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THE EFFECTS OF BEATING ON INDIVIDUAL FIBRES

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Summary

The effects of beating on the individual fibres are divided into four main groups — swelling, fibrillation, cutting and the removal of the primary wall.

Swelling takes place in the amorphous hydrophilic hemicellulosic interfibrillar material. It involves a loosening of the fibre structure. The fibre wall is plasticised by the imbibed water and the fibre becomes more flexible. In an advanced state of swelling, the hemicellulose molecules are supposed to be partially dissolved in the surrounding water.

Fibrillation is caused not only by the direct action of the bars, but also by other treatments such as simple agitation or ultrasonic radiation. It is pointed out that a certain amount of swelling is needed to allow fibrillation and that fibrillation may be regarded as a natural consequence of progressive fibre swelling. Fibrillation first takes place after rather a long beating time.

A method is described by which it is possible to determine quantitatively the amount of primary wall on the surface of the fibres. The primary wall is torn off rapidly at the very beginning of the beating process and it is shown that the fibre surface free from primary wall can be correlated with the tensile strength.

When beating wood fibres, the removal of the primary wall and the

swelling, in as much as it makes the fibre more flexible, seem to be the main effects in improving paper strength (fibre-to-fibre bonds), whereas fibrillation is of less or of no importance.

IN his very well-known chapter 'Properties and Treatment of Pulp for Paper' in Ott, Spurlin and Grafflin's book *Cellulose and Cellulose Derivatives*[1] d'A. Clark mentions six effects of beating on the fibres — swelling, rubbing, cutting, splitting, bruising and deformation. In the following paper, I should like to treat the subject entrusted to me at this symposium more or less along the same lines.

Beating does not in principle differ from other kinds of destructive mechanical action and, considering its action, the structure of the material in question is, of course, of the greatest importance. The structure of cellulose fibres has therefore already been carefully considered at this symposium: however, before going over to the main problems of my topic, I should like to summarise those aspects of the wood fibre structure that in my opinion ought to be kept in mind for a correct understanding of the effects of b eating on the individual fibres.

1. The microfibrils* of the primary wall form a cross-layered structure resembling plywood. As a consequence, the primary wall is isotropic from a mechanical point of view; furthermore, it will not fibrillate, it is brittle and behaves as a fragile skin and it will not swell.
2. The parallel orientation of the fibrils in the secondary wall, both in the outer and central layers, makes the fibre as a whole typically anisotropic. The fibre is strong in the longitudinal direction, but rather weak laterally. Swelling therefore takes place laterally and mechanical action causes fibrillation.
3. The interfibrillar material consists of hemicelluloses and is of an amorphous nature. Due to the strongly hydrophilic nature of the hemicelluloses, this material swells easily in water. The swelling behaviour can be classified as typical limited swelling.

During beating, the fibre is not only exposed to the mechanical action of the machine, but also to the action of stresses by the water in which it is suspended. As Barkas has stressed at the symposium on beating in 1951[2], the water is rapidly accelerated when entering the gap and retarded when

* The author prefers the term *cellulose strings*, but microfibrils is used here for uniformity amongst the symposium papers.

leaving. This results in stresses in the aqueous medium, stresses that must in turn react on every fibre in the suspension. The forces acting on the fibre during beating will therefore vary within a wide range, from weak ones, when the velocity or acceleration gradient of the water around the fibre is low, through stronger ones in regions of turbulent or vortex flow and cavitation, to the strongest for those fibres touched by the bars. The effect on the fibre will naturally depend on the intensity of these forces.

Swelling

A general feature of beaten fibres is that they are swollen. Swelling occurs at the beginning of beating and proceeds throughout the whole process. Measured in the cross-direction of the fibre, the dimensional swelling may be of the order of 20 — 30 per cent. without fibre damage. When the internal fibre structure is loosened, the fibres swell rapidly to twice their original diameter.

Swelling takes place in the hemicellulosic interfibrillar substance. It must be kept in mind that isolated hemicellulose — for instance, the gamma-cellulose fraction after an alpha-, beta- and gamma-cellulose separation — is water soluble. There, it must be expected that the native hemicellulose has not only a strong tendency to swell, but also to dissolve in water. As no such dissolution takes place, however, the swelling pressure must be restricted by other forces. In the hemicellulosic material itself, partial crystallisation and strong hydrogen bonds will limit the swelling. When the gel expands, the disordered chain molecules will be stretched until they prevent further swelling. The presence of lignin will act in the same way. Finally, the primary wall and the spiral outer layer of the secondary wall will restrain the swelling pressure of the interfibrillar material.

Obviously, when speaking of the bonds holding the whole fibre together as a unit, we have to consider a spectrum of bonds of quite different strength, from the strength of hydrogen bonds to the strength of the microfibrils or still larger units of the fibre wall. Any mechanical action on the fibre of such intensity that these bonds are broken will therefore cause further swelling. It seems likely that only rather weak forces are needed to break the bonds in the swollen gel, which already are under stress. There are no restrictions concerning the kind of mechanical action needed to perform swelling. The forces of any applied action, whether they be classified as stressing, pressing, bending, flexing, curling, bruising, kneading, rubbing, twisting, crushing etc., will be absorbed by the fibre, resulting in the breakage of internal bonds and thus in swelling. As shown by Steenberg,[3] some cracking of the fibre

structure is already produced in an early stage of beating which is not visible in the microscope but can be demonstrated by swelling the fibre in concentrated phosphoric acid.

Cellulose fibres also swell on ultrasonic treatment. Simpson and Mason showed that the effects obtained are similar to those of normal beating.[4] Typical features are shown in Fig. 1. Some fibres may swell appreciably and ballooning occurs similar to that observed in strong swelling liquids (cuprammonium, phosphoric acid, etc.). This behaviour demonstrates that, even in water, there is a significant swelling pressure inside the fibre.[5]

When speaking about swelling, attention is mostly paid to the dimensional increase of the fibre. The importance of the continuous swelling of the hemicellulosic gel must, however, not be overlooked. Even if it is difficult to follow the degree of swelling on highly beaten fibres, because of fibre destruction, there is no reason for not considering the gel swelling to increase through the beating process. The mechanical action will continuously break the restricting bonds in the hemicellulosic gel, which will swell more and more; when taking up water, its molecules will be more and more loosened from each other and, finally, as Campbell[6] pointed out, a state will be reached in which the molecules, though still anchored in the gel, possess a certain mobility in the surrounding water and thus can be considered to form a colloidal solution. This swelling is a part of the phenomena that among technical people is called *hydration.*

From a papermaking point of view, it seems quite likely that swollen hemicelluloses on the surface of the fibre will take an active part in the formation of bonds between the fibres in the paper sheet. It will simply act as a glue. It also seems likely that the more this surface material is swollen and partly dissolved in the water, the greater is the chance for bonding, the stronger are the surface tension forces when drying (Campbell;[6] Barkas and Hallan[7]) and the larger will be the areas of contact.

In the native fibre, the cellulose strings are held together laterally by the interfibrillar substance. When this becomes swollen, the bonding capacity is obviously decreased. Thus, swelling of the interfibrillar material will involve a loosening of the fibre structure and prepare the fibre for more far-reaching destruction. This loosening of the structure together with the plasticising effect of the imbibed water will influence the stiffness of the fibre and make it more flexible, and this flexibility will doubtless involve an increasing contact area between the fibres when the sheet is dried. (The effect of beating on fibre flexibility is treated in Mason's paper).

The importance of the fibre swelling as a result of beating has been given prominence in papers by Steenberg,[3] Lewis[8] and Gallay[9] and special attention has been paid to the increased flexibility and internal lubrication of the fibre walls as a central feature of the beating process in a recent paper by Emerton.[11]

The actual measurement of the degree of swelling has been found extremely difficult and, although many methods have been proposed, none is as yet generally accepted. All of them show that swelling increases with beating. It has, however, not been possible to correlate the degree of swelling with the properties of the paper, especially the strength properties, in such a way as to demonstrate that swelling, although it is a necessary condition to obtain paper strength, is the most important result or one of the most essential features of beating from a fibre-bonding point of view.

Among the different approaches for measuring the degree of swelling, the following may be mentioned. At the same time, it should be kept in mind that the ordinary freeness testers of different design, to some extent, also measure the degree of swelling of the fibre suspension, even if other properties influence the result simultaneously.

