Preferred citation: I.T. Pye, O.V. Washburn and J.G. Buchanan. Structural changes in paper on pressing and drying. In **Consolidation of the Paper Web**, *Trans. of the IIIrd Fund. Res. Symp. Cambridge*, *1965*, (F. Bolam, ed.), pp 353–367, FRC, Manchester, 2018. DOI: 10.15376/frc.1965.1.353.

STRUCTURAL CHANGES IN PAPER ON PRESSING AND DRYING

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Synopsis—The scanning electron microscope has been used to observe changes in the structure of paper at different stages of pressing and drying.

In the first experiments, beaten kraft pulp handsheets were subjected to various pressing and drying treatments. The structure at the solids content achieved was stabilised by freezing and drying by sublimation under vacuum. Photomicrographs show the collapse of the fibres and consolidation of the paper structure during processing.

Samples of the web were obtained at positions from the wet end to the reel of operating kraft, bond and newsprint papermachines. As soon as the specimens were sampled, they were quickly frozen and later dried under vacuum in the laboratory.

The influence of water removal on the web and fibre structure by pressing and drying is illustrated. The relative importance of fibre conformity and fine material differs for the three paper grades. Under pressure, fibres are deformed plastically, particularly at crossing points and asperities. Collapse of fibres on removal of water from the lumen and the fibre walls by drying can usually be distinguished from that produced by mechanical pressure.

Introduction

THE scanning electron microscope has been used in previous studies to examine the surfaces and tensile fractures of handsheets made from kraft, sulphite⁽¹⁾ and groundwood⁽²⁾ pulps, which had been prepared at various levels of yield, degree of beating and freeness. Teder has compared bleached and unbleached kraft and sulphite spruce pulps⁽³⁾ and examined the effect of rewetting on the structure of dried pulp sheets.⁽⁴⁾ These investigations have now been extended to study changes in the structures of paper webs at various stages of pressing and drying.

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Fig. 1a—Kraft handsheet freeze-dried from 8 per cent solids: low magnification area showing the open web structure and uncollapsed fibres

Fig. 1b—Uncollapsed fibre from the same specimen as Fig. 1a at higher magnification



Fig. 2a—Kraft handsheet freeze-dried from 8 per cent solids: fibre crossing with loosened primary wall

Fig. 2b—Fibrils attached to two fibre fragments form a weak bond: this is a higher magnification of Fig. 2*a*



Fig. 3a—Air-drying a kraft handsheet from 8 per cent solids produces severe collapse (compare with Fig. 1)

Fig. 3b—Fibre crossing from the centre of Fig. 3a shows the typical air-dried collapse of the lumen: the edges of the fibre are visible as ridges



Fig. 4—Kraft handsheet freeze-dried after pressing at 100 lb/linear in: fibres are severely plastically deformed

Fig. 6—Kraft handsheet pressed at 200 lb/linear in and dried at elevated temperature: both plastic deformation and drying collapse can be seen

The presence of water prevents direct examination of wet webs by the scanning electron microscope, because the specimen must be observed in a high vacuum. Therefore, to prepare a sample for examination, the water must be removed without changing the geometry of the fibrous material in the wet web. It is well known that simple drying alters this radically. Water may be removed from a structure by solvent exchange drying,⁽⁵⁾ which largely preserves the microstructure of the fibrous material, but, as Teder mentioned,⁽³⁾ loose fibrous and fine material is removed during the exchange. In the alternative method of freeze drying, the water is removed from the frozen specimen by sublimation. This technique preserves much of the gross structure of a wet web,⁽⁶⁾ but there is some shrinkage of the fibres and changes in the microstructure. Because of the simplicity of freeze drying, coupled with encouraging results from preliminary tests, this method was adopted for the present experiments.

In this study, the surfaces of paper webs have been examined at various stages of water removal from 8 per cent solids to air-dry. In the first part, handsheets made from moderately beaten softwood kraft pulp were subjected to varying amounts of pressing followed by freeze, heat or air drying. In the second part, the changes occurring along the papermachine during manufacture of three different grades of paper were examined.

