Strength development through refining and wet pressing

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ABSTRACT: At a given level of sheet densification, it is usually found that refining and wet pressing yield a higher tensile strength than wet pressing alone. An attempt was therefore made to examine this behavior within the framework of linear elastic fracture mechanics. It was hypothesized that at a given level of densification, differences in pore size distribution would be evident, and that this would be a factor in strength development, i.e., the larger pores would give rise to greater stress concentration or critical crack size. Although a correlation of the data based on Griffith's equation was not successful, a good correlation was found between specific tensile strength σ^* *and the product of specific fracture resistance* G_c^* *and specific modulus* E^* *of the form* $\sigma^* = A(E^*G_c^*)^r$. *This relationship seems to imply that tensile strength is not dependent on crack size. However, tensile strength does show a sensitivity to single-edge notches particularly at the highest level of refining. Curiously, the size, shape, and distribution of the pores did not appear to be important factors.*

KEYWORDS: Bibliographies, cracks, densification, elastic strength, fracture, mechanical properties, paper properties, Pinus, pressing, properties, refining, softwoods, tensile properties, tensile strength, wet pressing.

Refining and wet pressing are important process steps of papermaking. The primary function of refining is to modify the structure of the fibers in order to enhance their papermaking potential, while wet pressing removes water and consolidates the web prior to drying. In this paper, we will consider how the tensile strength of paper is influenced by refining and wet pressing.

Generally, at a given level of bonding or sheet densification, refining will produce a paper having a higher tensile strength than one which has only been wet pressed, e.g., see **Fig. 1**.

A satisfactory explanation for this difference has yet to be given. Although refining offers positive benefits to the papermaking process, it nevertheless has an adverse effect on water removal. In contrast, wet pressing is viewed as an effective means of removing water mechanically and, as a consequence, many of the strength properties of the sheet are enhanced. A better understanding of strength development by refining and wet pressing may therefore lead to the achievement of a better balance between these processes in order to better optimize strength properties and productivity. Is it possible that some grades of paper may be made with little or no refining if the water removal and consolidation process can be improved?

Fracture mechanics is the main approach we have employed toward an understanding of this problem. In other areas of material science, a common goal is to understand the nature of flaws and stress concentration. This has led to strength improvements in such materials as glasses, polymers, cements, and composites.

Literature review

In standard pulp evaluation procedures, refining is usually the only papermaking process variable which is varied, although there are a number of studies in the literature where both refining and wet pressing have been varied, e.g., *(1–4)*. In most cases, we typically see the tensile strength versus density—or bond area envelope *(1)*—shown in Fig. 1.

A number of theories have been developed for predicting the tensile strength of paper *(5–9)*, which have been reviewed by several authors *(7–9)*. Page's equation *(5)*, however, seems to be the one which is most often used

