beating increases the moisture content at which load starts to be transferred between fibre layers. Earlier load transfer between fibre layers further increases the residual stresses.

Since the presence of polyelectrolyte multilayers did not influence de-watering resistance or drying shrinkage (Larsson, Wågberg 2008), the increase in residual stresses with the addition of multilayers cannot have been because the multilayers increased the moisture and shrinkage gradients through the sheet. This suggests that the added polyelectrolytes mainly influenced the dry content at which fibres started to interact and transfer load and thereby the residual stresses. The fact that adding polyelectrolytes produced higher residual stresses than did beating for a given sheet modulus in combination with the fact that polyelectrolyte addition did not increase de-watering resistance and thus moisture gradients, means that the load transfer between fibre layers in sheets containing adsorbed polyelectrolyte must have started at a significantly higher moisture content than in sheets made of beaten fibres. The presence of adsorbed polyelectrolytes probably facilitated capillary forces, the development of van der Waals forces, and the interdiffusion of polymer chains between adjacent fibres, so that the fibres with adsorbed polyelectrolyte started to interact and transfer load at a significantly lower solids content.

The fact that the addition of polyelectrolytes to the fibres increased the sheet strength considerably without increasing the residual stresses as much as beating did, as shown in *Fig 6*, indicates that it is possible to produce paper with high strength but low residual stress. Whether or not this is advantageous, however, probably varies from case to case. In any case, the use of chemical additives when designing a paper grade increases the degrees of freedom and should be a useful tool for optimising mechanical properties and the residual stress state.

Conclusions

Both beating the fibres and adding polyelectrolytes increased the in-plane strength, stiffness, and residual stresses of the paper sheets prepared in this study. However, the increase in residual stress did not scale linearly with the increased stiffness. This suggests that both beating and the addition of polyelectrolytes induced better load transfer in the fibre/fibre joints at a lower solids content, such that stresses were transferred between sheet layers earlier in the drying, thus increasing the residual stresses. At a given sheet stiffness, the residual stresses were higher in sheets containing added polyelectrolytes, which suggests that the fibres with adsorbed polyelectrolyte started to transfer load at even higher moisture contents than did the beaten fibres.

The fact that beating and the addition of chemical additives had different effects on mechanical properties such as strength, stiffness, density, and residual stresses opens up new possibilities for tailoring paper qualities with respect to converting and end-use performance. For example, the use of polyelectrolytes should enable the production of paper with high strength and low residual stress.

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