Spruce kraft handsheets

A 49 per cent yield kraft pulp, prepared from a single log of black spruce, using pilot plant scale equipment, was beaten to 450 CSF in a Valley beater. Handsheets were formed on a Williams sheetmachine. After further processing to remove water from the handsheet to the desired extent, a small piece was carefully cut out and placed blotter side up on a thermoelectric cooling device, which froze the water of the specimen.⁽⁷⁾ The freezing was performed inside a bell jar, after which the ice was sublimed in the vacuum produced by a rotary pump. In all cases, a temperature of -30° C to -35° C was achieved and a sublimation time of at least 12 min was used. After freeze drying, the specimen was mounted on a stub, blotter side up and prepared for examination in the scanning electron microscope.

The appearance of a handsheet that has been freeze-dried from 8 per cent solids is shown in Fig. 1a.* It was prepared by taking the web directly from the wire before couching. The fibres are mostly uncollapsed and it is obvious that there is very little opportunity for direct bonding to take place. An uncollapsed fibre in the lower central part of Fig. 1a is shown at higher magnification in Fig. 1b. In many cases, such as shown in Fig. 2a, fibrillation and

^{*} The magnification indicated on all the micrographs is that across the picture; the vertical magnification is $1/\sqrt{2}$, this value being due to the 45° inclination of the specimen⁽¹⁾

lamellae on fibres still remain loose and not collapsed on to the surface; however, fibrils may be attached to more than one fibre fragment, forming a weak bond, as shown in Fig. 2b, which is a higher magnification picture of the centre of Fig. 2a. Longitudinal wrinkling in the lower fibre can be seen. Jayme & Hunger attributed wrinkles of similar appearance, noted in their own work, to differential shrinkage between the outer and inner layers of the wall structure.⁽⁸⁾

These micrographs may be compared with those (Fig. 3a and 3b) of a similar specimen that had been air-dried after removal from the wire. The fibres and fragments have collapsed, producing a much more closely bonded structure. As water is removed from between fibrils and fibres and from within the lumens of fibres, surface tension pulls the bounding surfaces into contact with great force as the menisci recede.⁽⁸⁻¹⁰⁾ In later stages of water removal, by drying at room or elevated temperatures, water is lost from within the fibre walls, reducing their thickness.

Specimens taken from a web couched from the wire on to a blotter in the standard manner and reaching 15 per cent solids content, appeared very similar to those from the web at 8 per cent solids. Most of the fibres were uncollapsed and many fibrils and fibre fragments were hanging loosely from the surface.

Standard handsheets were pressed by placing them, blotter side up, on top of a felt that was passed between a pair of 12 in diameter rolls, operating at 30 peripheral ft/min. Pressures from zero to 1 000 lb/linear in could be obtained.

Pressing at 50 lb/linear in increased the solids content to 29 per cent and caused the web structure to compact. The fibres have been brought into close contact and collapsed, particularly at crossing points. Much less fibrillar material projects from the main body of the fibres than before.

At 100 lb/linear in, the pressing compacts the sheet even further and deforms intertwined fibres quite severely, as can be seen in Fig. 4. Compaction and fibre deformation becomes much more severe at increased pressures. At 500 lb/linear in, the surface of the handsheet is very smooth in places, owing to the close fibre-to-fibre and fibre-to-roll contact achieved (Fig. 5a). This is more evident from Fig. 5b, which is a higher magnification of the same area. Most of the fibres have been forcibly collapsed and plastic flow has occurred, so that individual fibres cannot always be distinguished.

The progressive removal of water by pressing causes the web to contract from a loosely connected structure of swollen fibres into a close network of flat, ribbon-like fibres. The collapse of fibres produced by pressure gives an appearance to the web very different from that produced by the surface tension forces of the receding menisci in the drying web.^(9,10)

24—c.p.w. i

In using mechanical pressure, deformation occurs at asperities and contact points, which experience high loads sufficient to exceed the yield strength of the material, causing plastic deformation. Additional elastic constraints may then be applied by the surface tension of the residual water. In air-drying, the web and individual fibre structures are compacted by their mechanical failure under the action of the surface tension forces of the water alone.



