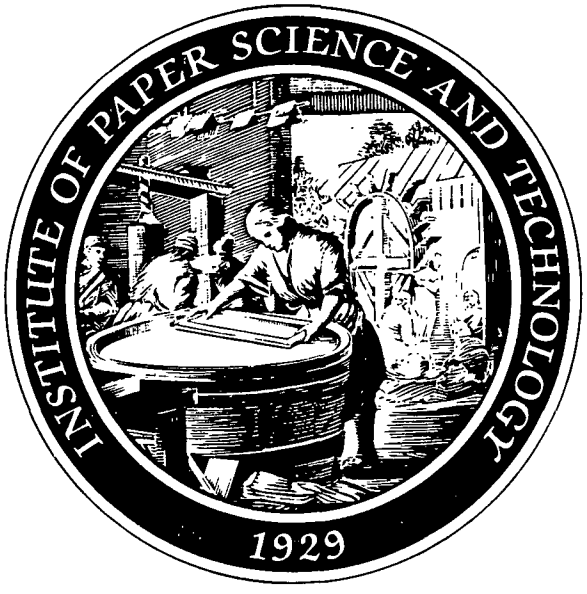

STATUS REPORTS

To The

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

March 21, 1990
Institute of Paper Science and Technology
Atlanta, Georgia

March 1, 1990

TO: MEMBERS OF PAPER PROPERTIES AND USES PROJECT ADVISORY COMMITTEE

Attached for your review are the Status Reports for the projects to be discussed at the Paper Properties and Uses Project Advisory Committee meeting scheduled for March 21, 1990, in Atlanta. A meeting agenda can be found inside the booklet.

We look forward to seeing you on March 21, 1990. Best regards.

Sincerely yours,

RL Ellis

Richard Ellis, Director
Engineering & Paper Materials Division

RE/at
Enclosure

Institute of Paper Science and Technology, Inc.

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Appleton, Wisconsin

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AGENDA

PAPER PROPERTIES AND USES
PROJECT ADVISORY COMMITTEE

March 21, 1990
Institute of Paper Science and Technology
Georgia Power Technology Applications Center
Atlanta, Georgia

WEDNESDAY -- March 21

8:00AM	Registration with coffee and doughnuts	
8:30	Welcome/Introductions	Betz/Ellis
8:45	Anticipated Changes in PAC'S	Yeske
9:45	PROJECT REVIEWS	
	Strength Improvement and Failure Mechanisms (3469)	Waterhouse
	On-Line Measurements of Paper Properties (Project 3332)	Hall
	Process, Properties, Product Relationships (Project 3467)	Brodeur
12:00	LUNCH provided on-site	
1:15	PROJECT REVIEWS	
	Internal Strength Enhancement (Project 3526)	Stratton
	Fundamentals of Paper Surface Wettability (Project 3646)	Etzler
	Board Properties and Performance (Project 3571)	Ellis
	Utilization of Recycled Fiber (Project 3681)	Ellis
4:00	Reception	

Thursday -- March 22

8:00AM	COMMITTEE MEETING (IPST Conference Room)	
11:00	ADJOURNMENT	

Paper Properties and Uses -- Project Advisory Committee

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*date of retirement
02/22/90

ON-LINE MEASUREMENT OF PAPER

STATUS REPORT

FOR

PROJECT 3332

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

March 21, 1990

PROJECT SUMMARY

DATE: February 23, 1990

PROJECT: 3332 - ON-LINE MEASUREMENT OF PAPER PROPERTIES

PROJECT STAFF: M. Hall, P. Brodeur
K. Lorenz, T. Jackson

PROGRAM GOAL:

Develop ways to make on-line measurement of paper properties and process parameters in order to control manufacturing processes.

PROJECT OBJECTIVE:

Shared support with DOE Project to develop sensors and instrumentation capable of measuring the velocity of ultrasound in the in-plane and thickness directions of paper while the paper web is moving at line speed.

PROJECT RATIONALE, PREVIOUS ACTIVITY, AND PLANNED ACTIVITY FOR FISCAL 1989-90:

See attached 1989-90 Project Form.

SUMMARY OF RESULTS LAST PERIOD: (March 1989 - October 1989)

(1) The necessary electronics have been set up and the software has been completed to integrate the in-plane and ZD measurements to provide simultaneous data collection on a moving web.

(2) Mounting yokes for the new IPST, PVDF wheels were completed. These can be bolted to the same frame used to hold the fluid-filled wheels.

(3) Two new fluid-filled wheels were purchased from a second vendor. These wheels offer improvements in ease of transducer exchange, greater latitude in transducer spacing, and in arrangement for installation of a thermocouple to monitor temperature.

(4) We have identified reflection from the fluid-rubber interface as a probable cause of signal interference affecting the accuracy of our determination of transit times. We believe thicker "tires" will provide improved signal cleanliness.

(5) An unwind/rewind system has been ordered. This web handling system will be capable of running in the reel-to-reel or endless loop mode.

(6) We have abandoned diamond coating for bender transducers. Instead, a technique has been developed to adhere a metal wear surface to the tip of the bimorph transducer without interfering with its operation. All indications to date are that this provides excellent wear resistance.

SUMMARY OF RESULTS THIS PERIOD: (October 1989 - March 1990)

(1) Completed set up and check out of hardware and software after move.

(2) Obtained thicker tires for the new fluid-filled wheels. Confirmed that these provide clean rubber-paper interface reflection signals without overlapping interference from fluid-rubber interface reflections.

(3) With cleaner signals, we are examining various techniques to measure the transit times of the direct and reflected pulses.

GOALS FOR NEXT PERIOD: (March 1990 - October 1990)

(1) Complete installation and start up of web handling system.

(2) Mount transducers and interface integrated data collection system on web handling system.

(3) Check out and debug operation of data collection system on moving web.

(4) Procure sample rolls of paper prepared with known processing conditions and selected processes parameter changes.

(5) Collect moving web data on rolls with known properties, determine response of instrumentation to selected process changes.

PROJECT TITLE:	ON-LINE MEASUREMENT OF PAPER	Date: 11/1/88
		Budget: $50,000
PROJECT STAFF:	M. Hall	Period Ends: 6/30/90
		Project No.: 3332

PROGRAM GOAL:

Develop ways to make on-line measurement of product properties and process parameters in order to control manufacturing processes.

CURRENT OBJECTIVE:

To develop the capability to measure mechanical properties on moving paper web. Current emphasis is on out-of-plane measurements. This project is concerned with the development of a laboratory instrument using wheel-type ultrasonic transducers. A related DOE-sponsored project is concerned with development of sensors suitable for making measurements on the paper machine and subsequent control of the papermaking process.

PROJECT RATIONALE:

The ability to measure mechanical properties on the paper machine will provide a means to continuously monitor product quality related to end-use performance. It also provides data needed to relate product characteristics to process variables for paper machine control.

RESULTS TO DATE:

A theory for the propagation of ultrasound in paper was developed. Devices were constructed to make on-machine measurement of the in-plane elastic parameters of paper and board. These devices were successfully tested in mill environments. Another version of the equipment for in-plane measurement was constructed and tested. This design used two receivers located at different distances from a transmitter, all mounted in a drum. Thus, this version was self-calibrating and could be used for on-machine measurement of light weight paper grades.

A cross correlation technique was implemented to improve the accuracy in measuring the transit time of an ultrasonic pulse for in-plane velocity measurements. Equipment was developed for measuring the effects of moisture and temperature on paper elastic properties. The feasibility of ZD signal transfer between rubber-faced, ceramic transducers at high paper speeds was demonstrated.

The background information and experience provided by this project was the basis for obtaining a Department of Energy contract to develop "On-Machine Sensors to Measure Paper Mechanical Properties."

A high-frequency, low impedance, ultrasonic transducer was developed for out-of-plane measurements using a plastic (PVDF) piezoelectric material. This type of transducer is superior to commercial ceramic transducers for our applications.

Wheel-type transducers for ZD measurement have been constructed with a continuous PVDF piezoelectric film around the circumference. A practical laboratory instrument for profiling caliper and ZD velocity by feeding a paper sample through the nip between two wheel-type transducers was completed. This rotary instrument gives results that compare well with the standard lab instrument that uses longitudinal flat transducers and is about twice as fast in evaluating samples.

Preliminary dynamic testing of the IPST PVDF wheels have demonstrated that their use for on-line ZD measurements is a feasible approach.

Preliminary investigations of commercial fluid-filled wheels for on-line ZD measurement have demonstrated this also to be a feasible approach.

In-plane, on-line bimorph transducers operate well, but work must be done to reduce sensitivity to tension and improve wear resistance.

PLANNED ACTIVITY FOR THE PERIOD:

Most of the research activity in this program area will be performed under the closely related DOE project. The DOE project will be concerned primarily with transducer design selection and

hardware design and construction to integrate in-plane and thickness-direction ultrasonic measurements into a system that can be used on a moving web. Principle planned work includes:

Construct improved IPST PVDF wheels for on-line operation.

Construct improved immersion transducers for operation in fluid-filled wheels.

Develop appropriate electronics hardware and software to do in-plane and out-of-plane on-line testing.

Construct the mechanical mounting apparatus necessary to do simultaneous on-line operation both in-plane and out-of-plane. Provide a viable calibration technique.

Refine the mounting of on-line bimorph transducers and improve their wear resistance.

Examine whether it may be feasible to determine on-line the angle of the principle axis of the plane polar plot.

ON-LINE MEASUREMENT OF PAPER MECHANICAL PROPERTIES
Project 3332/3613

The primary objective of this project is to develop sensors capable of measuring the velocity of ultrasound in the thickness and in-plane directions of paper while the paper web is moving at paper machine speed. The measurement of the velocity of ultrasound provides a nondestructive technique to characterize the mechanical properties of paper, allowing continuous monitoring of product quality as well as data for process control. A further objective is to develop hardware and techniques using these sensors to control the paper manufacturing process. Successful implementation of the results of this project will provide more effective utilization of raw materials and energy while producing products with improved uniformity and quality. A major part of the support for this project is provided by funding from the U.S. Department of Energy, Contract No. DE-AC05-86CE40777.

STATUS

Two approaches for making on-line measurement of ultrasonic velocity in the thickness direction (ZD) of paper have been developed. One uses IPST-made, elastomer-faced, PVDF wheel transducers; and the other uses commercially available fluid-filled wheel transducers. Our work to date has demonstrated that each of these has the potential of on-machine use. Current work is concentrating on the approach using fluid-filled wheel transducers.

For in-plane measurements, surface-hardened, bimorph or bender transducers have been developed. These offer potential advantages over transducers used in previous on-line, in-plane work in that they are broadband and have a much smaller area of contact with the web.

A 386-type computer is being used for our on-line work. Software necessary to support the on-line operation of the fluid-filled wheels has been developed. This requires determining the transit times for three discrete pulses, followed by printout of calculated results. The necessary electronics have been set up and the software completed to provide simultaneous data collection of both the in-plane and ZD measurements on a moving web.

An unwind/rewind system is being built and the vendor expects to have the system ready for delivery by mid-March. This web handling system will be capable of speeds up to 2500 fpm, running in either an endless loop or a reel-to-reel mode. It will be able to handle webs up to 14 inches in width with tension controllable from 0.5 to 4 pounds per linear inch. This system will enable us to test commercial and pilot-machine prepared webs in the laboratory.

