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Mathematical Analysis Section
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April 15, 1976

Progress Report Covering the Period
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U.S. DEPARTMENT OF COMMERCE, Elliot L. Richardson, *Secretary*
James A. Baker, III, *Under Secretary*
Dr. Betsy Ancker-Johnson, *Assistant Secretary for Science and Technology*
NATIONAL BUREAU OF STANDARDS, Ernest Ambler, *Acting Director*

CONTENTS

1. SUMMARY

Generally, the approaches to solving problems associated with paper are empirical in nature. Hypotheses are formulated on an intuitive basis and experiments are designed to verify the hypotheses. All too frequently, however, the experimental results can be used to verify conflicting hypotheses. Frequently the empirical approach results in an illusory solution to a specific problem. The system functions smoothly until changes are made. Problems surface because the solution to the original problem was not real. At that time there are three courses to take: 1) determine the real solution to the original problem, or 2) revert back to the original scheme of things and lose all flexibility in the system, or 3) obtain another illusory solution which will suffice until a desired or necessary change is made on the process at some future date.

One of the most powerful tools that can be used for solving problems involving the mechanical properties of paper is a mathematical model. A practicable mathematical model would further a better understanding of the relationship between the microstructure of paper and its mechanical properties. Ultimately the model would greatly assist in selecting the structural parameters important to the circulation life of currency. With this in mind an attempt was made to formulate a mathematical model for paper.

In order to interpret macroscopic data with microscopic observations, a decision must be made as to what should be observed and how the massive amount of data generated for each microvariable is to be treated. After all, the viability of any microvariable is contingent on the ability to analyze and interpret the data corresponding to that variable. It is important to be able to analyze and interpret microscopic data as a function of time. The two most popular approaches to analyze these data are through the well-known log-normal and gamma distributions. However, they are unsatisfactory as both distributions are always asymmetric to the left. There is no *a priori* reason to assume a microvariable has only one way to change its distribution with time.

The statistical theory proposed by Weibull was selected to formulate a mathematical model for paper. Three parameters describe a Weibull distribution and the curve can skew to the left or right. When the shape parameter of the distribution curve is 3.6, the curve is almost indistinguishable from the well known Gaussian distribution.

An analysis of the extensive data obtained on the flexing of currency paper was made. Important parameters such as fiber length distribution, microscopic strength data, pore size distribution and the elastic and sonic moduli were analyzed rigorously. Two mechanisms for the microscopic degradation of paper were proposed. One of the mechanisms involved fiber failure; the second involved the distribution of "equivalent" void diameter in paper. Both mechanisms account for a rapid loss of modulus at the early stages of flexing of paper and a gradual leveling of the modulus during

subsequent stages. The actual problem is probably much more complicated than postulated by either mechanism. All processes causing micro-structure degradation are probably occurring simultaneously albeit at substantially different rates.

Additional data, such as the shear modulus and Poisson's ratio of currency paper as well as the elastic properties of cotton and linen fibers was needed. Since this data had not been determined previously, a thorough search of the literature was made in an effort to get useful data which would allow completion of even a crude mathematical model. Only meager data was found in the literature and much of the data that was available appeared to be unsound.

In conclusion, a novel approach to the problem of modelling the degradation of paper has been found. The 3-parameter Weibull distribution is adequate to characterize paper micro-structure as well as the change in micro-structure with time or number of cycles. Two mechanisms for the loss of modulus due to repeated flexing have been proposed. However, additional data is needed for the eventual completion of the mathematical model.

The technique required to obtain data on the shear modulus and Poisson's ratio for paper are complex, while the single fiber techniques are tedious and time consuming. This could account for the limited and incomplete data of this kind in the literature. A significant effort should be made to obtain the necessary data required for formulating a mathematical model for paper. Development of a mathematical model would be beneficial to the solution of a large number of paper problems related to its mechanical properties. The mathematical model could be especially useful for the design of a durable currency paper containing all or part wood pulp fibers.

2. INTRODUCTION

The statistical nature of microscopic parameters such as the fiber length [1], the fiber breaking load [2], the pore size [3], etc., has been recognized by paper research workers for quite some time. However, mathematical modelling of the mechanical behavior of paper as related to its micro-structure has been limited to the case of a "quasi-static" process of deformation, where the microvariables were either endowed with a "random" statistical distribution [4,5,6] or stripped of their statistical content in formulating a variety of empirical relations [7]. To account for the physical phenomena such as anisotropy [8], fatigue [8], yield, and rate-dependent modulus [9], it is necessary to introduce either the time variable or the number of cycles of deformation explicitly into the statistical model. Clearly, any such modelling effort must be coupled with an experimental program where the variation of the micro-structure is monitored in conjunction with the change of macro-strength characteristics.

Recent advances in automatic scanning devices and their application in metallurgy [10] have convinced us that the same devices may be applied to the study of the micro-structure of paper. As more experimental data become available, a new mathematical framework other than random statistics is needed to interpret the data and guide the development of a time-dependent model. The purpose of this report is to provide a preliminary analysis of the data developed on currency paper at NBS over the past several years.

3. ANALYSIS OF FLEXING DATA

In Fig. 1 a sheet of currency paper, 15.2 cm wide, 30.5 cm long, and 0.13 mm thick is shown undergoing a flexing test. The rollers used for testing the strength properties of the paper in the cross direction are 3.18 mm in diameter. Assuming plane sections to remain plane when the paper is bent around a roller, and assuming further that the neutral axis is found half-way between the thickness of the paper, the radius of curvature is estimated to be 1.655 mm, and the maximum strain at extreme fibers is about ± 3.9%. It is, of course, understood that the estimated strain, whether tensile or compressive, is a macroscopic quantity. With the constraints supplied by the roller and the specimen geometry, macroscopic buckling simply cannot take place. In Fig. 2, is shown how the motion of the pair of rollers with a period of 2.0 sec. induces four bursts of bending strain at a typical point "E" along the flexing specimen D-F as shown in Fig. 1. Theoretically, as the paper is brought to contact with a roller, the rate of strain is infinite, i.e., the loading and unloading processes are both instantaneous. The duration of the maximum strain (± 3.9%) for the extreme fibers is estimated to be of the order of 0.02 s.

The load-elongation curves for a tensile test for currency paper in the cross direction before and after 80,000 flexes is shown in Fig. 3. The initial modulus of the quasi-static loading test (1.67×10^{-3} s^{-1}) dropped from a mean value of 246 kgf/mm^2 (2,410 N/mm^2) before flexing to 167 kgf/mm^2 (1,640 N/mm^2) after 80,000 flexes. Since the flexing test involves both loading and unloading, some previous data [12] is plotted in Fig. 3 on currency paper when it underwent a cyclic strain of tension in the cross direction. The initial portion of the unloading curve is seen to be elastic with an estimated modulus of 400 kgf/mm^2 (3,900 N/mm^2). To complete the picture, the dynamic modulus (≈ 850 kgf/mm^2 or 8,300 N/mm^2), based on wave propagation measurement is also plotted [11]. In Fig. 4, the dynamic modulus and the quasi-static modulus of currency paper in the cross direction is plotted as a function of the number of flexes. The dramatic drop in both moduli during the first 5,000 flexes with the subsequent flat lines in Fig. 4 characterizes the macroscopic phenomenon of fatigue of paper and will be the central problem in constructing a microscopic degradation model.

To complete this analysis, two additional points must be emphasized. In the first place, the load-elongation curve in Fig. 3 is given only for a <u>slow</u> rate of <u>tensile</u> loading. Unfortunately, the strain induced by the flexing machine is not only alternating between tension and compression, but also at a very high rate. This implies that this is insufficient experimental data to launch a rigorous stress analysis which is a prerequisite to a rational assessment of fatigue damage. The second point is related to the fact that, while the flexing test imposes a non-homogeneous strain distribution in the paper sample, the loss of modulus phenomenon as shown in Fig. 4 was observed on samples with a homogeneous strain distribution in the paper sample, the loss of modulus phenomenon as shown in Fig. 4 was observed on samples with a homogeneous strain distribution. Caution must be exercised in proceeding with the formulation of a degradation model where there is not a one-to-one correspondence between loadings that cause damage and strength tests that assess damage. Direct fatigue tests will have to be conducted in the future.