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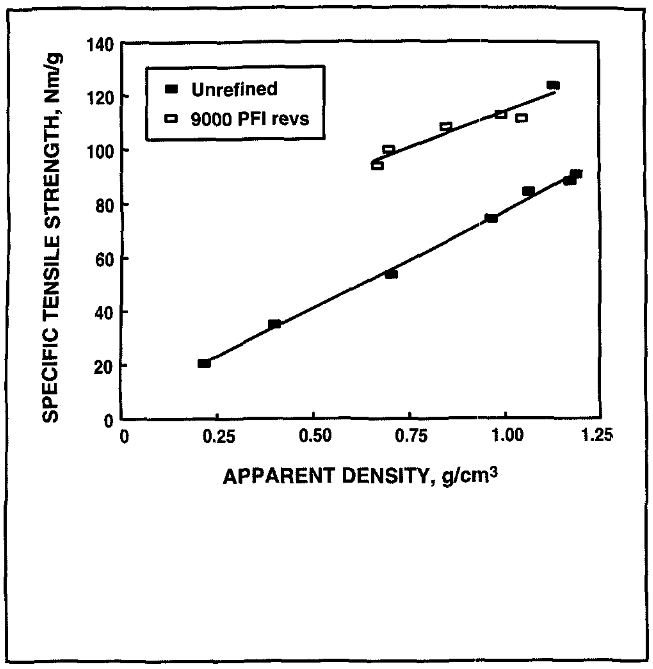
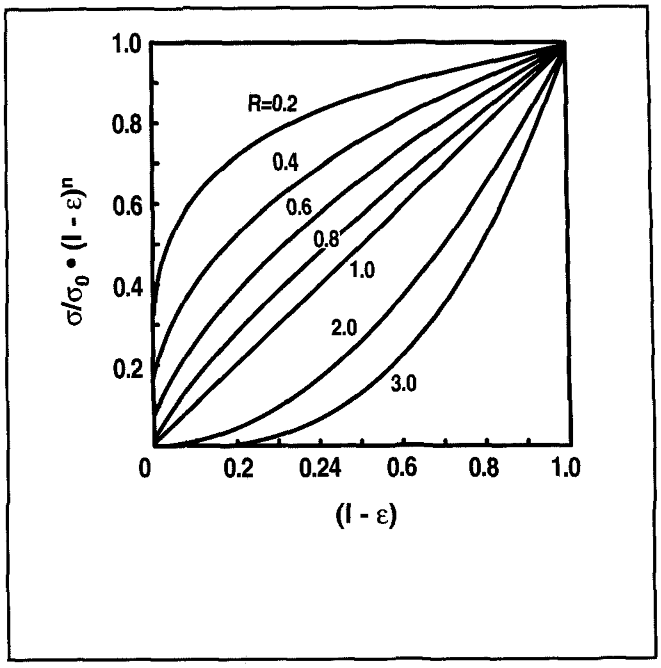
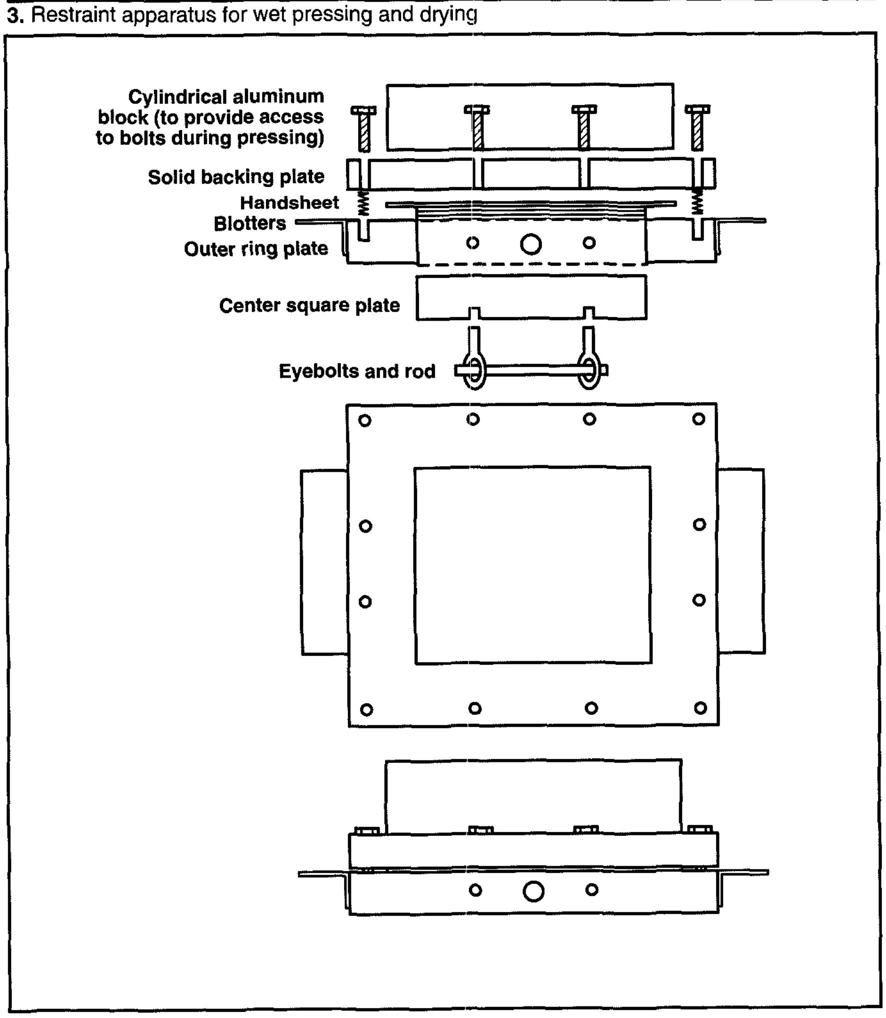
1. Specific tensile strength versus apparent density



