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# Crack Propagations and Fatigue Characteristics of Some Handmade Papers*

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**Abstract:** In this work, a number of handsheets made from unbeaten and unbleached kraft, refiner mechanical and cotton linter pulps and crack propagations on paper samples under monotonic loading, were analyzed and possible links with the properties of fibrous networks were investigated. Crack initiation and propagation on the tested paper strips were also investigated under a scanning electron microscope to determine visually the state of the paper matrix under applied loads. Furthermore, paper samples were subjected to a series of mechanical tests, such as tearing resistance, folding endurance and tensile strength. The primary goal of this study concentrated on determining the fatigue life responses of paper samples under cyclic loading. An empirical equation for the fatigue life responses of paper samples tested was developed. It was found that paper, being a heterogeneous composite material, responds differently to applied loads depending on the properties of its constituent individual fibers and the nature of interfiber bonding in its structure. Kraft handsheets, for instance, showed the highest mechanical properties, followed by samples made from refiner mechanical pulps and cotton linters respectively. Overall, the equation developed proved to be a good way of predicting the fatigue life of paper materials subjected to variable loads.

**Key Words:** Fibrous network, interfiber bonding, crack propagation, fatigue life.

## Bazı El Kağıtlarına Ait Çatlama ve Yorulma Karakteristikleri

**Özet:** Bu çalışmada, dövme ve ağartma işlemi yapılmamış olan sırası ile kraft, rafinör mekanik ve pamuk linters kağıt hamurları kullanılarak bir takım standart laboratuar kağıtları yapılmış ve monoton yüklenme altında kağıtlarda oluşan çatlamalar izlenerek bunlar ile lifsel yapıların özellikleri arasındaki bağlar incelenmiştir. Uygulanan yük altında kağıtların durumunu daha iyi anlamak için, test edilen kağıt şeritlerde oluşan çatlamanın başlangıcı ve ilerlemesi de taramalı elektron mikroskobunda incelenmiştir. Kağıtların ayrıca yırtılma, çekme ve katlanma dirençleri gibi bazı mekaniksel özellikleri de test edilmiştir. Bu çalışmada esas amaç kağıtların devam eden kademeli yükleme altındaki yorulma sonuçlarını bulmak üzerine yoğunlaşmıştır. Test edilen kağıtların yorulma karakteristiklerini gösteren deneysel bir formül geliştirilmiştir. Kağıtların, bir hetorejen malzeme olarak, onu oluşturan bireysel lifler ve bu lifler arasındaki bağların durumuna göre uygulanan yüklenmelere karşı farklı davrandıkları bulunmuştur. Örneğin kraft hamurundan elde edilen standart elkağıtları en yüksek mekanik özellikler gösterirken bunu sırasıyla rafinör mekanik ve pamuk linters hamurundan yapılan standart elkağıtları izlemiştir. Genel olarak, geliştirilen formulasyonun, değişen yüklenmelere maruz kalan kağıtların yorulma değerlerini vermesi açısından iyi bir yöntem olduğu gösterilmiştir.

**Anahtar Sözcükler:** Lifsel ağ, lifler arası bağlanma, çatlamanın ilerlemesi, yorulma ömrü.

## Introduction

Paper is a composite material that consists of a number of various ingredients of different morphologies from different origins. Paper strength is governed by various factors in different ways. The physical properties of the fibers themselves and the bonds formed between them are the 2 factors that most affect paper properties (Nissan, 1962; Page, 1969; Seth and Page, 1975). However, there are also many other factors, which have some complex interactive relationships.

### Factors Affecting the Mechanical Properties of Paper

Paper cannot be made unless there is a high degree of fiber bonding in the sheet, which depends on the chemical and physical nature of the fiber surface and upon the manner in which the fibers have been formed into the sheet of paper. In other words, the bonded area of the network is determined by the fiber conformability-flexibility, collapsibility and total surface area. The total surface area is a combination of the number of fibers in

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the paper, such as fiber dimensions, degree of external fibrillation and number of fines (Corte et al., 1962; Paavilainen, 1991).

The local number of fibers in paper, of a certain basis weight, alters the bonded area, if the presumptions regarding the formation of fiber-to-fiber bonds are fulfilled. Paavilainen (1991) showed that bonding strength increases linearly with increased summerwood content, and that bleached fibers have higher bonding strength than unbleached fibers. Low lignin, high total hemicelluloses and low xylan content on the fiber surfaces improve bonding strength. Regarding the importance of fiber bonding, the geometrical characteristics of the fibers themselves must also be included: length, width and thickness if the fiber is described as a flat ribbon, or length, diameter and wall thickness, if the fiber is described as a cylindrical tube (Kallmes and Eckerr, 1964). External fibrillation as a result of beating changes the physical structure of the fiber surface and creates new surfaces, which might have different abilities to form hydrogen bonds than those of the unbeaten fiber. Sheet formation and the uniformity of fiber distribution are other factors that are extremely important for paper quality. The strength of individual fibers constituting paper is also a governing factor in determining paper strength. In the presence of sufficiently strong fibers, paper strength can be increased by improving interfiber bonding. Furthermore, the presence of aluminum, sizing, fillers and other additives in the paper has a great effect on inter fiber bonding (Krkoska and Hanus, 1988; Eroglu, 1990; Niskanen, 1993; Tank, 1998).