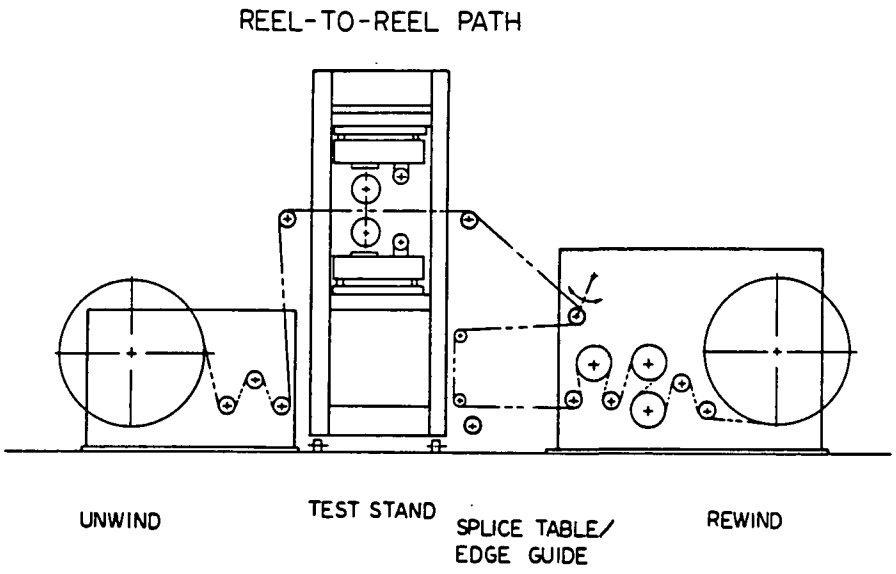


Figure 1.

A portable test stand for mounting either fluid-filled wheels or the IPST-built wheels is being built by the same vendor and will be delivered with the web handling system. This test stand will have cross web positioning, wheel-to-web speed matching, portability, and adjustable nip pressure.

FLUID-FILLED WHEEL TRANSDUCERS

Work continues with modified, commercial, fluid-filled wheels to make out-of-plane ultrasound velocity measurements and caliper measurements for moving paper webs. Here a transducer is mounted to a rigid frame which is encapsulated in a fluid-filled, rubber tire. The paper sample runs in the nip between two such wheels, and the caliper and velocity of ZD ultrasound in the paper are calculated from a comparison of the arrival times of ultrasonic pulses between the two transducers with and without the sample present.

A 386-type computer is being used in the development of our on-line system. One of our principle tasks has been to write the software necessary to support the on-line operation of the fluid-filled wheels. This must first analyze the reference signal pulses obtained without a sample in place. Three discrete pulses must be detected, individually gain controlled, signal averaged, and digitized. These reference signals, along with the oscilloscope delay used in their digitization, are stored for comparison with the sample signals. After the wheels are applied to the web, the three sample pulses are consecutively digitized, averaged, and cross-correlated with the corresponding reference signals. The three time differences between the sample and reference pulses are determined and reported by the computer. Also, the time-of-flight through the sample (FF delay), the caliper (FF caliper), and the ZD ultrasonic velocity (FF velocity) in the sample are calculated from the three time differences and printed out. The software development for the above was complete before the move to Atlanta.

In our earlier evaluation of fluid-filled wheels, we demonstrated that under static conditions it is possible to obtain reasonable values of velocity and caliper using this technique. We were also able to obtained good signal quality in dynamic tests on a moving web using the IPST Web Strainer. It was observed that temperature changes encountered in moving-web operation affect the velocity of sound in the fluid and consequently the transit time and caliper measurements. In order to monitor and correct this, we obtained a thermocouple to mount in one of the wheels. This is part of a digital thermometer that is interfaced to the computer.

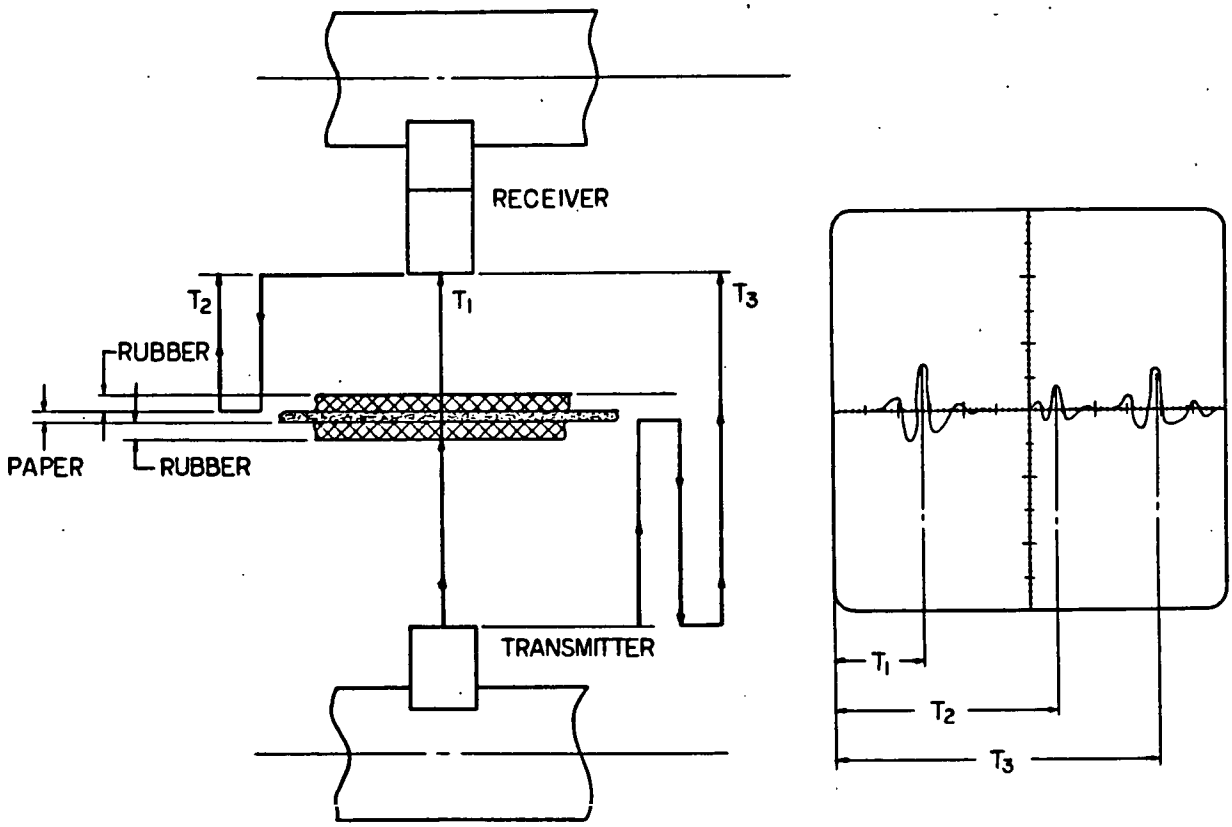


Figure 2.

We were getting lower values for fluid-filled wheel calipers and velocities than with standard laboratory measurements. We believed this to be caused by interference of the signals reflected at the fluid-rubber and rubber-paper interfaces. Experiments with added rubber thickness, indicated that these signals could be separated by using thicker "tires" on the fluid-filled wheels.

We purchased two new fluid-filled wheels from a second vendor. These new wheels are an improvement in three areas: (1) They are easier to use. The time it takes to service and exchange transducers has been reduced significantly. (2) The transducer spacing, which affects the time delay between signals, can be increased by more than 0.5 inch over that possible in the old wheels. This increase in range of adjustment gives us greater latitude in setting the transducer spacing to effectively eliminate interference encountered when one signal begins before another signal ends. (3) The yoke design allows the installation of a thermocouple into the center of the wheel without affecting the seal arrangement of the wheel.

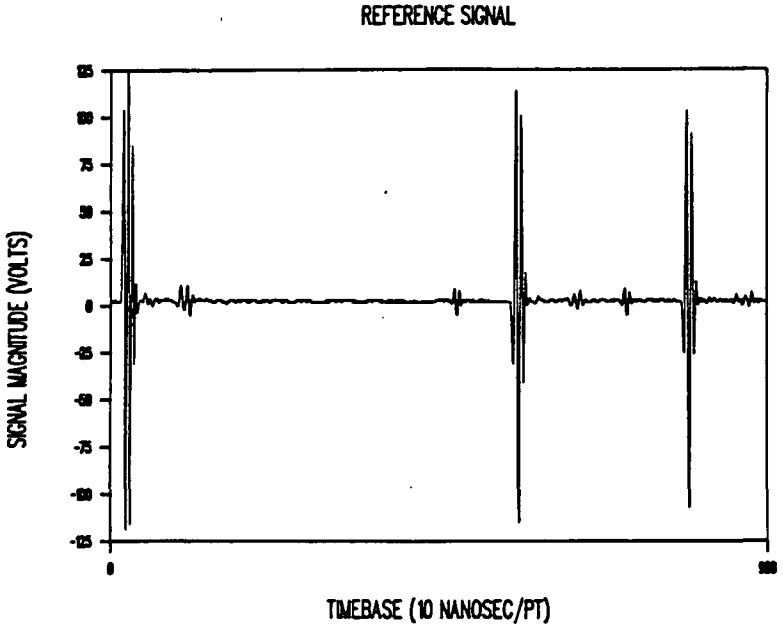
STATUS REPORTS

To The

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

March 21, 1990
Institute of Paper Science and Technology
Atlanta, Georgia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SIGNAL MAGNITUDE (VOLTS)
TIMEBASE (10 NANOSEC/PT)

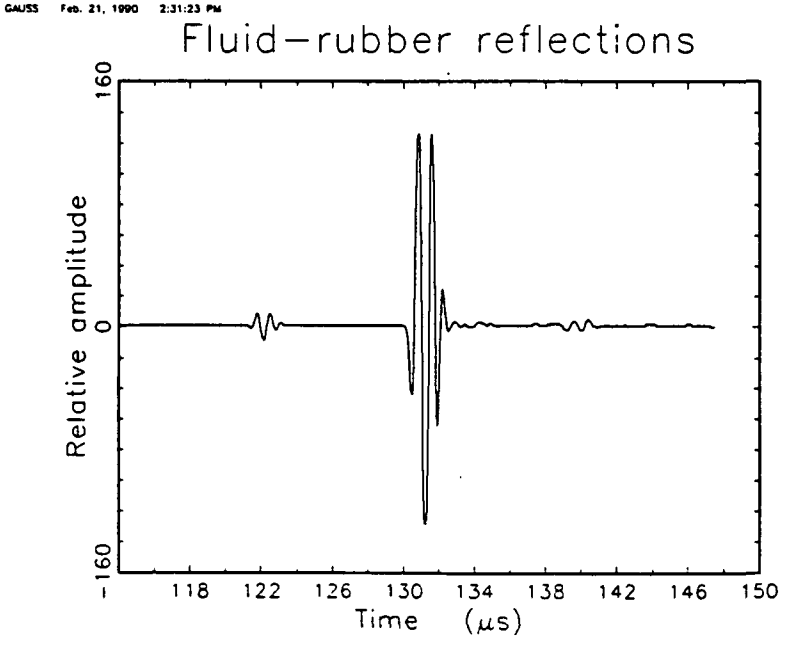

GAUSS Feb. 21, 1990 2:31:23 PM
Fluid–rubber reflections
Relative amplitude
160
0
-160
118
122
126
130
134
138
142
146
150
Time (μs)

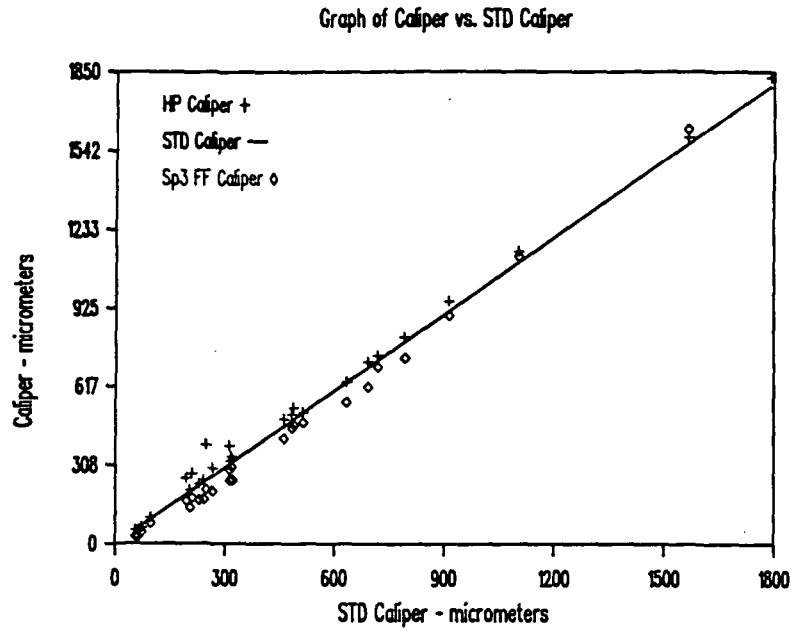
Figure 3.

