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INFLUENCE OF FIBER-FIBER BONDING ON THE TENSILE CREEP COMPLIANCE OF PAPER

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ABSTRACT

In this study, two sets of sheets were made with differing levels of specific bond strength and relative bonded area. One set of sheets were wet pressed using a high press load and the other set of sheets were wet pressed using a low press load. Within each set, the sheets were treated with either a debonder or a bonder or received no treatment. Creep compliance data showed that creep curves for the debonder, bonder and untreated sheets were the same for the sheets wet pressed at the high press load and different for the sheets wet pressed at the low press load. Creep failure time was influenced by the treatments in both the high and low load wet pressed sheets; sheets treated with debonder failed first and the sheets treated with bonder failed last. It was concluded that at high levels of bonding as is the case with the high load wet pressed sheets, differences in specific bond strength due to the treatments do not influence creep deformation because fiber-fiber bonding is at a level where the sheets are efficiently loaded structures. The low load wet pressed sheets showed differences in creep deformation when specific bond strength was changed with treatments because fiber-fiber bonding was at a lower level where the sheets were inefficiently loaded. As the loading efficiency of the paper structure is improved through increased fiber-fiber bonding

(either by increasing specific bond strength or relative bonded area), an efficiently loaded structure can be achieved where fiber-fiber bonding no longer affects deformation. This allows creep compliance to reach a minimum level which is dictated solely by the fibers. An efficiency factor can be used to describe deformation behavior where an efficiency of "1" indicates an efficiently loaded structure and lower values indicate a less than fully efficient structure, one in which fiber-fiber bonding influences deformation behavior. In this study, efficiency factors were used to scale the low load wet pressed sheet results and several sets of lesser refined and pressed sheets (thereby "removing" fiber-fiber bonding influence) and the data superimposed onto the high load wet pressed sheet results.

INTRODUCTION

Although research by Hill [1, 2] found that fibers are the structural element where creep compliance originates in paper, it is still unclear exactly how bonding of these fibers may influence this behavior. Beginning with the research of Byrd [3, 4] the focus of creep research shifted to accelerated creep without fully understanding the role of fiber-fiber bonding. This has resulted in a limited base of available research regarding fiber-fiber bonding and creep in the literature.

Brezinski [5, 6] showed that as wet pressing and level of refining were increased, higher initial applied stress levels were required to get the same amount of strain after 24 hours of creep testing. This implies that as fiber-fiber bonding is improved either through wet pressing (densification) or refining (making more conformable fibers), that creep compliance decreases. Schulz [7, 8] showed that increased levels of wet straining leads to a decrease in creep compliance. He hypothesized that wet straining has the affect of changing the way stress is distributed within the paper structure. In other words, wet straining makes paper more efficient in distributing stress, causing a drop in creep compliance. Unfortunately, it is unclear if this is the dominant reason for the drop in creep compliance. For example, fiber kinks and micro-compressions could be pulled out causing a strain hardening effect.

With regard to relative bonded area change, Sanborn [9] showed that light scatter increased as strain during creep testing increased. This implies that there is some relative bonded area loss during creep testing. It does not, however, imply that creep deformation is caused by bond breakage, only that bond breakage occurs concurrently. On the other hand, Byrd [10] showed in his research that light scatter decreased as strain during creep testing increased. His data implies relative bonded area is increasing during creep deformation. Most likely, there was minimal relative bonded area loss and the decrease in light scatter was due to fibers being drawn into optical contact from lateral contraction and longitudinal straining.

Overall, there are no definitive answers and some possible contradictions in the existing body of paper creep literature. By comparison, the role of fiber-fiber bonding with regard to elastic modulus, stress-strain behavior and tensile strength has been extensively researched. This work is best summarized in research by Seth and Page [11]. Seth and Page [11] were able to show that by either decreasing specific bond strength (with a debonder) or increasing specific bond strength (with a bonder), elastic modulus and the shape of the stress-strain curve remained constant as long as there was an adequate level of fiber-fiber bonding to maintain an efficiently loaded structure. The only difference between the cases was there were different strain to failures and tensile strengths. Page [12] showed in earlier work that tensile strength in paper can be affected by changing relative bonded area and specific bond strength. In addition, Seth and Page [11] measured changes in light scatter and found a loss of relative bonded area after sheet straining. They showed that relative bonded area decreased at differing rates depending on the treatment applied. The debonder treated sheets have the highest rate of loss followed first by the sheets with no treatment and finally by the bonder treated sheets.

In addition, Seth and Page [11] introduced the concept of the efficiency factor. Their premise is that deformation behavior within paper originates within the fibers and a common efficiency factor can be used to show the influence of fiber-fiber bonding whether the deformation behavior is elastic or viscoelastic. Specifically, they showed that if stress-strain curves in paper did not overlap, they could be superimposed by dividing the stress component of the stress-strain curve by an efficiency factor. The efficiency factor is calculated by dividing the elastic modulus of the more compliant stress-strain curve (inefficiently loaded structure) by the elastic modulus of the stiffest stress-strain curve (efficiently loaded structure). Seth and Page [11] were able to show that the whole stress-strain curve would superimpose based on an efficiency factor calculated from relating the elastic moduli of the sheets. Simply put, deformation behavior in the viscoelastic regime could follow an elastically derived efficiency factor. This would hold true as long as fiber-fiber bond breakage was not severe enough to reduce the efficiency factor during straining; an issue that could be a factor with creep deformation.

EXPERIMENTAL

Pulp & preparation

NIST standard reference material 8495 Northern Softwood Bleached Kraft Pulp was used in this study. The pulp arrived in dry lap sheets in a hermitically sealed package. The pulp has remained sealed for approximately 13 years. Unless otherwise specified, the pulp was refined in a valley beater at a charge of 300 O.D. grams per batch for 30 minutes. The final pulp Canadian standard freeness was targeted at 400 ml. The pulp was prepared in such a manner to create straight, conformable fibers that would easily bond. Prior to making handsheets, the pulp slurry was treated with either debonder or bonder or received no treatment. The debonder used was a surfactant (Incrosoft AS-55), while the bonder used was locust bean gum. The dosages used for the treatments were 0.45% by weight of bonder or 0.11% by weight of debonder.

Handsheets

Handsheets were made using a 8”× 8” Williams handsheet mold. A 100 mesh screen was used as the forming wire. The handsheets made from the treated pulp slurries were targeted to an oven dry basis weight of 90 g/m^2. Sheets were wet pressed at 150 psi, 25 psi, or at 10 psi. Handsheets were dried on a drum dryer at 20psi steam pressure for 5 minutes. All sheets were immediately bagged and transported to a 23°C and 50% RH room for conditioning prior to testing.

Physical & creep testing

A full battery of physical testing was conducted including, handsheet grammage, hard caliper, ultrasonic velocities, formation, light scatter, zero-span tensile strength, z-directional tensile strength and Instron tensile strength. Instron measurements recorded stress-strain curves as well as tensile strengths. Although no direct measurement of specific bond strength is made in this study, differences in z-directional tensile strength will indicate a change in specific bond strength when relative bonded area remains constant. Within each set of handsheets, relative bonded area was held constant by careful control of refining, pressing and drying. Creep testing was conducted using the IPST tensile creep tester under a constant 23 °C and 50% RH condition. Samples were cut into 170mm × 25 mm wide strips, mounted and conditioned for 24 hours at 23 °C and 50% RH condition prior to application of load. A series of different magnitude dead loads (initial applied stress levels) were evaluated. Displacements and failure times were recorded using LVDT sensors with the output signals sent to a computer based data acquisition system. Light scatter of creep test strips were measured prior to and after creep deformation testing to measure relative bonded area change. The basis for using light scatter as means to measure changes in relative bonded area in paper has previously been demonstrated[13, 14].

RESULTS

High load wet pressed sheets

The first of two sets of results presented are sheets treated with debonder, nothing (control) or bonder that were wet pressed at high load (150 psi) resulting in high density, highly bonded sheets. A small portion of these results were presented by DeMaio and Patterson[15] previous to this paper. Table 1 shows the physical testing results for these sheets.

The data from physical testing presented in Table 1 shows that sheets treated with debonder and bonder did not show significant differences from the control with regard to grammage, hard caliper, density, formation and zero-span tensile strength. Deformation behavior, as indicated by the ultra-

Table 1 Physical testing results from the high load wet pressed sheets.