## Failure of Paper Strength

Every solid will break if a sufficiently large load is applied to it. The value of this load as well as the shape and other characteristics of the resulting break strongly depend on the material and on how the load has been applied. In the case of elongation of paper, microscopic or submicroscopic failures in the paper structure occur. These may involve the internal structure of fibers, leading to small, permanent elongations, while only a few fiber segments fail completely. The fiber-to-fiber bonds also rupture, either in a brittle manner or gradually through the cutting of fibrils and polymeric molecules (Cetin et al., 2003). One may even regard the entire failure process as corresponding to the rupture of the hydrogen bonds (Nissan, 1957; Corte et al., 1962). It was reported that fiber strength is a much more important property in tear strength or fracture toughness than it is in the tensile strength of paper (Seth and Page, 1987). Fracture toughness measures the general ability of paper to resist crack growth. In tensile loading, the failure of a paper strip usually starts from the edge due to the large stresses at the corner of a strip (Seo et al., 1992).

During crack propagation in paper, there are usually fibers behind the crack tip that extend across the crack and hold the network together. When a load is applied to a sample, potential elastic energy is stored in the system. If a new crack forms or an existing crack grows, part of this elastic energy is released. The formation or growth of a crack implies the creation of free surfaces, normally the crack surfaces that are energetically less favorable than the bulk because chemical bonds have to be broken up (Griffith, 1921). It was stated that a crack grows if, and only if, the release of potential energy is equal to or greater than the surface energy that is required for the crack to grow.

Fatigue is in essence the progressive, localized, permanent structural change that occurs in materials subjected to fluctuating stresses and strains that may result in cracks or fracture after a sufficient number of fluctuations. Fatigue fractures are caused by the simultaneous action of cyclic stress, tensile stress and plastic strain. If any 1 of these 3 is not present, fatigue cracking will not initiate and propagate. The cyclic stress starts the crack, and the tensile stress produces crack growth or propagation (Howard, 1986).

The progress of fatigue is in general divided into 3 main stages (Howard, 1986; Callister, 1997):

- Initial fatigue damage leading to crack nucleation and crack initiation,
- Progressive cycling growth of a crack (crack propagation) until the remaining uncracked cross section of a part becomes too weak to sustain the loads imposed,
- Final, sudden fracture of the remaining cross section.

Fatigue cracks initiate and propagate in the region where the strain is most severe. Since most engineering materials and also fibrous networks contain defects, where strain is intensified under stress, most fatigue cracks initiate and grow from these structural defects. Defects in fibrous materials may arise from the nature of the fiber and/or the nature of interfiber bonds (Callister, 1997; Cetin et al., 2003).

Interest in single fiber fatigue testing has becom part of the research into low consistency refining. This comes from research that characterizes refiners by the number of impacts, N, and the energy expended per impact, I (called an intensifier of impact). This description of refining as a fatigue type process has led to studies examining the development of fiber mechanical properties as a function of the number of straining cycles (Kerekes, 1990; Wild and Provan, 1996).

The most significant work in measuring the mechanical properties of single fibers under monotonic and cyclic load was published in a series of papers in which details of the measurement techniques were given (Page et al., 1977; Mott at al., 1996; Conn and Batchelor, 1999).

Research into paper fatigue requires a large number of fatigue experiments, due to its inhomogeneous and composite structure. Fatigue of fiber reinforced composite materials is quite complex phenomenon, and the fatigue behavior of these heterogeneous materials is fundamentally different from the behavior exhibited by metals (Paepegem, 2000).

## Materials and Methods

### Preparation of Paper Sheets

Laboratory handsheets made of unbeaten and unbleached kraft softwood, refiner mechanical softwood and cotton linter pulps were obtained from the Paper Science Department's Pulp Store at UMIST in a commercial grade. Handsheets were evaluated by a British sheet maker instrument according to the Technical Association of Pulp and Paper Industry (TAPPI) standard test method T205, then conditioned at 23 ± 1 ºC and 50 ± 2 % relative humidity and used for testing.

### General Properties of Handsheets

Thickness, grammage and densities of paper samples were determined according to TAPPI test methods T411 om-84, T410 om-88 and T411 m44, respectively. Ten measurements were taken for each sample.