Jayme introduced the centrifugation method,[12-14] according to which the water retained by the pulp after centrifuging it under controlled conditions is taken as a measure of the degree of swelling. Mason and co-workers[15, 16] worked out the water permeability method, in which a pad is formed of the pulp in water and the rate of viscous flow is measured at different pad consistencies. By using a modified Kozeny-Carman equation, the *effective fibre volume* and *specific surface* can be calculated. In contrast to these methods, in which the degree of swelling is measured in a pure physical way, attempts have also been made to measure the amount of *chemically bound*

TABLE 1

The influence of beating on the amount of water retained after centrifuging (Quellwert), according to Jayme[20]

Unbleached sulphite pulp beaten in the Jokro mill		
Beating time, min.	*Freeness,* °S.R.	*Retained water, g./g. O.D. fibre*
0	16	1.870
10	22	2.231
15	30.5	2.375
22	43.5	2.554
32	56	2.689
40	72.5	2.870

water in the swollen fibre. One way has been to use the thiosulphate method of Champetier[17, 18] and another the freezing method of Magne, Portas and Wakeham.[19] Some results of these four methods are given in Tables 1 — 3.

As swelling takes place in the hemicellulosic interfibrillar substance, it seems quite natural that it depends on the pulp quality. Hemicellulose-rich, strong sulphite pulps are known to swell easily. The softer the pulp (that is, the more the hemicelluloses have been hydrolysed during the sulphite cook), the lower is the tendency to swell.[5, 18] High lignin content seems to restrict swelling and therefore bleaching may improve the swelling power

TABLE 2

The influence of beating on the 'effective fibre volume' measured by the water permeability method according to Mason[16]

Slush sulphite pulp beaten in the Valley beater

Beating time, min.	*Freeness (Canadian standard), ml.*	*Effective fibre volume, ml./g. O.D. fibre*
0	669	4.40
7	559	4.90
11	498	4.86
15	416	5.25
18	320	5.12
21	242	5.15

TABLE 3

The influence of beating on the amount of 'bound water' according to Bhargava, Giertz and Wiklund[21]

The amount of 'bound water' has been estimated according to the thiosulphate method (2 per cent. $Na_2S_2O_3$ solution) and the freezing method, in the latter case calculated as the amount of non-freezing water at — 10°C

Strong unbleached sulphite pulp (slush) beaten in the Lampen mill

Revolutions	*Freeness, °S.R.*	*'Bound water', g./g. O.D. fibre*	
		Thiosulphate method	*Freezing method*
0	14	0.31	0.352
1 500	20	0.34	0.361
3 000	24	0.37	0.371
7 000	37	0.42	0.386
15 000	59	0.50	0.415

of the fibre.[20] Sulphate pulps swell less than sulphite pulps,[18, 20] a fact that has been attributed to certain bonds in the sulphate fibre, postulated to have been formed during cooking.[22] It is a well-known fact that drying reduces the tendency to swell appreciably.

Fibrillation

When beating proceeds or when the fibre happens to be exposed to extra strong mechanical action — which in reality is mostly one and the same thing — the fibre splits in the longitudinal direction and forms fibrils. These fibrils, which are visible under ordinary magnification in a light microscope, consist of longitudinal sections of the secondary wall and bundles of microfibrils. These are the fibrils as known to the papermaker for a very long time. Today, however, it is also known from investigations with the electron microscope that single microfibrils and thin bundles consisting of some few microfibrils are separated at the same time, a fact that could be expected. The largest part of the fibrils in technically beaten pulps seems to originate from the outer layer of the secondary wall. Some fibrils may also be ruptured from the fibre wall and, together with fragments of the primary wall, form debris in the water suspension.

Among technical people, it is a generally accepted opinion that splitting and fibrillation occurs when the fibre is more or less crushed in the gap of the beater and that the high pressure between the bars is a prerequisite for obtaining fibrillation. This is, however, not the case. Other kinds of mechanical treatment also cause fibrillation — for instance, simple agitation with a propeller or ultrasonic treatment. In the latter case, no mechanical action comparable with that of the bars in the beater takes place, but the fibres are easily fibrillated in any case.[4, 5, 23] Such fibrillation by ultrasonic treatment is shown in Fig. 2.

When discussing the mechanism of fibrillation, the swelling pressure of the interfibrillar substance must be kept in mind. When enough bonds are broken, this internal pressure will expand the fibre laterally and split it longitudinally during the formation of fibrils. The fibre more or less explodes by itself.

The fibre ruptures between the strings in the interfibrillar material. As was mentioned earlier, the more this material is swollen and thus softened, the more easily will the rupture take place. It seems, therefore, likely that a certain, and perhaps rather advanced, swelling is needed to allow fibrillation. When the swollen fibre is exposed to extra strong forces, it will easily split. It may, however, also be possible that fibrillation should only be looked upon as a natural consequence of proceeding fibre swelling. In this connection,

it may be remembered that fibrillation first appears after rather a long period of beating and that beating in non-swelling liquors involves very little or no fibrillation, but mainly cutting.[24] The typical feature of fibrillation obtained with ultrasonic treatment may also be taken as a criterion that fibrillation should be regarded as a natural consequence of swelling.

As a consequence of the rupture within the interfibrillar areas, the separated fibrils and microfibrils will be covered with a thin layer of hemicellulose, which can be expected to swell when liberated from its bonded state in the fibre. Fibrillation will therefore not only increase the external fibre surface, but it will also liberate internal hemicellulose, making it available for fibre bonding.

It is quite natural that papermakers have always paid the greatest attention to the fibrillation of the fibre when trying to understand the effect of beating and fibrillation is also integrated in the wide-ranging expression *hydration*. It is striking, however, that up to now it has not been possible to show a convincing correlation between degree of fibrillation and paper strength. It is difficult of course to measure the degree of fibrillation quantitatively. When estimated in the microscope, fibrillation first appears after a rather long period of beating — in general, when the wetness has reached 30° S.R. or higher. Strength properties, however, are developed from the very beginning of beating and have already reached a high value before 30° S.R. At the Fibre Chemistry Section of the Swedish Forest Products Research Laboratory, a serious attempt was made some years ago to investigate quantitatively the influence of fibrillation on paper strength.[25] Keeping in mind the difficulty in evaluating this kind of observation in the microscope or on the photomicrographs, the conclusion was drawn that the strength of paper could not be related to the amount of fibrillation or to the skin-like formations in the angle between two crossed fibres in the paper, formations which obviously are formed by the fibrils. In dense papers, however, fibrils and debris certainly play an important role.

The tendency of the fibre to fibrillate depends on the chemical composition and pulp quality. In general, however, variations in fibrillation closely follow those of swelling. There is no method of measuring fibrillation as an isolated phenomenon. Many suggestions have been made, however, to measure the external fibre surface and these have recently been reviewed by Emerton.[26]

Cutting

If the strain in the fibre is strong enough, it will break or be bent and deformed. The former phenomenon is generally called cutting, because it is

believed that the bars cut the fibre like scissors. It is questionable, however, if cutting occurs only as a result of shear forces applied on the fibre by direct action of the bars or if the strain in the fibre, when the velocity or acceleration gradient of the surrounding water is high, can be strong enough to break the fibre. Judging from Rance and co-workers' studies on the beating process, the latter seems also to be the case, especially when beating soft pulps under hard setting conditions.[27]

A general trend seems to be that strong pulps, such as strong sulphites and kraft pulps, are preferentially fibrillated and not so much shortened during beating, whereas soft pulps are mostly cut and not very much fibrillated. The explanation might be that, of all the forces applied on the fibres under certain beating conditions when beating strong pulps, the strongest ones are enough to cause fibrillation but not strong enough to rupture the whole fibre, whereas the same forces when beating a soft pulp, structurally weakened by hydrolysis during the sulphite cook, are strong enough to cut most of the fibres.

The primary wall

The foregoing discussion has only been devoted to the effect of beating on the fibre proper and nothing has been said about the primary wall. It is obvious that the surface properties of the fibre and thus the existence of the primary wall and its removal during beating is of the greatest interest to the papermaker. As the primary wall is extremely thin, however, it is very difficult to detect its presence in the microscope and therefore very little is known about the fate of the primary wall during beating.

It is quite natural that the primary wall, being very thin and brittle, is removed by beating and this has also been proved by, for instance, d'A. Clark[1, 28] and Bucher and Widerkehr.[29] A general impression is that the primary wall is stripped off easily in the very beginning of beating.[1, 28] It has been shown that the rubbing action of the beater bars is not necessarily needed to remove it. It is just as readily torn off by ultrasonic treatment, for instance.[5] Up to now, however, no quantitative measurements regarding its removal have been published.