Sheet Treatment	Grammage (g/m^2)	Hard Caliper (mm)	Density (g/cm^3)	Formation Number	Ultrasonic Modulus (km^2/s^2)
Debonder	96.0	0.118	0.814	32.8	10.3
Control	95.8	0.117	0.819	33.8	10.3
Bonder	95.7	0.115	0.832	33.4	10.6
Variation	0.3%	2.6%	2.2%	1.8%	2.9%

Sheet Treatment	Light Scatter (m^2/g)	Z-Tensile (N/mm^2)	Tensile (N/mm)	Failure Strain (%)	Zero-Span (N/mm)
Debonder	21.4	0.672	9.12	3.39	15.5
Control	20.5	0.798	10.2	3.84	15.8
Bonder	19.8	0.927	10.7	3.99	16.0
Variation	8.1%	37.9%	17.3%	17.7%	3.2%

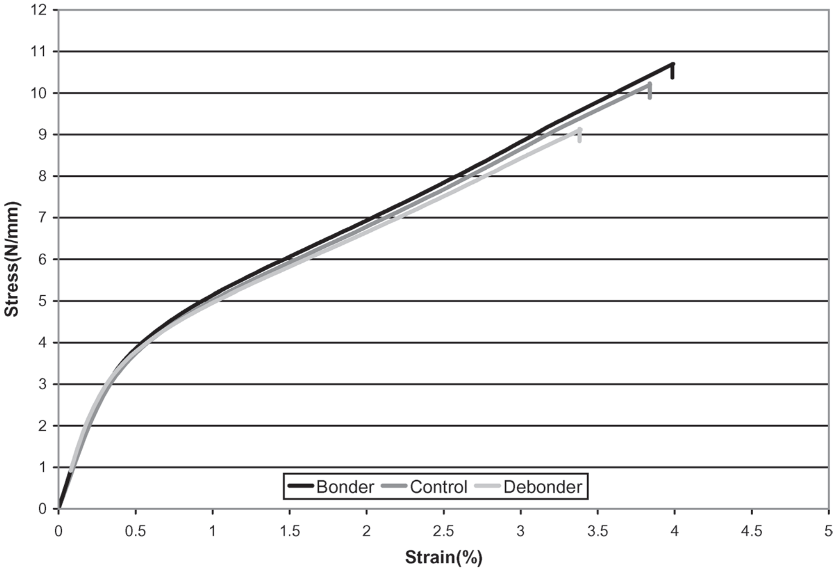

Figure 1 Stress-strain curves from Instron tensile testing of high load wet pressed sheets.

sonic elastic modulus data in Table 1 and stress-strain curves shown in Figure 1 were similar for all three sets. The differences in the sheets were in z-directional tensile strength, tensile strength and strain to failure, caused predominantly by differences in specific bond strength. Figure 1 shows that sheets treated with debonder were the weakest, while the sheets treated with bonder were the strongest and illustrates how deformation between the three sets of sheets remain similar, only differing in the failure point.

These results demonstrate that it is possible to create three sets of handsheets with similar deformation behavior and differing specific bond strengths. These results also confirm the work of Seth and Page [11] where at high levels of bonding, a fully efficient paper structure can be created where elastic modulus is maximized, and differences in specific bond strength do not affect deformation behavior, but do influence failure behavior.

Creep compliance results follow the same trend with regard to deformation as the elastic modulus data, and stress-strain behavior. This would indicate that even though the time duration for a creep test is much longer than that of a stress-strain measurement, its influence was not a factor. Overall, as illustrated in Figure 2, the creep curves generated at several different initial

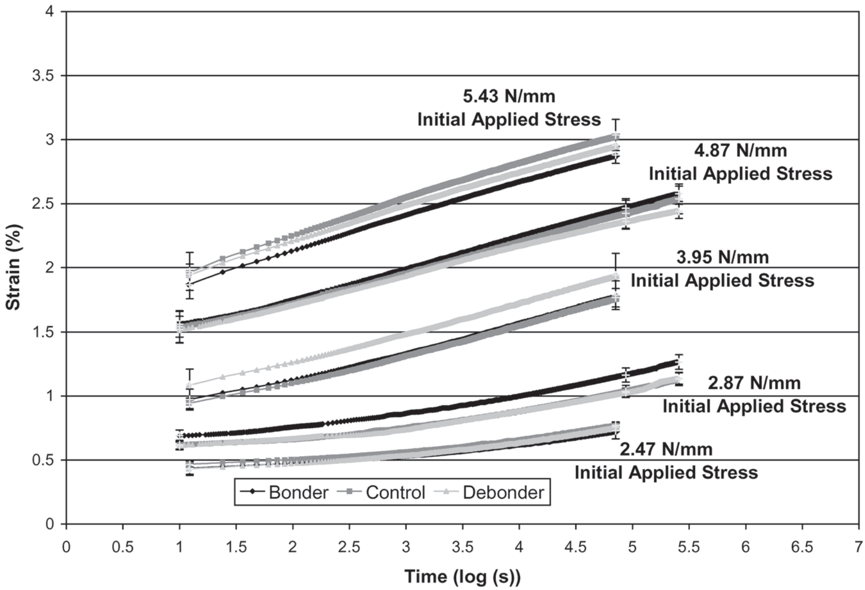

Figure 2 Creep curves from high load wet pressed sheets.

applied stress levels show good overlap and fall within the standard error bars, indicating they have creep compliances that cannot be differentiated from each other.

Further proof for this is obtained by constructing isochronous stress-strain curves from the data. Isochronous stress-strain curves, plotting strain versus the initial applied stress at various snapshots in time, are another way of comparing creep data. In Figure 3, strain after 10 seconds and 24 hours of creep testing are plotted versus initial applied stress. As illustrated in Figure 3, the isochronous stress-strain curves derived from the creep curves in Figure 2 for the debonder, control and bonder sheets do not show any significant difference between the cases. The curves overlap and fall within the standard error bars. The data was fit with power function trend lines with R^2 values all greater than 0.98. This is contrary to the expectation that the bonder treated sheets would be the least compliant, and the debonder treated sheets would be the most compliant.

Furthermore, light scatter data indicated that the loss of relative bonded area occurred at differing rates. Figure 4 shows that at a given level of strain, the bonder treated sheet showed the smallest change in light scatter and

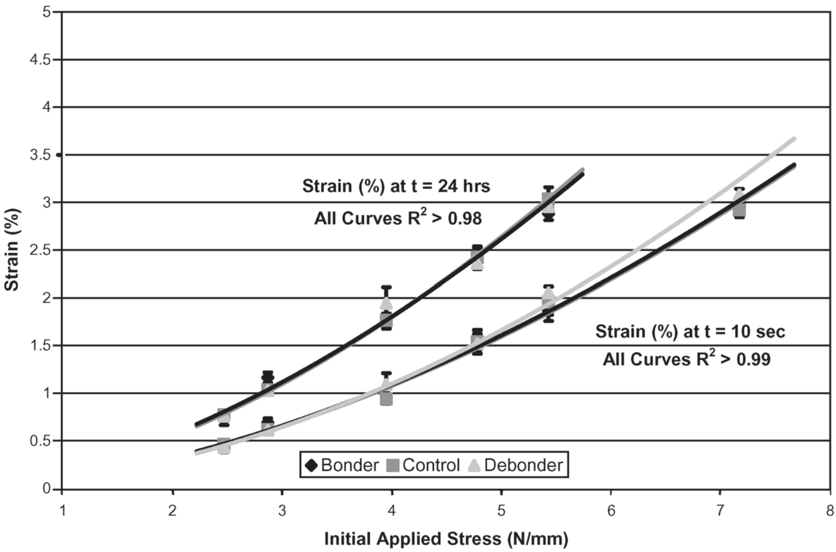

Figure 3 Isochronous stress-strain curves from creep testing of high load wet pressed sheets.

debonder treated sheet the greatest. In order to more easily illustrate these differences in relative bonded area loss versus strain, the data points were fit with a second order polynomial function, all of which gave R^2 values greater than 0.90. Overall, this data indicated that creep compliance remains unaffected at high levels of bonding despite differences in specific bond strength and rate of relative bonded area decrease.

Additional creep testing was done at a higher initial applied stress level with the intent of causing failure, allowing a more detailed analysis of the behavior. Figure 5 illustrates the failure points and light scatter change versus time for debonder, control and bonder treated sheets.

The debonder treated sheets fails much sooner than the bonder treated sheets with the control failure points scattered in between. Average failure strain is not significantly different in the bonder treated sheets versus the control and debonder sheets. Light scatter change indicates that there is the greatest loss in relative bonded area with the debonder treated sheets and the change occurs fastest in those sheets. Table 2 best summarizes the data illustrated in Figure 5.