### Folding Endurance Test

Folding endurance was measured using a variable speed MIT folding endurance tester, adjusted to a speed of 175 double folds per minute through an angle of 270° and equipped with a cooling fan in order to eliminate the effects of temperature. Paper samples 15 mm in width were tested under 0.5 kg constant tension provided by a spring device. The method is described in detail in TAPPI standard test method T511.

### Tear Resistance Test

The internal tearing resistance of paper was measured on a pendulum type instrument (Elmandorf tear tester) that measures the amount of work done in tearing a piece of paper through a fixed distance, after the tear has started, by means of a standard cutting device attached to the instrument. The procedure used with this instrument is described in TAPPI standard test method T414.

### Tensile Strength (Monotonic Load Testing)

The tensile strength of paper samples was measured using an Instron Model 1122 universal testing instrument equipped with data acquisition, computer and printer. The testing procedure used involved a slight modification of that described in TAPPI T494. Paper samples were 15 mm in width and the distance between the jaws of the machine was set at 100 mm. The crosshead speed of the Instron was fixed to 20 mm min$^{-1}$ for all tests. The Instron Model 1122 used was a most versatile testing instrument, which also allows the calculation of stretch, tensile energy absorption (TEA) and Young's modulus directly from the tensile test data.

### Fatigue Testing (Cycling Load Testing)

The Instron Model 1122 universal testing instrument was used for applying a direct (axial) stress mode of loading to the prepared samples. The shape and dimensions of the test specimens used in the study are illustrated in Figure 1. Samples were cut so that the span between the jaws of the Instron could be set at 100 mm with a 25 mm width. A sharp 4 x 4 mm V notch, as a stress raiser, was induced at the center of the specimen, normal to the loading direction where the crack propagation was initiated as a result of cycling loading. The distance between the jaws of the machine was adjusted to 100 mm and the crosshead speed of the machine was set at 20 mm min$^{-1}$ for all samples.

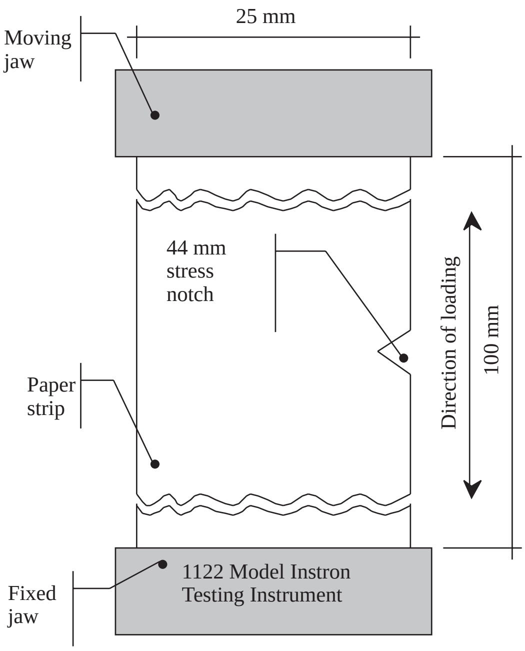

Figure 1. A simplified drawing of test specimen and the test apparatus used. A distance of 100 mm was set between the jaws, one of which was fixed and other mobile, applying the preset loads.

After carrying out preliminary tensile and cyclic tests on each sample, the maximum breaking loads of the paper samples were determined, as well as some idea about fatigue behavior and the frequency effects being obtained. Then the maximum breaking loads for each sample were gradually reduced and applied at a constant cycling action. At least 5 tests were repeated for each load application. Results were plotted as applied maximum load (Newton) versus number of cycles in a logarithmic scale that gives the S-N curve of the tested paper. Some of the samples were recorded with a video camera during cycling for further analysis. Furthermore, scanning electron microscopy (SEM) was used for analyzing the crack path for some handsheet specimens within the fracture region. Samples mounted under the microscope were examined in several regions and a photomicrographic record was obtained.

## Results and Discussion

### Basic Physical Properties of Paper Samples

The properties of the handsheets tested are presented in Table 1. The differences in the test results of papers are normally attributed to the properties of fibers and fiber networks. Pulps used in papermaking received no further chemical or mechanical applications, except for the pulping process, as well as no filler or papermaking ingredients. Depending on the pulping process, cellulose fibers can undergo mechanical and chemical modifications to different extents that directly affect the properties of the resultant papers. Therefore the pulping methodology should be taken into account. In the kraft pulping process, white liquor containing the active cooking chemicals, sodium hydroxide (NaOH) and sodium sulfide ($Na_2S$) is used for cooking the chips, where the lignin in the wood chips is first swollen and then chemically split into fragments by the hydroxyl ($OH^-$) and hydrosulfide ($SH^-$) ions present in the pulping liquor. The lignin fragments are then dissolved as phenolate or carboxylate ions. Carbohydrates, primarily hemicelluloses and some celluloses, are also chemically attacked and dissolved to some extent. It is reported that during a typical cook, approximately 80% of the lignin, 50% of the hemicelluloses and 10% of the celluloses are dissolved and removed from the pulp (Kleppe, 1970; Smook, 1992). It can be seen from Table 1 that kraft paper was stronger than the other papers tested.

