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## DETERMINATION OF ALL NINE ORTHOTROPIC ELASTIC CONSTANTS FOR MACHINE-MADE PAPER

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Determination of all nine orthotropic elastic constants for machine-made paper

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#### Abstract

Ultrasonic methods for determining all nine orthotropic elastic constants for machine-made paper are discussed. Experimental results for a heavy milk carton stock are presented, along with estimated measurement uncertainties. Low z-direction stiffness coefficients and high out-of-plane Poisson ratios were found.

#### Introduction

In a previous paper we demonstrated that machine-made paper may be considered a three dimensional homogeneous orthotropic medium and that orthotropic plate wave theory is successful in describing the propagation of ultrasonic waves in paper at wavelengths much greater than fiber dimensions.  $(\underline{1})$ \*. This paper describes the measurement of all nine elastic constants using ultrasonic techniques on a heavy milk carton stock.

This paper has been submitted for publication in Tappi.

<sup>\*</sup>An orthotropic material has elastic properties which are symmetric about each of three mutually perpendicular symmetry planes. It requires nine independent elastic constants for complete characterization of its elastic properties [e.g., see Reference (2)]. In paper the three principal axes are taken as the machine (X or 1) direction, the cross machine (Y or 2) direction, and the thickness (Z or 3) direction.

Nonresonance mechanical methods for measuring the elastic properties involve the measurement of stress and strain. For paper this means measuring small displacements, especially in the z-direction. Ultrasonic measurements, on the other hand, involve the determination of wave velocities which relate directly to the elastic constants of the material. Compared to the mechanical methods, ultrasonic elastic constant measurements are made at high frequencies and low strain. Paper is a viscoelastic substance, and since there is less time for viscoelastic relaxation, ultrasonic stiffness coefficients should always be larger than their mechanical counterparts. This has been observed in our laboratory and reported by a number of authors. A definitive investigation of the relationship between mechanical and ultrasonic elastic coefficients would require a detailed knowledge of the viscoelastic behavior of paper. The viscous character requires that complex stiffness coefficients be defined, and these must be known as functions of frequency and temperature. Some work has been done in this area (3).

#### Background

In the previous publication (1), ultrasonic techniques were described for determining 7 of the 9 orthotropic stiffness constants for heavy board samples. These are reviewed briefly here. At frequencies where the wavelength of ultrasound in the z-direction is less than or comparable to the caliper, paper must be treated as a plate when describing in-plane propagation of elastic waves. In this case there are an infinite number of plate wave modes. These modes are dispersive, that is, the velocities are functions of frequency. Paper can be considered a bulk (three-dimensional) material when propagating waves in the z-direction, however, and three of the stiffness coefficients are easily determined by measuring z-direction bulk wave velocities:

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$$C_{33} = \rho V_{Lz}^2$$
 (1)

$$C_{44} = \beta v_{Sy-z}$$
(2)

$$S_{SS} = \rho v_{Sx-z}$$
 (3)

where

 $V_{Lz}$  = velocity of bulk longitudinal wave,

 $V_{Sy-z} =$  velocity of bulk shear wave polarized in the y-direction,

- $V_{Sx-z}$  = velocity of bulk shear wave polarized in the x-direction, and
  - $\rho$  = apparent density.

The constants  $C_{11}$  and  $C_{22}$  can be determined by propagating bulk longitudinal waves in the machine (x) and cross-machine (y) directions, respectively. In the case of paper, this requires the construction of paper stacks (<u>1</u>). A more accurate technique has been developed, however, whereby bulk velocities are measured on single-thickness specimens. The basis for this technique will be discussed later.

At low frequencies, where the wavelength is large compared with the plate thickness, the zeroth order symmetric (SO) mode is essentially nondispersive. The stiffness coefficients  $C_{13}$  and  $C_{23}$  can thus be determined by measuring these low frequency SO velocities, and using:

$$C_{13} = [C_{33}(C_{11} - \rho V_{S0x}^2)]^{1/2}$$
(4)

$$C_{23} = [C_{33}(C_{22} - \rho V_{S0y}^2)]^{1/2}$$
(5)

where  $V_{SOx} =$  velocity of low frequency SO wave propagated in the x-direction

V<sub>SOy</sub> = velocity of low frequency SO wave propagated in the y-direction

Several of the plate wave modes for x-direction propagation in the milk carton stock are shown in Fig. 1. These dispersion curves represent

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theoretical predictions of the relationships between phase velocity and frequency, and are obtained using an orthotropic plate wave theory (4). For x-direction propagation, the parameters which must be specified are density, plate thickness,  $C_{11}$ ,  $C_{33}$ ,  $C_{13}$ , and  $C_{55}$ . Similar results were obtained on linerboard samples (5).