4. WEIBULL STATISTICS

To undertake a task of interpreting macroscopic data with microscopic observations, one is immediately confronted with two difficult problems, namely, what to observe and how to treat the massive amount of data that is generated for each microvariable. The two problems are not completely independent of each other, since the test of the viability of any microvariable includes the requirement that the data corresponding to that variable can be analyzed and interpreted. However, a three-parameter probability density function due to Weibull [13] is a natural tool for analyzing and interpreting microscopic data as a function of time.

To begin with, it is assumed the reader is familar with the notion that a probability density function known as Gaussian or "normal" is given by two parameters, namely, the mean and the variance, and is symmetric about the mean. If any microvariable is describable by a normal distribution at a specific time, say, t_o, and if that variable changes with time explicitly due to physical reasons, then it is conceivable that the distribution may become non-Gaussian at any time before or after t_o. This creates a need to look for a distribution which in general is asymmetric and has an extra parameter that measures the asymmetry.

Two obvious candidates, which seem most popular in the current literature (the well-known log-normal and gamma distributions), have been rejected. It is a mathematical fact that both distributions are asymmetric always to the left, i.e., the vertex of the density curve lies to the left of the mean. For the specific purpose in mind, a distribution that could skew either to the left or to the right is needed since there is no *a priori* reason to demand that a microvariable has only one way to change its distribution in time.

In his pioneering work on a statistical theory of the breaking strength of materials, Weibull [13] proposed a distribution which appears to be ideally suited for paper. The three parameters, m, x_o, x_u, can be given a physical meaning in every application involving some ultimate property of a material. Since the mathematical properties of a Weibull distribution are nothing new [13, 14] its graphical behavior as we change one of its three parameters will be discussed. In Fig. 5 is shown not only the exact mathematical expression of the distribution but also its shape as the shape parameter m is varied. In Fig. 6, the scale parameter x_o is varied while m remains fixed. There is an apparent change in the shape of the curve. In both figures, the location parameter x_u has been ignored, simply because its variation merely corresponds to a horizontal shift of any curve for specific values of m and x_o.

It is worth mentioning that for m = 3.6 (approx.), the Weibull distribution is almost indistinguishable from the Gaussian (normal) distribution. For m less than 3.6, the distribution is asymmetric to the left, and for m greater than 3.6, the distribution skews to the right, as shown in Fig. 5. When m approaches infinity, the Weibull distribution approaches that of a Dirac-delta function with the spike located at $x = x_u + x_o$. This is a useful property particularly when we know that in applications we are constantly faced with a judgmental question in choosing either a deterministic model or a probabilistic model. With the use of a Weibull distribution, both models can be formulated without asking the question whether m is finite or infinite.

5. ANALYSIS OF SPECIFIC STRUCTURAL PARAMETERS

5.1 Fiber Length Distribution

Having settled on a specific tool for the statistical treatment of microscopic data, attention was turned to an obvious micro-variable, namely, the fiber length.

The data was obtained on 600 fibers from currency paper before and after 80,000 flexes in the cross direction [21]. The histograms and the appropriate Weibull curves are plotted in Fig. 7. Physically, the fibers in the neighborhood of 2 to 3 mm lengths disappear and those around 1 mm in length increase after 80,000 flexes. Mathematically, through the Weibull parameters, the shape parameter m hardly changes, while both the scale parameter x_o and the location parameter x_u suffer considerable changes. This coincides with the discussion associated with Fig. 6 of the previous section, where a change in scale parameter can change the apparent shape of a distribution curve when the shape parameter remains fixed.

5.2 Microscopic Strength Data

Since Weibull [13] intended his distribution for application to the breaking strength of material, our next illustration is motivated by the curiosity whether the microscopic strength parameters of wood and cotton fibers can also be analyzed with Weibull statistics. Two quantities are of direct interest, the fiber breaking load, and the fiber-to-fiber bond shear strength. In Fig. 8 is shown some typical data on those two quantities. Even though we can always fit some 3-parameter distribution to a given histogram by calculating its first three moments, in the case of the two examples listed in Fig. 8, the fit is not necessarily "smooth". Nevertheless, the availability of an extra parameter in our approach speaks well of the analysis as compared with the conventional mean-and-variance analysis given in both references 15 and 16. For example, with m = 2.0, the breaking load distribution given in Fig. 8(b) is asymmetric to the left with the implication that any degradation model based on fiber breaking as a mechanism should consider x_u, the minimum breaking load as a critical microvariable. On the other hand, if we extrapolate Weibull's result [13] on cotton yearn and assume that for cotton fibers, the shape parameter m is about 5.5, we conclude that the breaking load distribution for cotton fibers may be asymmetric to the right (as illustrated in Fig. 5 for m = 5.5), and the critical microvariable may be $x_u + x_o$ rather than x_u.

In conclusion, the Weibull distribution is useful for representing micro-strength data. Unfortunately, for currency paper, no data on fiber breaking load or fiber-to-fiber bond is available for analysis and model-building.

5.3 Porosity of Paper

So far, only one micro-geometric variable has been discussed, i.e., the fiber length, and two micro-strength parameters, the fiber breaking load, and the fiber-to-fiber bond. To complete the crude formulation, we need to introduce a second micro-geometric variable, namely, the concept of a pore radius distribution as expounded by Corte [3]. Once again no pore size data on currency paper, before or after flexing, is available. Presumably, the data are not difficult to get, and will be available in time to guide the modelling effort in the near future. For the time being, an extrapolation based on air permeability measurements will have to be used.

The porous structure of paper is one of its fundamental characteristics. Microscopic examination suggests that an adequate description of this complicated structure is not presently obtainable. However, a simple picture of the structure which is acceptable for many purposes is one in which the voids are described by circular pipes, or spherical cavities. Specification of statistical distribution of these voids as a function of their diameter then defines the porous structure.

Air permeability can be used to provide a measure of the paper porosity. The permeability relates the rate at which air flows through a sample to the small pressure drop imposed across it. At normal pressures the volume flow of air is found to follow the theoretical predictions for Poiseuille flow through long capillary tubes. When a Poiseuille law is followed and the simple model of circular holes is used to describe porosity, the permeability data specifies the product of the mean number of voids and the mean area of a void over the paper sample. In Fig. 9, air permeability data [8] is plotted as a function of the number of flexes in the cross direction for currency paper. The air permeability of paper is measured in units of cubic centimeters of air per second through an area of one square meter when the air pressure difference is 98.08 N/m^2. The commonly accepted notion of permeability of a porous medium differs from that given in Fig. 9 by merely a multiplicative constant.

To interpret Fig. 9, we note that there is a dramatic increase of air permeability from the initial state to the intermediate state corresponding to either 5,000 or 10,000 flexes. This corresponds to the sharp drop in modulus as shown in Fig. 4. The increase in permeability can be interpreted as an increase in a single parameter called the equivalent void diameter. If the initial void diameter is assumed to be known, then the air permeability data and this simple model enable us to estimate the new void diameter by multiplying the initial one with the square root of the ratio of the new permeability to the initial permeability. For example, there is a five-fold increase in permeability after 5,000 flexes. This indicates an increase in the equivalent void diameter by a factor of 2.2. There is a less dramatic increase in permeability between 10,000 and 80,000 flexes. With the square root relationship, the increase in equivalent void diameter in the same cycle interval is even more gradual. The fact that the modulus of a flexed paper also changes very slowly during that cycle interval as shown in Fig. 4 provides us with a reasonable argument to adopt the equivalent void diameter as a second microgeometric variable.