Fig. 5a—Kraft handsheet freeze-dried after pressing at 500 lb/linear in: severe and extensive plastic deformation

Fig. 5b—High magnification of the centre of Fig. 5*a*: severe plastic flow has obscured the fibre boundaries

When both pressing and drying occur, a combination of phenomena may be expected; thus, a specimen pressed to 200 lb/linear in, then dried at elevated temperatures shows both induced mechanical flattening and shrinkage collapse (Fig. 6).

Changes during commercial papermaking

Most previous studies have been concerned with papers produced and treated in the laboratory. Such investigations have the merit of maintaining close control of the pulp furnish and processing conditions during the production of the finished sheet. These sheets can be made to simulate commercial paper in many ways, but it is never possible to reproduce exactly all the conditions of a dynamic commercial operation. To get confirmation of the laboratory handsheet experiments and to examine the appearance of the web at various stages in a commercial process, specimens were obtained from a bag kraft, a bond and a newsprint machine.







BEFORE COUCH

AFTER CO



јсн

AFTER FIRST PRESS



AFTER SECOND PRESS

AFTER SMOOTHING PRESS





REEL

Changes on pressing and drying

To maintain the web structure at the various points of sampling, the samples were immediately frozen in dry ice and preserved in a frozen condition until the ice was sublimed from the samples, under vacuum, in the laboratory. Subsidiary experiments had shown that this technique preserved the gross structure of the sheet, although some shrinkage of individual fibres did occur. Solvent exchange methods were not satisfactory, because of redistribution of fibres and the washing out of fines during immersion in the solvent.

At the same time that the samples were taken from the machine for microscopic examination, further parts of the web were retained in cans for moisture determination.

Bag kraft

A DIAGRAM of the kraft machine, indicating the points at which samples were taken and their measured solids contents, is shown in the diagram of Table 1. The sheet substance was $50 \text{ lb}/3 000 \text{ ft}^2$.

TABLE 1—BAG KRAFT MACHINE DATAFibre in furnishUnbleached softwood kraft, lightly refinedMachine speed1 000 ft/minSubstance50 lb/3 000 ft²



The samples 3, 4 and 5 were taken from narrow strips that had passed alone through the previous section. Samples 3 and 4 were probably representative of the whole web, because the pressure distribution is mainly controlled by the felt, but the pressure experienced by the web in the smoothing press may have been excessive. The samples at all the other positions were removed from the full web.

Representative scanning electron micrographs for the positions, both on the top side and wire side of the sheets, are shown in Fig. 7. Differences



Fig. 8—Fibrils from the wet web are free and may contact surfaces without bonding (kraft)

Fig. 9—After the first press, fibrils and lamellae aggregate or collapse on fibre surfaces (kraft)



Fig. 10—End of a tracheid: both plastic deformation and drying collapse are evident in the dried paper (kraft)

Fig. 12—Fibre collapsed and bonded at a crossing after the first press (bond)



BEFORE COUCH

AFTER FIRST P



RESS

AFTER SECOND PRESS



AFTER SMOOTHING PRESS



BEFORE SIZE PRESS



AFTER SIZE PRESS

BEFORE (





ALENDER

REEL

between the top side and wire side are quite obvious and persist throughout the whole series of pictures. Many more fines are visible on the top side. The gradual compaction of the sheet through the machine is evident from these pictures.

The first positions of sampling show that most of the fibres are completely swollen, although many are partially or completely collapsed. Stereomicrographs (not suitable for reproduction here) were valuable for estimation of the degree of fibre collapse. Presumably, these collapsed fibres are partly a consequence of the failure of the once-dried broke fibres in the furnish to reswell on slushing and partly due to the collapse of thin-walled fibres on air intrusion of the web. Much of the fibrillar material that can be seen in the wet end specimens is free and does not stick when it touches other surfaces (Fig. 8). After the first press, this material is not as evident (Fig. 9). The fibrils and fibre lamellae have either aggregated or collapsed on to the surface of larger fibres. At the same position also, far more of the fibres show some degree of collapse and the whole structure is becoming more compact. There are some signs of pressure-induced distortion of fibres at crossings. It is only after the second press that large numbers of fibres are brought into really intimate contact.