As indicated by the percentage differences in Table 2, there are large

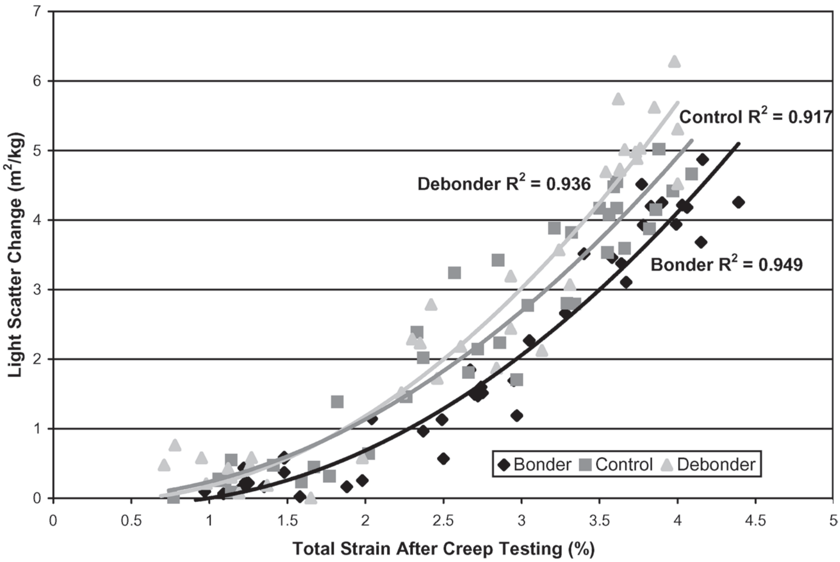

Figure 4 Light scatter change versus maximum strain during creep tests of high load wet pressed sheets.

Table 2 Failure strains, times and light scatter changes for high load wet pressed sheets.

Sheet Treatment	Failure Strain (%)	Failure Time (log(s))	Failure Time (min)	Light Scatter Change (m^2/g)
Debonder	3.77	2.86	12.1	5.16
Control	3.73	3.94	144	4.22
Bonder	3.94	4.43	449	4.04
Variation	5.6%	54.9%	>>100%	27.7%

differences in failure time and light scatter change between the three differently treated sets of sheets. The bonder treated sheets last over an order of magnitude longer than the debonder treated sheets. The debonder treated sheets have an almost 30% higher change in light scatter than the bonder treated sheets. Overall, the creep compliance data is consistent with Seth and Page [11]. The creep curves and isochronous stress-strain curves indicate that creep compliance is the same for sheets wet pressed at high load, despite

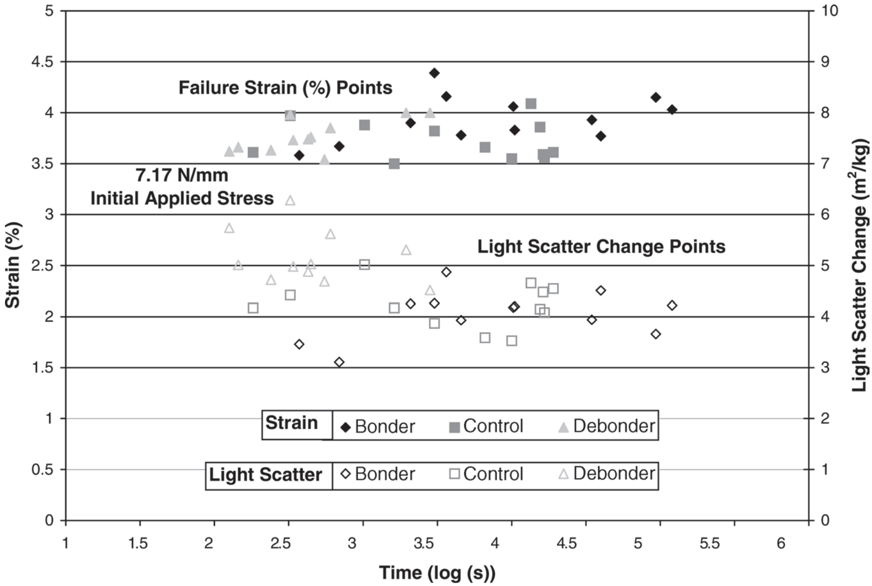

Figure 5 Creep testing failure strain and light scatter change versus time for high load wet pressed sheets.

differences in specific bond strength. The differences in specific bond strength manifests itself only by a change in failure behavior and change in light scatter.

Low load wet pressed sheets

The second set of results presented here are sheets treated with debonder, nothing (control) or bonder and wet pressed at low load (25 psi), resulting in lower density and lower bonded sheets than the highly pressed case. Table 3 shows the physical testing results for these low load wet pressed sheets.

As with the high load wet pressed case, the data from physical testing presented in Table 3 showed that sheets treated with debonder and bonder did not show significant differences from the control with regard to grammage, hard caliper, density, formation and zero-span tensile strength. The major noticeable difference between the low load wet pressed sheets and the high load wet pressed sheets are that the low load wet pressed sheets have more bulk than the high load wet pressed sheets. This is seen by comparing the

Table 3 Physical results from the low load wet pressed sheets.

Sheet Treatment	Grammage (g/m²)	Hard Caliper (mm)	Density (g/cm³)	Formation Number	Ultrasonic Modulus (km²/s²)
Debonder	95.2	0.145	0.657	31.7	9.67
Control	94.9	0.143	0.664	31.8	10.1
Bonder	95.0	0.141	0.674	31.3	10.4
Variation	0.3%	2.8%	2.6%	1.3%	7.5%

Sheet Treatment	Light Scatter (m²/g)	Z-Tensile (N/mm²)	Tensile (N/mm)	Failure Strain (%)	Zero-Span (N/mm)
Debonder	27.6	0.474	8.57	3.54	15.4
Control	26.9	0.567	9.18	3.59	15.7
Bonder	26.1	0.631	10.2	3.85	15.3
Variation	5.7%	33.1%	19.0%	8.8%	2.6%

sheet densities from Table 1 and Table 3. Deformation behavior, as indicated by the ultrasonic elastic modulus data in Table 3 and stress-strain curves illustrated in Figure 6 were more dissimilar for all three sets than in the case of high load wet pressed sheets. Light scatter in the low load wet pressed sheets is higher, indicating a lower relative bonded area than the high load wet pressed sheets. The same differences as with the high load wet pressed sheets existed with regards to z-directional tensile strength, tensile strength and strain to failure. Sheets treated with debonder were the weakest, while the sheets treated with bonder were the strongest.

Overall, the low load wet pressed sheets have lower moduli, more compliant stress-strain curves and are weaker than the high load wet pressed sheets. These results also relate to the work of Seth and Page [11]; at lower levels of bonding, an inefficient paper structure is created where elastic modulus is not maximized, and differences in specific bond strength and relative bonded area do affect deformation behavior and failure behavior.

Creep compliance results again follow the same trend with regard to deformation as the elastic modulus data, and stress-strain behavior. Overall, as illustrated in Figure 7, the creep curves generated at several different initial applied stress levels show poor correlation as the curves do not overlap and do not have overlapping standard error bars. In all cases, the bonder treated

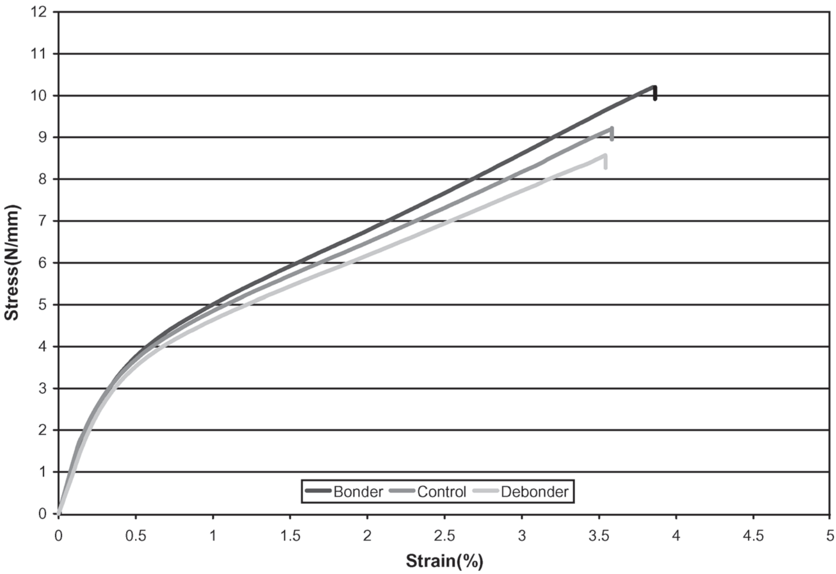

Figure 6 Stress-strain curves from Instron tensile testing of low load wet pressed sheets.

sheets are the least compliant and debonder treated sheets are the most compliant.