Compared with chemical pulping, refiner mechanical pulping (RMP) can transform the wood chips into wood fibers without changing their chemical nature much. Therefore fibers can preserve most of their ingredients as they were in the wood, and hence the yields can go up to 95%, despite consuming prodigious amounts of energy. The pulp forms a highly opaque paper with good printing properties, but the sheet is weak and discolors easily on exposure to light, which is a clear indication of high lignin content (Arif, 1996). Under mechanical pulping, cellulose fibers inevitably undergo some cutting action that results in a pulp with short and broken fibers. In RMP, the process usually involves the use of 2 refining stages operating in series and therefore produces a longer-fibered pulp than does conventional groundwood (Leask and Kocurek, 1987).

Cotton linters, classified as seed fibers, are already cellulose fibers that do not need any processing for separation and bleaching. The only process they need are refining and beating to produce shorter and flexible fibers, depending on end-use. The length and diameter of softwood and cotton linters fibers were reported to be on average 4 mm and 40 µm for softwood and 20 mm 20 µm for cotton linter, respectively (McGovern et al., 1987;

Table 1. The basic physical properties of paper samples used in the experiment (RMP: Refiner Mechanical Pulp, Cotton L: Cotton Linter, SD: Standard Deviation).

| Paper Properties | Kraft Handsheet | | RMP Handsheet | | Cotton L. Handsheet | |
|---|---|---|---|---|---|---|
| | Mean | SD | Mean | SD | Mean | SD |
| Caliper, μm | 115 | 1.49 | 170 | 2.10 | 170 | 1.15 |
| Grammage, g $m^{-2}$ | 62.70 | 1.42 | 65.70 | 1.58 | 65.65 | 0.97 |
| Sheet density, kg $m^{-3}$ | 545 | 14.90 | 386 | 6.31 | 386 | 6.71 |
| Max. breaking load, N | 22.64 | 2.03 | 22.43 | 3.08 | 7.42 | 0.52 |
| Tensile index, (N m) $g^{-1}$ | 24.07 | 2.16 | 22.75 | 3.12 | 7.54 | 0.53 |
| Stretch, % | 1.69 | 0.12 | 1.13 | 0.28 | 2.01 | 0.26 |
| Tear index, (mN $m^2$) $g^{-1}$ | 18.98 | 1.35 | 7.78 | 0.91 | 6.78 | 0.92 |
| Folding endurance, $Log_{10}$ No. | 2.53 | 0.07 | 1.49 | 0.05 | 0.25 | 0.02 |
| TEA index, mJ $g^{-1}$ | 337 | 7.92 | 169 | 5.37 | 120 | 6.13 |
| Young modulus, MPa | 1719 | 41.81 | 1074 | 23.15 | 355 | 16.01 |

Smook, 1992). Considerable differences in length are apparent here between wood and cotton fibers. Since cotton linter received no chemical or mechanical treatments before papermaking, its fibers are thought to be undisturbed. Therefore external and internal fibrillations on the cotton linter fibers used here are not thought to be present. In fact, due to the lack of hemicelluloses, fibrillation effects on cotton fibers occur with difficulty, even if beating is applied. Furthermore the fibers are expected to protect their original shapes as long, thin cylindrical tubes in the paper structure. That will have major negative effects on the contact area between fibers, and hence on the strength of interfiber bonding. Therefore the resultant paper will be expected to be not very strong, as seen in Table 1.

Figure 2 demonstrates the basic physical properties of the tested papers. Kraft paper showed the highest strength, followed by samples made from RMP and then cotton linters. This was attributed to the lower lignin content in kraft paper, compared with RMP paper, and cooking conditions. Furthermore, fines and short fibers are thought to be absent from the fact that no harsh mechanical action was imposed on the fibers. The higher density of kraft paper should be also taken into consideration, as it partly reflects the firm formation of the paper structure and larger contact area between fibers. Tearing results (Table 1) clearly demonstrate the different effects of pulping methods on individual fiber strength. Tearing index figures, 18.98 $(mNm^2)g^{-1}$ for kraft paper and 7.78 $(mNm^2)g^{-1}$ for RMP paper, reveal