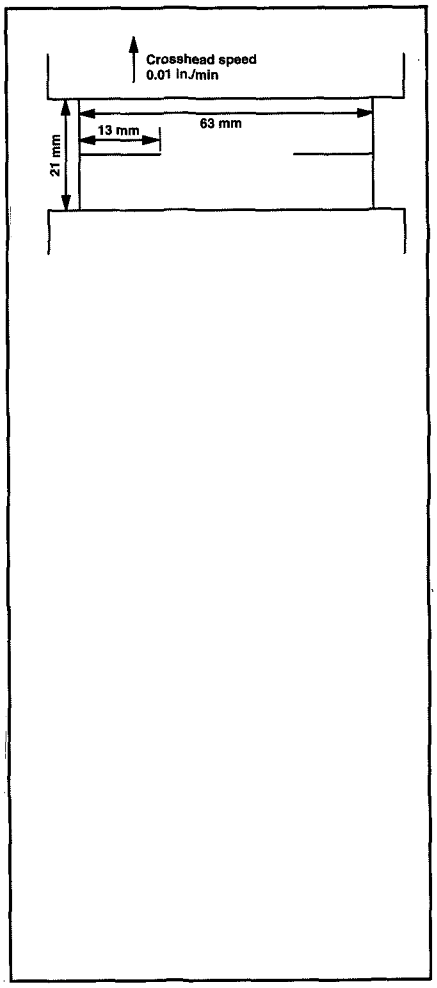
2. Modeling the tensile strength of paper as a voidal continuum



3. Restraint apparatus for wet pressing and drying



4. Single-edge-notch sample and dimensions



to explain the tensile strength of paper. It involves six independent variables: namely, fiber strength, bond strength, fiber length, fiber perimeter, fiber coarseness, and relative bonded area.

Single interfiber bond studies, mainly by Stratton *(10)* and Mohlin *(11)*, have shown that specific bond strength does not change with refining, while the work of McIntosh *(12)*, Button *(13)*, Stratton *(10)*, and Hieta *et al.* *(14)* suggest that bond strength may be dependent on bond geometry, i.e., the results of McIntosh and Stratton show that latewood fibers yield a higher interfiber bond strength than earlywood fibers. Of course, we have yet to determine if single interfiber bond strength results are relevant to paper. One problem with single interfiber bond tests which has not yet been addressed is the influence of fines, although Nanko's *(15)* recent findings indicate that this might be an important consideration. Retulainen and Ebling *(16)* have evaluated a number of methods for measuring bond strength and found it to increase in some instances with refining.

Button *(13)* investigated the strength of lap joints using cellophane model fibers. He found that as the bond width-to-thickness ratio decreased, specific bond strength increased, and these findings were in close agreement with theoretical predictions based on linear elastic fracture mechanics (LEFM). It is interesting to speculate on whether these findings explain the strength differences between earlywood and latewood single interfiber bonds *(12)*.

Bond strength changes due to wet pressing are less clear-cut, although the indications are that bond strength based on energy measurements is reduced, according to the findings of Nordman *(17)* and Skowronski *(18)*.

Increases in fiber strength due to refining have been found by a number of researchers, including Alexander *et al.* *(3, 4)* and Kim *et al.* *(19)*. The increase is justified on the basis of increased fibril orientation and the reduction of cell-wall defects such as microcompressions which occur during restrained drying of the fiber.

Tensile strength is also dependent on formation, and as the uniformity in mass density deteriorates, so does tensile strength. Presumably, Page's equation would interpret this as a loss in bonding strength, although loss of uniformity suggests an increase in stress concentration within the network.

In summary, Page's equation suggests that refining and wet pressing may be more effective in improving strength than wet pressing alone, since both fiber and interfiber bond strength may be improved by refining, while wet pressing alone may actually reduce interfiber bond strength. Nevertheless, bond strength remains something of an enigma, since there is some contradiction between the inferences made from network and single interfiber bond strength determinations.

A tensile strength model for paper where it is viewed as a voidal continuum has also been proposed by Waterhouse *(8)* and is illustrated in **Fig. 2.** The interesting question which arises is how do we explain the difference in strength at a given level of density or solidity?

At a given level of densification, we have the same void volume irrespective of how it was produced. However, there must be a difference in size and shape of the pores constituting that void volume. Furthermore, these dissimilarities may result in stress concentration differences which could be responsible for the strength differences found. Dissimilarities in pore size, shape, and distribution should, at the appropriate level of resolution, result in differences in mass density distribution. This result has been confirmed in more recent work *(20)*.

For brittle materials such as glass, cement, some metals and polymers, the ideas of fracture mechanics have been used to examine the impact of flaws or defects on their ultimate strength. Equation 1, the classical Griffith equation for tensile strength, shows that strength deteriorates as the inverse square root of the critical crack or flaw size:

$$\sigma = [(E \times G_c)/(\pi \times c)]_{0.5} \qquad (1)$$

where
E = modulus of the material
G_c = fracture roughness
c = critical crack size.

Nissan *(21)* was one of the first researchers to apply fracture mechanics to paper and to conclude that LEFM was not applicable to paper. Since that time there have been a number of papers and reviews *(22–33)* on the subject, where consideration has been given to such topics as whether paper may be treated as a brittle material, its fracture behavior when gross flaws such as edge cuts are present, measurement of fracture resistance for both regular and very tough papers, and measurement of out-of-plane fracture resistance.