Using the staining technique of Bucher,[30] it is possible to identify the primary wall in a simple way, making quantitative investigations possible.[31, 37]

The fibre is first stained with Victoria blue and then left to swell in a diluted cuprammonium solution under observation in the microscope. In the alkaline solution, the blue stain is changed to a brown pigment and, as the primary wall is much more heavily stained than the secondary wall, it is

easily recognisable. Using a suitably diluted cuprammonium solution (the solution used in CCA 16 diluted 1 : 1 or 1 : 2), the secondary wall — both the outer and central layers — will swell and dissolve completely, whereas the primary wall, probably because of its crossed fibrillar texture, will neither swell nor dissolve. It is left on the slide as a continuous skin or as fragments. This is shown in Fig. 3. If the primary wall has been removed, the fibre dissolves completely, leaving no residue.

There can be no doubt that the stained skins and fragments observed originate from the primary wall.* On some, residues from the middle lamella can be detected (Fig. 3*b*). Furthermore, they cannot originate from the outer layer of the secondary wall, because this can also be identified with certainty (Fig. 3*d* and *e*). The outer layer of the secondary wall does not take up the stain as heavily as does the primary wall and dissolves completely, together with the main part of the secondary wall.

By following the swelling and dissolving process carefully, it is possible to estimate the amount of primary wall that covered the fibre and the observations can easily be classified in the following four groups —

1. Fibres covered all over with primary wall.
2. Fibres mainly covered with primary wall or with distinct parts of it.
3. Only fragments of the primary wall left.
4. Fibres without primary wall.

Using this technique, it was found that unbleached, strong pulp fibres — both sulphite and sulphate — in general, are covered with the primary wall, whereas in the soft sulphite pulps and rayon pulps many fibres have partly or totally lost it. During bleaching, a partial removal also takes place.

The effect of beating on the primary wall is shown in Table 4. A bleached sulphite pulp of medium strength and an ordinary unbleached kraft pulp, both dried, were beaten in the PFI beater, about a hundred fibres examined in the microscope at different beating times and the observations classified in the above four groups.

Typical for the sulphite pulp is that the primary wall is removed very quickly. Even after 500 revolutions, which means very little beating (16°S.R.), more than half of the fibres has lost parts of the primary wall and, after 2000 revolutions (22° S.R.), there are practically no fibres that have their primary wall undamaged. At the same time, the tensile strength of the paper increases.

* At the Cambridge symposium, the author queried whether these skins might be identical with the membranes observed by Emerton. Subsequent discussion has made it quite clear that this is not so: they are the primary wall and the outer layer of the secondary wall, respectively.

After 6000 revolutions, which corresponds to optimum tensile strength, the majority of the fibres have lost their primary wall completely and only fragments of the rest can be detected.

The same removal of the primary wall takes place when beating the kraft pulp, but the removal in this case goes much more slowly, which corresponds to the well-known slow increase in tensile strength compared with sulphite pulps. The same observation was made by d'A. Clark.[1]

An attempt has been made to calculate an average figure from these grouping data, giving a quantitative value for the fibre surface area from

TABLE 4

The removal of the primary wall during beating, according to Giertz and Nisser[37]

Revolutions in the PFI mill	*Number of fibres in group*				*Exposed fibre surface, per cent.*	*Tensile strength, m.*
	1	2	3	4		
Bleached sulphite						
0	84	12	4	0	8	1 200
500	35	42	21	1	38	2 850
1 000	5	72	19	3	50	3 900
2 000	4	24	28	44	79	5 800
4 000	0	4	33	63	94	7 200
6 000	0	2	25	73	96	7 800
Unbleached sulphate						
0	100	0	0	0	0	1 500
1 000	92	8	0	0	0	3 650
5 000	43	36	20	0	32	6 250
16 000	15	31	50	6	61	8 400
32 000	7	25	45	23	74	10 000
64 000	2	7	29	62	91	11 350

which the primary wall has been ruptured as a percentage of the total fibre surface. This area will be called *the exposed surface* in what follows.

It can be said immediately that the exposed surface for group *1* is 0 and, for group *4*, 100 per cent. The border between groups *2* and *3* was estimated to lie at about 80 per cent. exposed surface. To a first approximation, the average for group *4* is therefore 40 per cent. exposed surface and, for group *3*, 90 per cent. Based on these estimated average values for each group, the weighed mean for the total amount of fibres can be calculated. This exposed surface value is also given in Table 4 and it is believed to give a fairly correct idea about the amount of primary wall on the fibres.

Using these exposed surface values, it is easier to follow the effect of beating on the removal of the primary wall. If the percentage of exposed surface is plotted against the logarithm of revolutions of the beater, straight lines are obtained. In Fig. 4, this relationship is given for the kraft and sulphite pulps mentioned above. Values for the corresponding unbleached sulphite (slush) and the slush bleached pulp are also included. From this diagram, it can be seen that the primary wall is much more quickly torn from the sulphite pulps than from the kraft pulp and, among the sulphite pulps, the primary wall of the dried pulp is more resistant than that of the corresponding slush pulp. That bleaching weakens the primary wall has already been mentioned.

The fact that straight lines are obtained in Fig. 4 is in accordance with the theory for crushing and milling. It should be kept in mind that the primary wall can only be torn off once. At the beginning of beating, the chance is rather high that some fibre passes the zone of beating in such a way that the primary wall is rended. As beating proceeds, however, the chance is less that some of those fibres still having the primary wall intact will come into a suitable position. The removal of the primary wall must therefore decrease exponentially. The fact that straight lines are obtained from the experimental data in Fig. 4 may be taken as a criterion that the estimations made and the way of calculating the exposed surface are likely to be correct.

It can be easily imagined that the removal of the primary wall could influence the bonding capacity of the fibre drastically. This possibility was stressed by d'A. Clark[28] and is generally pointed out when the paper bonding capacity of the fibre is discussed (*cf.* Jayme,[32] Steenberg,[3] Lewis,[8] Wardrop and Dadswell [33]).

Very little is known about the chemical composition of the primary wall in technical pulps and especially about its hemicellulose content. In ordinary sulphite and sulphate pulps, it seems probable that the surface of the fibre is rather free from hemicellulose because of its exposed location and accessibility to the cooking liquor. This assumption is confirmed by a limited number of published electron micrographs showing the surface of technical pulp (*cf.* Jayme and Hunger[34] and Svensson[35]). Furthermore, if some hemicellulose has been left on the surface, most likely it has been widely depolymerised for the same reasons and thus lost its stickiness and binding capacity.

If the hypothesis is right that a certain amount and certain type of hemicellulose is needed to form strong fibre-to-fibre bonds, then chemical

pulps taken directly from the digester must be expected to possess rather poor papermaking properties. As soon as the primary wall is torn off, however, a fresh hemicellulose-rich layer will be exposed and this hemicellulose will swell when liberated from its bonded state in the outer layer of the secondary wall as described earlier. The primary wall of a papermaking pulp fibre can therefore be likened to the protecting cover of sticking plaster. Furthermore, as pointed out and shown by d'A. Clark,[1, 28] when the primary wall is torn off, the fibrils of the outer layer of the secondary wall may be split by the mechanical action of the rupturing and a fuzzy fibrillation is obtained on the fibre surface. A correlation between the exposed surface and paper strength could therefore be expected.

d'A. Clark has shown that many properties of the paper, such as density, tensile strength and opacity, follow straight lines when plotted against the logarithm of beating time.[28] This relationship is quite clear if the beating is performed in the PFI beater. An example is given in Fig. 5.

Thus, if tensile strength is plotted against exposed surface, as in Fig. 6, straight lines must be obtained. This simple correlation between exposed fibre surface free from primary wall and developed strength seems quite natural and convincing, but it must seriously be questioned if this relation only happens to be a coincidence or if it really implies a matter of cause and effect. It must indeed be kept in mind that all other effects of beating are of the same destructive nature, involving rupture and breaking of bonds, thus also have to proceed exponentially. This holds for swelling and flexibility as well as for fibrillation and other effects discussed earlier. The development of paper strength during beating can therefore not be related with any certainty to the removal of the primary wall, despite the nice correlation in Fig. 6. On the other hand, it has been pointed out by d'A. Clark, the removal of the primary wall is the most obvious effect of beating that can explain the rapid increase in strength at the very beginning of beating.