One problem was observed with the tires supplied with the new wheels. The shape of the signals received, though strong, did not appear as clean as previous signals. Measuring the old tire thickness and new tire thickness revealed that the new tires are thinner than the old. This observation reinforced our conclusion from the added-rubber experiments noted above. Arrangements were made to have thicker tires made for the new wheels.

That thicker tires provide cleaner pulses may be seen in a scope trace of the first reflected signal with no paper sample. The small signals on each side of the first reflected pulse are due to the fluid-rubber interface. If the tires are thin, reflections from the fluid-rubber interfaces overlap and interfere with the rubber-paper interface signal of interest.

With the thicker tires installed, we reexamined the comparison of fluid-filled wheel caliper measurements (FF Caliper) with hard platen caliper (HP Caliper) and with caliper measured with our standard ZD laboratory instrument (STD Caliper). Data was recorded for a series of samples with caliper ranging from 80 to 1800 microns (3 to 70 mils). This data was taken with distance between the wheel axles or transducers fixed by spacers (SP2, SP3, SP4). Changing from SP2 to SP3 decreased the axle and transducer separation by 1/8 inch. Similarly, changing SP3 to SP4 decreased separation by another 1/8 inch.

The data show the expected differences between hard platen and soft platen caliper. We observed variations in the FF Caliper data that required further examination. We found that data recorded immediately after taking a calibration reference appeared to be in better agreement than data for subsequent samples using the same reference. This was attributed to temperature changes in the fluid. The thermocouple for temperature compensation had not as yet been installed in the new wheels. Taking a new reference before each sample measurement improved the caliper comparison results. We also are recording caliper data by an alternate technique that does not involve dependence on a no-sample reference. This is discussed in Project 3467.


Graph of Caliper vs. STD Caliper
HP Caliper +
STD Caliper —
Sp3 FF Caliper ◇
Caliper - micrometers
STD Caliper - micrometers
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308
617
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1542
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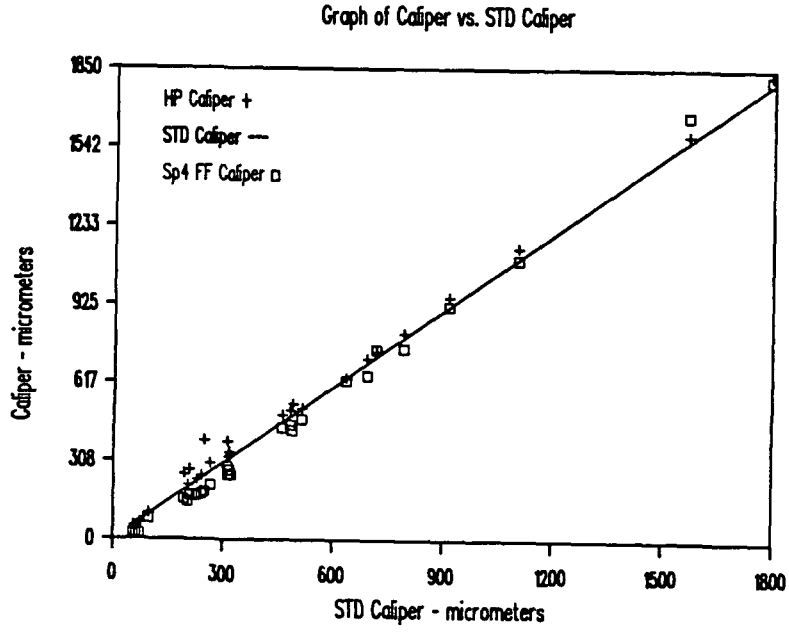
Graph of Caliper vs. STD Caliper
HP Caliper +
STD Caliper —
Sp4 FF Caliper □
Caliper - micrometers
STD Caliper - micrometers
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Figure 4.

Dispersion causes the shape of the ultrasonic pulse received through a sample to differ significantly from the shape of the reference pulse with no sample. This is particularly apparent for thicker samples. This raises a question regarding the most appropriate way to compare the pulses received with and without sample in order to determine the transit time of the pulse in the sample. An examination of this question is presented in Project 3467.

We are reviewing the speed and capacity of our instrumentation. Our present setup has an analog-to-digital data acquisition window of 1024 points. In order to record the three-pulse signal for the fluid-filled wheels at high resolution, it is necessary to record the data at three separate times, once for each pulse location. Although this takes more time, it is adequate for static measurements. However, for moving web operation it would be desireable to capture the three-pulse signal with one measurement at sufficient resolution for accurate transit time measurement.

ELASTOMER FRONT-FACED, PVDF WHEEL-TRANSDUCERS

A second set of wheel-transducers, constructed with piezoelectric polyvinylidene fluoride (PVDF or Kynar) films, were completed before our move to Atlanta. These wheels incorporate harder, more durable polyurethane front-faces, and higher temperature, more sensitive experimental copolymer PVDF film (VF2-VF3 from Pennwalt Corp). In addition, improvements were made in the uniformity of both the piezoelectric element and the front-face.

Yokes for mounting these wheels on the portable test stand being constructed for the fluid-filled wheels were also built. The bottom yoke will be rigid, and the top one will be mounted on a plate attached to a linear bearing. This mounting arrangement will permit the use of a Linear Variable Differential Transformer (LVDT) to measure caliper. The top wheel will be loaded with a spring-dashpot combination to hold it in position with constant force against the paper and bottom wheel.

These wheel-transducers are ready for mounting and testing when the web handling system is available. However, initial check out of the web handling system and evaluation of the integrated ZD and in-plane system on moving webs will be done with the fluid-filled wheels.

ON-LINE BIMORPH TRANSDUCERS

If the use of bimorph transducers for in-plane ultrasonic velocity measurements on-line is to be practical, two problems needed to be solved. The first is to provide a durable contacting surface. A technique has been developed to adhere a metal wear surface to the tip of the bimorph transducer. Several transducers have been modified in this way and tested. Their performance makes us hopeful that this problem is solved.

The other major challenge is to mount the bimorph transducers in the roll housing, so that the results obtained with the three-transducer velocity measurement technique are not sensitive to web tension. A new roll for mounting the bimorph transducers was designed and built before the move to Atlanta. This roll is made of aluminum and is lighter than the original steel roll. It has a groove pattern that should be appropriate for achieving strong, narrow acoustic pulses. It is more amendable to experimentation with different transducer mounting procedures. A custom-manufactured spring mounting fixture has been built to reduce variability in the loading of the transducers to the web and thus reduce tension sensitivity. Provision has been included in the new web handling system for mounting this roll. Evaluation of the bimorph transducers and in-plane measurements on moving webs will continue when installation and start up of the web handling system is completed.

PROCESS, PRODUCT, PROPERTIES RELATIONSHIPS

STATUS REPORT

FOR

PROJECT 3467

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

March 21, 1990

PROJECT TITLE: PROCESS, PROPERTIES, PRODUCT RELATIONSHIPS

Date: 11/1/88

Budget: $175,000

PROJECT STAFF: M. Hall

Period Ends: 6/30/90

Project No.: 3467

PROGRAM GOAL:

Develop relationships between the critical paper and board property parameters and how they are achieved in terms of raw material selection, principles of sheet design, and processing conditions.

CURRENT OBJECTIVE:

Improve our ability to characterize the physical properties of paper and board materials.

Relate measured parameters to end-use performance.

Relate measured parameters to machine and process variables.

PROJECT RATIONALE:

It is important to understand the relationships between process variables, end-use performance, and paper and board properties in order to improve these products or maintain performance within close tolerances while effectively utilizing available raw materials, minimizing energy requirements, and minimizing environmental impacts.

RESULTS TO DATE:

The major activities and results can be divided into three areas: development of instrumentation; the impact of process variable on elastic properties; and the relationships between elastic properties and the end-use performance of paper.

A soft-rubber platen caliper system was developed under this project to provide accurate caliper and density measurements. A microwave technique for determining fiber orientation was also developed.

A major accomplishment of this project has been the development of ultrasonic techniques and instruments which enable us to measure the in-plane and out-of-plane elastic stiffnesses of paper. This equipment has been automated, so that the measurements are carried out under computer control with appropriate calculations made and results printed out in a report format. An automated in-plane ultrasonic tested based on a standard laboratory robot and off-the-shelf electronics has been designed and constructed and the associated software development has been completed. Technology transfer for commercialization is in progress.

The ability to measure the three-dimensional, elastic behavior of paper has led to an extensive study of how process variables effect the elastic responses. To date, process variables examined include wood species, pulping method, yield, refining, jet-to-wire speed ratios, wet pressing, wet stretching, drying restraints, and calendering. Several useful relationships between elastic stiffnesses and these process variables have been identified. The effects of coatings fillers, sheet composition, sheet structure, and environmental factors (temperature and relative humidity) have also been investigated.

The elastic stiffnesses have been shown to be related to a number of parameters now used to predict end-use performance and/or convertibilty. The elastic stiffnesses are fundamental properties of a material, and are good indicators of operating conditions on the paper machine and of final product quality.

In addition to completion to the robot-based tested, recent results include:

A special pulse-echo transducer has been developed for measuring reflections of ultrasonic waves at transducer-paper interfaces.

Techniques have been developed for measuring out-of-plane loss in ZD wave propagation in paper.

Techniques for measuring out-of-plane ultrasonic properties of tissue are being developed and look promising as a possible way to characterize "softness."

A method for using longitudinal polar plots to calculate non-orthotropic elastic constants is in place.

The construction of an instrument (Board Optical Transmission Meter) that measures specific scattering coefficients of heavy paper board has been completed.

PLANNED ACTIVITY FOR THE PERIOD:

Cooperate with the licensee to insure the manufacture of a high-quality, commercial, in-plane robotic ultrasonic tester.

Develop the software to use the pulse-echo transducers to automatically make measurements of loss and reflection coefficients.

Do a fundamental study of out-of-plane loss processes in paper to determine the meaning of our measured loss coefficients.

Develop special PVDF immersion transducers and construct an improved ultrasonic sizing instrument.

Use the measurement of non-orthotropic constants to characterize paper.

Test and apply the Board Optical Transmission Meter (BOTM) for measurements of scattering coefficients of high basis weight papers.

RELATED STUDENT RESEARCH:

B.F. Berger, Ph.D.-1988; B.J. Berger, Ph.D.-1988; J.E. Biasca, Ph.D.-1988; R.R. Willhelm, M.S.-1988; W.E. Myers, M.S.-1989

PROJECT SUMMARY

DATE: March 1, 1990

PROJECT NO: 3467 - PROCESS, PROPERTIES, PRODUCT RELATIONSHIPS

PROJECT STAFF: P. BRODEUR, M. HALL, T. JACKSON

PROGRAM GOAL:

Develop new and improve existing techniques to characterize the physical properties of paper and board and relate measured parameters to end-use performance and to machine and process variables.

PROJECT OBJECTIVES:

- Improve our ability to characterize the physical properties of paper and board materials.

- Relate measured parameters to end-use performance.

- Relate measured parameters to machine and process variables.

SUMMARY OF RESULTS LAST PERIOD: (March 1989 - October 1989)

(1) The in-plane ultrasonic robot tester was programmed to average multiple measurements and record polar data at five degree intervals to carefully characterize a paper sample. A study was made of the trade off between evaluation time and accuracy resulting from decreasing the number of polar directions measured in recording the polar diagrams. The use of thirty degree intervals was selected as a good compromise for determining the polar angle (angle of deviation of maximum stiffness from the machine direction) for a series of paper samples.