The plateau regions on the symmetric (S) modes in Fig. 1 are quite sensitive to the z-direction stiffness. If  $C_{33}$  is very small compared to  $C_{11}$  or  $C_{22}$ , well defined plateaus are obtained. These frequency, independent regions disappear if  $C_{33}$  is comparable to  $C_{11}$  or  $C_{22}$ .

The existence of the plateaus for paper suggests that pulse propagation techniques can be employed in these regions. It is possible in fact to determine  $C_{11}$  and  $C_{22}$  by propagating the S1 mode at the appropriate frequency. In Fig. 1 a line drawn at the bulk longitudinal velocity,  $V_{Lx}$ , is shown crossing the S1 and S2 modes in the middle of the plateau regions. The frequency which corresponds to  $V_{Lx}$  on the S1 mode can be determined from orthotropic plate wave theory (5):

$$f_{1} = \frac{V_{LZ}}{T} \left[ \frac{1}{1 + C_{13}(C_{13} + 2C_{55})} C_{11}C_{55}} \right]^{1/2}$$
(6)

where T = plate thickness.

The radical term in this expression is very nearly one so that  $f_1$  can be estimated by measuring  $V_{L_2}$  and T. The bulk longitudinal velocity,  $V_{L_x}$ , is then obtained by propagating an Sl plate wave at this frequency. A similar equation and procedure is used to estimate  $V_{L_y}$ . The velocities  $V_{L_x}$ and  $V_{L_y}$  may then be used to compute  $C_{11}$  and  $C_{22}$  from:

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$$C_{11} = \rho V_{Lx}^2 \tag{7}$$

$$C_{22} = \rho V_{Ly}^2 \tag{8}$$

and  $C_{13}$  and  $C_{23}$  may be found from Equations (4) and (5). The values of these coefficients can be improved by substituting the estimates for  $C_{13}$ and  $C_{11}$  ( $C_{55}$  is measured directly) back into Equation (6) and recalculating  $f_1$ . An iterative procedure is thus used to arrive at final values for  $C_{11}$ ,  $C_{22}$ ,  $C_{13}$  and  $C_{23}$ . This procedure is superior to the technique described earlier (<u>1</u>) which requires the construction of paper stacks.

With the above four coefficients known, and with  $C_{33}$ ,  $C_{44}$ , and  $C_{55}$ measured directly [Equations (1)-(3)], only  $C_{12}$  and  $C_{66}$  remain to be found. The coefficient  $C_{66}$  is easily determined by measuring the velocity of a shear wave propagated in either the x or y-direction with polarization in the y or x-direction, respectively. The expression for  $C_{66}$  is:

$$C_{66} = \rho V_{Sx-y}^2 \tag{9}$$

This shear velocity can be measured on either plate or bulk materials.

The constant  $C_{12}$  is obtained by propagating a shear wave, polarized in the x-y plane, at a direction 45° to both the x and y-axes. The expression for  $C_{12}$  in this case is:

$$C_{12} = ((2\rho V_{S}^{2}(45^{\circ}) - 1/2(C_{11} + C_{22}) - C_{66})^{2} - ((C_{11} - C_{22})/2)^{2})^{1/2} - C_{66}$$
(10)

where  $V_{S}(45^{\circ})$  = velocity of the in-plane shear wave propagated in a direction 45° to the x and y-directions.

Equation (10) applies to bulk materials. It has been shown ( $\underline{6}$ ) that Equation (10) is also valid for low frequency wave propagation in plates.

In this case the  $C_{ij}$ 's are planar stiffness coefficients, which have different values from the bulk  $C_{ij}$ 's.