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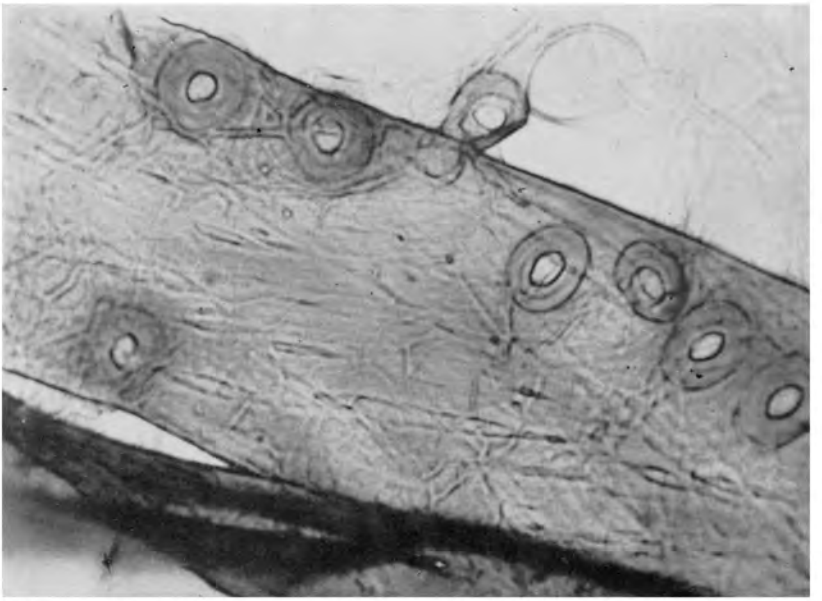

Fig. 1 — Ultrasonic treated spruce fibres, according to Algar and Giertz[5] — (*a*) Swollen springwood sulphite fibre

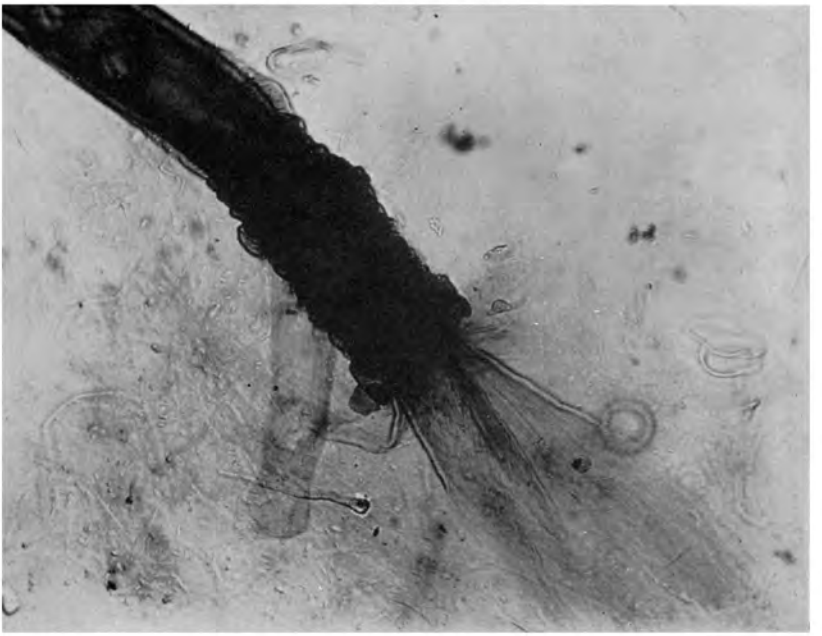

Fig. 1 — Ultrasonic treated spruce fibres, according to Algar and Gietz[5] — (*b*) Primary wall rolled up and intense swelling of the secondary wall (holocellulose)

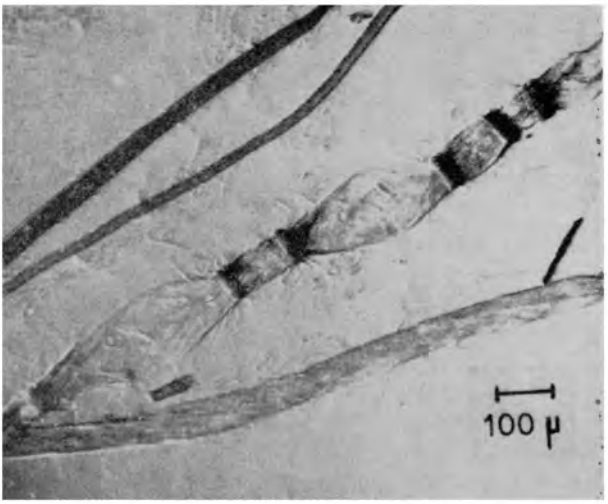

Fig. 1 — Ultrasonic treated spruce fibres, according to Algar and Giertz[5] — (*c*) Holocellulose fibre with balloon-like swelling

Fig. 2 — Fibrillation by ultrasonic treatment, according to Algar and Giertz[5] — (*b*) Soft sulphite pulp

Fig. 2 — Fibrillation by ultrasonic treatment, according to Algar and Giertz[5] — (*a*) Spruce sulphate fibre

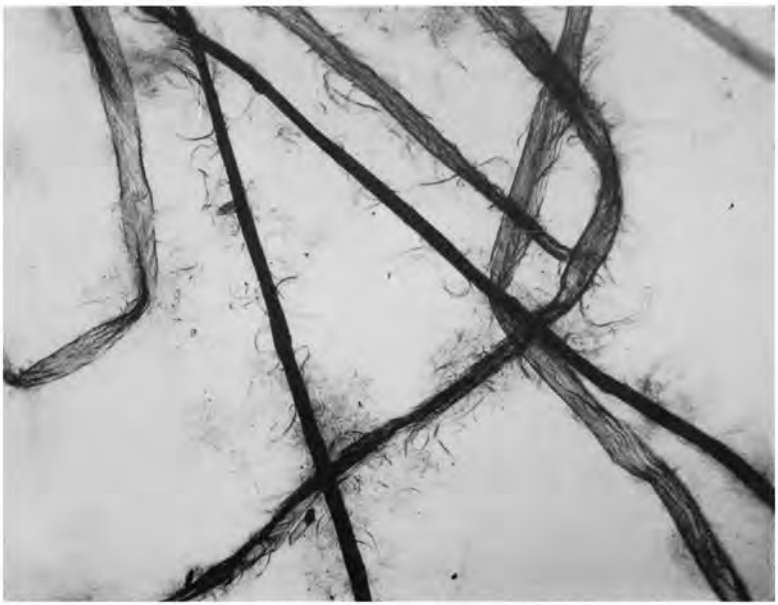

Fig. 2 — Fibrillation by ultrasonic treatment, according to Algar and Giertz[5] — (*c*) Kraft pulp

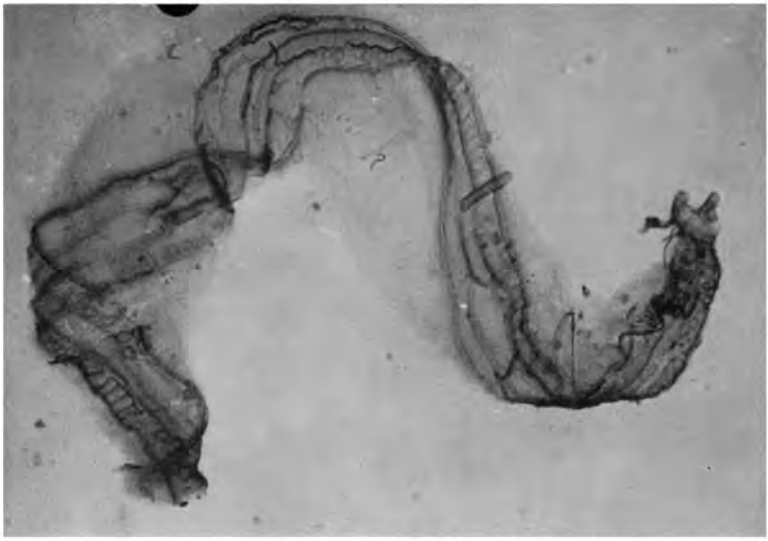

Fig. 3 — Swelling in cuprammonium solution of fibres stained with Victoria blue according to Dymling and Giertz[31] —