Again, isochronous stress-strain curves can be generated from the creep data to further illustrate differences in the creep behavior. In the Figure 8, strain after 10 seconds and 24 hours of creep testing are plotted versus initial applied stress. As illustrated in Figure 8, the isochronous stress-strain curves derived from the creep curves in Figure 7 for the debonder, control and bonder sheets show that the creep behavior is different between the cases. Again, the bonder treated sheets are the least compliant and the debonder treated sheets are the most compliant. The curves do not overlap or fall within the standard error bars. All data were fit with power function trend lines, all with R^2 values greater than 0.99.

As with the high load wet pressed sheets, light scatter data indicate that the loss of relative bonded area occurred at differing rates. Figure 9 shows that at a given level of strain, the bonder treated sheet shows the smallest change in light scatter and debonder treated sheet the greatest. In order to more easily illustrate these differences in relative bonded area loss versus strain, the data

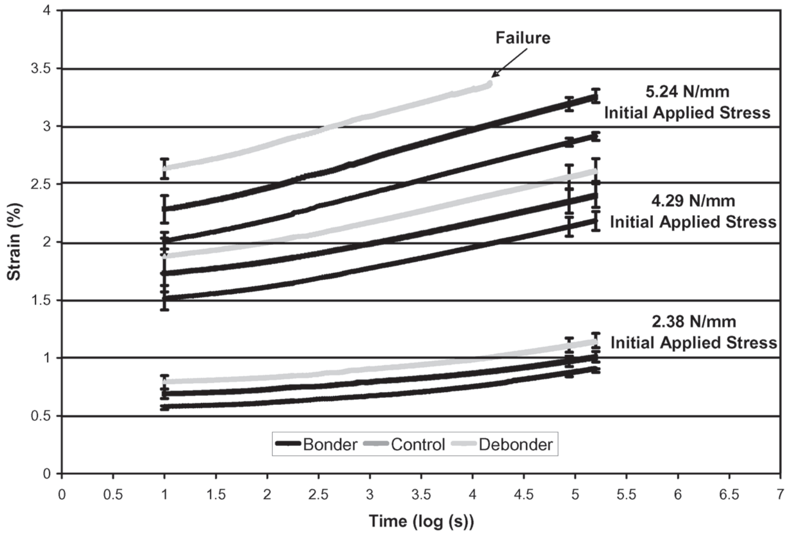

Figure 7 Creep curves from low load wet pressed sheets.

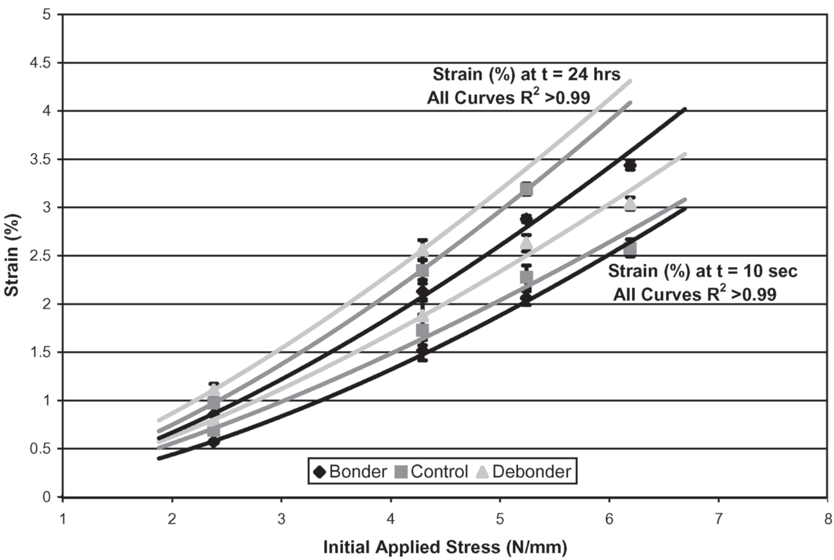

Figure 8 Isochronous stress-strain curves from creep testing of low load wet pressed sheets.

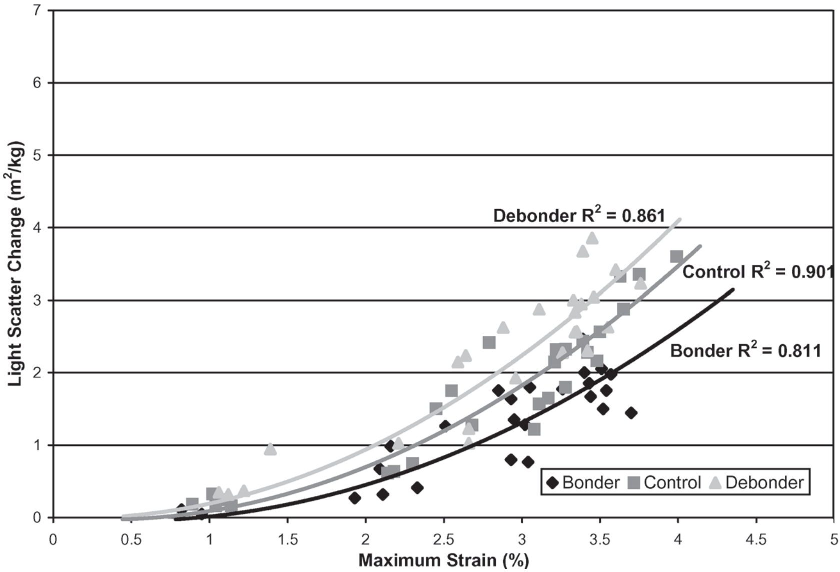

Figure 9 Light scatter change versus maximum strain during creep tests of low load wet pressed sheets.

points were fit with a second order polynomial function, which gave R^2 values all over 0.80. Overall, the change in relative bonded area is smaller for the low load wet pressed sheets than the high load wet pressed sheets. This is due to the fact that there is less initial relative bonded area in the low load wet pressed sheets versus the high load wet pressed sheets. Therefore, a small change in light scatter amounts to a much larger percentage of relative bonded area loss in the low load wet pressed sheets.

Additional creep testing was also done at a higher initial stress levels with the intent of causing failure, allowing a more detailed analysis of the behavior. Figure 10 illustrates the failure points and light scatter change versus time for debonder, control and bonder treated sheets.

The debonder treated sheets fails much sooner than the bonder treated sheets with the control failure points scattered in between. Average failure strain is not significantly different in the bonder, control and debonder sheets although they all are lower than the failure strains from the high load wet pressed sheets. Light scatter change indicates that there is the greatest loss in relative bonded area with the debonder treated sheets and the change occurs

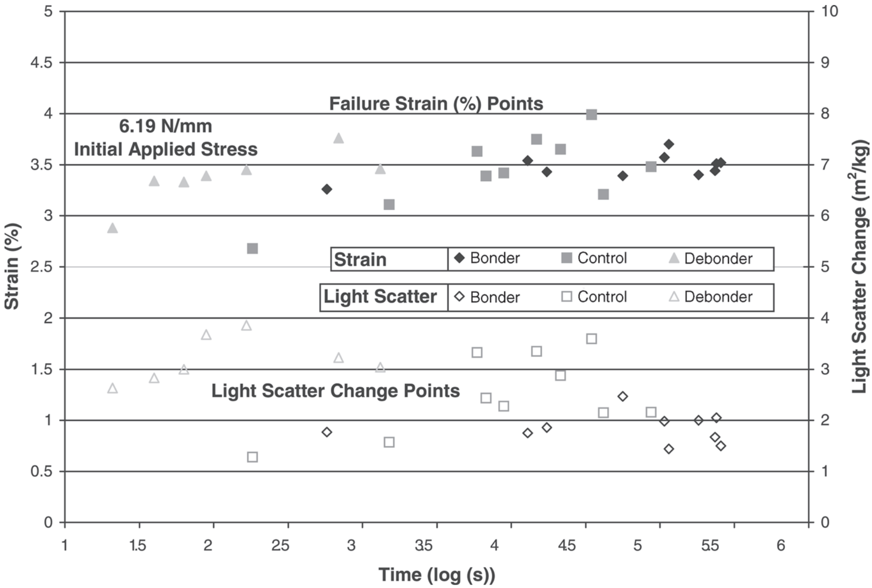

Figure 10 Creep testing failure strain and light scatter change versus time for low load wet pressed sheets.