SUMMARY OF RESULTS THIS PERIOD: (October 1989 - March 1990)

(1) Ultrasonic transient signals recorded with the ZD fluid-filled wheels experimental setup are found to be dispersive. As a result, the standard analysis based on cross-correlation of two waveforms is unable to provide accurate caliper and transit time measurements.

(2) Two techniques have been proposed to overcome the dispersion effect. While the first attempt involves a limited range cross-correlation to provide less dispersion sensitive measurements (XCHW method), the second approach is based on the dispersion effect analysis in order to apply a correction on cross-correlated waveforms (XCFS method).

(3) A frequency domain analysis of the transient signals has been initiated to understand their dispersive nature. This study is aimed at finding new relationships with paper properties.

(4) A mathematical analysis of the in-plane ultrasonic robot tester lean angle accuracy as a function of the stepping angle (number of polar directions used for measurements) has been done. It is found for a simulated ellipse shape polar diagram that calculated angles close to 0 degree can be off by 15% from expectation (underestimated) if measurements are collected every 30 degrees. The study shows that for a constant number of polar directions, accuracy is improving as the lean angle is increasing. A stepping angle of 15 degrees provides a minimum 1% accuracy for the full span of lean angles.

PROCESS, PROPERTIES, PRODUCT RELATIONSHIPS
Project 3467

INTRODUCTION

As stated in the program goal, the long-term objective of this project emcompasses the study of paper and board physical properties and their relationships with process variables and end-use performance. This is accomplished via existing and yet to be developed new techniques. However, based on the unique capabilities available at the Institute for the non-destructive ultrasonic inspection of paper materials in machine, cross-machine and out-of-plane directions, the current research activities are focused around ultrasonic related applications. It is aimed in the near future that other topics will be investigated as well.

Caliper and ZD sound velocity measurement techniques for the fluid-filled wheels setup.

The development of a new on-line apparatus combining in-plane and ZD ultrasonic instruments to measure elastic properties of paper in a paper mill is the primary objective of project 3332/3613. As such, it is not our intention to duplicate the work in this project. Our main interest is to develop a reliable signal analysis technique to handle data sets collected with the Z-direction two fluid-filled wheels instrument. As it stands now, this instrument provides static simultaneous measurements of the caliper, Δd, transit time, Δt, and out-of-plane sound velocity, v_p.

The fluid-filled wheels setup using rubber tires has been introduced in previous PAC reports. As a reminder, the transmitting and receiving transducers are clamped inside the wheels in a face-to-face arrangement. Water is used as the coupling medium between transducer's surfaces and tire's inner edges. Unavoidable discontinuities at rubber-water interfaces and rubber-rubber (without paper sheet) or rubber-paper interfaces produce time delayed transient signals with respect to the main transmitted pulse signal. Relative arrival time measurements of some of these signals provide the basis for caliper and transit time measurements. As the same measuring technique is used in both cases, accurate caliper measurements should allow indirect calibration of the transit time and hence, sound velocity.

Assuming that rubber-water interfaces dependent signals do not interfere with rubber-rubber or rubber-paper/paper-rubber dependent signals, the transit times between the transmitter and receiver for waveforms of interest are described as follow and in agreement with Fig. 1:

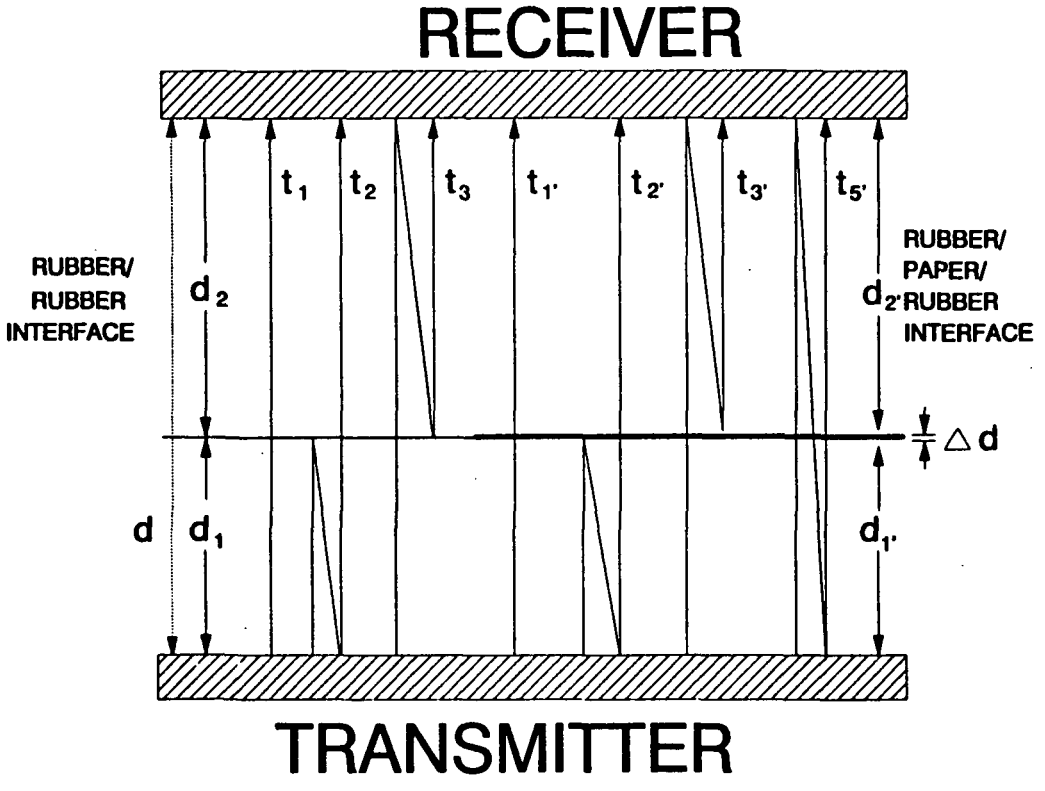

Figure 1.

Reference signals (captured without paper):

$$t_1 = d/v_f \quad (1)$$

$$t_2 = (d + 2\,d_1)/v_f \quad (2)$$

$$t_3 = (d + 2\,d_2)/v_f \quad (3)$$

Paper signals:

$$t_{1'} = \Delta t + (d - \Delta d)/v_f \quad (4)$$

$$t_{2'} = \Delta t + (d - 2d_{1'} - \Delta d)/v_f \quad (5)$$

$$t_{3'} = \Delta t + (d - 2d_{2'} - \Delta d)/v_f \quad (6)$$

$$t_{5'} = 3\, t_{1'} \quad (7)$$

where $d = d_1 + d_2 = d_{1'} + d_{2'} + \Delta d$, and v_f is the apparent velocity of sound in the fluid (combined water and rubber contributions). The so-called fifth signal corresponds to the "second occurence" of the transmitted signal. In agreement with the optimized distances $d_1 = 0.4d$ and $d_2 = 0.6d$, this signal is delayed with respect to the "second occurence" of signal #2 (fourth signal not shown in Fig. 1). Assuming a known separation distance d, the apparent fluid velocity at constant temperature can be determined from eq. 1, 2 and 3:

$$v_f = (2d)/(\Delta t_{21} + \Delta t_{31}) \quad (8)$$

where $\Delta t_{yx} = t_y - t_x$.

The caliper can be deduced from 1, 2, 3, 4, 5 and 6:

$$\Delta d = v_f(\Delta t_{1'1} - (\Delta t_{2'2} + \Delta t_{3'3})/2) \quad (9)$$

or from 4, 5 and 6:

$$\Delta d = d - v_f(\Delta t_{2'1'} + \Delta t_{3'1'})/2 \quad (10)$$

The transit time is computed by using 1, 4 and 9,

$$\Delta t = \Delta t_{1'1} + \Delta d/v_f \quad (11)$$

or 4, 7 and 10:

$$\Delta t = \Delta t_{5'1'} + (\Delta d - d)/v_f \qquad (12)$$

The sound velocity in paper is:

$$v_p = \Delta d/\Delta t \qquad (13)$$

As it can be seen, there are more than one way to compute Δd or Δt. While the caliper calculated from (9) requires 3 reference signals and 3 paper signals for a total of 6, the caliper calculated using (10) requires only 3 paper signals. Due to its reference-free measurements, the second option is more attractive. The transit time calculated by either (11) or (12) requires only 2 signals, once the caliper is known. Again, (12) appears more attractive due to its reference-free feature. There are two reasons to prefer reference-free measurements: they are independant of the d_1/d_2 ratio (see Fig. 1) and they are less dispersion sensitive. In practice, the d_1/d_2 ratio is found to fluctuate because the tires are not perfect discs. A full set of equations taking into account temperature dependency is available.

Fig. 2 shows a full length recording of the first 3 reference signals (denoted #1, #2 and #3). As one should expect from impedance mismatch at the rubber-paper interfaces, #2 and #3 are inverted with respect to #1. Approximate arrival times for #2 and #3 agree with distances d_1 and d_2. The weak signals are due to rubber-water coupling discontinuities; in any case, they do not interfere with signals of interest. Fig. 3, 4 and 5 display typical recordings for paper samples J (LVDT soft-platten caliper = 96.4 μm), Y (792.4 μm) and Z (1592.6 μm) respectively. A comparative analysis of the reference and paper signals leads to the following observations:

a) Paper signals are more and more delayed as the caliper is increased. This effect is the basis for caliper and transit time measurements.

b) They are more and more dispersed as the caliper is increased (especially obvious for sample Z).

c) The signal-to-noise ratio is degrading for thicker samples. This is of course related to sound absorption in paper.

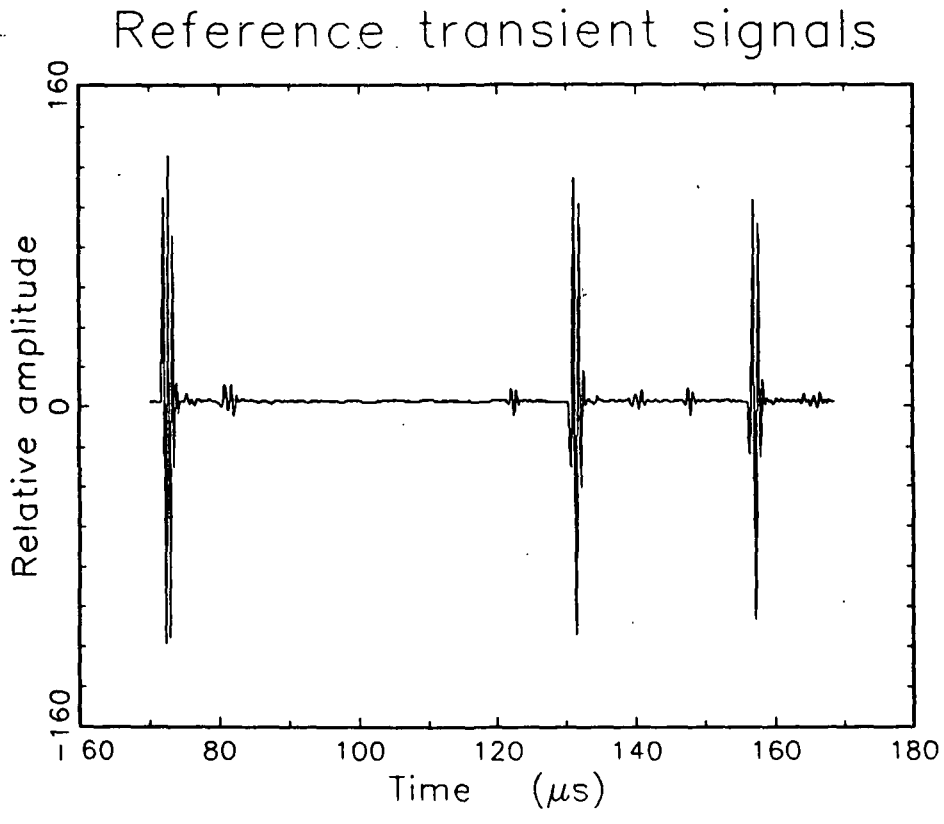

Figure 2.