(*a*) Final stage of swelling — the primary wall has split longitudinally as a pea shell and the secondary wall is just about to dissolve (holocellulose fibre)

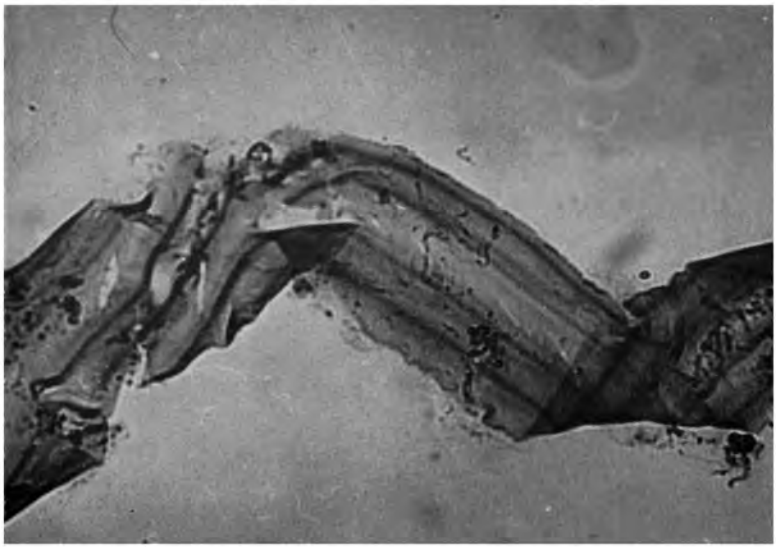

Fig. 3 — Swelling in cuprammonium solution of fibres stained with Victoria blue, according to Dymling and Giertz[31] —

(*b*) The primary wall of a strong sulphite fibre after the secondary wall has been dissolved completely — the four sides of the fibre are easily recognised — the longitudinal dark formations originate from the middle lamella in the corner between four adjacent fibres

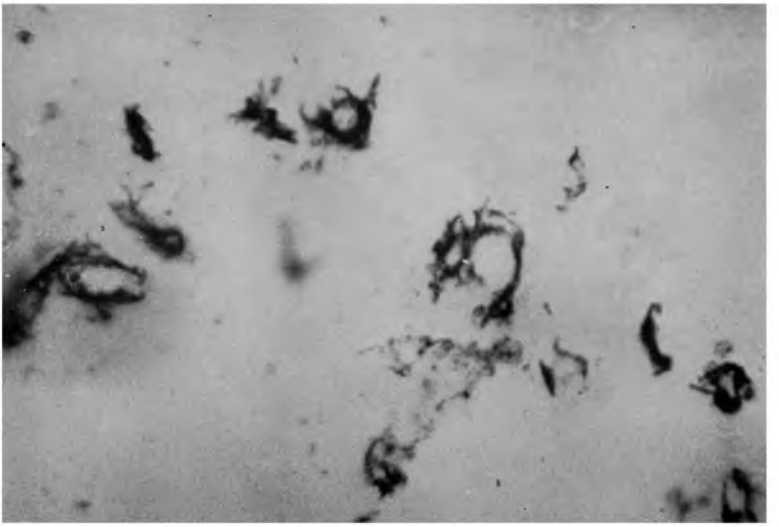

Fig. 3 — Swelling in cuprammonium solution of fibres stained with Victoria blue, according to Dymling and Giertz[31] —

(*c*) Fragments of the primary wall (soft sulphite)

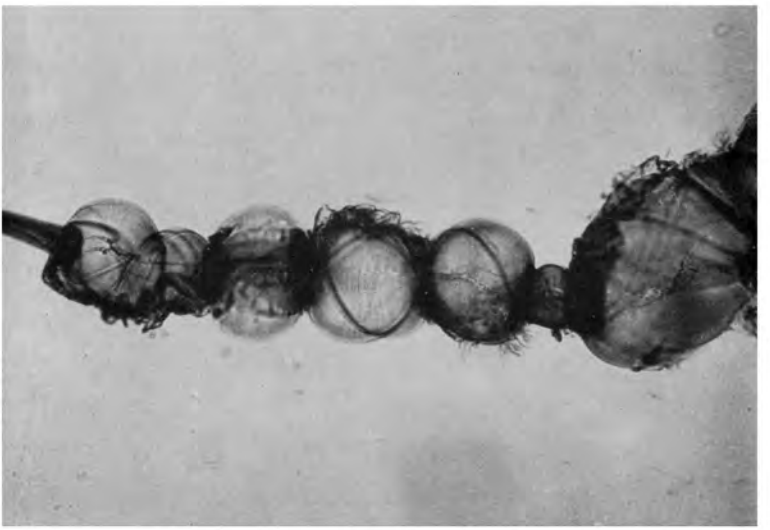

Fig. 3 — Swelling in cuprammonium solution of fibres stained with Victoria blue, according to Dymling and Giertz[31] —

(*d*) Ballooning — the stained primary wall can be seen as a broken skin around the fibre. The z-formed loops, which also constrain the fibre at the nodes, belong to the outer layer of the secondary wall and will dissolve in the cuprammonium solution.

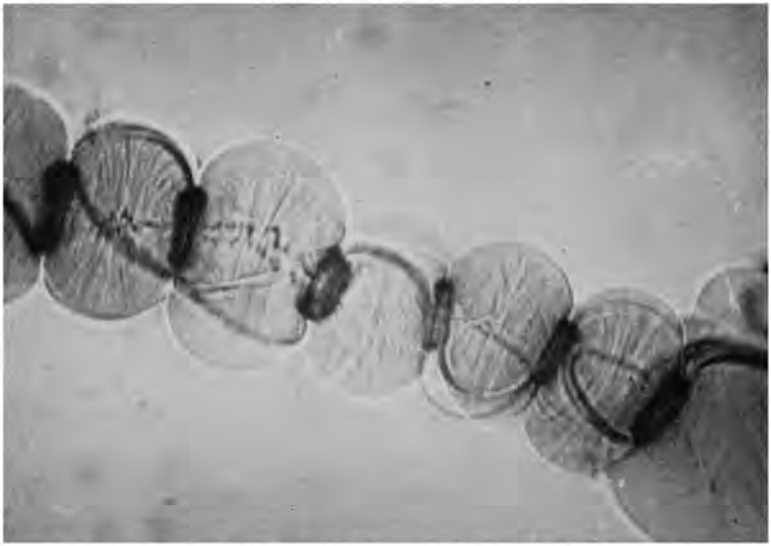

Fig. 3 — Swelling in cuprammonium solution of fibres stained with Victoria blue, according to Dymling and Giertz[31] —

(*e*) Beaten sulphite fibre without primary wall — the dark parts at the nodes are optical artefacts; when this fibre had dissolved, there was no remainder of the primary wall to be detected and it is clearly seen that ballooning is caused by the outer layer of the secondary wall, as pointed out by Wardrop and Dadswell,[36] not by the primary wall.

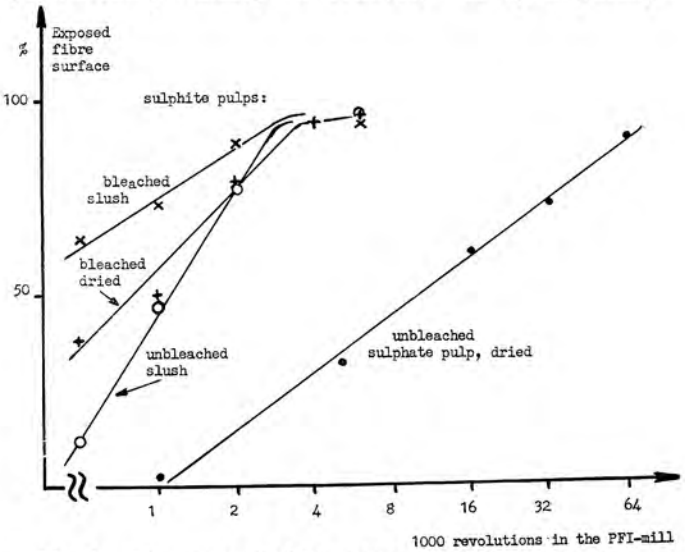

Fig. 4 — The removal of the primary wall during beating

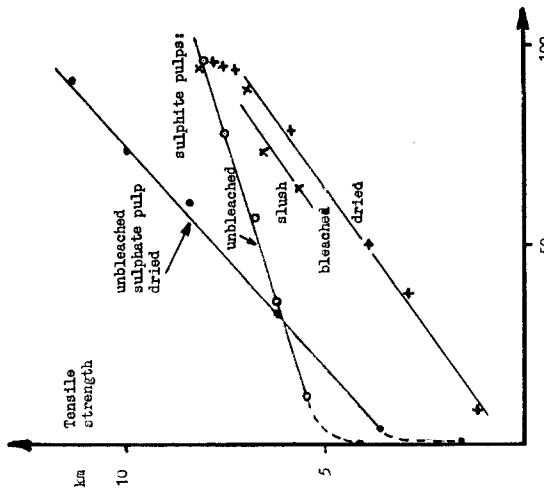

Fig. 6 — The correlation between tensile strength and 'exposed fibre surface'