Determining  $C_{12}$ ,  $C_{13}$ , and  $C_{23}$  from ultrasonic velocities requires taking a square root [see Equations (4), (5), and (10)]. For results reported in this paper, we have used the positive square root in order to make  $C_{12}$ ,  $C_{13}$ , and  $C_{23}$  positive.

#### Experimental

The ultrasonic methods described above were used on a heavy bleached kraft milk carton stock. The 26.7 mil thick sample had an average basis weight of 107.8 lb/1000 ft<sup>2</sup>, and an apparent density of 0.78 g/cm<sup>3</sup>. As received, the 14 x 28-inch sheets were preconditioned at low humidity for 48 hours before being placed in a controlled environment (73°F, 50% RH) for subsequent testing.

The experimental techniques for determining  $V_{Lz}$ ,  $V_{Sy-z}$ ,  $V_{Sx-z}$ ,  $V_{S0x}$ , and  $V_{S0y}$  have been previously described (<u>1</u>). These velocities are determined by measuring the transit time of a short burst of sine waves (pulse) through the specimen. Two piezoelectric transducers are used in the measuring system depicted in Fig. 2. The output pulse from the function generator is amplified and fed to the sending transducer. Coincident with the first positive peak in this pulse is the positive slope of the SYNC OUT square pulse. The main time base of the oscilloscope is triggered by this positive slope. At the same time, the scope generates a pulse which starts a time interval counter. The mechanical disturbance transmitted through the particular specimen is converted back to an electrical signal, which is amplified and dis-

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played on the oscilloscope. By adjusting the delay time multiplier knob on the scope, the instant of triggering of a second (delayed) time base is controlled by the operator. The scope provides visualization of the precise point of triggering. Finally, coincident with the triggering of the delayed time base is the delayed GATE OUT which stops the counter. Delay time intervals are averaged by the digital display counter.

Delay times were measured out to a positive peak in the received signal, typically near the middle of the pulse. The measurements were corrected for delays in the transducers and electronics. It was found that by time averaging the time intervals, delay times could be measured to the nearest nanosecond.

The two in-plane shear velocities,  $V_{Sx-y}$  and  $V_S(45^\circ)$ , are also measured using this pulse propagation technique. In this case, pulses were propagated through three-dimensional stacks, comprised of individual sheets of the milk carton stock glued together with rubber cement. The effect of the glue was determined to be small. These measurements can also be made on loose, slightly compressed, stacks.

Three stacks were assembled from the milk carton stock. Each was about 3 inches long, 2 inches high, and 1-1/2 inches wide. Opposite sides were machined smooth, using a belt sander, for eventual placement of the piezoelectric contact transducers. Propagation was through the width of the stacks, with propagation directions  $0^{\circ}$ ,  $45^{\circ}$ , and  $90^{\circ}$  from the xdirection. Shear velocity measurements were made at three locations along the length of each stack, using shear transducers at a frequency of about 400 kHz. A small amount of honey was used to efficiently couple the 1/2-inch diameter transducers to the specimens.

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The established technique for accurately measuring  $V_{SOx}$  and  $V_{SOy}$  for thick board samples employs contact transducers at low frequencies (10-50 kHz). The transducers are affixed to opposite edges of the specimen, and low frequency pulses are propagated across the specimen width. Because the SO waves have negligible z-direction stress at these low frequencies, this technique is valid for propagation through loose stacks and, therefore, sheets of any caliper can be used.

The bulk longitudinal velocities,  $V_{Lx}$  and  $V_{Ly}$ , are also determined on single thickness specimens, or loose stacks, by propagating longitudinal pulses at the appropriate S1 plateau frequency,  $f_1$ . This technique is applicable only when  $f_1$  is less than about 750 kHz. For a value of  $V_{Lz}$  of 0.25 mm/µsec, the minimum specimen thickness would be about 0.33 mm, or 13 mils.

Experimental S1 velocities are plotted in Fig. 3 along with a portion of the theoretical S1 dispersion curves for x-direction propagation in the milk carton stock. The experimental data, representative of only one specimen, follows the plateau region closely. The small slope of the S1 plateau regions means that  $V_{\rm Lx}$  is quite insensitive to errors in estimating  $f_1$ .