Experimental

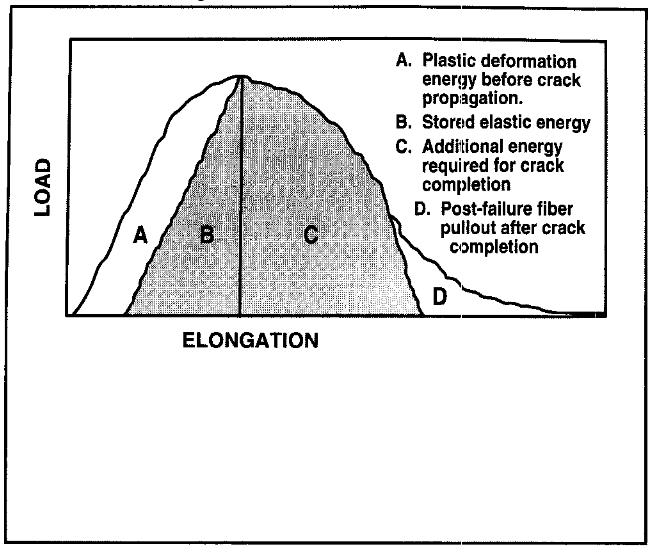
Pulps and handsheet making

The two low-yield softwood bleached kraft pulps used in this study were a northern pine and a southern pine. The pulps were never dried and were thickened to 30–35% consistency and were stored with formaldehyde in the cold room at 4°C until required.

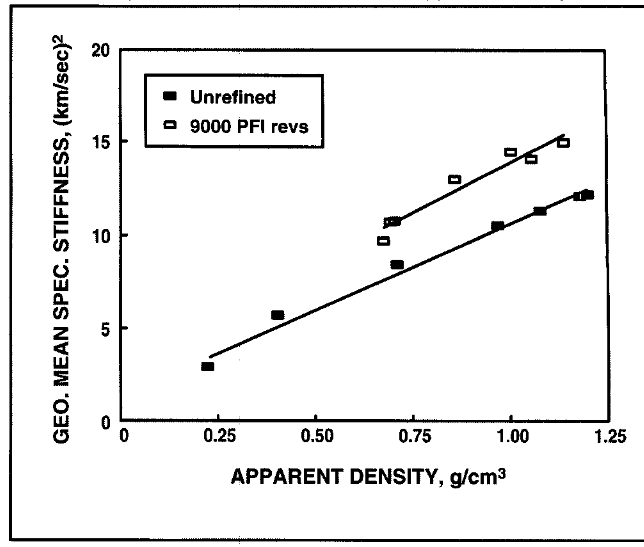
The pulps were beaten in a PFI mill using a gap setting of 0.4 mm, and a series of runs were made to provide sufficient pulp for sheetmaking in the Formette Dynamique. The two refining levels were 0 revs and 9000 revs.

Handsheets having close to a random orientation of fibers were made on the Formette Dynamique *(34)* using an H 1/4 U2510 nozzle and a wire speed of 820 m/min. After forming, the handsheets were centrifuged by increasing the wire speed to 1900 m/min which resulted in a couched solids of approximately 23%. After forming and couching, the handsheets were lightly marked with indelible pencils a known distance apart. The spacing between these marks was checked after pressing and drying in order to detect any changes in sheet dimensions. Following this procedure, the sheets were placed between wet blotters of approximately the same consistency and stored

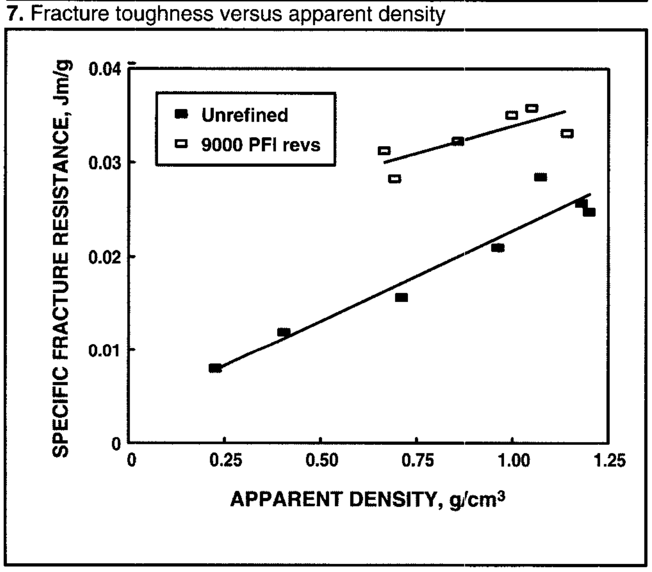
5. Tensile load elongation curve for fracture