The smoothing press flattens the fibres and brings them into still closer contact. They appear to be slightly swollen and incompletely collapsed. Water removal in the dryers causes appreciably more collapse of the fibres, so that they become flattened, wrinkled ribbons. The final paper shows a mixture of pressure-induced conformation and that resulting from water removal collapse (Fig. 10). No change to the surface attributable to heat deformation or decomposition could be detected up to \times 1 300 magnification.

The series of pictures confirms the handsheet investigations, though this commercial pulp was less beaten than the experimental pulps studied. The fibres were not as highly collapsed and they did not conform to each other as closely.

Lightweight bond

A DIAGRAM of the bond machine selected and relevant data appear in Table 2. A series of scanning electron micrographs at various positions on the machine, on both the top and the wire side, is shown in Fig. 11. As in the kraft samples, a difference between the top side and wire side is very evident. The basic furnish is a mixture of several different types of fibre, some of which had been dried, plus fillers and additives. Sheet substance was 19.5 lb/2 600 ft². Even before the couch, quite a number of fibres are already collapsed. Non-fibrous components of the furnish can be seen easily on the top side, but are not common on the wire side. There appear to be fewer fibrils and lamellae



* This value appears to be in error

than in the kraft paper and they seem to be much more closely associated with fibres. This could be, at least partially, due to the alum and rosin treatments.

It was not possible to obtain a sample between the couch and the first press. After the first press, it is seen that many of the fibres are deformed badly and most of them are collapsed, though not always completely. Fig. 12 shows how a fibre has been deformed into close contact with an underlying fibre. Clay particles and probably the rosin can be seen on the top side of the paper in Fig. 13.

The second press produces further deformation and this is accentuated slightly after the smoothing press. The first part of the dryers has little effect on the gross appearance of the sheet, although the fibres walls appear to collapse more completely, giving some wrinkling.

It is possible that some of the fibres are reswelled after the size press, but details of the structure are obscured by the coating of starch. At the end of the dryer section, the film of starch is not so obvious, but details of the fibres are still obscured. Through the calender stack, mechanical deformation can be detected and, at low magnification, the paper surface appears fairly smooth; at high magnification (Fig. 14), the particles of clay and starch coating make it look very rough, particularly on the top side.

In neither the bond nor the kraft does fibrillar material appear to play a



BEFORE COUCH

AFTER COUCH



AFTER FIRST PRESS

AFTER SECOND



PRESS

DURING DRYING

BEFORE



CALENDER

REEL

Changes on pressing and drying

very significant part in bonding, except in the immediate neighbourhood of fibre crossings. Most of the fine material appears to collapse on to fibres before compaction has taken place. The fibres are quite broad and fairly flat, but are forced into close contact at crossing points in pressing.



Fig. 13—Clay particles and rosin flakes attached to fibres on the top side of the bond web after the first press

Fig. 14—A film of starch covers the fibres and filler obscuring details of the structure: top side of the bond sheet at the reel

Newsprint

THE machine diagram and data are shown in Table 3 and the scanning electron micrographs in Fig. 15. Sheet substance was $32 \text{ lb}/3 000 \text{ ft}^2$. The



appearance of these micrographs is very different from the others, owing to the far larger quantity of fine material that is visible. Once more, there is much more fine material on the top side than on the wire side. The fine material in these pictures appears quite different from that in the other two grades of paper; there are many more fibre fragments and quite large, thin lamellae.

The structure of the web is compacted significantly on passage through the machine and the fibres collapse to some extent, but not so completely as in the other grades of paper. The stiff groundwood fibres, which have structural support from the lignin, do not collapse very readily on removal of water. Under the influence of pressure, however, they can bend to conform with each other and viscoelastic deformation can be seen. The chemical pulp fibres are much more flexible and ribbon-like.