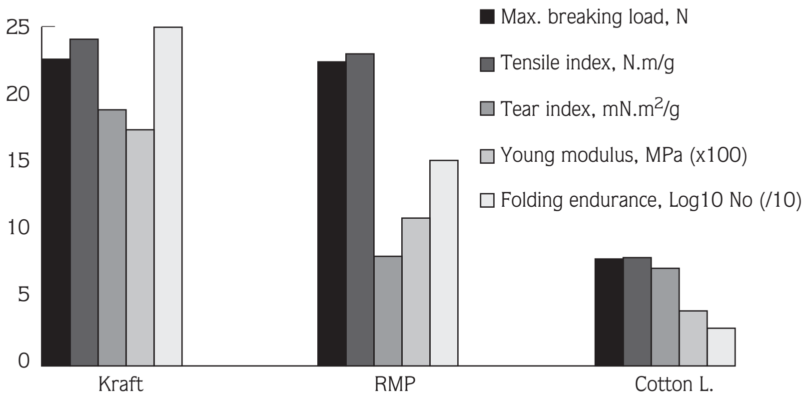

Figure 2. Some key results of the paper samples tested. Kraft paper was superior in terms of the properties compared.

that fibers undergo enormous mechanical stress and ruptures in mechanical pulping, which produces pulps with weak fibers and fiber fractures. In the kraft method, however, fibers can preserve their uniform structures, and hence their strength.

### SEM Images

SEM images of tested papers were taken of their fractured zones in order to see their formations and fibers more clearly. Unfortunately, the pictures taken at higher magnifications of papers made from kraft and cotton linter fibers cannot be presented here due to a technical accident. However, images at 500 μm magnification of the aforementioned samples and at 100 μm of paper made from RMP are shown in Figures 3, 4 and 5. Figure 4 is the best image, offering a more detailed view of RMP paper. This image clearly supports the discussion in the previous section about RMP fibers and paper. Broken and fibrillated fibers are apparent with some fiber fragments. Fibers are also flat, which indicates the fibers collapsed as a result of mechanical action. These are all normally expected to create a larger contact area between fibers, better conformation and stronger resultant paper. The tensile strength of RMP paper was largely attributed to relatively stronger interfiber bonding. Tearing and folding tests, however, indicate that individual fibers in RMP paper were not as strong as kraft fibers.

Figure 3 shows the state of kraft paper at the fractured zone. Compared to RMP fibers, more uniform, rather longer as well as collapsed fibers can be seen. This explains why kraft paper has the highest tearing resistance, as it mainly depends on the strength of individual fibers.

Cotton linter paper is presented in Figure 5. Compared with Figure 3, cotton linter paper can be seen to consist of rather longer and thinner walls of fibers. The fibers seem to be twisted and entangled rather than stretching out in different directions. If one compares the width of fibers in Figures 3 and 5, then the fibers in Figure 3 are, on average, 3 to 4 times wider. This means that the kraft fibers were in a more collapsed state and that the cotton linter fibers preserved their original shape. Probably for that reason, cotton linter paper had the weakest interfiber bonding and in the lowest tensile strength. However, tearing resistance was slightly higher than in RMP paper. That is presumably due to the undamaged cotton linter fibers.

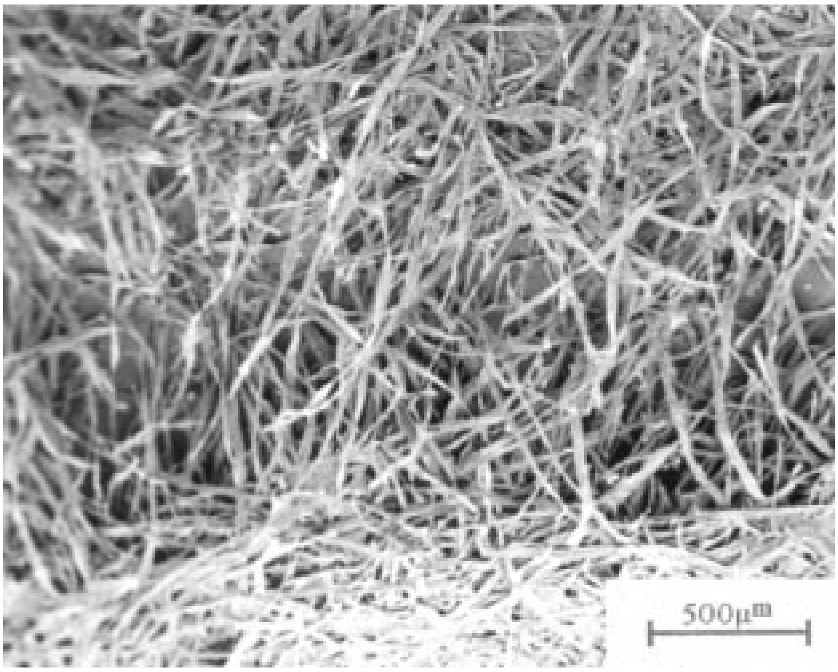

Figure 3. Scanning electron microscope image of the fracture region of paper made from the kraft pulp.