6. TWO MECHANISMS FOR MICROSCOPIC DEGRADATION OF PAPER

Two micro-geometric variables, the fiber length and the "equivalent" void diameter, and two micro-strength parameters, the fiber breaking load and the fiber-to-fiber bond have been singled out as the basic elements for a microscopic degradation model of paper. Since the only degradation data available are based on uniaxial tensile testing, the modelling effort can only be as primitive as a one-dimensional balance of forces and strains. Unfortunately, even that is too much to ask for, because both fiber length and void diameter (through air permeability) measurements yield no information about the anisotropic character of the mechanical behavior of paper. To resolve this dilemma, the new notion of an "effective" fiber length equal to the length of a fiber as seen under a microscope and projected along an axis that is parallel to the direction of loading and unloading of a given specimen is introduced. The "effective" fiber length distribution is obtainable by taking advantage of the capability of an automatic scanning device as discussed by Moore [10]. For our purposes here, it shall be assumed that all four microvariables, namely, the "effective" fiber length, the "equivalent" void diameter, the fiber breaking load distribution for some critical fiber length, and the fiber-to-fiber bond for some typical fiber width, have all been estimated as a function of time or number of cycles. Within the framework of a one-dimensional degradation model, two mechanisms are proposed to relate the change of microvariables to the loss of modulus as shown in Fig. 4.

6.1 <u>Failure Mechanism Type "A"</u>

In Fig. 10, is shown a sequence of events leading to a loss of load-carrying capacity of a single fiber in tension, if there is experimental evidence that the fiber-breaking load distribution, as a material property, changes with time or number of cycles more rapidly than the fiber-to-fiber bond distribution. In that case, the fiber-to-fiber bond strength, as a first approximation could be represented by the mean of the observed strength distribution mentioned earlier. With this simplification, the true tensile load distribution curve in a typical single fiber could be represented with an idealized one as shown in Fig. 10(a).

To illustrate this concept, let us construct a numerical example based on the bond shear strength data of white fir summer-wood as shown in Fig. 3(a). The mean shear strength of the fiber-to-fiber bond is given as 2.516×10^6 N/m^2 (or 0.26 kgf/mm^2 which is also0.00026 gf/μm^2). Assumming a typical fiber having an oblong cross-section of 20 μm by 5 μm, the perimeter is approximately 50 μm. The estimated bond strength per line dimension of the fiber may be given by $0.00026 \times 50 = 0.013$ gf/μm. By assuming that the fiber is bonded to its neighbors and the matrix in the same effective way, and further assuming that the average distance between neighboring fibers is given by 100 μm, we can estimate that the force differential between two crossings can be at most 1.3 gf.

Let us now assume that the minimum breaking load for the single fiber is 5.2 gf. According to the above calculation, it takes 4 crossings to fully develop that force. Since this takes place at both ends, it requires a minimum length of 800 μm or 0.8 mm for a fiber to fully develop a load of 5.2 gf between D and E as shown in Fig. 10(b). It is reasonable to expect that there are a good portion of fibers longer than 0.8 mm. Every cycle of loading will then subject those fibers longer than 0.8 mm to break with a probability given by the fiber-breaking load distribution similar to that given in Fig. 8(b). On a statistical basis, a measure of the load carrying capacity of the specimen can be constructed from the area under the curve given by the load distribution of Fig. 10(b). In Fig. 10(c), we see how an application of the fiber-breaking criterion can reduce the load-carrying capacity of the same fiber after a break is assumed to take place mid-point between D and E. This mechanism produces a loss of modulus by keeping the strain constant, the load-carrying capacity decreasing, and the "effective" fiber length distribution altered in favor of shorter fibers. Fig. 10(d) shows how the fiber load distribution changes during unloading with the implication that the damage per cycle as caused by microscopic fiber-breaking is irreversible. Since the same calculation is carried out for a microscopic state with a time- or cycle-dependent fiber length distribution, the damage per cycle must necessarily vary with each cycle. This non-linear damage-to-cycle result is consistent with the experimental data on currency paper as shown in Fig. 4.

6.2 Failure Mechanism Type "B"

If it turns out that the fiber-to-fiber bond distribution, as a material property, changes with time or number of cycles more rapidly than the fiber-breaking load distribution, then we must consider a mechanism where the bond strength is controlling rather than the fiber-breaking load as shown previously. Since bond strength is usually analyzed over several crossings, it does not make sense to emphasize a minimum value and its probability as was done for the fiber-breaking load. Our new mechanism, therefore, will be formulated for a critical bond strength to be given by the mean of a given distribution. In addition, the failure mechanism is considered as a process of bond transfer through a typical matrix containing a void which increases its diameter with time. In Fig. 11(a), we view the fiber as being loaded until the critical bond strength is reached. In Fig. 11(b) and 11(c), we describe how local shear yielding can take place as a consequence of the increase in void diameter. The mechanism is such that there is no loss in bond-transfer capacity, but there is an increase in fiber motion following the loss of the matrix stiffness. A loss of modulus is again a natural consequence of this failure mechanism because as the load capacity is maintained constant, the strain can increase and the "equivalent" void diameter distribution is altered in favor of larger holes. Assuming that unloading has no influence on the micro-geometry of the specimen, we see that the damage under this mechanism is again irreversible. Using a similar argument as given for the Type "A" mechanism, it is seen that the damage per cycle cannot be a constant and therefore the damage is a nonlinear function of the number of cycles.

7. DISCUSSION

In each of the two mechanisms of microscopic failures described above, there is a built-in algorithm for the feature of a rapid loss of modulus during the first few thousand cycles and a gradual leveling of the modulus during subsequent cycling. For different grades of paper, it is quite conceivable that either mechanism or both may be controlling during separate stages of degradation.

For currency paper, evidence from scanning electron photomicrographs of flexed specimens shows that the problem is much more complicated than postulated in the two mechanisms. For instance, all of the processes causing micro-structure degradation are probably occurring simultaneously, albeit at substantially different rates. Mathematical modelling with limited experimental data should never be pushed beyond the stage where there is no forseeable hope of verifying critical assumptions. At this stage, it is not unreasonable to conclude that a novel approach to the problem of modelling the degradation of paper has been found. It remains to be seen whether this approach is more profitable and physically more meaningful than those due to Corte [17], Page [18,19], Dodson [20] and others.

8. ACKNOWLEDGMENT

We wish to thank C. T. J. Dodson (University of Lancaster, England), R. Kirsch and H. J. Oser (both of the National Bureau of Standards), for helpful discussions and encouragement in the course of this investigation.