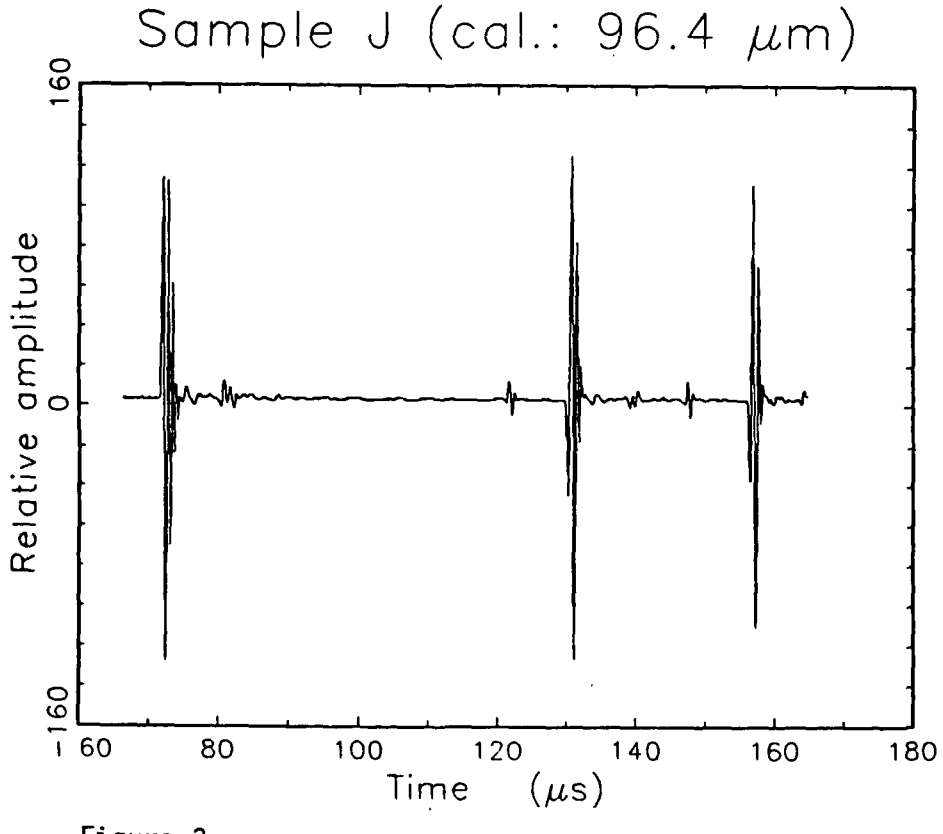

Figure 3.

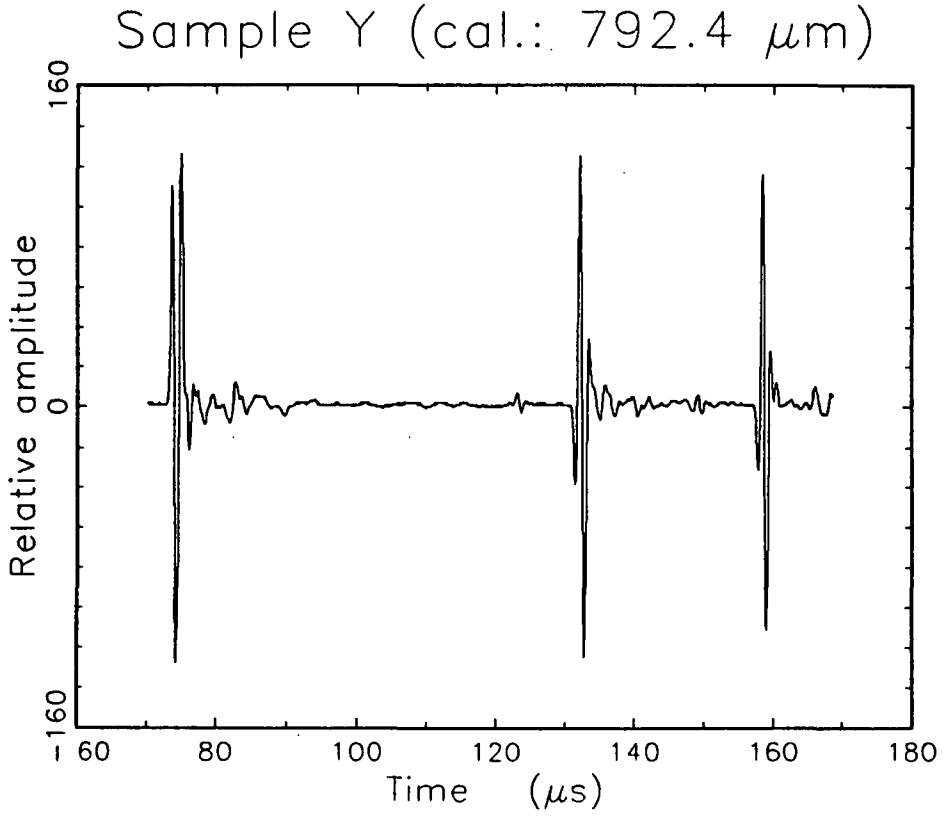

Figure 4.

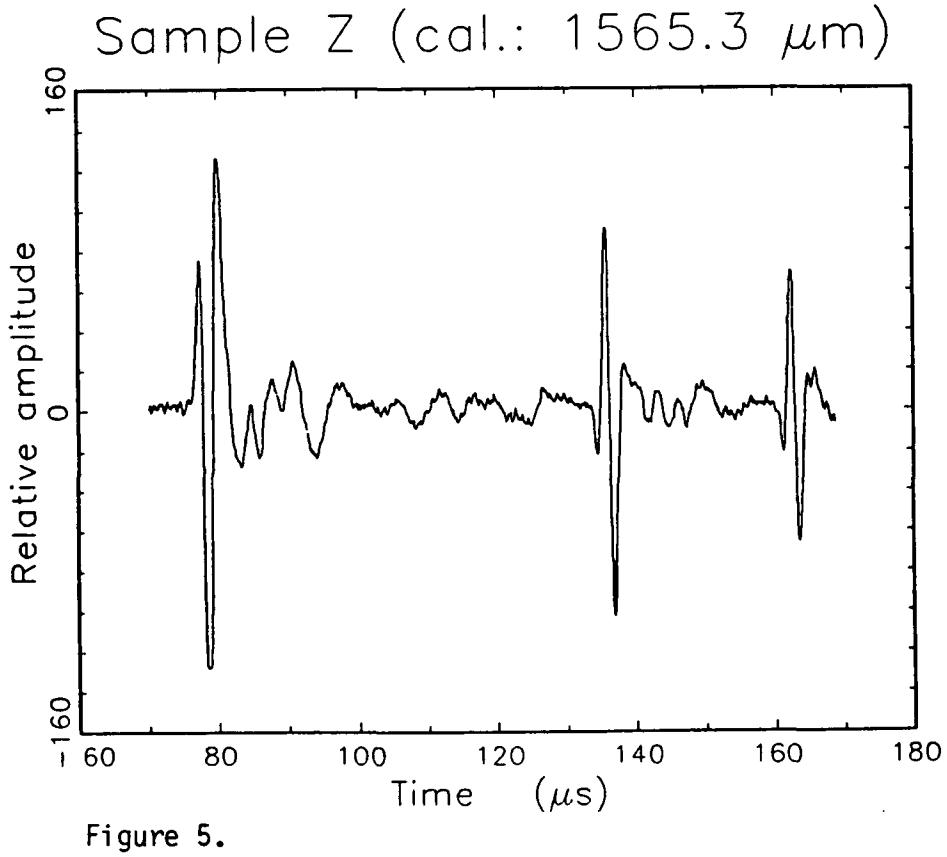

Figure 5.

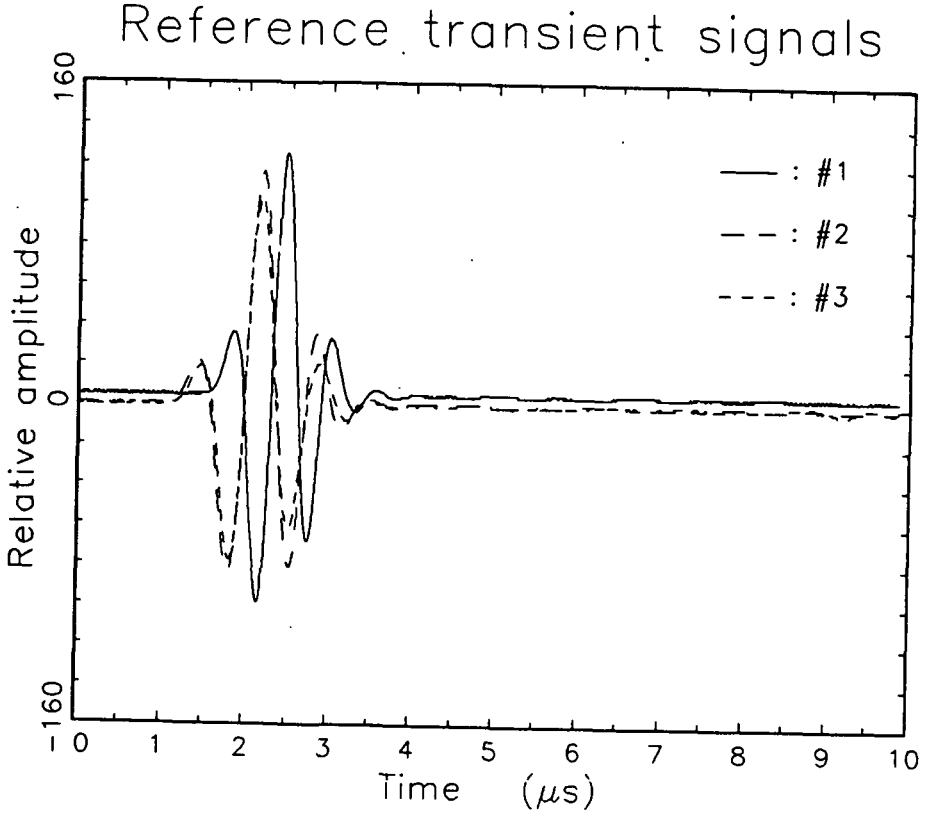

Figure 6.

Due to hardware memory limitations, recordings shown in Fig. 2 to 5 are collected at a reduced sampling rate to get an overall picture. In order to obtain meaningful raw data, each signal must be further recorded separately on a shorter time scale, but at an optimized sampling rate. This process allows signal averaging to improve the signal-to-noise ratio. While this time consuming data acquisition procedure has no immediate consequence for static measurements, this is unlikely to be successful in a dynamic environment. Fig. 6, 7, 8 and 9 show expanded views of signals from reference and paper samples J, Y and Z respectively. Please note that waveforms #2 and #3 are inverted for the purpose of analysis. The caliper dispersion effect is clearly visible. Although not a universal feature, waveforms from the same paper sample can be dispersive as well. These observations lead to the following question: can routine cross-correlation calculations be efficient if waveforms to be cross-correlated are dispersive? The answer is no, and caliper and transit time measurements may be way off.