Table 4 Failure strains, times and light scatter changes for low load wet pressed sheets.

Sheet Treatment	Failure Strain (%)	Failure Time (log(s))	Failure Time (min)	Light Scatter Change (m^2/g)
Debonder	3.37	2.53	5.69	3.18
Control	3.43	4.35	371	2.50
Bonder	3.48	5.09	2040	1.85
Variation	3.3%	102%	>>100%	71.9%

fastest in those sheets. Table 4 best summarizes the data illustrated in Figure 10.

Table 4 shows that there are large differences between the three sheet types in failure time and light scatter change. The bonder treated sheets last over two orders of magnitude longer than the debonder treated sheets. The debonder treated sheets show an over 70% increase in light scatter change from the bonder sheets. Overall, the creep curves and isochronous stress-strain curves indicate that creep compliance is different for the low load wet pressed sheets.

DISCUSSION

Deformation behavior

As paper reaches higher levels of fiber-fiber bonding, relative bonded area and specific bond strength will reach or surpass a point where only fiber deformation controls paper deformation behavior. This occurs because a sufficient amount of fiber-fiber bonding exists within the paper structure to effectively distribute load throughout the fiber network. The load distribution paths provided by the fiber-fiber bonds are redundant in the amount of relative bonded area and specific bond strength. Therefore, individual fiber-fiber bonds cannot control creep deformation. This can be considered to be a fully efficient structure. If a bonder is added to paper where fiber-fiber bonding has already reached or surpassed this point, the increase in specific bond strength will not result in a change in creep compliance. If a debonder is added to paper and it does not reduce specific bond strength to a point where fiber-fiber bonding is below this point, creep compliance will also remain unchanged. This can be correlated to work by Seth and Page[11] where they showed that the elastic modulus and stress-strain curve in paper remained unchanged at differing levels of specific bond strength as long as the paper's structure remained fully efficiently loaded. This was the case with the high load wet pressed sheets from this study.

If paper remains at a low level of fiber-fiber bonding as was the case with the low load wet pressed sheets in this study, the combination of relative bonded area and specific bond strength will be at a point where bonding will influence the paper deformation. This occurs because not enough fiber-fiber bonding exists within the paper structure to effectively distribute load through the fiber network. If a debonder is added to paper, specific bond strength will decrease, acting to further deteriorate the paper's ability to effectively distribute load through the fiber network during deformation. This will lead to increased creep compliance. This type of paper structure would be considered an inefficiently loaded structure. A bonder would act to increasingly improve the paper's ability to distribute load effectively, decreasing creep compliance. Eventually, enough bonder could be added to increase specific bond strength enough to result in a fully efficient structure.

Efficiency factor & deformation

When Seth and Page [11] introduced the concept of the efficiency factor, they hypothesized that deformation originates within the fibers and fiber-fiber bonding could influence deformation and be related to an efficiently loaded structure by means of an efficiency factor; a common efficiency factor that could be used for both elastic and viscoelastic deformation behavior. By using the efficiency factor to scale the stress magnitude, stress-strain curves with different efficiencies were superimposed on one another. This removed the fiber-fiber bonding. This was attempted with the data from this study. First, an attempt was made to superimpose all of the stress-strain curves generated from the low load wet pressed sheets and high load wet pressed sheets. Figure 11 shows the stress-strain curve before efficiency factors were applied.

It shows the three low load wet pressed stress-strain curves (debonder, control, bonder treated), the three high load wet pressed stress-strain curves (debonder, control, bonder treated) and three additional stress-strain curves (untreated-controls) at lower pressing and refining levels. Sheet treatments, freeness values, and press loads are indicated in Figure 11 and all subsequent figures and tables for the purpose of differentiation. Upon applying efficiency

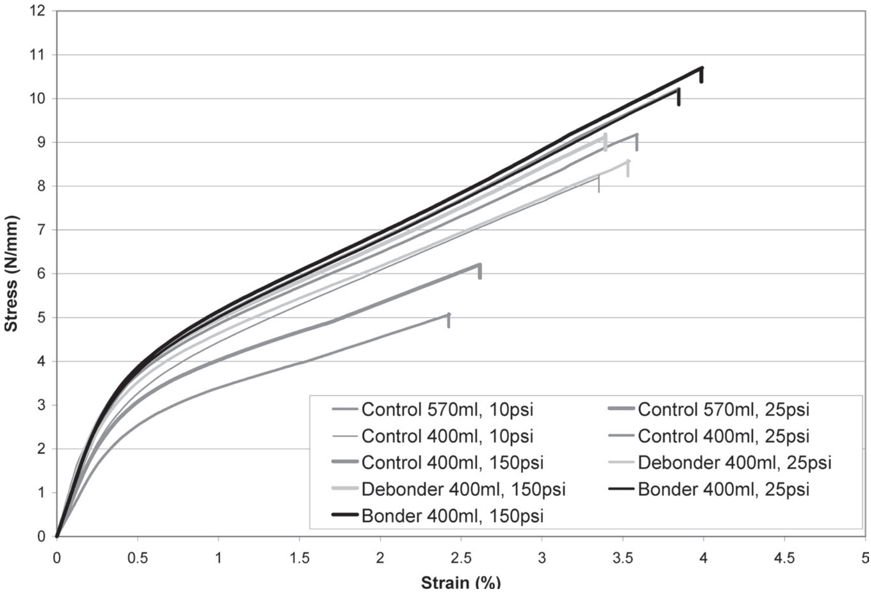

Figure 11 Stress-strain curves for all sheet conditions.

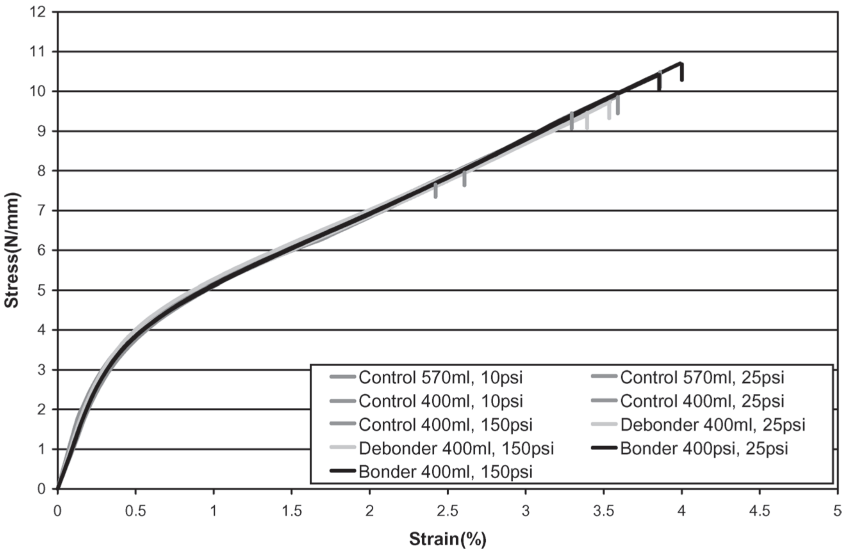

Figure 12 Stress-strain curves for all sheet conditions with efficiency factors applied.

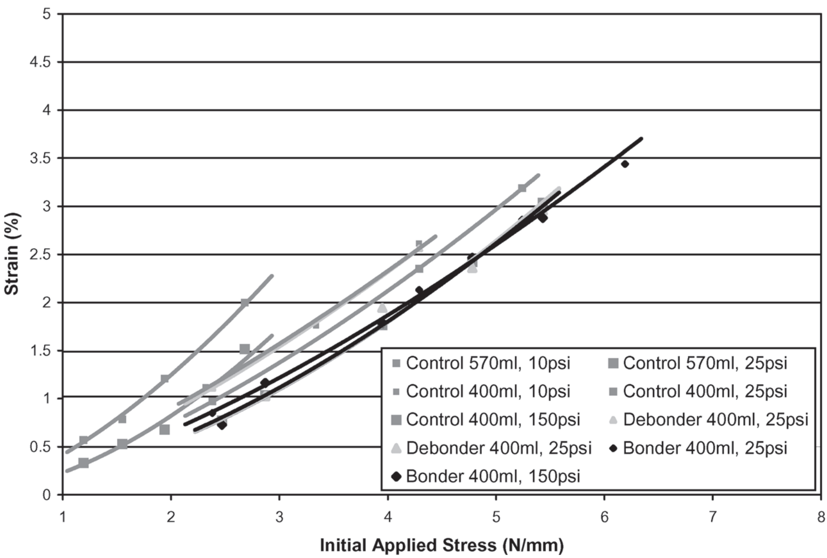

Figure 13 Isochronous stress-strain curves for all sheet conditions.

factors, the curves superimpose as shown in Figure 12. The efficiency factors used were approximated, to best superimpose the curves. These efficiency factors are compared to the efficiency factors calculated from the ultrasonic elastic modulus results later in this section.