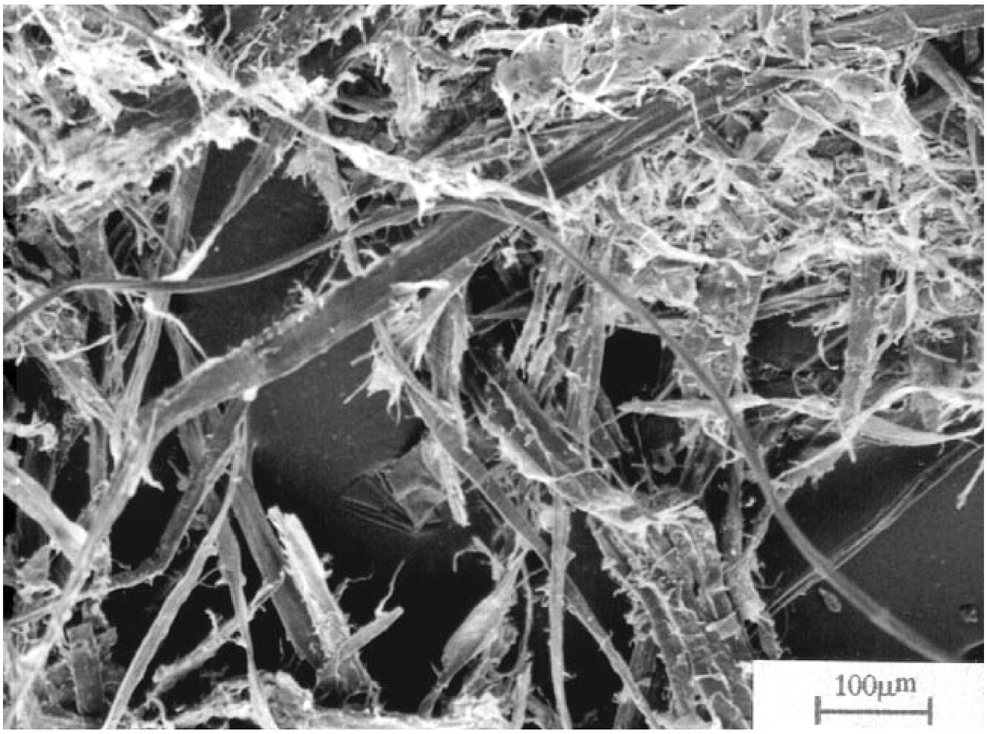

Figure 4. Scanning electron microscope image of the fracture region of paper made from refiner mechanical pulp (RMP).