Fig. 16—Adhesion of secondary wall fragments to a fibre surface: newsprint after the first press

Fig. 17—A large lamella, which spans several fibres, bonds securely to an uncollapsed groundwood fibre: the thin fibril on the left indicates post-bonding shrinkage of the underlying surfaces

As noted in previous studies,⁽²⁾ the fine material appears to play quite a large role in the adhesion of the web structure. The fibre débris sticks well to fibres (Fig. 16). Large quantities of this material can be seen and fragments may be continuous over several fibres. Although their cross-sections are very small, they are numerous and have very large contact areas in the bonded portions. Their fibrillar structure can usually be detected. A 'skin' adhering to a fibre is shown in Fig. 17. That shrinkage has occurred in drying can be seen from the small fibril bonded to both elements.

In passing through the dryers, most of the fibres collapsed to some extent, although some of the large groundwood fibres retained much of their large cross-section. Many of the fibres showed the collapsed lumen with raised edges typical of normal drying.

Before the calender stack, plastic deformations such as that seen at the presses on the other grades were not common. Passing through the stack, the newsprint was calendered to a much higher finish. Most of the fibres had obviously been forcibly compressed and deformed, the fibres and fibre fragments often losing their identity. In contrast with the smoother areas, particularly on the top side, the wire side had frequent rough areas where the fibres had not been greatly deformed. If mechanical pressure was not evident, close contact and bonding between fibres did not occur, most of the bonding being due to the fine material.

Discussion and conclusions

MERCHANT⁽⁵⁾ has shown that freeze-drying is not so effective as solvent exchange drying in preventing bonding and it is known that in small capillaries the freezing point is depressed. It is impossible to determine from pictures alone how much water actually froze and was sublimed, but the differences between specimens indicate that many of the gross aspects of the wet state structure are preserved. Some of the fibrils in the wet sheets are bonded and longitudinal wrinkling can be seen. It is probable that some of these phenomena are artefacts of freeze drying and that in the smaller capillaries the water is not being removed by sublimation. Within the limitations of the freeze-drying method, it is believed that the inferences made in this study are valid.

The change in the appearance of fibre surfaces as the solids content increased from 8 per cent to about 30 per cent can be explained on the basis of removal of free water from the surface of the fibres. Robertson⁽¹¹⁾ has shown that the critical solids content for pulps in an unbeaten condition is usually between 20 and 30 per cent and will be less if beaten. Thus, loose fibrils and lamellae would collapse back on to the surface of the parent fibre or make contact with a close neighbour as the solids content for each of the commercial grades studied is probably fairly close to 20 per cent. Most of the fibrils have collapsed on to surfaces after passage through the first press.

Progressive compaction of the sheet structure at each position of water removal can be seen for all grades. The differences between the top and wire sides of the web were striking in all the commercial samples. The quantity of fines and filler is much less on the wire side at the wet end and the difference in appearance persists to the finished paper, as was shown for several grades of paper by Emerton *et al.*⁽¹²⁾

The removal of water by pressing and the action of the calenders result in plastic deformation of the fibres and a blending of fibre material, so that the identity of the fibres is not always clearly defined.

No clear indication of thermal melting in the dryers could be seen. Increased conformability of fibrils could be explained by water removal alone, but the possible melting of the fibres and fragments was not sought specifically at high magnification.

For chemical pulp fibres, the contact area available for bonding is a function of their conformability. Beaten, low-yield fibres will collapse to very thin ribbons on drying and large interfibre contact areas are possible even without the assistance of pressing. In fairly lightly beaten or unbeaten and higher yield pulps, the fibres are much more rigid and less conformable and the fine material probably plays an increased role in bonding. At the same time, the influence of pressing is also greater⁽¹³⁾ by forcing fibres into close contact and ensuring large contact areas by deforming the fibres viscoelastically wherever pressures are sufficient to cause yielding of the fibre material.