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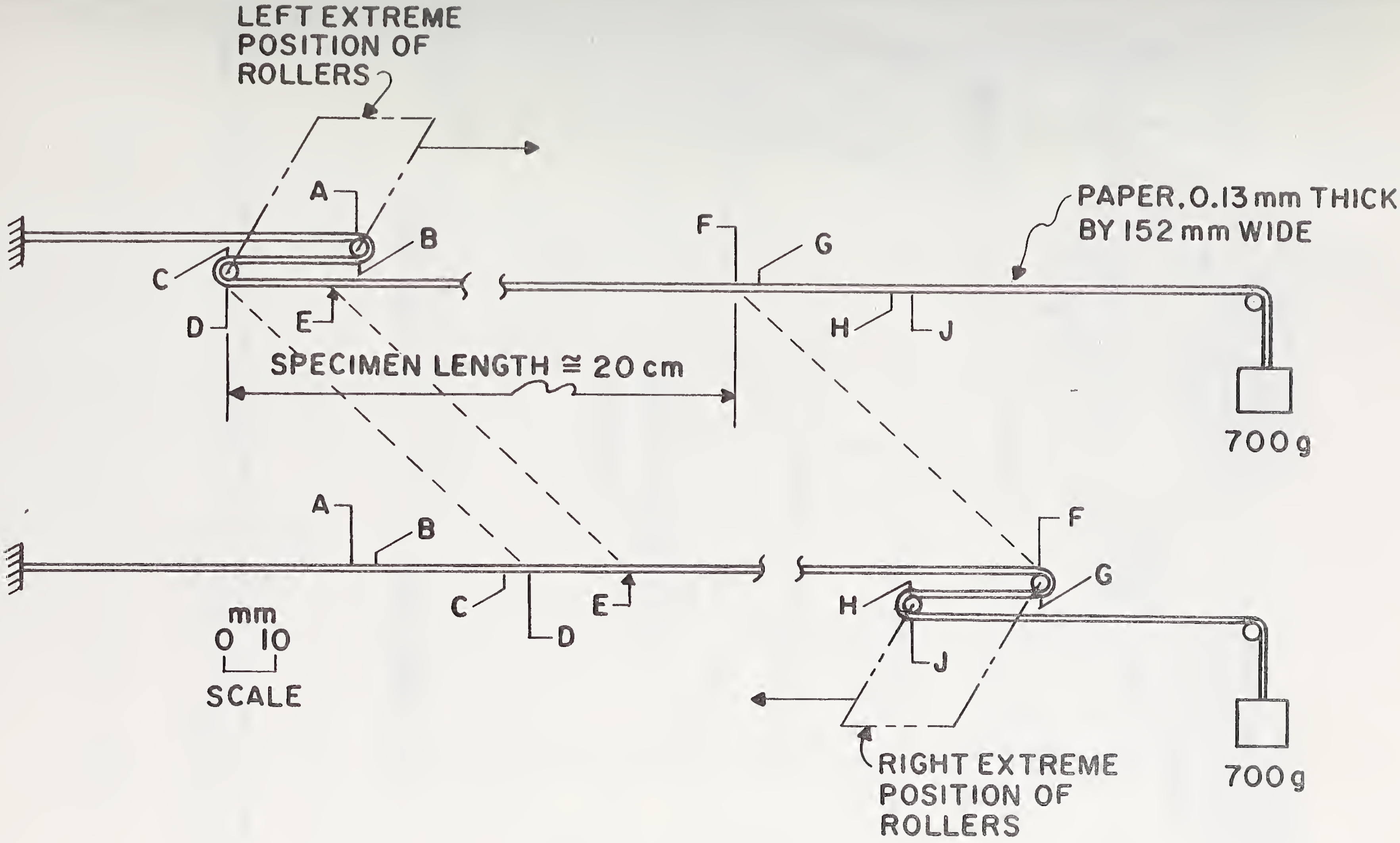


Figure 1: Elevation View of NBS Paper-Flexing Machine in Two Extreme Positions of Rollers (3.18 mm diameter).

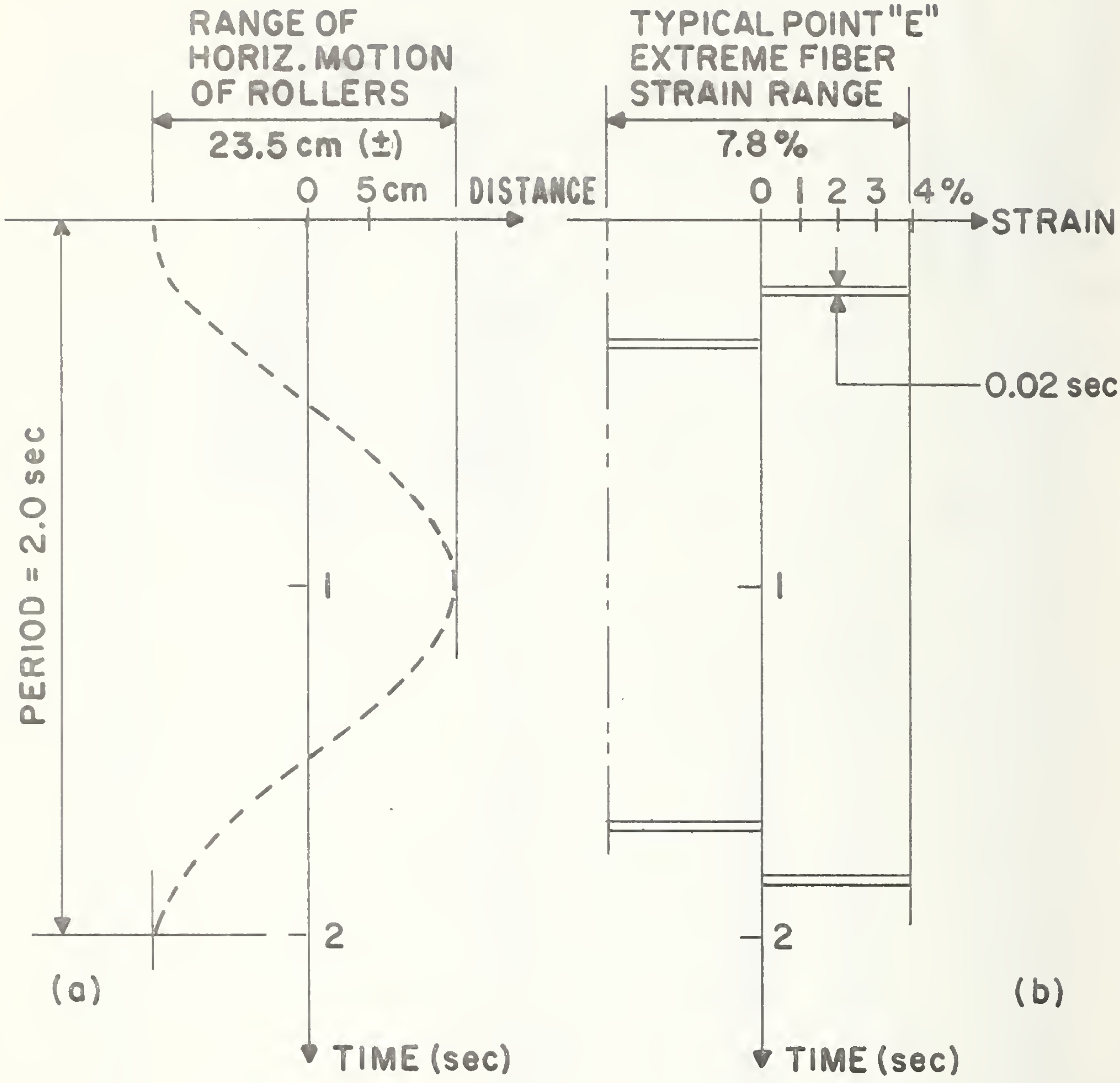


Figure 2: Time and Motion Plot for Rollers and Typical Point "E": (A) Horizontal Motion of Rollers vs. Time; (b) Extreme Fiber Strain vs. Time.