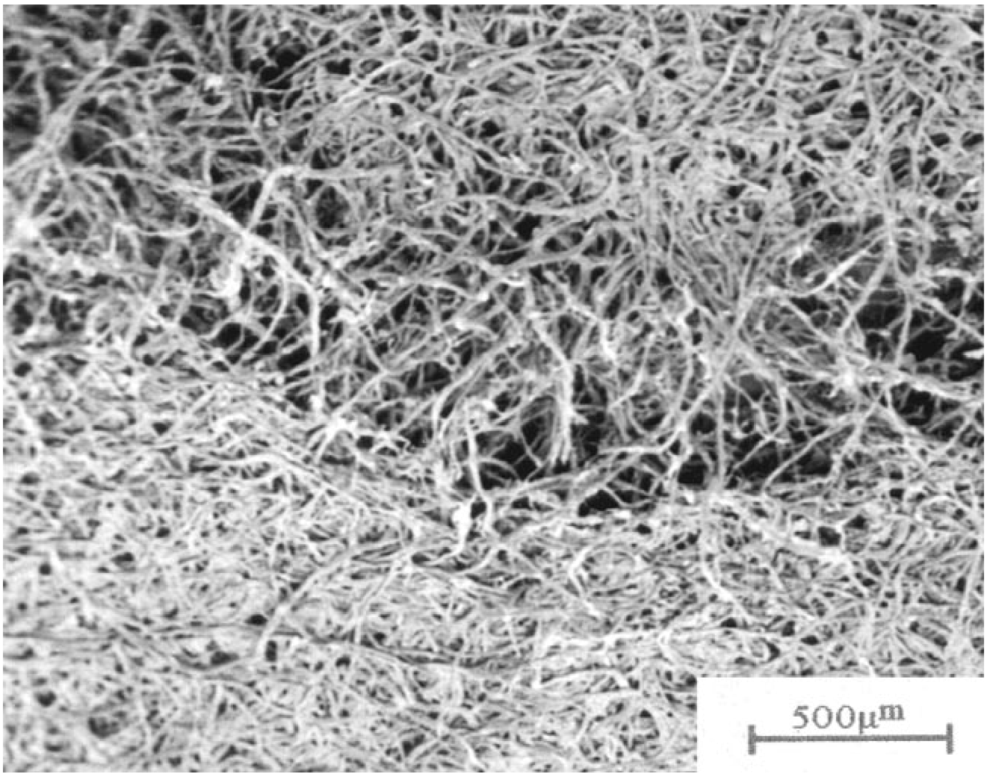

Figure 5. Scanning electron microscope image of the fracture region of paper made from cotton fibers.

### Fatigue Characteristics

Every material has an original fatigue life and character. Fatigue cracks for a single fiber may be characterized by 3 distinctive stages: (i) the initiation stage, followed by (ii) microcrack propagation, and (iii) the macro-propagation stage, including the transitory modes linking them (Hamad and Provan, 1995; Callister, 1997). The growth or extension of a fatigue crack under cyclic loading is principally controlled by the maximum load and stress ratio. However, as in crack initiation, there are a number of additional factors that may exert a strong influence, including environment, frequency, temperature and fiber direction.

The experimentally applied load in Newtons and the number of completed cycles for each handsheet sample, which were 25 mm in width and V notch induced, were recorded as experiment values. Then these experimental data were entered into a computer program (Hamad, 1991) based on least-square polynomial regression techniques. By using this special program the characteristic mathematical equation for each fibrous network was determined. A range of fatigue loads were calculated until the fatigue limit was reached (from 1 to 100, 000). Experimental and simulated data points for each kind of paper sample, i.e. applied load versus number of cycle curves, were drawn.

The characteristic mathematical equation for fibrous networks is

$S = A + [B / (N + D)]$ where,

S: Load (N),

N: Fatigue life (number of cycles),

A: Fatigue limite (N),

B, D: Empirical material constants given in Table 2.

Figure 8 presents the S-N curve of the tested papers. The abscissa contains the number of cycles (logarithmic scale), and the ordinate axis shows the force (Newtons), which was measured by the Instron machine during the fatigue tests. As explained in the experimental section, first the maximum breaking load of paper samples was determined. Then these loads were gradually reduced and imposed on paper samples under monotonic cycling action, which created the fatigue damage leading to crack nucleation.

Table 2. The empirical material constants for the paper samples used. The constant values were used in the formula developed for calculating the fatigue characteristics of the samples investigated (RMP: Refiner Mechanical Pulp, Cotton L: Cotton Linter, A : Fatigue limit, B and D : Empirical Material Constants).

| Materials | Empirical A | Material B | Constants D |
|---|---|---|---|
| Kraft Handsheets | 14.25 | 674.99 | 68.45 |
| RMP Handsheets | 16.61 | 288.31 | 89.27 |
| Cotton L. Handsheets | 4.75 | 514.15 | 155.04 |

The number of stress cycles of that a material can endure before failure increases with decreasing stress. Below this limiting stress, known as the fatigue limit or endurance limit, the material can endure an infinite number of cycles without failure. In general, fatigue properties are very sensitive to material conditions. Except in special cases where internal defects or case hardening is involved, all fatigue cracks initiate at the edge of the sample.

The data presented in Figure 8 illustrate several important points. Firstly, a fatigue limit is evident. Below a certain cyclic load, fatigue failure will not occur for an arbitrarily large number of cycles. Secondly, the load-number of cycle curves of each sample show various types of fatigue behavior. Finally, tensile strength is a significant factor affecting fatigue behavior. If the tensile strengths of samples and fatigue tests are compared, it can be seen that there is a very close relation between them. As seen in Figure 8, the experimental and the simulated data for paper samples fit perfectly, which implies that the formula developed can be used for determining the fatigue life of paper materials.

Figure 7 shows a fully failed paper strip as a result of cycling loading. Applied loads after some cycling actions initiated the crack at the tip of notch (Figure 6), and the crack the propagated in the cross direction of the paper strip. Fibers and fiber bundles in the direction of the crack would normally make propagation more difficult. In such a case, the crack would follow other directions and deviate from a straight line. That seems to be what happened in Figure 7 as the paper did not break straight in a cross direction.