The determination of  $C_{13}$  depends upon the relative accuracy of measuring  $V_{Lx}$  and  $V_{S0x}$  [see Equations (4) and (7)], since these velocities typically differ by only a couple of percent. Because of wide variations between specimens, it becomes imperative to measure these velocities on the same specimens. Low frequency pulses were first propagated across the width of a 12 x 28-inch specimen using longitudinal transducers at 50 kHz. Transit

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time measurements were made at 10 locations. The original specimen was then cut into two 6 x 28-inch specimens, and transit times were measured through each half, at the same 10 locations. Average transit times were calculated for each transducer separation (i.e., 12 and 6 inches) and the difference was divided into 6 inches (152.4 mm) to determine  $V_{SOx}$ .

This procedure was then repeated at 332 kHz (using different transducers), starting with the two 6 x 28-inch specimens. The S1 plate waves were propagated along the same paths. In this way,  $V_{Lx}$  and  $V_{S0x}$  should be representative of the same material. The velocities  $V_{S0y}$  and  $V_{Ly}$  were then determined in the same manner, using specimens oriented in the ydirection.

### Results and discussion

The nine independent velocity measurements for the milk carton stock are given in Table I, along with estimates of measurement accuracy.  $V_{Sx-y}$ was taken as the average velocity for the two stacks oriented 0 and 90° to the x-direction. The difference between the two velocities was slight, as expected.

Absolute accuracy refers to how well the particular velocity measurement characterizes all of the specimens of the sample. For instance, it is estimated that  $V_{Lx}$ , measured on a few specimens, is within 0.25% of the true average velocity for those specimens. However, considering the variation between specimens, it is estimated that this value of  $V_{Lx}$  is only within 0.5% of the true sample average. While the same argument applies to  $V_{S0x}$ , both  $V_{S0x}$  and  $V_{Lx}$  were measured on the same specimens making the accuracy of either measurement, relative to the other, higher. The same holds for the measurement of  $V_{S0y}$  and  $V_{Ly}$ , and for  $V_{Sx-y}$  and  $V_{S}(45^{\circ})$ .

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The velocities cited in Table I yield the orthotropic elastic constants given in Table II using Equations (1)-(5) and (7)-(10). The stiffness coefficients are normalized with respect to the apparent density, and have units of (mm/ $\mu$ sec<sup>2</sup>). Using the apparent density of the board of 0.78 g/cm<sup>3</sup>, the engineering constants have been calculated from the stiffnesses [e.g., see Reference (2)] and are also presented in Table II.

The estimated errors associated with the determination of the elastic constants are given in Table III. For those elastic constants which depend upon only one velocity measurement the analyses are straightforward. These constitute the first six entries in the table. The errors involved in determining the remaining constants are not so easily estimated. A computer program has been written which determines  $d(\ln C_{ij})/d(\ln V_k)$  coefficients for each of the elastic coefficients and each velocity. By multiplying these coefficients by the estimated accuracies of the measured velocities, and summing absolute values, the maximum errors associated with the elastic constants are estimated.

The elastic constants for the milk carton stock are typical of other (linerboard) samples tested. These constants indicate that the elastic properties of paperboard are highly unusual. Perhaps the most unique characteristic is the high degree of anisotropy between the in-plane and z-direction properties. This is indicated by the high  $E_{11}/E_{33}$  ratio of 190. In addition the two z-direction shear moduli are both greater than the z-direction Young's modulus.

The very high and low out-of-plane Poisson ratios are also unusual. A value of  $v_{31}$  greater than one means that a stress applied in the x-direction results in a greater strain in the z-direction than in the x-direction. The two out-of-

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plane Poisson ratios,  $v_{31}$  and  $v_{32}$ , are measured least accurately of all the elastic constants. With an accuracy of  $\pm$  61%,  $v_{31}$  is located in the range 0.59 to 2.45. The other Poisson ratio,  $v_{32}$ , lies between 1.32 and 2.36. The z-direction properties are undoubtedly traceable to the more or less parallel alignment of fibers in the plane of the sheet, although no model is proposed at this time to explain these results.

The strain level associated with the propagation of ultrasonic waves is very low. At the same time, the frequency of the oscillations is very high. Ultrasonic measurements allow less time for viscoelastic relaxations than standard mechanical tests. Therefore, the stiffnesses determined ultrasonically for paper always should be higher than the mechanical values.