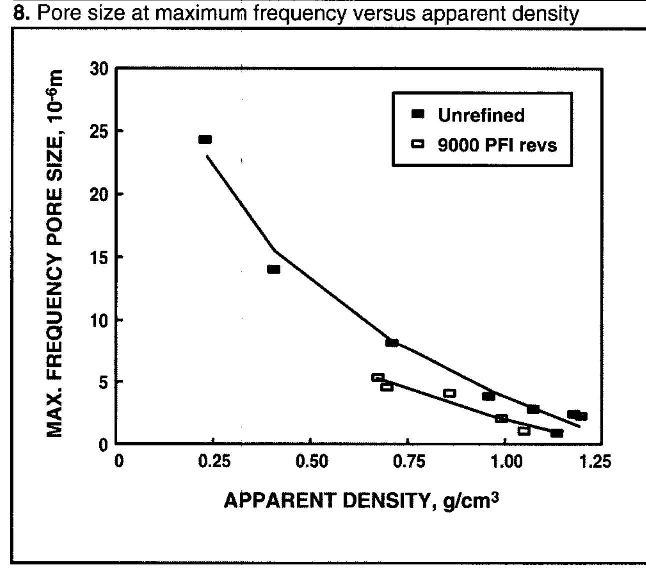
6. In-plane specific elastic constant versus apparent density



7. Fracture toughness versus apparent density



8. Pore size at maximum frequency versus apparent density



in the cold room to await pressing and drying.

We wished to achieve by refining and wet pressing high levels of densification, i.e., around 1 g/cm³ and at the same time maintain complete restraint until the sheet is dry. The traditional separation of wet pressing and drying which is used for handsheet preparation is not satisfactory for this purpose. In press drying, Setterholm and Kuenzi *(35)* showed that once water is removed to below the fiber saturation point then restraint on the sheet is required until it is fully dry to prevent shrinkage. It was found that when pressing to a high solids content, pressing and drying can no longer be separated. In order to achieve the required restraint conditions during wet pressing and drying, two methods were used, as discussed by the authors in a recent presentation *(36)*.

The method used for restraint during pressing and drying of the northern pine 200-g/m² handsheets is shown in **Fig. 3**.

The second method used for continuous restraint during pressing and drying was the IPST press and dryer combination.

After pressing and drying, the sheets are first preconditioned and then conditioned according to TAPPI recommended procedures.

Nondestructive testing

Nondestructive characterization of the handsheets consisted of measuring changes in sheet dimensions, hard and soft platen calipers *(37)*, grammage, and in-plane and out-of-plane elastic constants using ultrasonic wave propagation techniques developed at IPST *(38, 39)*.

Fracture resistance

Fracture resistance measurements are based upon the quasi-static method proposed by Gurney and Hunt *(40)*, and later used by Seth and Page *(25)* to measure the fracture resistance of paper.

A modified IPST in-plane tear tester *(41)* was used to perform these tests. Normally, the jaws of this tester are rotated through 6°, but for the purposes of having a pure mode I fracture they were kept at 0°. Seth and Page *(42)* have indicated that this should give agreement with their method. The double-edged-notch (DEN) sample geometry and dimensions are given in **Fig. 4**.

The fracture energy was measured from the appropriate areas under the load-displacement curve which is sketched in **Fig. 5**.

The energy involved in fracture is calculated from the sum of the areas A and B. The fracture resistance R multiplied by the sheet thickness t is obtained by dividing the energy of fracture by the crack length (units of J/m). The specific fracture resistance is obtained by dividing this quantity by the sample grammage. The crack tortuosity factor is defined as the ratio of the actual to geometric crack length, i.e., 37 mm. (See Fig. 4.) This factor appears to depend on formation, for example, trials with cellophane gave a tortuosity factor of 1.0, scratch pad paper 1.35, and Formette handsheets 1.1.

Mercury porosimetry

Pore size distribution of the 200-g/m^2 handsheets was measured using an American Instrument Co. Porosimeter, model 5-7121, 0–103 MPa (0–15,000 psi) range.

Tensile and notched tensile testing

Tensile testing of the 100-g/m^2 handsheets was done using rectangular samples 127 mm x 25.4 mm and a cross-head speed of 12.7 mm/min. Sample slippage, even with line clamps, was experienced when testing the 200-g/m^2 handsheets; therefore, dogbone-shaped tensile samples were used. The test section of the dogbone-shaped samples measured 58.9 mm x 12.7 mm, and the overall length of the samples was 114.3 mm.