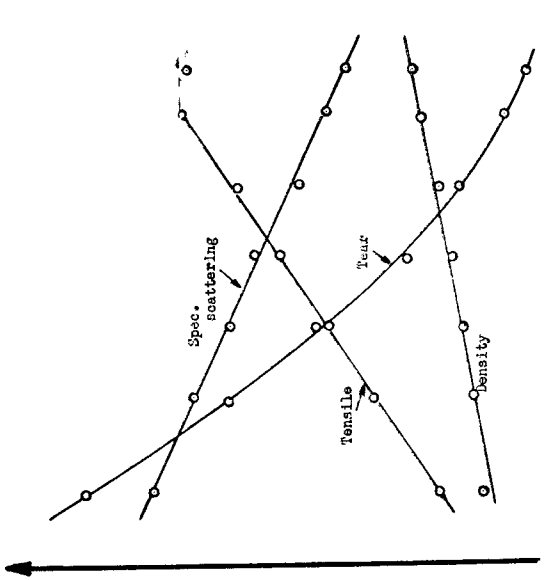

Fig. 5 — The effect of beating (PFI mill) with beating time plotted on a logarithmic scale

DISCUSSION

MR. G. HUNGER: Dr. Gallay, you said you had displaced the water in the sheets by different solutions — how did you prepare the sheet?

DR. W. GALLAY: The sheets were prepared from the pulp stock in normal fashion up to the couching stage. They were then removed from the blotters and immersed in alcohol, drained, immersed in ether and dried in a vacuum desiccator over calcium chloride.

MR. HUNGER: A sheet formed in water will have thin layers of water at its contact regions, where one fibre touches the other. This water is kept there by hydrogen bond forces and I doubt whether it might be possible to extract this water by the different organic solvents applied. It may be very interesting to have a sheet prepared by putting a wet pulp into the different solvents — that is, to have the pulp standing for one night in the first solution, this being removed the next day, then suspending the pulp in the next solution and so on, lastly, forming a sheet from the fibre suspension in the last organic solvent. I wonder if you would still get the high strength that you found then. A pulp totally surrounded by the organic solvent would not have such a high strength, I think.

DR. GALLAY: We considered the method you have proposed, but felt that there were objections that would detract from the general principle. Notably, a radical change might be expected in the mode of deposition of the fibres from suspension in the organic liquid. We therefore consider it preferable to use water as the medium of deposition. It is considered from our data that little difference exists between beaten and unbeaten sheets in so far as thoroughness of water removal is concerned. It should be remembered that a very small residual amount of water is always associated with cellulose and removal of this residue is virtually impossible without radical decomposition of the cellulose.

MR. L. G. COTTRALL: I found Dr. Gallay's paper very stimulating, indeed. He has given us some new ideas, coming as he does from the plastics industry fairly recently. Whether they are right or wrong, these ideas make us think on new lines.

When he carried out his test of removing water by solvents, did Dr. Gallay try pulp that had been beaten in a ball mill a very short time? Some years ago, there was much controversy over how we got such a very large

increase in strength in a Lampén ball mill by beating for only 2 – 3 min. and various people had different ideas. I think it was the start of the idea of internal fibrillation as distinct from external fibrillation, when the surface of the fibre or the body of the fibre itself is damaged (*Proc. Tech. Sect. P.M.A.*, 1932, **13** (1), 15), although I believe the term was originally due to W. B. Campbell. Really, I do not know whether we have at the present moment a satisfactory explanation of that very great increase in both tensile and bursting strengths with a very slight reduction in tearing strength. In the case of sulphate pulps, the tearing strength is in fact increased over this small range of beating.

Has Dr. Gallay tried testing sheets in that way and has he found that there is still that same great increase in strength when these sheets have been treated with alcohol in the ordinary way or that there is a great reduction in strength through that process?

DR. GALLAY: The direct answer to Mr. Cottrall's question is that we have been thus far using only the Valley beater in this work for strength development.

Certainly we must examine closely any unusual results obtained with specialised beating methods, since these must add to our knowledge of the potential resident in the fibres, if they are treated in better fashion. The sort of result Mr. Cottrall mentioned can be obtained by beating in a rubber-lined ball mill with rubber-covered balls. Clean-looking fibres with high bursting and tensile strengths and well-preserved tearing strength can be obtained.

It would be most interesting to examine such pulps from the point of view of separation of fibre friction and bonding.

MR. G. VAN NEDERVEEN: I think the question put forward by Dr. Gallay is one we also know in Holland. We met the problem how to beat fibres without decreasing the tearing strength too much. For beating experiments in the laboratory, we use the Lampén mill and an apparatus that is nothing but a kitchen mixer (we call it a Vimix), turning at 12 000 r.p.m. The tearing strength of laboratory sheets made from Lampén mill beatings is definitely lower than that from Vimix beatings. The breaking length is somewhat higher with the Lampén mill than with the Vimix for high freeness; but, at the end, the Vimix breaking lengths come up to the same level as those with the Lampén mill. I mean, the Vimix does really beat the fibres and is not only a disintegrator.

When you examine under the microscope fibres beaten in the Vimix, you will see that this mixer does not cut the fibres very much: the fibres are

not shortened by this beater. With cuprammonium solution one finds balloons in a few places on the fibre wall. This means that there is indeed some kind of external fibrillation proceeding in the Vimix.

With this apparatus, therefore, we succeeded in getting high tearing strength, the fibres being beaten but little damaged.

DR. GALLAY: I am very grateful for this additional contribution; it is typical of what I have noted and what may be expected if the integrity of the fibre is preserved.

As you will have gathered from what I said this morning, I dislike gross fibrillation as a component of the beating process. I wish to make my position on this matter quite clear. If the objective is to manufacture greaseproof or glassine or, in general, if permeability to gases and liquids is an objective, then undoubtedly extensive fibrillation is difficult to avoid or may indeed be necessary. If we restrict our objective to strength factors and associated properties, however, then I would wish to reduce this gross fibrillation to a minimum.

MR. P. E. WRIST: There is one particular aspect of fibre preparation that has not been mentioned so far and should not be forgotten. In practice, we are not dealing with fibres of uniform dimensions. When we form a sheet, we assemble fibres of differing shapes and sizes. The fibre length distribution influences the way in which fibres will pack together. We may get greater strength in a sheet, not by developing bonds, not by increasing the flexibility, but by adjusting the fibre length distribution in such a way that the fibres can form together better. I think this is what the papermaker is primarily trying to do with his machine Jordan. Unfortunately, he can only cut the fibre this way and that not selectively, thus he obtains the improved formation and the resulting increases of bursting and tensile strengths at the sacrifice of average fibre length and corresponding tearing strength.

Mr. Chairman, on Wednesday, you said that ten years ago there was much more beating done than today. I think this is probably due to the increasing blending of pulps, whereby some of the results of beating can be achieved without its use. I believe we should not overlook in our discussions the importance of optimum fibre distribution for optimum fibre packing.

MR. G. F. GLOVER: In the first few minutes of beating, even with quite a low roll pressure, there is a very rapid increase in strength properties. I think that agrees with the idea of internal fibrillation, but the main point is that the fibre is made more flexible in those first few minutes (even seconds) and the

surface tension forces cause the fibres to bed down much more securely in the sheet, giving far more possibility of bonding. There is an increase in sheet density almost immediately in the first few minutes of beating.