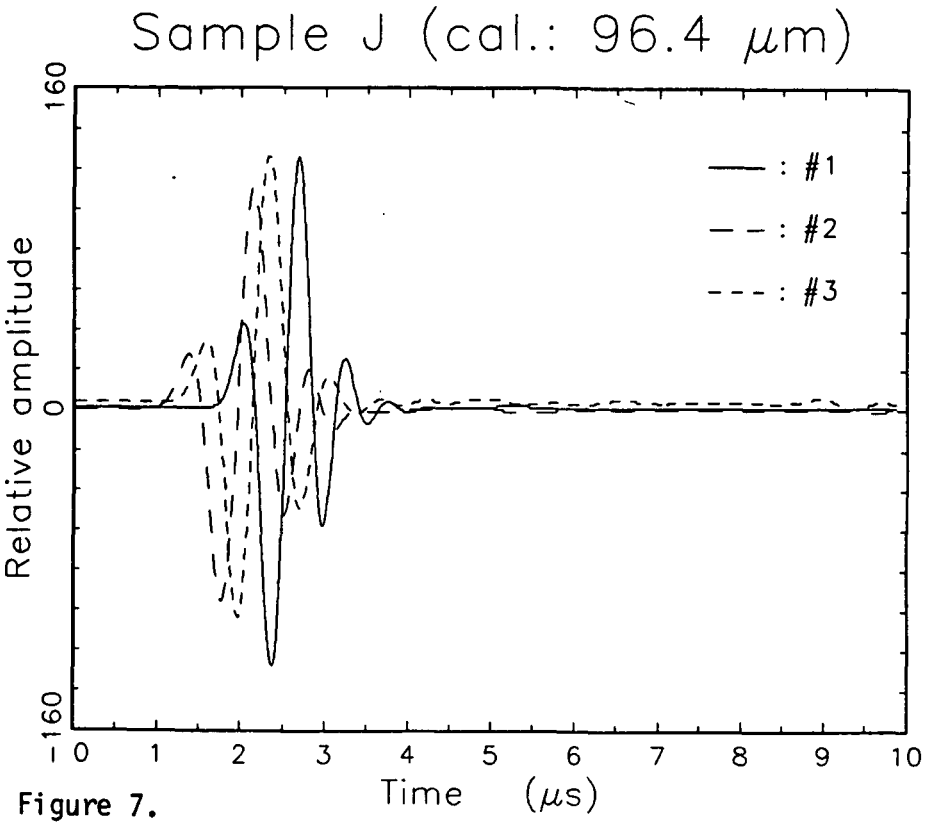

Figure 7.

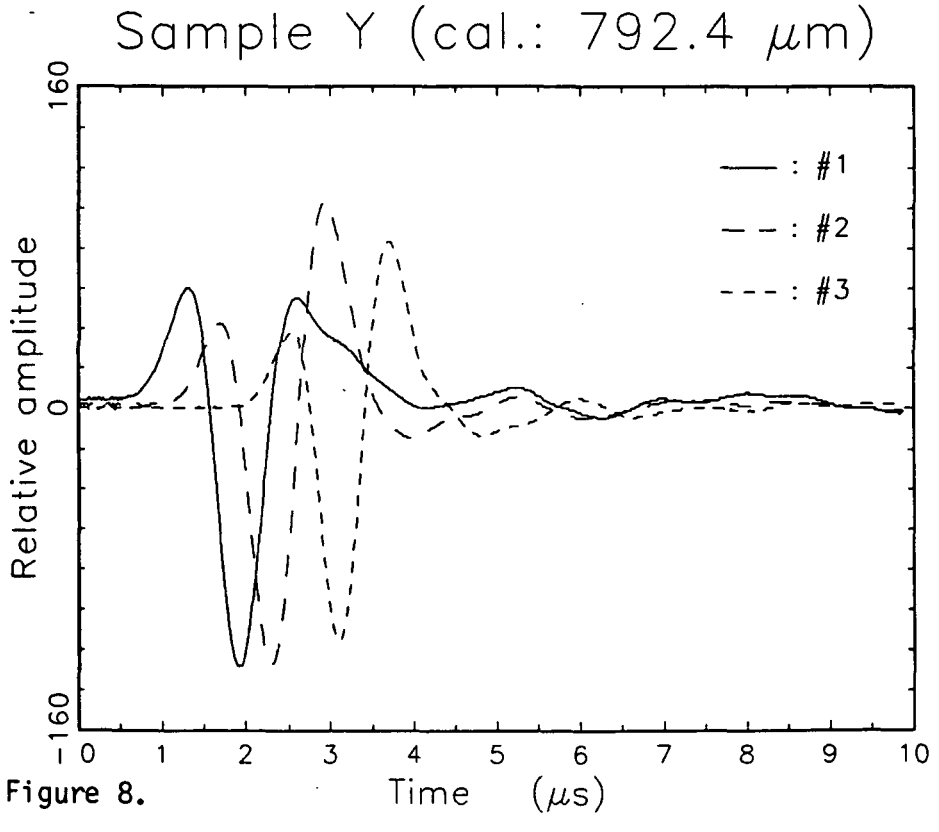

Figure 8.

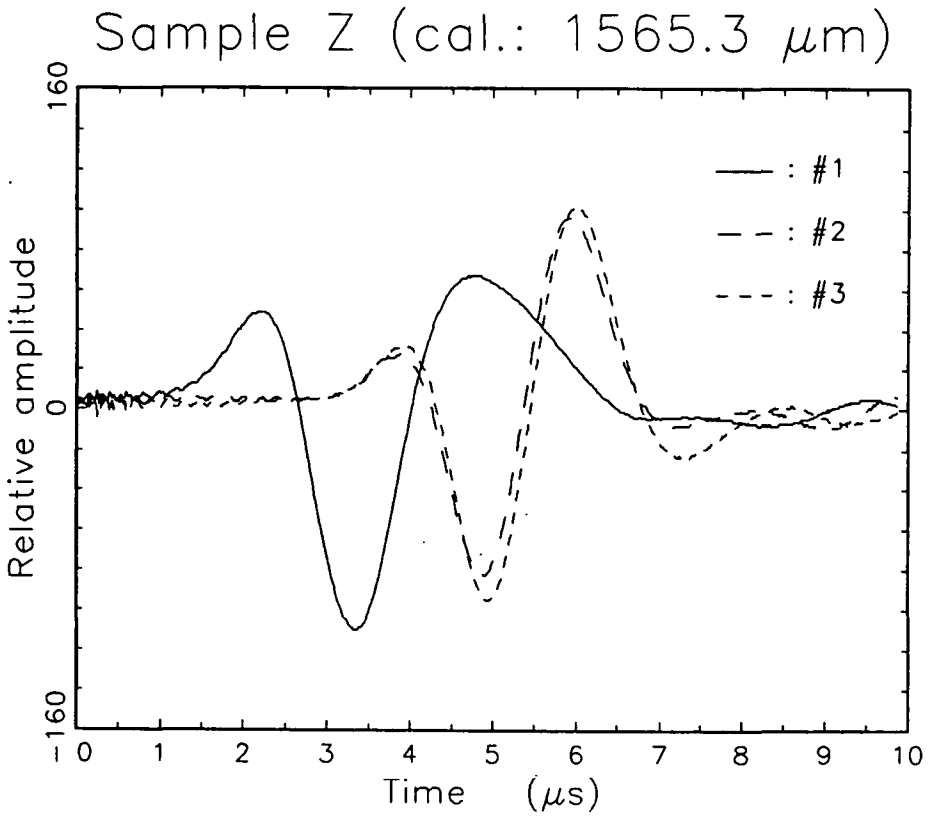

Figure 9.

Two methods have been proposed and partially developed to overcome the dispersion effect. Both are using cross-correlation as the core measurement technique. They are now described:

XCHW Method

This method is windowing measurements around the first half-cycle peak of each signal in order to limit the dispersion effect. The vicinity of this peak is first located. Then, the sampling rate is further increased in a stepwise manner so as to maximize the number of data points on a very narrow time interval centered around the peak. In this process, the time delay up to this interval is updated. The amplitude level is also maximized. Once this pre-treatment is completed for two signals, cross-correlation is applied. Signal analysis examples using the XCHW method are displayed in Fig. 10 and 11. In both cases, peaks are perfectly aligned and the precision is solely limited by the sampling rate.

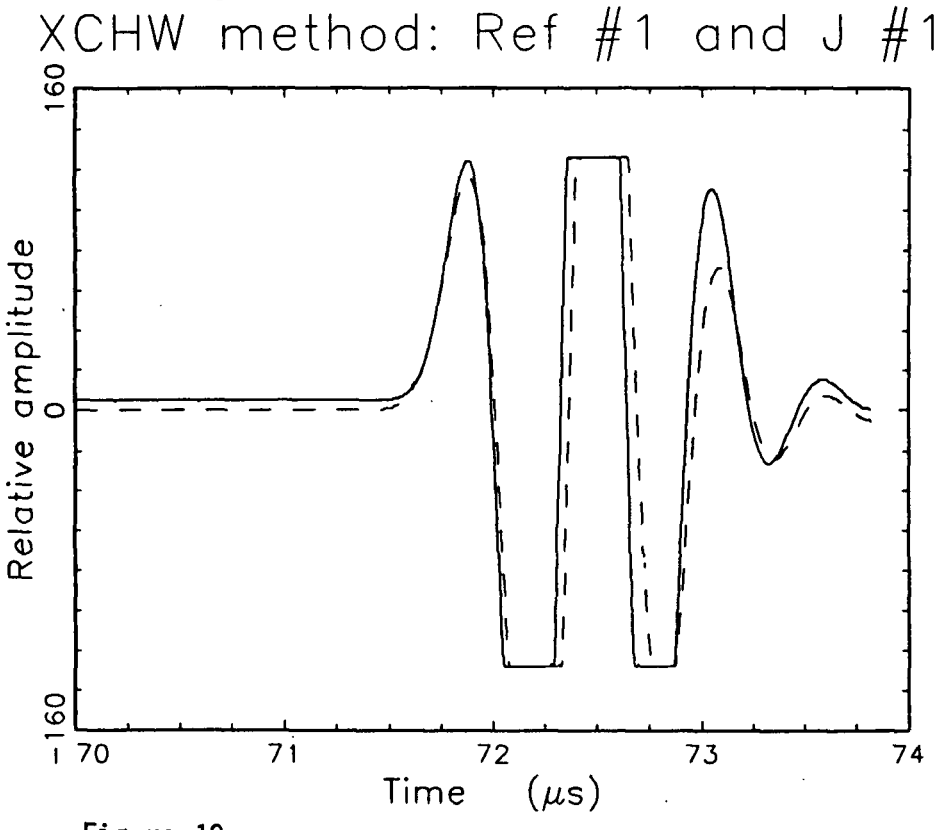

Figure 10.

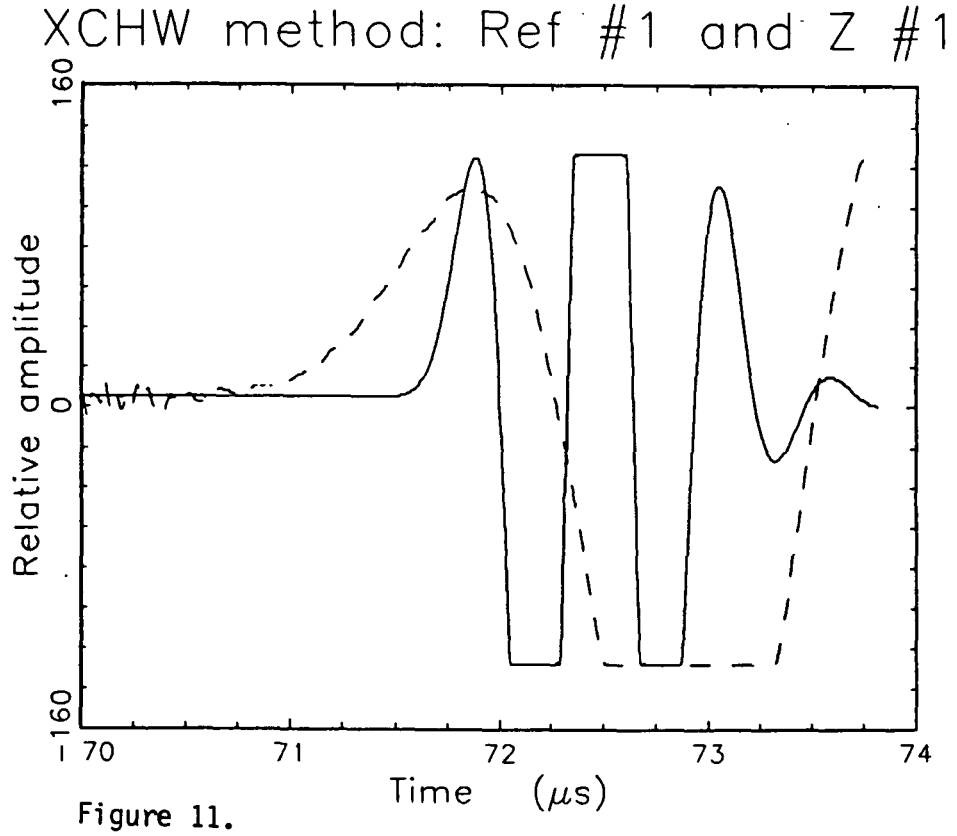

Figure 11.