The curves all superimpose indicating that none have severe enough fiber-fiber bonding loss during straining to reduce the efficiency factor. In other words, damage to the sheet is not severe enough to affect the deformation behavior during straining. It also confirms the work of Seth and Page[11]. The creep compliance data in Figure 13 shows the isochronous stress-strain curves for the three low load wet pressed cases (debonder, control, bonder treated), the three high load wet pressed cases (debonder, control, bonder treated) and three additional isochronous stress-strain curves (untreated-controls) at lower pressing and refining levels.

If efficiency factors are applied, the data points can be superimposed unto a common curve as shown in Figure 14. The data was fit to a power function trend line with an R^2 value of 0.988. Again, the efficiency factors used were approximated to best superimpose the isochronous stress-strain data.

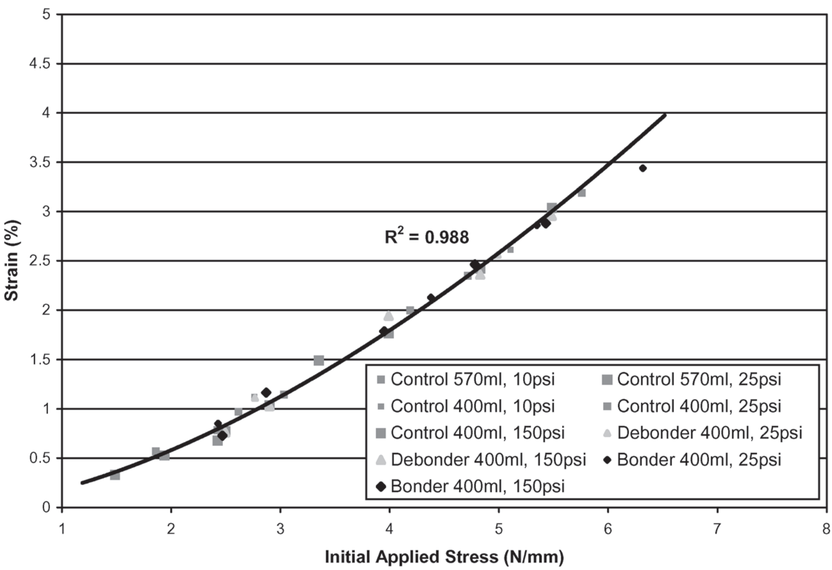

Figure 14 Isochronous stress-strain curve for all sheet conditions with efficiency factors applied.

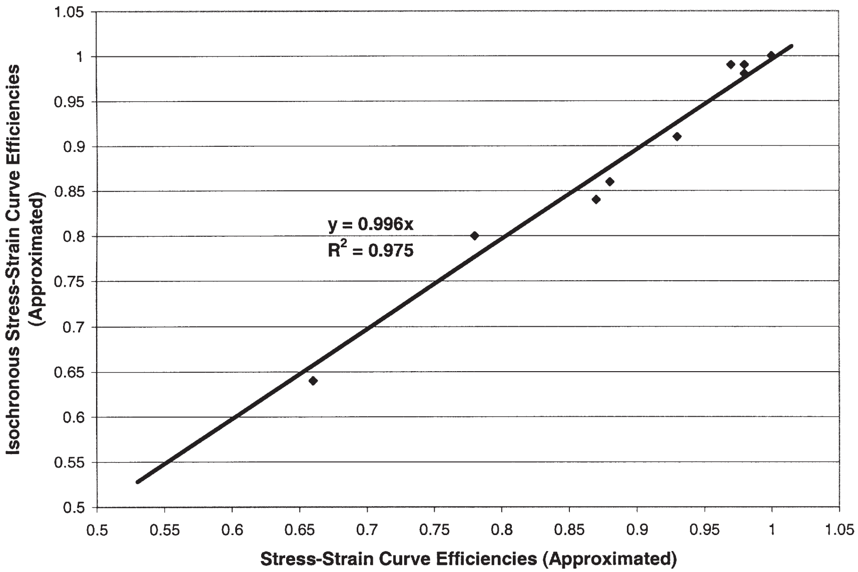

Figure 15 Isochronous stress-strain curve efficiency factors versus stress-strain curve efficiency factors.

Overall, the efficiency factors approximated to superimpose the stress-strain curves from Instron testing (shown in Figure 12) and the isochronous stress-strain data from creep testing (shown in Figure 14) were consistent with each other. Figure 15 shows the efficiency factors approximated for the isochronous stress-strain curves versus the efficiency factors approximated for the stress-strain curves.

Figure 15 shows the slope of the linear trend line at 0.996 with an R^2 of 0.975 indicating a one to one relationship between the efficiencies used to overlap isochronous stress-strain curves from creep testing and stress-strain curves from physical testing. This data indicates the isochronous stress-strain curves generated from 24 hours of creep deformation and the stress-strain curves generated from a less than 20 second Instron test can have common efficiency factors applied to them, meaning fiber-fiber bond breakage was not significant enough to reduce the efficiency factor for the creep compliance results. It however, does not prove that efficiency factor does not decrease over longer creep testing durations where damage to the sheet may occur from the decrease of fiber-fiber bonding. Nevertheless, efficiency factors can be applied to the creep data to make it superimpose and these factors are consistent with the Instron stress-strain curve efficiency factors.

In addition, efficiency factors were calculated using the ultrasonic modulus data in Tables 1 and 3. Figure 16 shows these calculated efficiency factors from the ultrasonic modulus data versus the efficiency factors approximated to superimpose the stress-strain curves from Instron testing (shown in Figure 12) and the isochronous stress-strain curves from creep testing (shown in Figure 14).

Figure 16 indicates that there is good agreement as the slopes of both trend lines indicate a one to one relationship between calculated efficiencies from ultrasonic modulus data versus the approximated stress-strain curve efficiencies (line slope of 0.992 and R^2 of 0.985), and the approximated isochronous stress-strain curve efficiencies (line slope of 0.988 and R^2 of 0.947). This first indicates good consistency between physical testing results and creep testing results. Having three sets of data (ultrasonic elastic modulus, stress-strain curves from Instron testing, and isochronous stress-strain curves from creep testing) relate so well as indicated in Figures 15 and 16 is excellent considering all possible sources of error. This also indicates that calculated efficiency factors from ultrasonic modulus data can be shown to still apply to deform-

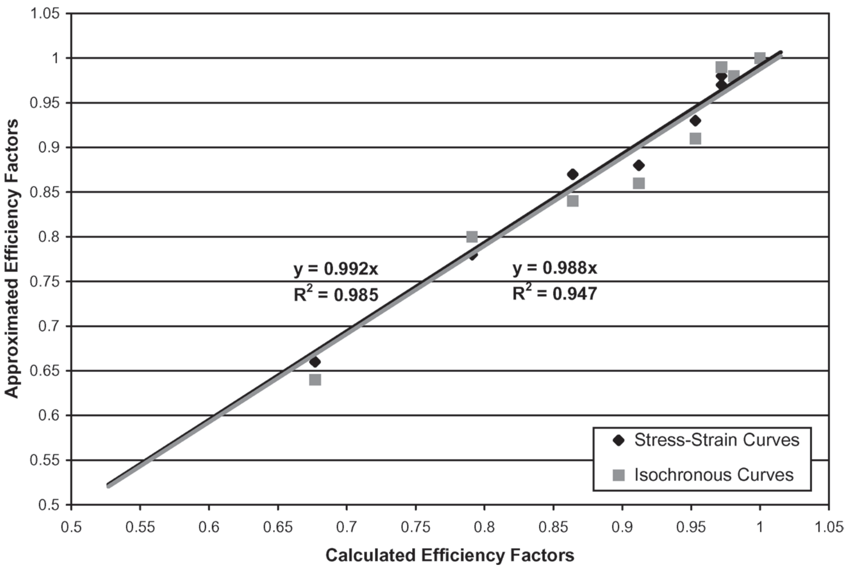

Figure 16 Approximated efficiency factors versus calculated efficiency factors.

Table 5 Calculated efficiency factors from ultrasonic modulus data and approximated efficiency factors for stress-strain curves and isochronous stress-strain curves.