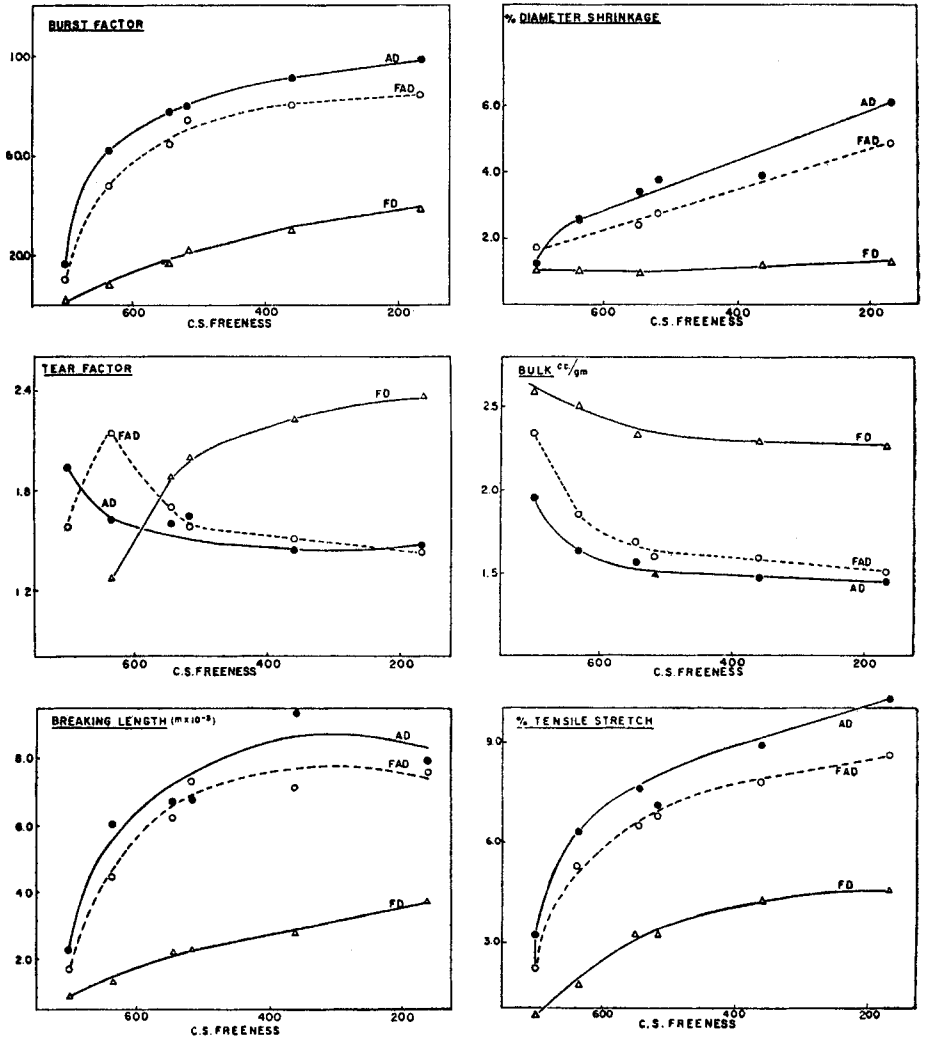

Fig. T — Variation of physical properties of freeze-dried (FD) and air-dried (AD) standard handsheets on beating — FAD designates a control series in which the wet pulp was frozen and then allowed to thaw before preparing air-dried handsheets

(From Marchessault, Lodge and Mason, *Svensk Papperstidn.*, 1956, **59**, 859)

DR. S. G. MASON: In his spoken remarks, Dr. Gallay speculated on the possibility of beating without sacrificing the tearing strength. It may be of interest to know that we were able to accomplish this by beating in the normal way in the Valley laboratory beater, forming standard handsheets and then freezing the wet sheets and evacuating them so as to remove the water by sublimation. This effectively eliminates the surface tension forces normally present when drying from liquid water. The results are illustrated in Fig. T.

It should be emphasised that by freeze-drying we have not completely eliminated interfibre bonding — that is, the freeze-dried sheets are not held together by frictional forces and fibre entanglement alone. In these experiments, sublimation was carried out at about – 10°C; however, once we get below a certain moisture content, which depends upon the water sorption isotherm, the 'freezing point' of the water associated with the cellulose is less than – 10°C. Thereafter, we may consider that the water is removed from a solution of 'liquid water' in cellulose; thus, the last stages of freeze-drying are basically no different from those in evaporation drying.

DR. GALLAY: I fully agree with the statement made by Mr. Wrist on fibre distribution. It would certainly appear logical that some filling of voids with fibres of suitable length should enhance entanglement.

I am reminded of the excellent work carried out by Prof. Brecht on the wet strength of mechanical pulp, from which he concluded that the highest possible wet density was required for maximum tensile strength.

I agree also with the remark by Mr. Glover and I interpret that by concluding that there simply has not been time for appreciable damage to occur during the first portion of the beating curve.

With regard to Dr. Mason's remarks, we have not made a study of the freeze-drying method. I am rather surprised at the relatively small loss in tensile strength shown, but hesitate to comment further in a field that is unfamiliar to me.

MR. G. F. UNDERHAY: I have two points. One is that I thought Dr. Gallay's unbeaten strength figures with which he compared the strengths of his pulp after the alcohol washing were very low indeed. It seemed to me that for that reason the ratio was rather higher than it should have been.

The other point was that I heartily endorse and agree with what Dr. Gallay said about the incidental properties that arise when you are beating pulps. Once again, does it not underline the fact that wetness development is not only an incidental property that you may not want, but that, if you do want it, it is very badly measured by the Schopper-Riegler instrument?

DR. GALLAY: The last part of Mr. Underhay's remarks I agree with heartily, but his first point requires clarification. The pulp used was a normal sulphite pulp of average strength. Due regard must be paid to the fact that these sheets were air-dried and not pressed after couching. The published paper will deal with this further.

THE CHAIRMAN: I think we must end the discussion on Dr. Gallay's paper; time is up. Mr. Cottrall, you wish to say something?

MR. COTTRALL: Dr. Gallay is assuming that filling in the spaces in the paper with little bits and pieces increases the strength of the paper. We heard, the other day, that fines in paper add nothing to the strength of the paper.

THE CHAIRMAN: Summing up this discussion, it shows that there is much more to it than is apparent. Personally, I think the important points that have been brought out by Dr. Gallay's paper can be stated in the following way.

In the last few years, we have had a deluge of hydrogen bonds over the paper industry; everything in paper is suddenly hydrogen bonds. This is probably due to the fact that so few people in the paper industry and in research really know what a hydrogen bridge is. There are many types of hydrogen bonds. We certainly know that the hydrogen bridge can be quantitatively calculated only in very few extremely simple circumstances. It is a nice concept, however, because it is easy to understand and to apply qualitatively. Thus, everybody jumps to it.

MR. HUNGER: I am afraid we have now built up a Tower of Babel in the nomenclature of the cell walls. According to the classic terminology of Kerr and Bailey, we have a primary wall and secondary walls nos. 1, 2 and 3. Bucher has reintroduced the term tertiary wall and Meier has introduced that of a transitional lamella. Today, it seems that the primary wall Prof. Giertz spoke of corresponds to the outer secondary wall referred to by Mr. Emerton. I strongly support Mr. Emerton's proposal published some time ago that, until there is definite proof to the contrary, we should use the terms of Kerr and Bailey only.

MR. VAN NEDERVEEN: I want to thank Prof. Giertz for this lecture, because I found most of our own experiences embodied in the ideas he put forward.

My interest, too, was aroused by J. d'A. Clark's article in the book by Ott, *Cellulose and its derivatives.*

At the Fibre Research Institute in Delft, our observations indicate that the values obtained with the Schopper-Riegler instrument are composite values, in which the influence of more than one property of the pulp suspension is hidden.

Swelling, fibrillation and shortening of the fibres are three phenomena that contribute to the beating degree found with this apparatus. We therefore use the Jayme swelling measurement and that of fibre length distribution with the HS apparatus. An impression of the fibrillation is obtained by determination of the specific surface of the fibres with the Robertson and Mason apparatus. In this way, the three factors that each contribute in their own way to the ultimate value of the beating degree are measured separately.

Our investigations on beating have been carried out with various laboratory instruments — a Lampén mill, a Jokro mill, a Valley beater and a modified Turmix (kitchen mixer). Applying these methods and also checking the effects of beating with the phase contrast microscope, we found the largest divergency in beating effect between the Lampén mill and the Turmix. We think it useful always to use these two instruments in studying the beating behaviour of an unknown pulp, just because of the very different effects they have.