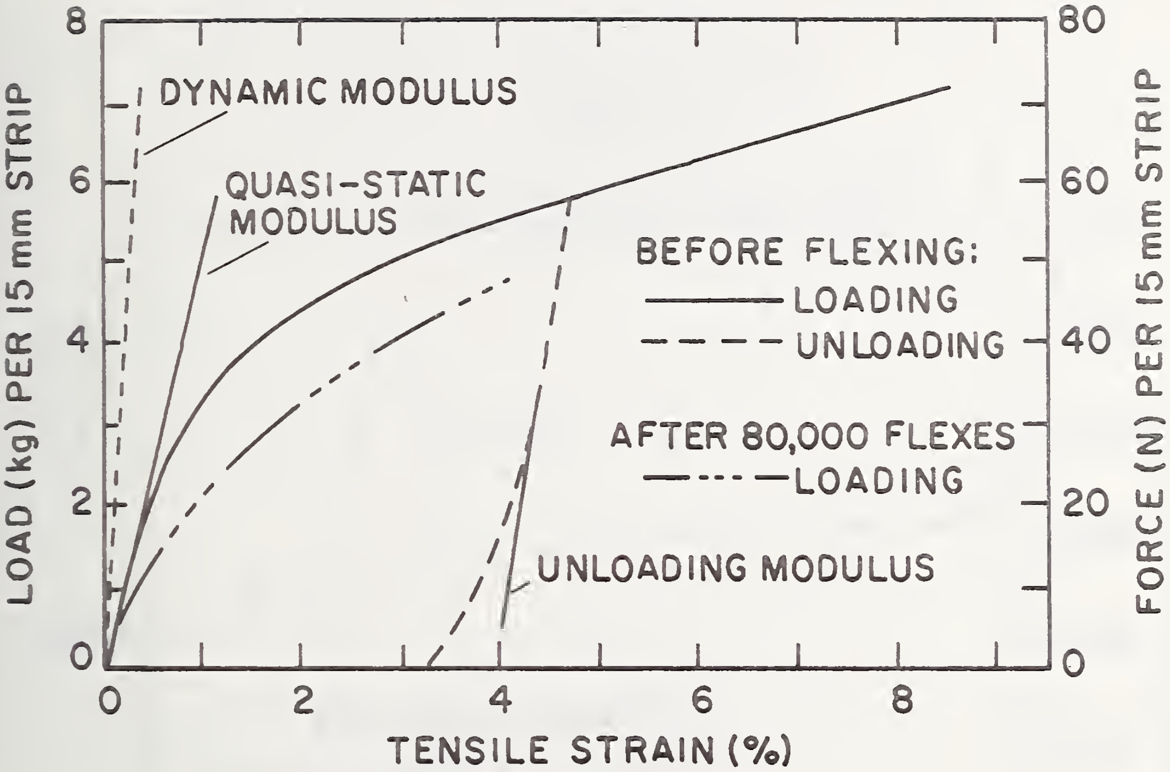


Figure 3: Load-Elongation Curves for Currency Paper in Cross Direction Before and After 80,000 Flexes.

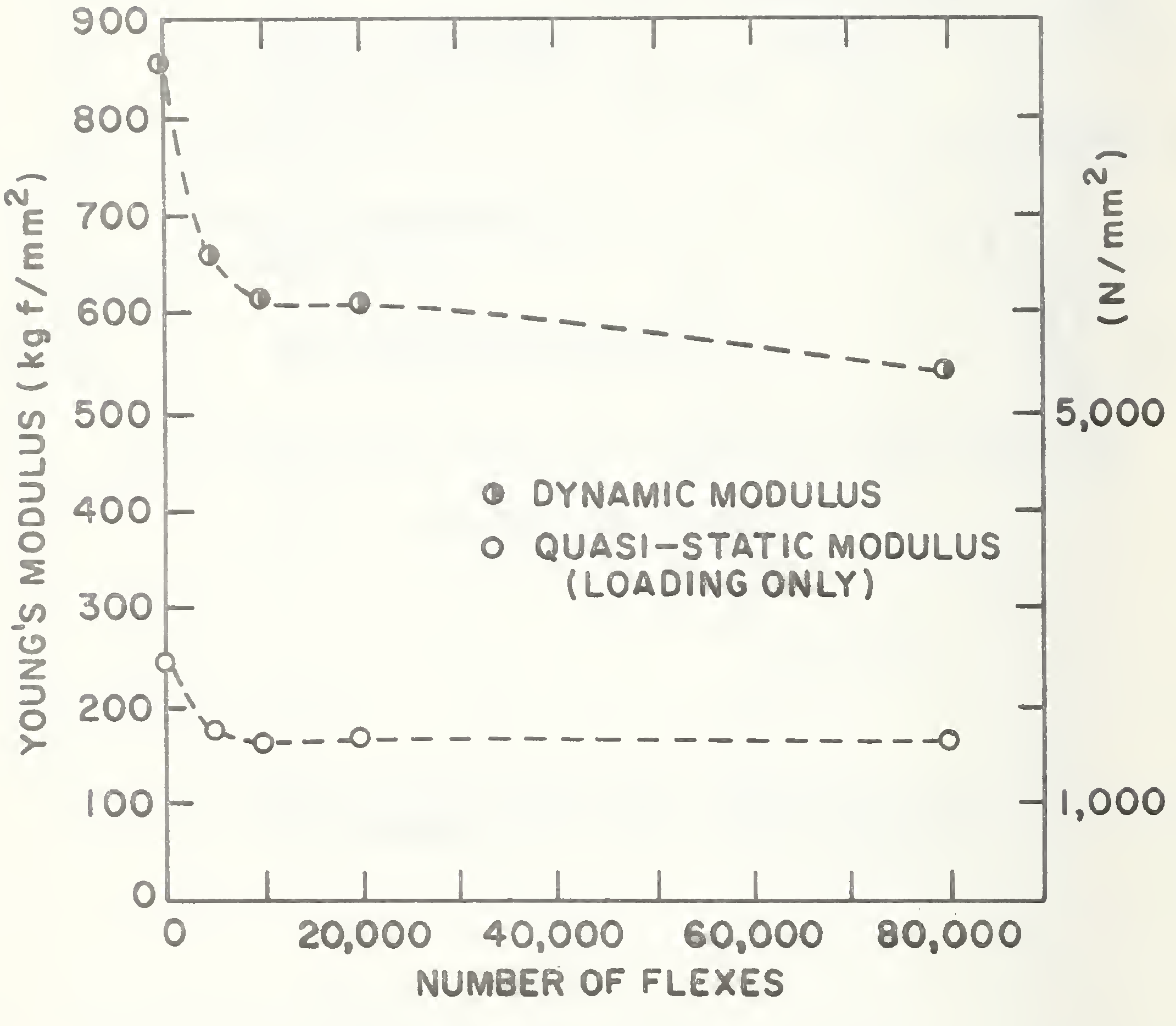


Figure 4: Change of Young's Modulus of Currency Paper in Cross Direction Due to Flexing over 3.18 mm. Diameter Rollers.