Single-edge notched tensile tests were also performed on the 100-g/m^2 handsheets using rectangular samples 127 mm x 25.4 mm and a cross-head speed of 12.7 mm/min. The single-edge notch was cut with an X-Acto knife normal to the left-hand edge at mid-span of the tensile specimen. The notch sizes were 0, 0.51, 1.27, 2.54, 3.81, 6.35, 8.89, 10.16, and 12.7 mm.

Results and discussion

The variation of in-plane elastic properties, fracture resistance, median pore size, and tensile strength with densification by wet pressing at two levels of refining for the northern softwood 200-g/m^2 handsheets is shown in **Figs. 1 and 6–8.**

According to Page and Seth *(43)*, there are three basic mechanisms to account for the variation of mean in-plane specific elastic constant with apparent density shown in Fig. 6. The first is that increasing densification at any level of refining will increase relative bonded area (RBA) and thus reduce the fiber end effect. The second mechanism proposed is that the fiber's effective modulus is increased through fibril realignment, i.e., reduction of microcompressions. The reduction of gross kinks and curl, particularly in market pulps, and concomitant increase in load bearing fibers is the third mechanism proposed. No measurements of fiber curl or the frequency of microcompressions were made during this study. However, in view of the fact that the pulp was never dried, it is tentatively concluded that refining has produced an effective increase in fiber modulus.

The variation of out-of-plane specific elastic constant with apparent density is shown in **Fig. 9**. An envelope in this property is much less distinct. A theoretical prediction for the out-of-plane elastic constant has been given by Berger *(44)* and is equal to the product of the fiber's transverse specific elastic constant multiplied by RBA. Therefore, if there is an increase in the out-of-plane specific elastic constant with refining at a given level of RBA, this would imply an increase in the transverse elastic constant of the fiber with refining.

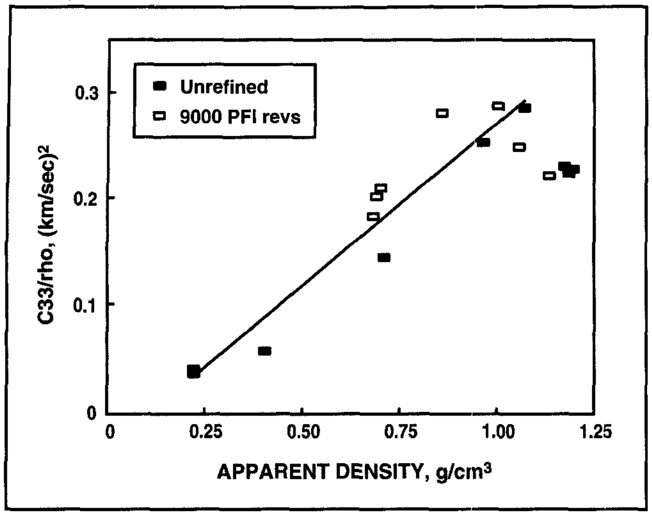
There is a scarcity of data on the transverse elastic properties of fibers. Berger found that even with low levels of refining, the transverse stiffness of the fiber decreased, and attributed this to a loss of the fiber's primary and outer secondary wall layers. We also see for a density greater than about 1 g/cm^3 that there is a reduction in out-of-plane elastic constant. No satisfactory explanation can yet be given for this effect, although some form of damage is suspected, i.e., a calendering effect.

A number of researchers have measured the fracture resistance of paper; however, the micromechanics of fracture have yet to receive attention, i.e., the role of fiber and interfiber bonding, etc. Such an understanding would be helpful in explaining the variation of fracture resistance with densification shown in Fig. 7. It is seen that refining is more effective than wet pressing in increasing fracture resistance.

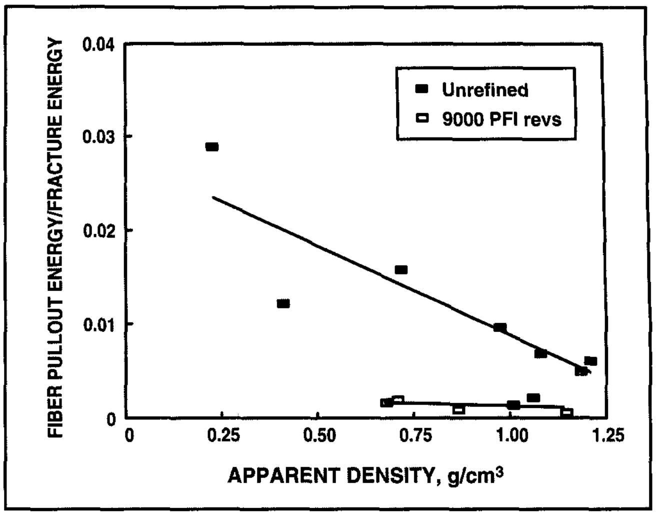
We have observed that there is a greater degree of fiber pullout at the lowest level of refining, while fiber failure is predominant at the highest level of refining. With reference to Fig. 5, we have also found that the work of separation, i.e., area D, is also greater for the lowest level of refining, as shown in **Fig. 10.** This suggests that the basic mechanisms operative in fracture are similar to those controlling tensile failure as, proposed by Page *(5)* and Kallmes *(6)*, i.e., predominantly bond failure at low levels of refining progressing to predominantly fiber failure at high levels of refining.