As efficient as it is to locate peaks, this method has a drawback in its current implementation. Assuming that arrival times for dispersive waveforms are best described by their leading edge (neglecting the surface roughness of paper), delay times will be overestimated. While this problem has limited consequences for caliper measurements (Eq. 9 or 10) because offsets in time delays cancel each other, this is not the case for transit time measurements (Eq. 11 or 12). In the extreme situation shown in Fig. 11, the delay time is overestimated by approximately 1 microsecond. This clearly demonstrates the difficulties encountered in dealing with the dispersion effect. An overestimated transit time in paper leads to an underestimated sound velocity. Preliminary calculations indicate that v_p can be underestimated by as much as 15 to 20% for large caliper samples. Design of an algorithm to locate the leading edge of the signals using the XCHW method is in progress.

XCFS Method

Realizing that dynamic measurements will be more subject to noise than off-line measurements (especially if signal averaging cannot be done), it may be preferable to do signal analysis over the entire length of the signals, i.e. to use as much information as possible from the waveforms. As already pointed out, the dispersion phenomenon excludes the sole use of cross-correlation to determine the proper delay time between two transient waveforms. In order to remedy to this problem, it was proposed to analyze the frequency content of the signals and to define a "dispersion related" correction that could be applied to cross-correlation measurements.

Using fast Fourier transform analysis (FFT), waveforms were converted to their frequency domain spectra. The magnitude and phase as a function of frequency for the reference and sample H signals are shown in Fig. 12, 13, 14 and 15. Some interesting observations can be denoted. As seen in Fig. 12, the transmitted reference signal (#1) peak frequency is approximately 1.6 MHz; this is the largest observed frequency with the current experimental setup. #2 and #3 peak frequencies are down to 1.3 MHz. A close look at the reference phase spectra (Fig. 13) indicates a slight non-linearity of the slopes. This is due to a slight waveform dispersion (frequency dependent velocity). Peak frequencies for sample H are down to approximately 0.4 MHz for all 3 signals (Fig. 14). The corresponding phase spectra (Fig.15) are linear. In that particular case, waveforms are dispersion-free.

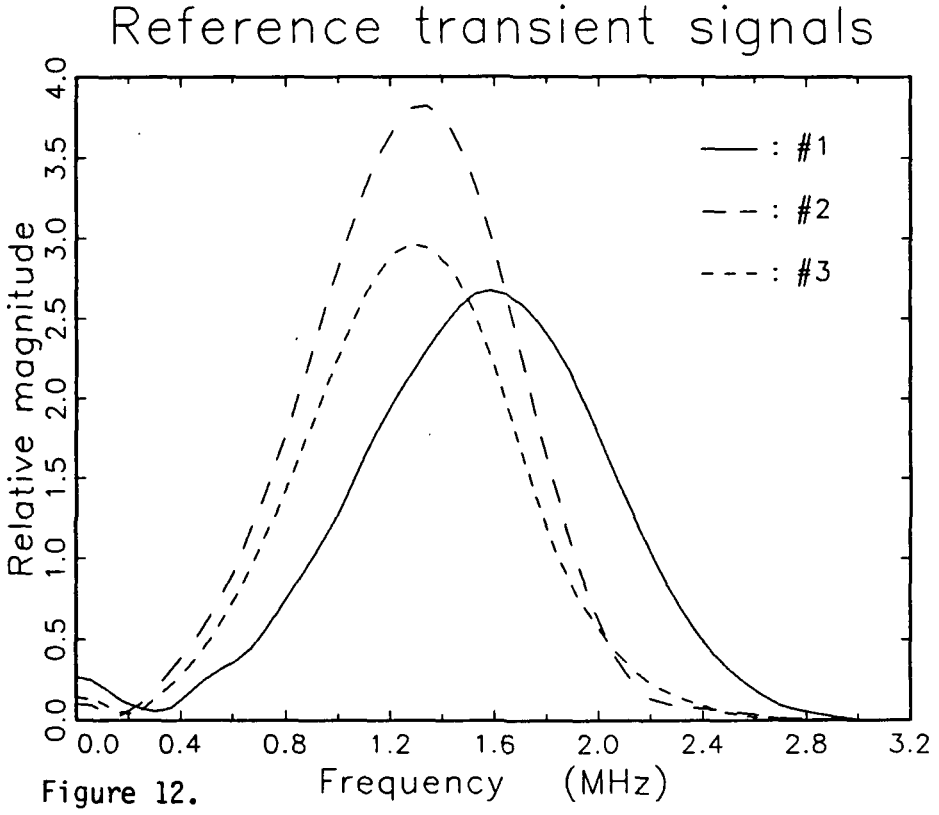

Figure 12.

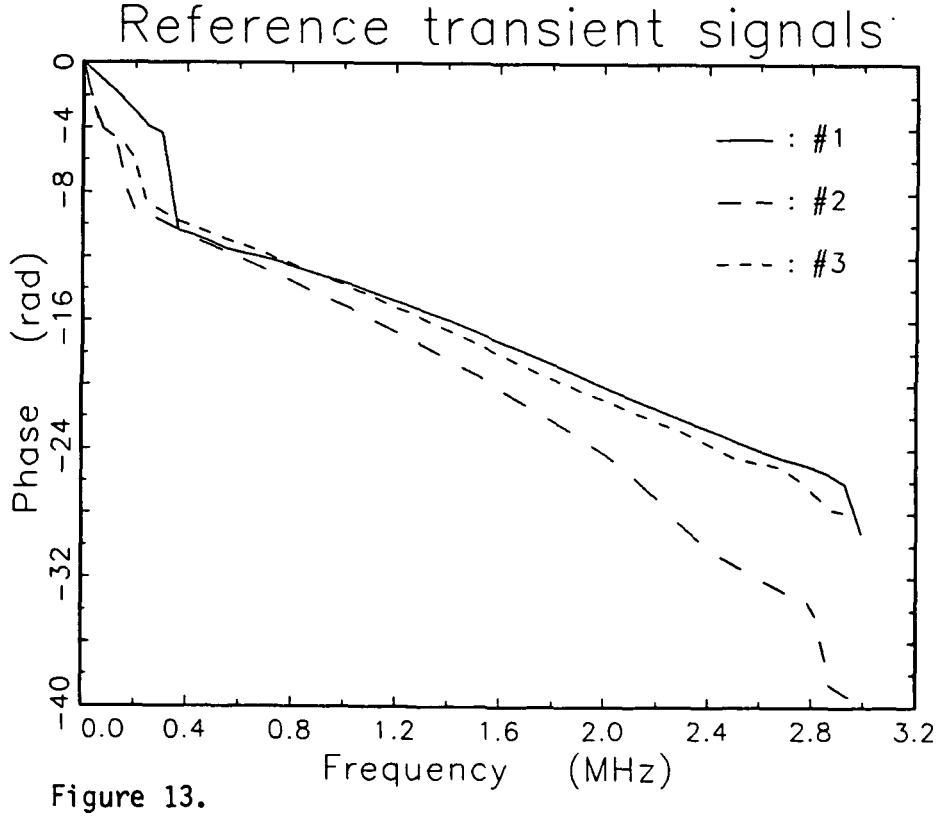

Figure 13.

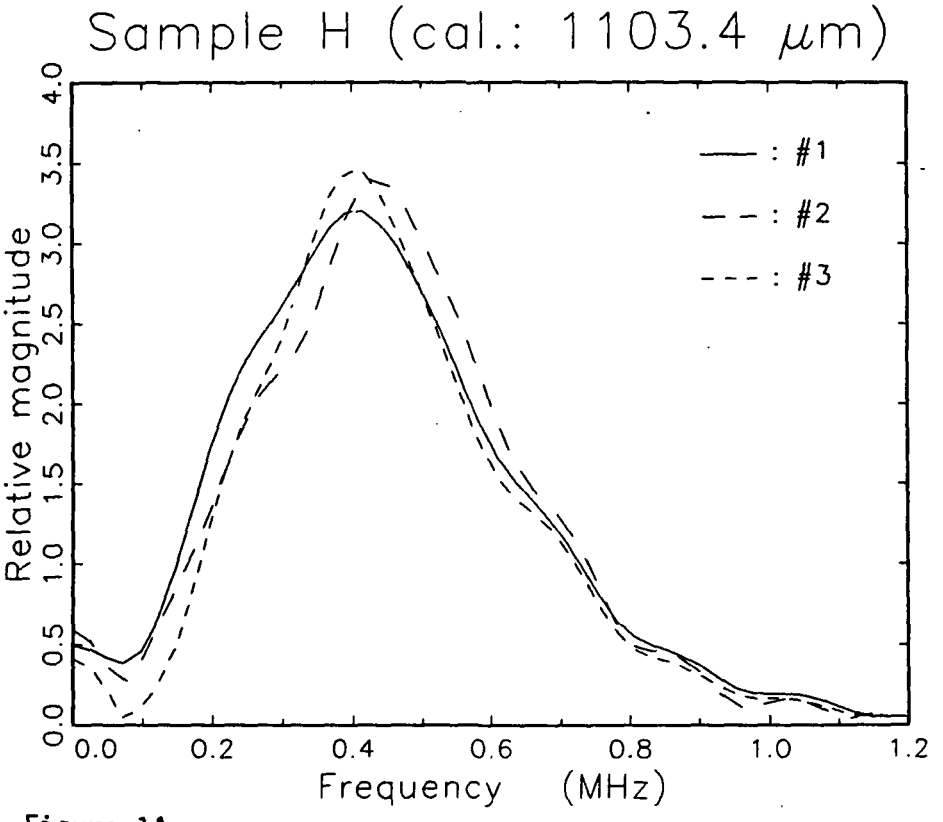

Figure 14.

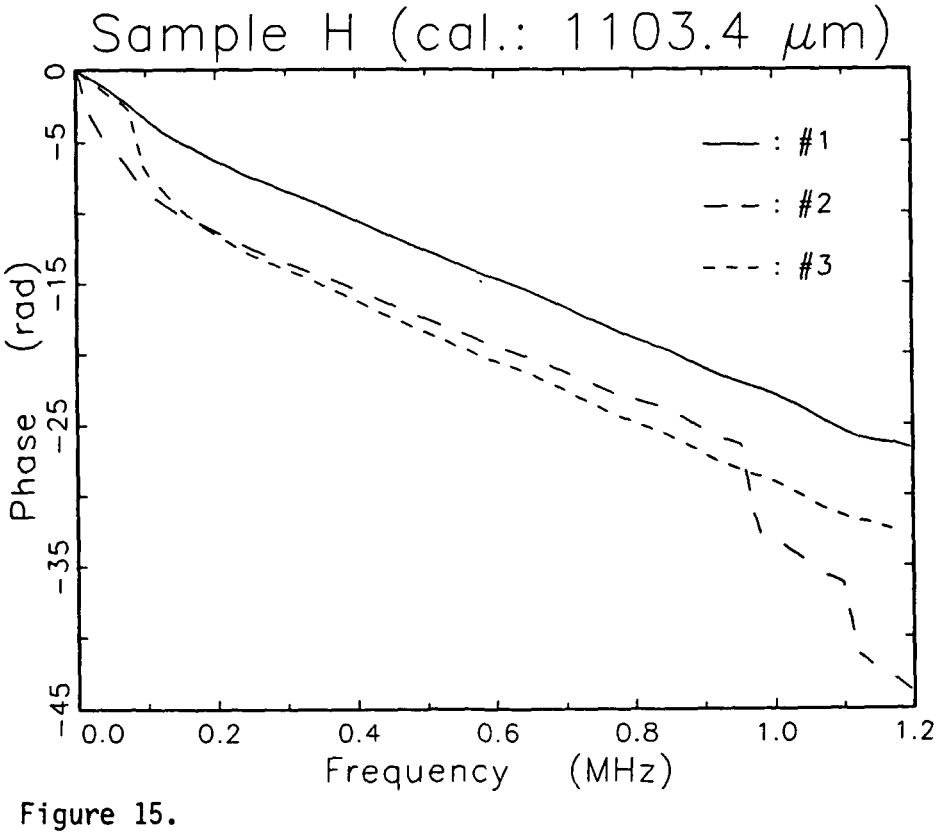

Figure 15.