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Velocity	/ (mm/µsec)	Estimated Accuracy
 V <sub>Lx</sub>	= 3.205	+ 0.5% absolute
V <sub>Ly</sub>	= 2.219	+ 0.5% absolute
V <sub>Lz</sub>	= 0.231	<u>+</u> 5.0% absolute
V <sub>SOx</sub>	= 3.164	$\pm$ 0.25% relative to V <sub>Lx</sub>
V <sub>SOy</sub>	= 2.161	<u>+</u> 0.25% relative to V <sub>Ly</sub>
V Sx−y	= 1.615	<u>+</u> 0.5% absolute
V <sub>S</sub> (45°)	= 1.596	$\pm$ 0.25% relative to $V_{Sx-y}$
V <sub>Sx-z</sub>	= 0.418	+ 5.0% absolute
V <sub>Sy-z</sub>	= 0.351	<u>+</u> 5.0% absolute

I. Nine velocity measurements for milk carton stock

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II.	Elastic	constants	for	the	milk	carton	stock

$C_{11}/\rho =$	10.27	Eı	 1 =	7.44 x $10^{10}$ dynes/cm <sup>2</sup>
$C_{22}/\rho =$	4.92	E <sub>2</sub>	22 =	$3.47 \times 10^{10} \text{ dynes/cm}^2$
$C_{33}/\rho =$	0.0534	Es	33 =	$0.039 \times 10^{10} \text{dynes/cm}^2$
$C_{12}/\rho =$	1.74	$v_{12} = 0.15$		$v_{21} = 0.32$
$C_{13}/\rho =$	0.118	$v_{13} = 0.008$		$v_{31} = 1.52$
$C_{23}/\rho =$	0.116	$v_{23} = 0.021$		$v_{32} = 1.84$
$C_{44}/\rho =$	0.127	G	.4 =	$0.099 \times 10^{10} \text{ dynes/cm}^2$
$C_{55}/\rho =$	0.175	G	<sub>55</sub> =	$0.137 \times 10^{10} \text{ dynes/cm}^2$
$C_{66}/\rho =$	2.61	Ge	56 <b>=</b>	$2.04 \times 10^{10} \text{ dynes/cm}^2$

\*Units of C.  $/\rho$  are  $(mm/\mu sec)^2$ . The engineering constants were calculated using the apparent density (0.78 g/cm<sup>3</sup>).

Elastic constant	Estimated maximum error
C <sub>11</sub> /ρ	<u>+</u> 1%
C <sub>22</sub> /p	<u>+</u> 1%
C <sub>33</sub> /p	<u>+</u> 10%
C <sub>44</sub> /p, G <sub>44</sub> /p	<u>+</u> 10%
C <sub>55</sub> /p, G <sub>55</sub> /p	<u>+</u> 10%
C <sub>66</sub> /p, G <sub>66</sub> /p	<u>+</u> 1%
<b>Ε</b> <sub>11</sub> /ρ	<u>+</u> 1.3%
E <sub>22</sub> /p	+ 1.4%
E <sub>33</sub> /ρ	<u>+</u> 12%
C <sub>12</sub> /p	<u>+</u> 7.7%
C <sub>13</sub> /p	<u>+</u> 36%
C <sub>23</sub> /p	<u>+</u> 19%
V <sub>21</sub>	<u>+</u> 10%
V <sub>31</sub>	<u>+</u> 61%
V <sub>32</sub>	<u>+</u> 28%

III. Estimated measurement errors for milk carton stock elastic constants



FREQUENCY, (kHz)

Figure 1. Typical theoretical dispersion curves for milk carton stock for X-direction propagation. Specimen caliper = 0.0679. The stiffness coefficients needed to compute the curves are  $C_{11} = 10.75$ ,  $C_{33} = 0.0534$ ,  $C_{13} = 0.177$ , and  $C_{55} = 0.175$ , all with units of  $(mm/\mu sec)^2$ .



Figure 2. System for making velocity measurements.

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Figure 3. Theoretical (dashed line) and experimental S1 plate wave velocities for x-direction propagation in milk carton stock.