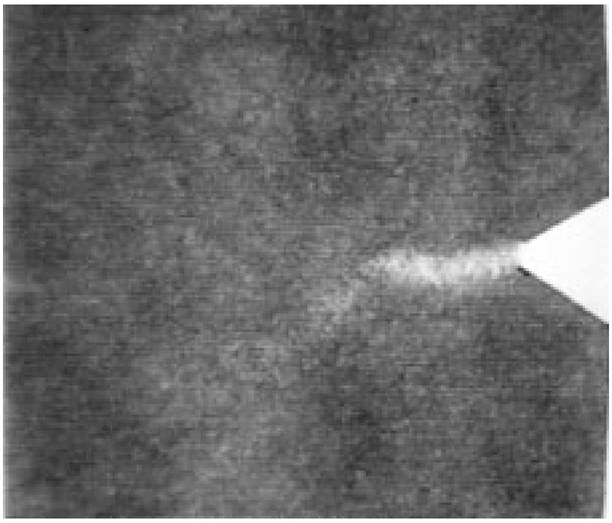

Figure 6. Crack nucleation at the tip of the notch and crack propagation on strained paper.

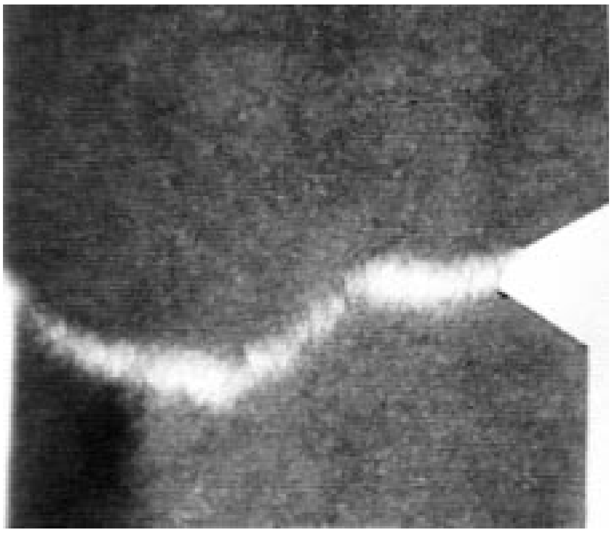

Figure 7. Fully failed kraft paper sample under cycling load. Due to strength differences in the paper structure, the crack was forced to follow a non-straight path.

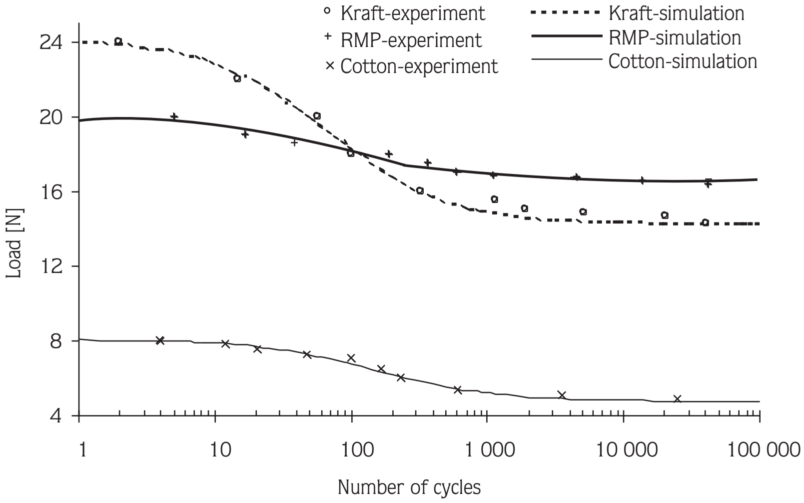

Figure 8. Fatigue responses of paper samples to variable cycling loads as real and calculated data. The calculated data illustrated by lines fit the experimental data. The paper made from cotton linter had the weakest resistance to the cycling load.

## Conclusions

Paper strength is largely dependent on the properties of its constituent fibers and interfiber bonding. Kraft paper was superior in terms of the properties investigated here to papers made from RMP and cotton linters. Kraft cooking was found to favor paper strength. In addition to delignifying the fibers, it produces fibers that are mechanically undamaged and flexible. Furthermore, kraft fibers collapsed on their lumens created stronger interfiber bonding, and hence stronger paper as well as a denser sheet. By contrast, paper made from RMP contained many damaged fibers and short fiber fragments. Although the fibers had some fibrillation, they preserved all the chemicals as in the wood. The strength of RMP paper was therefore lower than that of kraft paper. Paper made from cotton linters was the weakest of all. This is attributed to the properties of the cotton linter fibers. As the SEM images reveal, because no mechanical and chemical treatments were applied to cotton linters, the fibers were not able to create stronger interfiber bonding and sheets.

Some fatigue limits of the tested paper samples were experimentally determined. A full range of tested paper fatigue characteristics was plotted by the equation developed in this study. Experimental and simulated results were in good agreement. Therefore, it is likely that the equation developed in this study can be used in predicting the fatigue life of paper samples.

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