The effect of refining and wet pressing on pore size at maximum frequency is shown in Fig. 8. At a given level of densification, i.e., void volume, we see that increased refining results in a greater number of smaller pores. Since these pores are likely to be interfiber pores, then it follows that on average the size of interfiber bonds should be smaller. Therefore, following our earlier discussion of Button's *(13)* work, this should result in a reduction in

9. Longitudinal out-of-plane specific elastic constant versus apparent density



10. Work of fiber pullout



stress concentration and an increase in interfiber bond strength.

The variation of tensile strength with apparent density is shown in Fig. 1 for the northern pine 200-g/m² handsheets. We note that at constant density strength is improved by refining, as found by other researchers, e.g., *(1)*.

Acknowledging that conditions for brittle fracture may not be completely satisfied, we have, nevertheless, attempted to use Griffith's equation, i.e., Eq. 1, to reconcile tensile strength, modulus, fracture resistance, and pore size data. The relationship between tensile strength and the product of specific in-plane modulus E^* and fracture toughness G^* is shown in **Fig. 11** for the 200-g/m² handsheets. A good correlation is found which is given by:

$$\sigma^* = 2.73(E^*G^*)^{0.60}$$

$(R^2 = 0.99)$

where

σ = specific tensile stress.

This correlation is similar to that found by Waterhouse *(8)* using the data of Seth and Page for refining at a constant level of wet pressing for different pulps (the exponent was in the range of 0.67 to 0.8).

When pore size at maximum frequency is used as a measure of critical crack size c, i.e., (E^*G^*/c), and its variation with specific tensile stress is examined, a single correlation is no longer obtained.

The data we have presented so far have been for northern pine 200-g/m² handsheets, where the tensile tests were performed using dogbone-shaped samples as stated previously in the experimental section. In order to determine notch sensitivity, tensile tests were performed using samples having a grammage of 100 g/m². The variation of specific tensile strength with notch size is shown in **Fig. 12** for the various levels of refining and wet pressing.

We observe that at the lowest level of refining and wet pressing, tensile strength is relatively insensitive to notch size. However, at the highest level of refining and wet pressing, the sensitivity of tensile strength to notch size is much greater, and there is less influence of wet pressing except in the unnotched samples. This is similar to the findings of Birchall *et al. (45)* for cement.

The specific tensile strengths shown in Fig. 12 are calculated on the basis of total sample width. In **Fig. 13**, the extremes of refining and wet pressing are graphed on a log-log scale and include calculations based on load bearing area, i.e, sample width minus crack width. We see that even when specific strength is based on load bearing area, there is still a reduction in strength. This indicates that stress concentration effects are present. The dashed line shown in Fig. 13 represents the Griffith relationship, i.e., the slopes of the lines are –0.5. It appears that significant deviation of the data from this line occurs below a notch size of around 2 mm.

The property trends which were seen for the northern pine handsheets were in evidence for the southern pine handsheets. The correlation between specific tensile strength σ^* and the product of specific modulus E^* and the fracture toughness G_c^* for the two pulps are compared below:

Northern pine:

$$\sigma^* = 2.73(E^*G_c^*)^{0.607}$$

$$R^2 = 0.989$$

Southern pine:

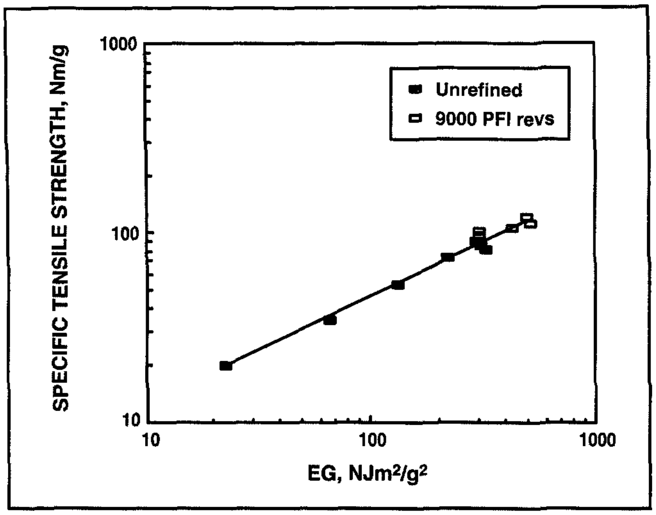
$$\sigma^* = 2.51(E^*G_c^*)^{0.687}$$

$$R^2 = 0.982$$

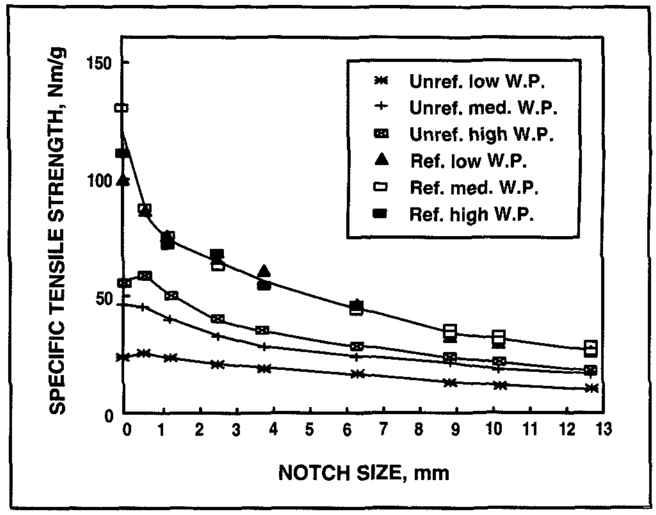
Conclusions

We have attempted to examine the tensile strength of paper within the framework of linear elastic fracture mechanics. An envelope for all of the properties examined, i.e., in-plane elastic constant, fracture resistance, pore size at maximum frequency, tensile strength, and notched tensile strength was found, with the possible exception of out-of-plane elastic constant, when

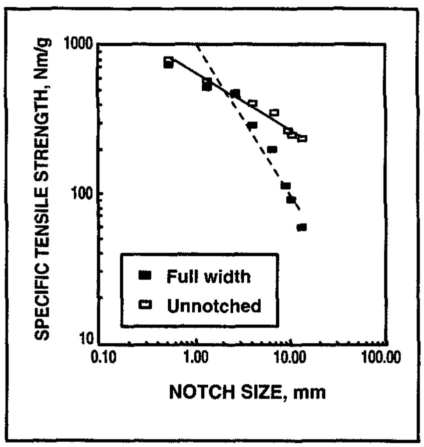
11. Specific tensile strength versus (E x G)



12. Specific tensile strength versus notch size



13. Log specific tensile strength versus log notch size (high level of refining and wet pressing)



paper was densified by different combinations of wet pressing and refining.

Fracture resistance was determined using a modified IPST in-plane tear tester, and values were found to be at the same level as those determined by Seth and Page *(25)*. At the lowest level of refining and wet pressing, a small but significant amount of work is consumed in post fracture fiber pullout.

Attempts to use tensile strength, fracture resistance, modulus, and pore size data to form a correlation based on Griffith's equation were not successful. However, a good correlation was found between tensile strength σ^* and the product of fracture resistance G_c^* and modulus E^* of the form $\sigma^* = A(E^*G_c^*)^n$, where A is constant. This relationship would seem to imply that tensile strength is not dependent on crack size. On the other hand, the notched tensile results do show a sensitivity to single-edge notches particularly at the highest level of refining.

Griffith's equation was also used to estimate crack size using fracture resistance, modulus, and tensile data, and was found to be in the range of 1 mm to 2.5 mm. This might be interpreted as a formation effect or some form of distributed cracking as proposed by Zubelewicz and Bazant *(46)* for cement, i.e., a certain amount of pre-cracking of the material occurs before a crack can propagate. Tortuosity of the fracture path is one indication that formation might be an important factor. On the other hand, one would expect that the size, shape, and distribution of the pores in paper would be important determinants or, alternatively, that interfiber bond stress concentration is important, i.e., the Button effect. Further work is obviously required to resolve this paradox.

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The authors would like to acknowledge the assistance of Betty John for Formette handsheet making; Don Gilbert and Norman Colson for assistance with mercury porosimetry; Roger Van Eperen, Carl Smith, Albert Van Beuningen, and David Brennan in measuring paper properties; and Susan Clark and Angela Colar in the preparation of this paper.

Portions of this work were used by T. B. as partial fulfillment of the requirements for the Ph.D. degree at the Institute of Paper Science and Technology.

Received for review June 28, 1991.

Accepted May 29, 1992.

Presented at the TAPPI 1991 Paper Physics Conference.