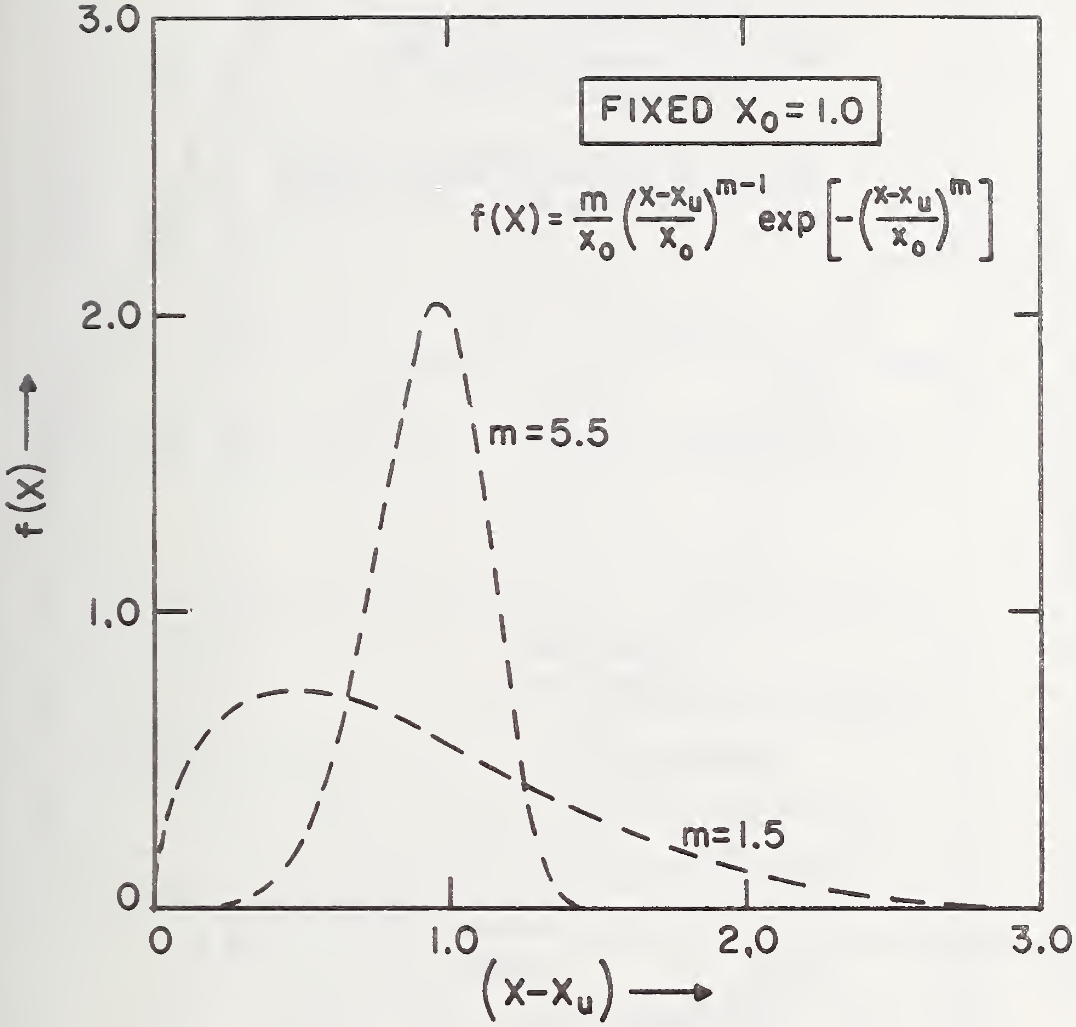


Figure 5: Weibull Probability-Density Function with Fixed x_o and Varying m.

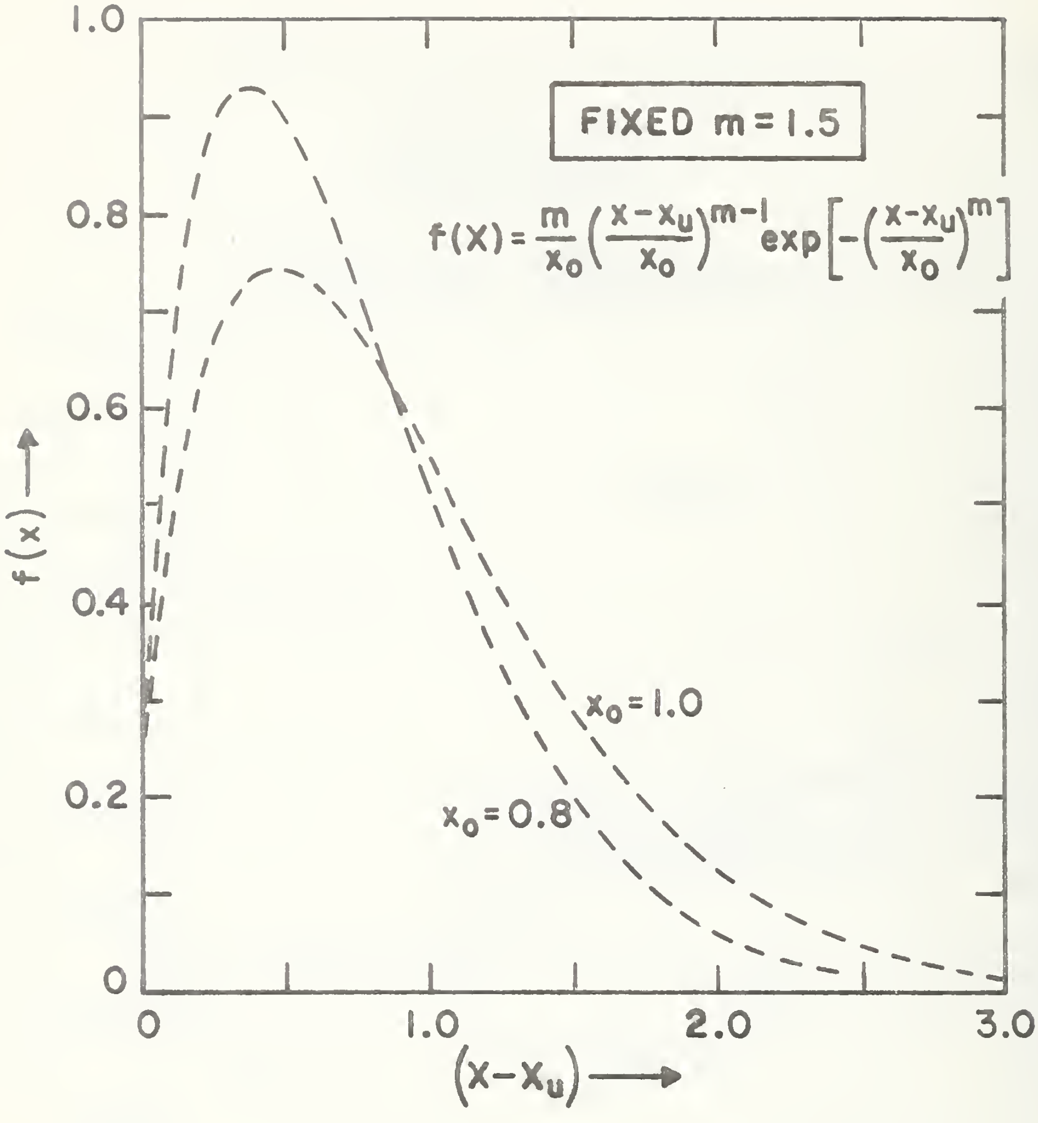


Figure 6: Weibull Probability-Density Function with Fixed m and Varying x_o.