For mill control, the determination of swelling, the fractionation with a classifier and the measurement of specific surface would take too much time. Now Ivanov (Leningrad) and Imset (Oslo) have developed rather simple instruments for getting a quick impression of the fibre shortening and the swelling separately but in one operation. We think both of these instruments would be very valuable tools in mill practice.

Has Prof. Giertz had any experience with these two instruments?

PROF. H. W. GIERTZ: I can very rapidly answer the question about the different apparatus. I do not know about the Ivanov apparatus, but I am fairly familiar with the Imset one, which measures the shortening of the fibres and at the same time the swelling degree of the long fibres. I say swelling, but, as the method is based on centrifugal forces, it is more or less the same kind of retained water that is measured with Jayme's method. As I know that only a small part of such retained water has to do with swelling and that the main part more accurately ought to be called entrapped water (held by the fibre in the lumen and other pockets), I do not think it is a suitable instrument for laboratory work. Whether it is a good apparatus in the papermill, I do not know.

MR. COTTRALL: I would really like to know something about this swelling. In his paper, Prof. Giertz makes a reference to me in relation to the considerable importance I attach to swelling. I certainly do, so far as the unbeaten part is concerned, but I am not quite clear how to get information on the amount of swelling when the fibre is beaten. Let us take a dry fibre: put into water, it swells considerably. I think you will all agree with that. Then you beat this fibre, either by internal fibrillation by breaking the bonds between some of the cellulose fibrils or by external fibrillation by taking pieces off the surface or by splitting the fibre or by all three methods. My point is, if you add the volumes of all these pieces together, is the sum of these very much greater than the volume of the soaked, unbeaten fibre — or are you, when you are considering swelling, taking the external boundary of the fibre, including all the voids derived from the internal and external fibrillation of the fibre? Assuming the length of the fibre is unaltered, is the volume of the fibre (obtained by multiplying the length by the average cross-section comprised of the sum of the body of the fibre plus the sum of the areas of all the external fibrils) any different or very much greater than the volume of the soaked, unbeaten fibre (*cf.* Kress, O. and Bialkowsky, H., *Paper Trade J.*, 1931, **93** (20), 42)? It is indicated in Prof. Giertz' paper and in other papers that the volume is very much greater and I should like the point to be clarified.

Another point in connection with the effect of hemicelluloses referred to in Prof. Giertz' paper, is what would happen in the case of a fibre that contains no hemicellulose, such as a rag fibre?

I am not suggesting that external fibrillation is an advantage for most papers, but most swelling measurements have been made when the fibre is appreciably beaten and there is considerable external fibrillation. It is when there is appreciable external fibrillation that we get the high figures recorded for so-called swelling. I do not regard these as representing measurements of true swelling.

PROF. GIERTZ: But you get this fibrillation at such a very late stage of beating that it cannot play any role for ordinary papers. If you make a bond paper or a greaseproof paper, however, it may play quite an important part. I have explained this in my paper.

MR. H. W. EMERTON: In his paper, Giertz says, "the fibre splits in the longitudinal direction and forms fibrils." I should like to underline a fact that I have repeatedly emphasised, that, to a much greater extent than is realised, external fibrillation is not in the form of longitudinal strings, but is in the form of sheets or membranes.

The bulk of the cellulose in mature fibres is in the middle secondary wall in the form of co-axial layers of microfibrils, the tangential surfaces of which are encrusted with hemicelluloses. Such lignin as is present in the cell wall occurs between these co-axial polysaccharide layers. Thus, the removal of lignin by cooking predisposes the structure, when mechanically treated, to disintegrate into thin membranes. It is, to a large extent, the effect of surface tension drawing these together, also folds and turned up edges, that lead to the impression that long, thin fibrils are seen.

THE CHAIRMAN: On Mr. Emerton's paper, I must say that in transmission electron micrograph experiments carried out in Stockholm by Dr. S. Asunmaa on bonding in paper — studied by the serial cutting of pieces of paper to about 100 Å thickness — you can see that quite large bonding areas occur. You can also, because of the osmium penetration, easily identify the S1 layer. Consequently, it is frequently possible to identify the part of the cell wall in such fibre-to-fibre contact areas. We only consider electron-optical contact areas. Whether such contacts mean bonding or not is uncertain, but the fibres frequently appear welded together even in high resolution micrographs. The probability that we are here dealing with bonds is much higher of course than in the experiments of Nordman discussed yesterday. Such welds are found between all the layers. We have observed such areas between S1 and S1 layers; we have them between S1 and S2 layers; we have them between S2 and S2 layers. We also frequently observe a collapsed lumen when it is impossible, even at high resolution, to observe anything but an electron-optical contact.

Consequently, I think it is an oversimplification to say that we want to take away S1 in beating to have the benefit of S2 bonding. I think there is no evidence that bonding is better between S2 and S2 layers. I think that all types of bonding occur between all the variables and, after all, if we still believe in the hydrogen bond, all surfaces are profuse with hydrogen bonds, so there is no reason from that point of view to assume that bonding could not occur between all layers.

Furthermore, I think Mr. Emerton does not intend to say what could be read into his paper, that you can carry out beating so nicely that you remove just the S1 layer. As a matter of fact, in micrographs of all beating products, sheaves or flakes, loose fibrils or whatever they are named are formed by splitting fibres more or less at random. Only seldom does the split occur at or near the S1/S2 boundary.

That brings me to the second point about the fibrils. All fibrils discussed in the papers are the fibrillar portions of a fibre. This is what you first see in

the microscope, because, when you focus, you will focus on the fibre, but not on the slide below it. On that slide, you would see loose fibrils, also separate entities that I will call chips (or sheaves or something like that), which are free. Many fibre microscopists never see them, especially when using Herzberg staining solution, because this agent swells these loose particles so that they are practically dissolved.

By pouring pulp into a shallow trough with a bottom made of teflon foil and allowing the water to evaporate at 60°c, examination under the light microscope shows a great quantity of fine material, woolly and hairy — just like the hairs in a horse's tail — all over the picture. This material was lost in the whitewater in the process of making a sheet of paper from the pulp.

I think that Mr. Emerton may be right. He thinks that the material coming off the fibre in the beating process is flakes — the material looks hairy to me. It may, of course, be possible that the flakes have rolled up into bundles in drying, owing to surface tension forces. Cross-sectioning of such material has, however, not forced such a concept on us.

If we take the whitewater from the sheet made on the wire and evaporate it on the teflon foil, we see it is a mass of small particles intermixed with a few long fibres that have passed through the mesh.

The point I have tried to make is that, in beating, even at its early stages, a great mass of fibrils is torn off the fibre. Very probably there are more loose fibrils than those still adhering to the fibres. Van den Akker's paper on the forces required for tearing loose a fibril makes it understandable that the probability may be higher for the particle to be completely torn off than left partially intact, once the tearing process has begun.

If we discuss fibrillation in beating and paper properties, we cannot ignore this material.

MR. EMERTON: I think there is no real disagreement between us — just a question of emphasis — on this matter of longitudinal fibrillation. We must not accept too readily the idea of fibrillation being in the form of long, thin fibrils.

I did not mean to imply that it is desirable during beating to remove S1 in order to get bonding between S2 layers in adjacent fibres: that may or may not be so. My argument was that it may be desirable — I do not state dogmatically that it is — to remove S1 simply to enable the fibre to imbibe plenty of water and become more plastic.

THE CHAIRMAN: I think I was not the only one who thought that you stressed the point too much, so I just wanted to give you a chance to put it right.

DR. H. MEIER: I do not quite agree with Mr. Hunger that the primary wall of Prof. Giertz has become synonymous with the secondary wall of Mr. Emerton. As far as I understood it, Prof. Giertz really meant that only the primary wall is removed during beating, whereas Mr. Emerton meant that S1 is removed, too.

Probably, the only technique with which to resolve this question is that of metal shadowing used by Mr. Emerton. It is possible by this means to distinguish clearly between the crossed fibrillar structure of the primary wall and the parallel structure of S1.

DR. B. G. RÅNBY: Firstly, I should like to say that the S1 layer in the cotton fibre is called the *winding layer* and that term is used rather extensively in the U.S.A.

Secondly, I should like to fulfil an intention of two days ago and speak this afternoon about recent work on hydrogen bonds, especially in cellulose. What we know now about them gives some explanation how it is possible at all to beat a pulp containing no hemicellulose and to make such strong paper from it. I should also like to show how amorphous cellulose can react with water in the same way as hemicellulose.