Sheet Treatment	Ultrasonic Modulus Data	Stress-Strain Curves	Isochronous Stress-Strain Curves
Control 570ml, 10psi	0.68	0.66	0.64
Control 570ml, 25psi	0.79	0.78	0.80
Control 400ml, 10psi	0.86	0.87	0.84
Debonder 400ml, 25psi	0.91	0.88	0.86
Control 400ml, 25psi	0.95	0.93	0.91
Bonder 400ml, 25psi	0.98	0.98	0.98
Debonder 400ml, 150psi	0.97	0.97	0.99
Control 400ml, 150psi	0.97	0.98	0.99
Bonder 400ml, 150psi	1.00	1.00	1.00

ation behavior that is neither elastic nor rate independent in behavior, supporting Seth and Page[11]. Table 5 shows the efficiency factor data used to generate Figures 15 and 16.

Further evidence that efficiency factors can apply towards creep compliance comes from data reported by Brezinski[5]. Figure 17 shows isochronous stress-strain curves from a series of sheets made at differing wet pressed and refining levels; wet pressing levels ranging from 10 psi to 800 psi and refining levels that gave a pulp freeness ranging from 425 ml to 775 ml.

Upon applying approximated efficiency factors, the data points all fall unto a common isochronous creep curve as seen in Figure 18. This curve was generated using a power function trend line with an R^2 of 0.991. The Brezinski[5] data is more dramatic than the data from this study as there was greater spread between the isochronous stress-strain curves. There was no available elastic modulus data to compare the efficiency factors approximated (to create Figure 18) to efficiency factors that would have been calculated from elastic modulus data. Still the Brezinski[5] data confirms that other creep data using a different pulp and processing techniques follow the same trend; a trend where efficiency factor's can be applied to the data of inefficiently loaded structures to create a data set that behaves like an efficiently loaded structure.

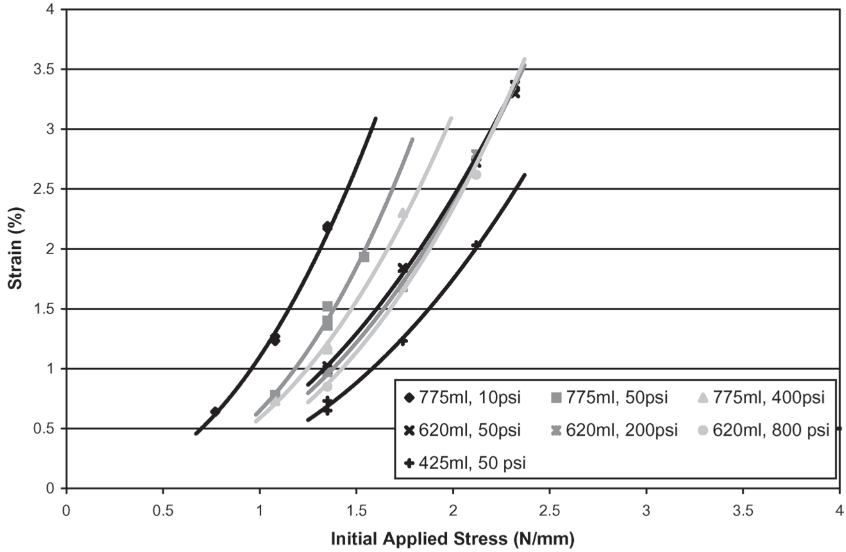

Figure 17 Brezinski[5] isochronous stress-strain curves.

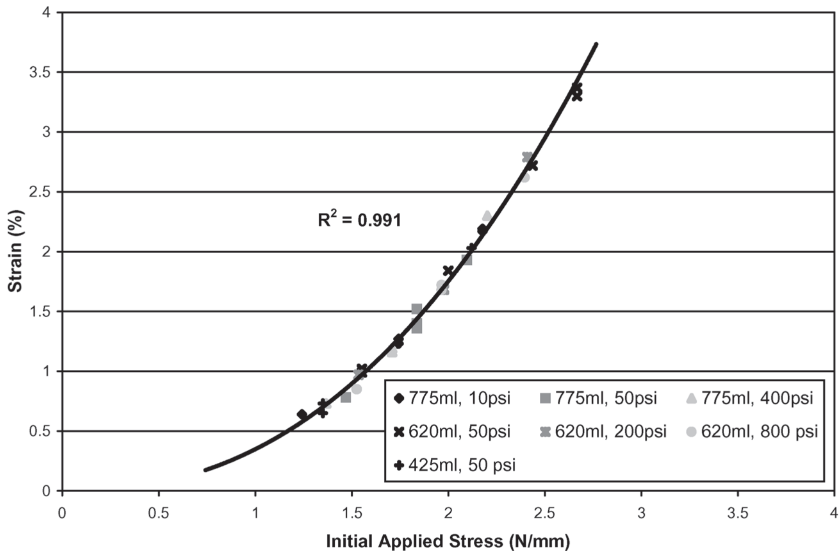

Figure 18 Brezinski[5] isochronous stress-strain curve with efficiency factors applied.

Failure behavior

With regard to creep failure time, it was influenced by fiber-fiber bonding for both the low load wet pressed and high load wet pressed sheets. In a general sense, ultimate failure occurs when localized bond and fiber failure (damage) becomes significant enough to cause part of the structure to partially or completely stop bearing load. The remainder of the paper redistributes that load continually to compensate until it can no longer bear it and fails. If a bonder is added to the paper, specific bond strength increases and the rate at which relative bonded area decreases during creep deformation is diminished. As a result, the time when failure occurs is increasingly due to the fibers themselves. The higher the specific bond strength or relative bonded area, the less influence bonding has on failure. If a debonder is added to the paper, the opposite would occur. Therefore, bonds would play an increasingly important role in the time of creep failure, acting to diminish it. The Page Equation [12] offers an empirical explanation of how fiber strength and fiber-fiber bonding influence the tensile strength in paper. This can be seen as a corollary to failure time in creep. Theoretically, according to the Page Equation [12], relative bonded area and specific bond strength could be increased past a point where even bonding will have no influence on failure. This could be called a "super" efficiently loaded structure. None of the sheets made in this study achieved that level of fiber-fiber bonding.

Fiber-fiber bonding regimes

Overall, one can imagine that paper can be placed in one of three regimes depending on the level of fiber-fiber bonding. The first regime would be where fiber-fiber bonding is at a level where deformation behavior and failure behavior are influenced. This would be considered an inefficiently loaded structure. This is what occurred in the low load wet pressed sheets where creep compliances were different and failure times were different. The second regime would be where fiber-fiber bonding is high enough where deformation behavior is unaffected but failure behavior is influenced. This would be considered an efficiently loaded structure. This is what occurred in the high load wet pressed sheets, where creep compliances were the same and failure times were different. The third regime would be where fiber-fiber bonding reaches a "super" efficiently loaded state and neither deformation behavior nor failure behavior would be influenced. Again, no sheets were made in this regime. A conceptual relationship between structural efficiency and fiber-fiber bonding is shown in Figure 19.

With reference to Figure 19, two aspects of fiber-fiber bonding and effi-

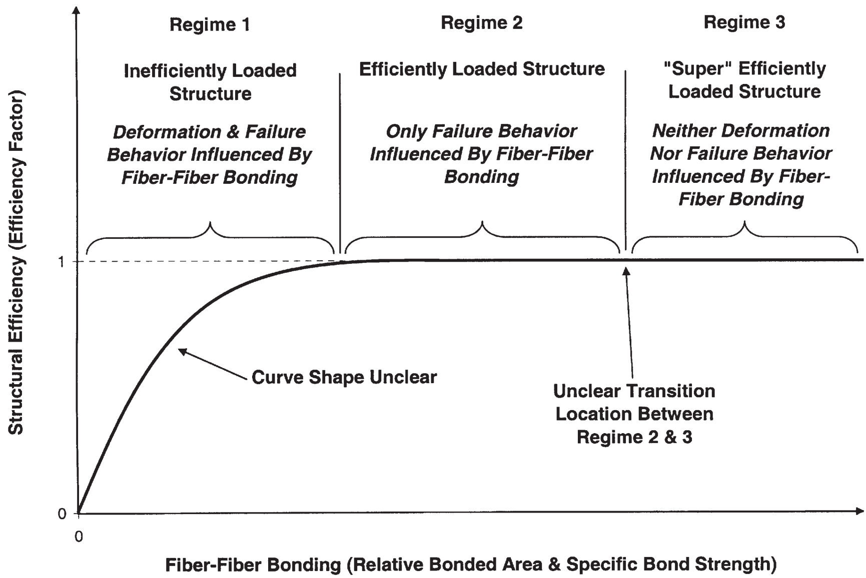

Figure 19 Conceptual relationship between structural efficiency and fiber-fiber bonding.

ciency are unclear. First, it is unclear what the exact relationship between structural efficiency and fiber-fiber bonding is when efficiency is less than one. Most likely, the curve on Figure 19. is a good approximation. It is likely that fiber-fiber bonding has more of an influence on efficiency when it is close to "0" and has a diminishing influence as efficiency approaches "1". More lab work would need to be conducted to quantitatively determine the shape of this relationship. Second, it is unclear exactly where the fully efficient regime ends and "super" fully efficient regime begins. It would be theoretically possible to quantitatively determine this if more laboratory work is done. This would most likely be accomplished by making high load wet pressed sheets with high levels of bonder.