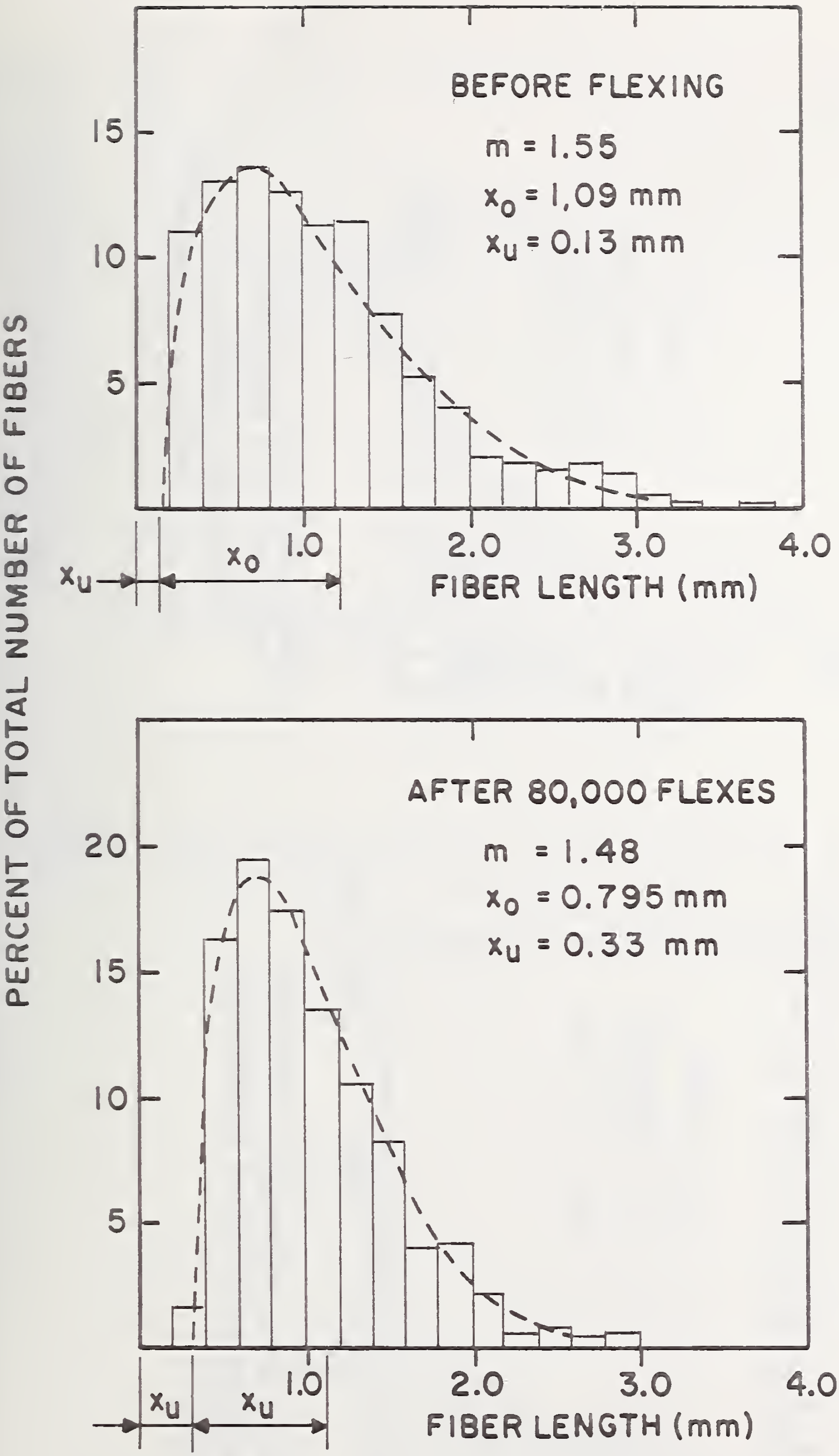
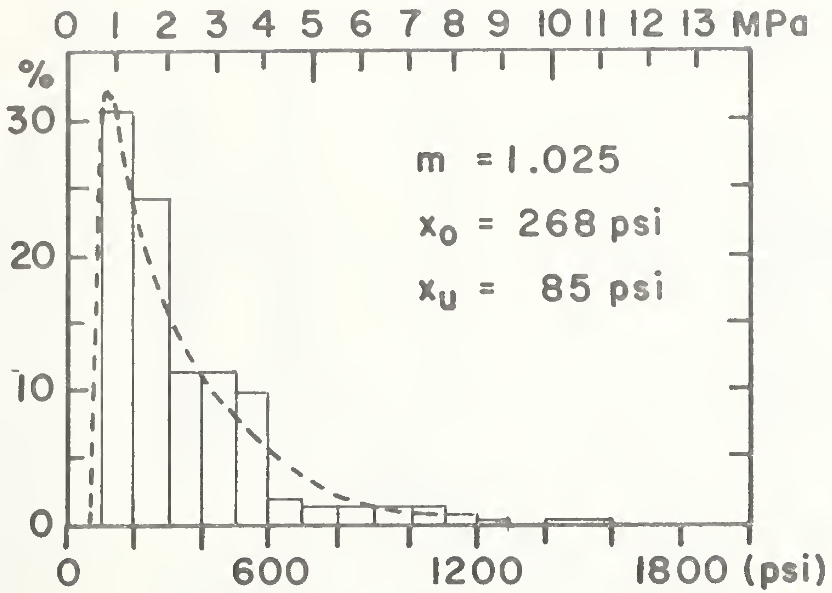
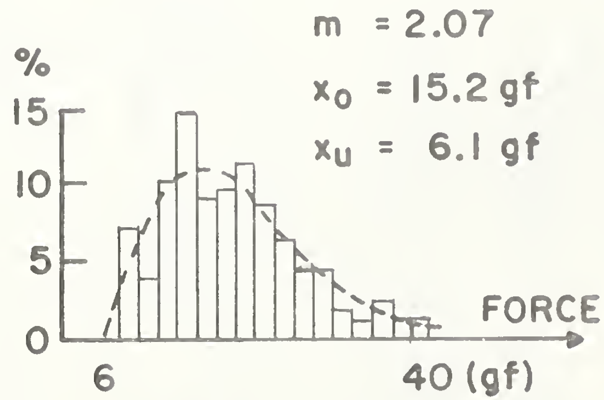


Figure 7: Frequency Distribution of Fiber Lengths for Currency Paper Before and After 80,000 Flexes.



(a) Bond shear strength of white fir Summerwood (after Schniewind, et al[16])



(b) Breaking load of Scots pine Summerwood fibers from unbleached unbeaten kraft pulp (after Dunker & Nordman[15])

Figure 8: Typical Frequency Distribution of Micro-Strength Parameters for Summerwood Fibers.

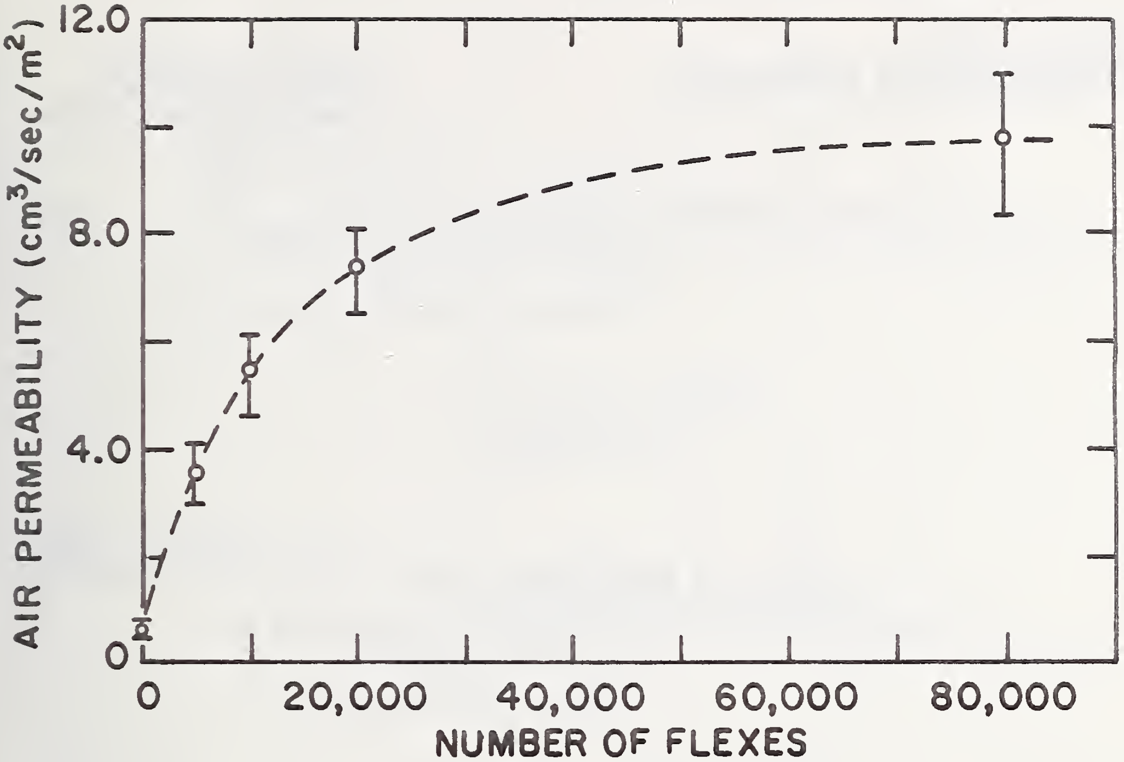


Figure 9: Air Permeability vs. Number of Flexes for Currency Paper in Cross Direction.