Before pressing, as the free water between the fibres has been removed, fibrils and fragments have been drawn close to neighbouring surfaces. The expanded network reduces the probability of the fine material contacting more than one fibre. This suggests that, as the network is compacted and the web becomes reflooded on compression at a press nip (perhaps also at the presser roll), the fibrils may be resuspended and increase the chances of forming short, strong links in the web structure.

Acknowledgements

It is a pleasure to acknowledge the co-operation and assistance of the management and personnel of Domtar Pulp and Paper, East Angus, P.Q.; Domtar Howard Smith, Cornwall, Ont.; James Maclaren Co. Ltd., Buckingham, P.Q., Canada in obtaining the papermachine web samples.

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Discussion

Dr A. A. Robertson—This is a remarkable series of electron micrographs, even more so viewed stereoscopically. They provide a pictorial record of the consolidation process, thus with illustrations of a great many of the phenomena that have so far been inferred rather than directly observed.

I recognise that this is so for my own paper. Because of the importance of this record, we should carefully consider the reliability of the method. Pye has suggested in his paper that there are some distortions in the fibres because of the freeze-drying technique used and a certain amount of collapse, which Stone also referred to. This is apparently not too serious and one sees expanded fibres in specimens taken from very early in the papermachine.

There is one other possible source of unreliability about which I would like to ask. The samples from the couch or the presses are taken while the paper is being pressed either by surface tension forces of the water or by the press itself. They are then frozen and, under these conditions, one has a stressed specimen being frozen—the stresses are literally frozen in. What happens to these stresses when the ice is sublimed from the freeze-dried specimen? Does the caliper of the specimen decrease, increase or remain the same during the process of preparation for observation?

Mr I. T. Pye—We acknowledge that there are such stresses within the structure of the specimens. We regard laboratory tests as appropriate for study of fundamental fibre and web behaviour. These experiments were designed specifically to compare commercial operation with the laboratory tests. There is evidence (as in Fig. 4) that a fibre has been pressed into another and that separation has occurred afterwards. Whether or not this is due to shrinkage or stress relaxation, it is very difficult to tell: both undoubtedly occur to some small extent. If this is recognised when viewing the pictures, we consider the differences between specimens to be significant and valuable. The reduction of surface area on freeze-drying reported by Stone occurs within the cell wall. The external configuration of the fibre is not affected too greatly.

The thickness decreases noted on freeze-drying the wet specimens were not measured accurately, but were of the order of only a few per cent.

Discussion

Mrs R. Marton—With S. Alexander, we recently did some work on the effect of very strong wet pressing and severe beating on the properties of fibres. We knew in advance (as this had been stressed at previous meetings here and in Oxford) that the density is the most fundamental property of the sheet, also that there is a linear relationship between breaking length and density. However, when we wet press sheets to very high densities (over 0.9) or if we beat a pulp to very low freeness levels and later press the wet sheets, we obtain very strange curves for the correlation of breaking length and density. Each curve has a marked maximum. This phenomenon was repeatedly observed on different types of pulp (Fig. C).



We observed also that, as the yield of the pulp decreased or when hardwood pulps were used, the location of the maximum moved towards a lower density. These results confirm exactly what Page and Pye reported about fibre collapse on pressing. Why is it that, after reaching a certain density, the curve declines so sharply? The explanation is that the ascending branch of the curve represents the region in which fibres are collapsed. As the pressure is increased or the beating extended, the fibre collapses more and more. After reaching a maximum that is characteristic for every type of pulp, however, the fibre walls are crushed, the binding of the fibrils is disorganised, the fibre structure is loosened, there is no more cohesion between the fibrils and, despite the increase in density, no more strength is achieved and the breaking length of the sheet decreases. Micrographs made in polarised light show that the fibres are collapsed as a result of moderate pressing and beating, but are crushed and internally disorganised with stronger action. The changes in the structure with fibres treated with cupriethylenediamine (a very strong swelling agent) illustrates this point even better. It shows the sites where the fibres are damaged or weakened. The crushing of fibres on heavy pressing also occurs in practice, because a sheet of paper is not perfectly flat and the elevated particles in the sheet profile are exposed to much larger pressure concentrations than are the deeper spots.