CONCLUSIONS

Creep compliance in paper will reach a minimum as higher and higher levels of fiber-fiber bonding are achieved. This is because fiber-fiber bonding can be improved until an efficiently loaded structure is created; a structure which can effectively distribute load. Once the point is passed where the paper structure becomes fully efficient, only creep failure time can be increased with increased levels of bonding. This substantiates the premise that a fully efficient structure is not influenced by fiber-fiber bonding with regards to deformation behavior, only failure behavior. This again correlates with what Seth and Page[11] saw with elastic modulus and stress-strain behavior.

In addition, it is possible to apply efficiency factors to the creep data of inefficiently loaded structures and create a data set that superimposes with creep data from an efficiently loaded structure. This efficiency factor can be calculated by relating elastic modulus data and still applies to the time dependent viscoelastic deformation seen with stress-strain behavior and creep behavior. The efficiency factor in effect relates how well the existing fiber-fiber bonding allows the structure to effectively distribute load throughout the sheet; a structure where deformation originates within the fiber and fiber-fiber bonding can only influence deformation at a less then fully efficient loaded condition. This relation will hold true as long as efficiency factor does not decrease with strain due to excessive relative bonded area loss (sheet damage). This did not occur with the data in this study but has been shown to occur in work by Seth and Page[11].

Furthermore, it has been shown that differences or similarities in deformation behavior do not necessarily correspond to differences or similarities in failure behavior with regards to creep or other physical properties. Depending on which one of three regimes fiber-fiber bonding is in, it has been shown that it is possible to create sheets with differing levels of fiber-fiber bonding (either in relative bonded area or specific bond strength) that have:

- The same deformation behavior and different failure behavior.
- Different deformation behavior and different failure behavior.
- While not achieved in this study, the same deformation behavior and the same failure behavior.

ACKNOWLEDGMENTS

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Transcription of Discussion

INFLUENCE OF FIBER-FIBER BONDING ON THE TENSILE CREEP COMPLIANCE OF PAPER

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Lars Wågberg KTH (*prepared contribution*)

I want to show you a graph (see Figure 1 Page 1490). Mats Rundlöf and I have measured the light scattering of filter papers with different hole diameters and as you see in the figure when the diameter is below 0.2 *µm*, the light scattering drops dramatically. This is expected, because you cannot determine these distances with white light. I do not necessarily disagree with your overall conclusions, but your conclusions are based on a method that cannot determine fibre-to-fibre bonding in detail and I want to stress that and if you want to determine it in the future, you have to use other methods. I also want to draw your attention to the statement you make on page 752 that no fibre-fibre bonding has been determined in this work. This means that the title of your paper is a bit misleading.

Andrew DeMaio

As part of that thought, I understand that there is this debate with light scattering behaviour. That, of course, is one of the main reasons why I am going to do microscopy work to try to visualise these fibre-fibre bonding areas. Also, you can infer that there are differences in fibre-fibre bonding between the sheets, because the z-directional strengths are tremendously different, and granted, the failure mechanism for z-directional tensile strength is more out of plane than it is a shear action. Even so, I think you can say that, if it is stronger in this direction, we can assume that the bonding is actually different between these samples.

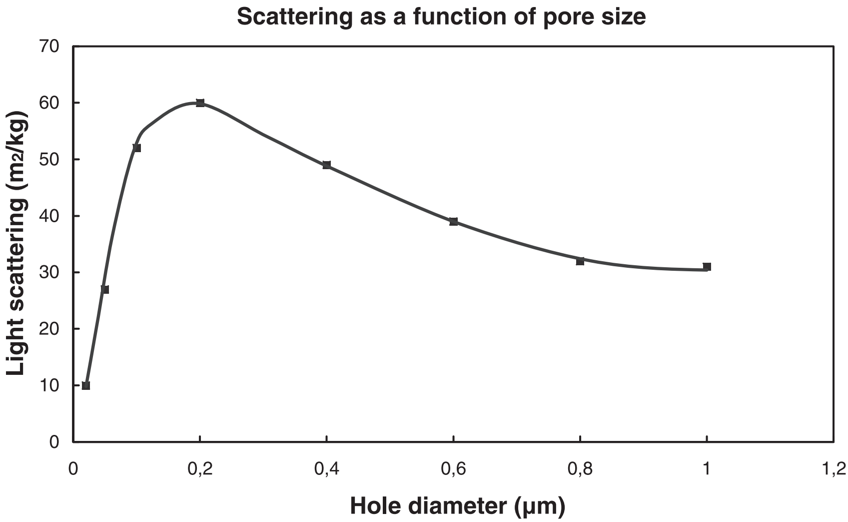

Figure 1 Light scattering coefficient as a function of hole diameter for a series of filters with well-defined cylindrical holes of a very narrow size distribution. The scattering properties of the filters were obvious to the eye, from milky white to transparent in the case of the two filters with the smallest hole size. An attempt to quantify this was made by calculating the Kubelka Munk light scattering coefficient from measured reflectance factors over a white and a black background according using the CIE-Y filter (peaks at 557 *nm*) and conforming to ISO standard methods (d/0° geometry, C2 illumination). Figure 1 shows a decrease in the *s*-value corresponding to the observed transparency and the smallest hole sizes, as expected.

Each type of filter was examined using scanning electron microscopy which showed that the holes were indeed cylindrical and of uniform size; the microscopy also showed that the number of holes per unit area was comparable and certainly not smaller for the smaller hole sizes, which excludes the possibility of too few scattering sites as a reason for the low scattering ability. The filters, except for the filters with 0.8 *µm* holes, were made of polycarbonate and delivered by Nucelopore. The 0.8 *µm* filters consisted of mixed cellulose esters and were delivered by Millipore. The holes were prepared by laser etching and the filters were of similar grammage, around 10 $g\ m^{-2}$.

Joel Panek Iggesund Paperboard

Very interesting work. I have a question about the isochronous curves that you had superimposed with the efficiency factor. What timeslice were the curves from and would you expect to see a different result, say, if all these curves were from 24 hours versus they were all from, say, 10 days. Does that not make a difference?

Andrew DeMaio

In the bulk of my data, most data points are from the 24-hour regime, that is why I use that regime, but it does not mean I did not try other times. I also looked at 48 hours or up to 72 hours but I do not have as many points, so it is not as impressive, but they also overlap quite nicely. Even if I go backwards inside 24 hours and look at 12 hours, 10 hours, whatever, I still see this overlap.

Wolfgang Bauer Graz University of Technology

I think you used a locust bean gum as a bonder. Yesterday, we heard in other presentations that there are many different mechanisms of bonding. If you used another mechanism for bonding, would you also get other results?

Andrew DeMaio

That is a good question. I looked at that same paper yesterday and the reason I chose locust bean gum was because I was under the impression that this particular agent would not affect the other properties. Particularly, it would not change the density, would not significantly change relative bond area and according to that paper, they actually cite that there were studies using locust bean gum that show exactly what locust bean gum predominantly does. It just improves bond strength.

Yes, I tried other bonding agents. I tried some pretty fancy bonding agents and what happened was the first one I tried had retention aid in it. The retention aid meant that I got completely different results because I changed my fibre properties, I believe, because I was keeping fines in the sheet that I did not have in my other sheets. So, the answer to your question is that you have to be really careful which bonding agent you use for this to work. I went through about 20–30 iterations of handsheets before I got the procedure and the method down perfectly enough, so that I could isolate this one variable.

Bob Pelton McMaster University

I guess perhaps the most spectacular de-bonding agents are calcium carbonate fillers. I have not heard very much discussion about fillers at this meeting and so I have a question to you, or perhaps the last speaker. What do we know about the effect of fillers on the creep properties of paper?

Andrew DeMaio

Well, I do not think I have seen much information about it. If I were to try to do this particular study with filler, the effect would be to replace fibres with fillers. This would mean that I would not be able to do this study because, in effect, I have changed the makeup of the sheet.