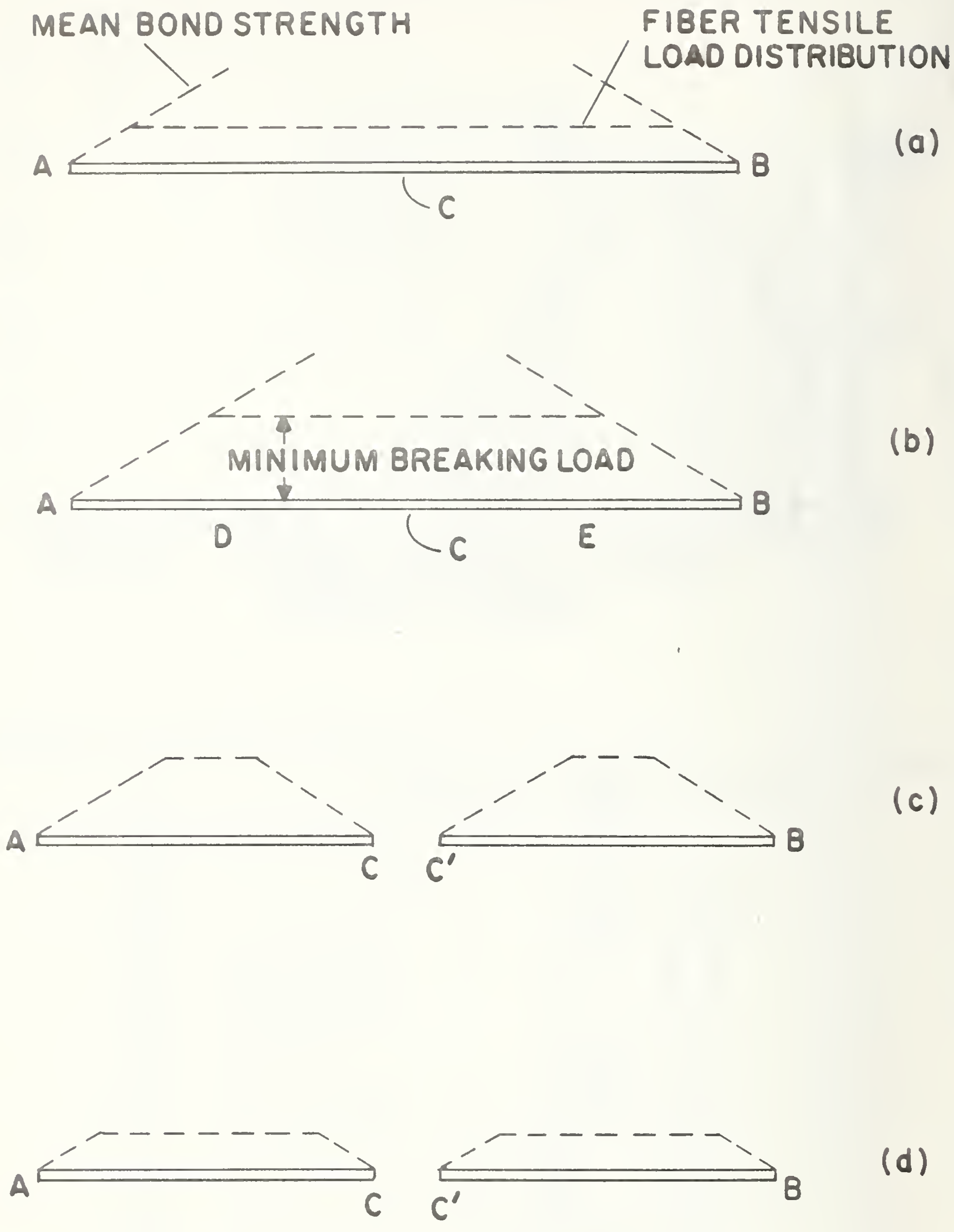


Figure 10: Failure Mechanism Type "A" Due to Fiber-Breaking.

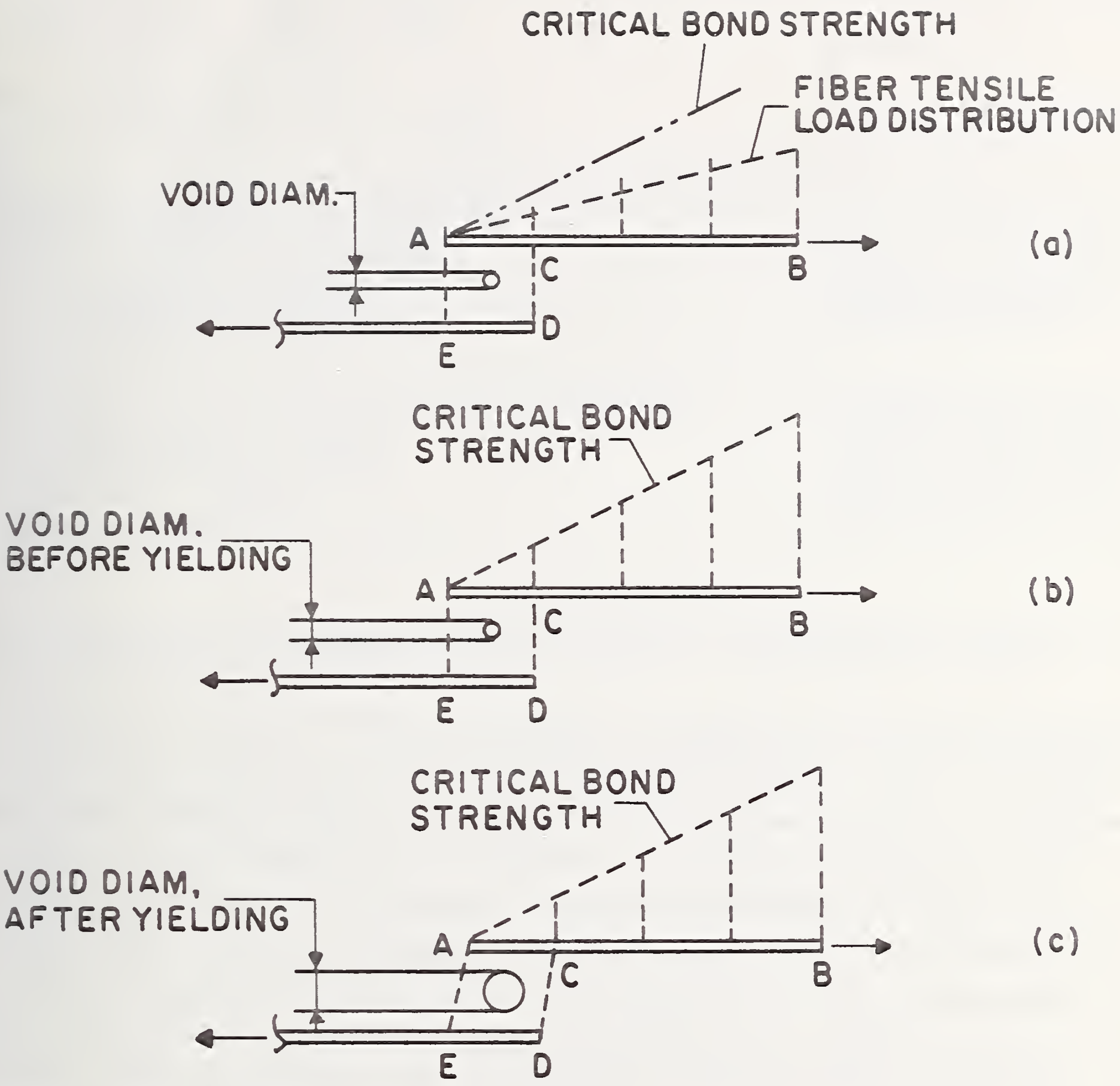


Figure 11: Failure Mechanism Type "B" Due to Loss of Matrix Stiffness.

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15. SUPPLEMENTARY NOTES

16. ABSTRACT *(A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)*

Motivated by scanning electron micrographs showing micro-structural degradation of several grades of paper under repetitive loadings (Graminski, 1973), we present a crude statistical approach in defining two micro-variables, namely, an "effective" fiber length, and an "equivalent" void diameter. The approach is based on the mathematical properties of a 3-parameter statistical distribution originally due to Weibull (1939). Two micro-strength parameters, namely, the fiber breaking load, and the fiber-to-fiber bond strength, are also given a Weibull statistical analysis. Two distinct mechanisms for explaining the loss of modulus due to repetitive flexing are presented for constructing a self-consistent microscopic degradation model of paper.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)* Durability; fatigue; fiber length; flexing; low cycle fatigue; mechanical properties; microstructure; modelling; paper; pore size; statistical analysis; Weibull distribution.

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