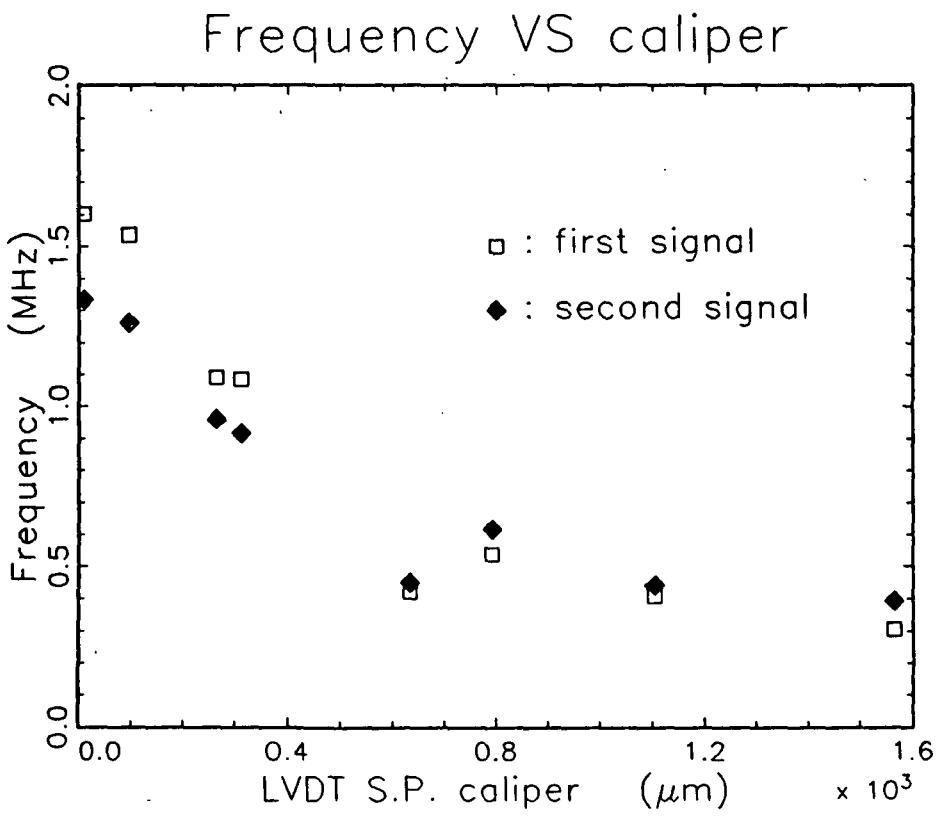

Figure 16.

The shift toward lower frequencies for sample H waveforms with respect to the reference frequencies suggests relationships between dispersion and paper properties. In fact, this trend was investigated as a function of caliper. Preliminary results are summarized in Fig. 16 for signals #1 and #2 (frequencies for #2 and #3 are found to be nearly identical). This graph confirms the low frequency shift as the caliper is increased. Relationships are not linear in both cases and it is interesting to note that at large caliper, #2 frequencies are higher than #1 ones. Hence, there is a caliper value for which no frequency change is observed between the transmitted and reflected waveforms (sample H caliper is actually very close to this value). At least one set of data points (cal.: 633.4 μm) does not match the general relationships, indicating that dispersion is not strictly related to caliper. One last word concerns the measured frequency values for the reference signals at zero caliper. According to the expected curve trends at low frequency, these frequencies should be higher than they are. That might be due to a non-negligible air gap when no paper sheet is present between the tires. Since sound velocities in air and in paper (Z-direction) are of the same

order, it looks as if the instrument was sensing a fictive thin paper caliper. No satisfactory explanation has been found to predict the range of these frequencies.

The difference in peak frequencies between two signals was calculated and such a measurement formed the basis for the dispersion correction. Several cross-correlation schemes were studied in order to provide dispersion-free results before applying the dispersion correction. None have been fully successful so far. Nevertheless, one can find a general idea of the technique in Fig. 17. Waveforms are first cross-correlated and the cross-correlation function maximum predicting the one-cycle zero-crossing points best correlation is selected. A one-cycle frequency shift is then applied. This two-steps treatment produces a close match between the waveform leading edges and one should expect a fairly good accuracy of the delay time.

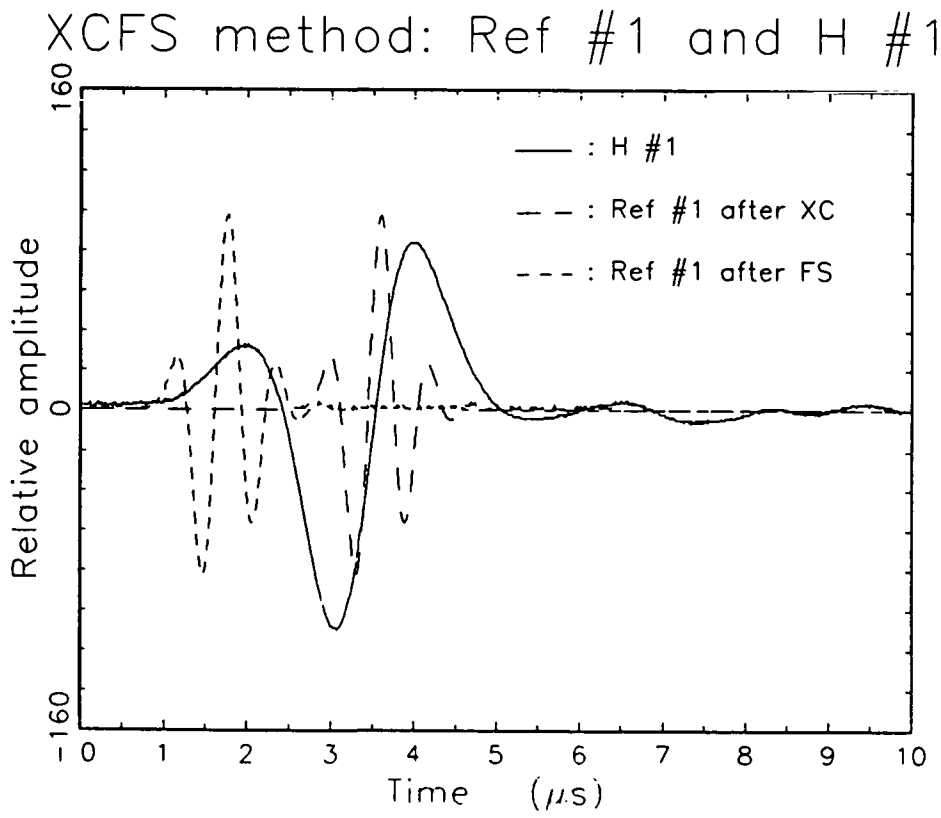

Figure 17.

In-plane lean angle accuracy

A simple mathematical analysis of the in-plane ultrasonic robot tester lean angle accuracy as a function of the number of polar directions was conducted. Typical polar diagrams were simulated by a perfect ellipse. The major and minor radii were 1 and 0.33 respectively. For a given lean angle, polar plot points were computed for every selected stepping angle (for example 30 degrees). Then, the lean angle was computed using the maximum moment of inertia algorithm. The difference in both predicted and calculated angles was computed and divided by the predicted angle. Results are presented in Fig. 18. At expected 0 degree lean angle, the calculated angle is underestimated by 15% at 30 degree stepping angle. At constant stepping angle, the accuracy is improving as the lean angle is increasing. This analysis suggests that a 15 degree stepping angle should provide measurements with at least 1% accuracy.

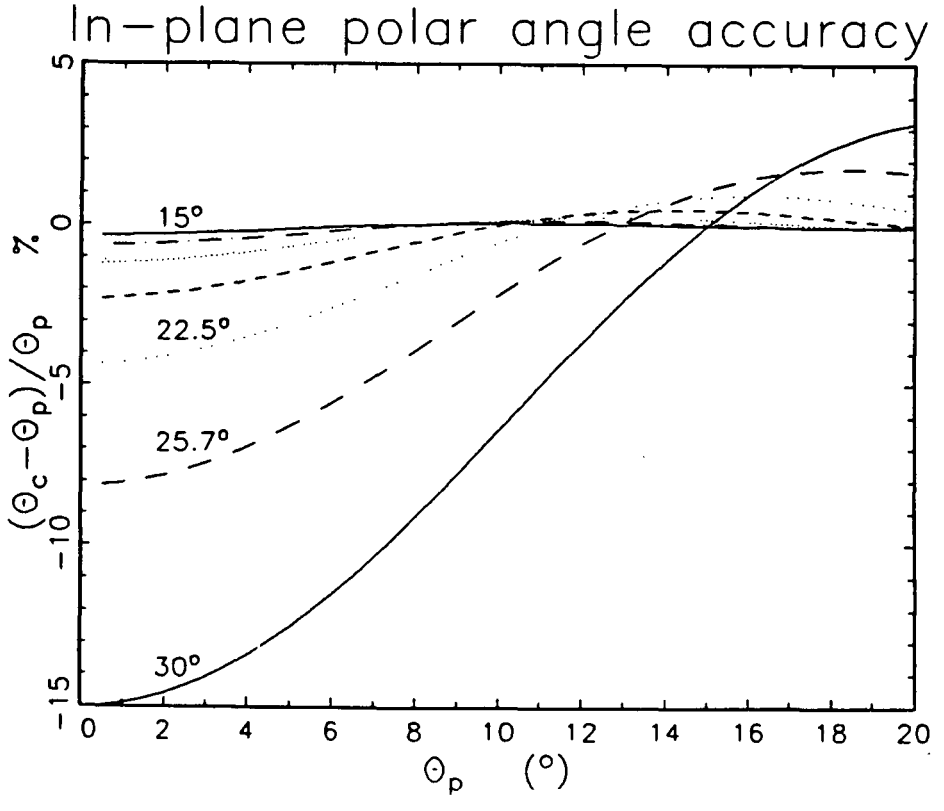

Figure 18.

FUTURE WORK

It is planned to:

a) Continue the development of a cross-correlation based technique to properly analyse recordings from the fluid-filled wheels instrument. Alternative methods like the phase-slope method and the correlation of two waveforms using FFT will be examined.

b) Continue to perform frequency domain analysis of transient waveforms collected with the fluid-filled wheels setup and investigate relationships with paper properties.

c) Investigate amplitude attenuation of fluid-filled wheels setup transients and determine possible relationships with paper properties.

d) Improve the microwave attenuation laboratory instrument to allow polar diagram measurements. Design an experiment to compare in-plane ultrasonic and microwave polar diagram measurements.

e) Complete a theoretical/numerical analysis of acoustic radiation pressure effects on fluid suspended fibers.

f) Explore the possible use of acoustic radiation pressure effects on fluid suspended fibers as a mean to control fiber orientation during the formation process.

g) Explore the development of a research project aimed at studying changes in elastic properties of paper due to plasma etching.

STRENGTH IMPROVEMENT AND FAILURE MECHANISMS

STATUS REPORT

FOR

PROJECT 3469

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

March 21, 1990

PROJECT SUMMARY

DATE: March 1, 1990

PROJECT: Strength Improvement and Failure Mechanisms

PROJECT STAFF: J. Waterhouse

PROGRAM GOAL:

Identify critical parameters which describe converting and end-use performance and promote improvements in cost/performance ratios.

PROJECT OBJECTIVE:

Establish practical methods for enhancing strength properties (especially compressive strength) during paper manufacture and to evaluate deformation behavior as it relates to sheet composition and structure.

SUMMARY OF RESULTS LAST PERIOD: (October 1989 - March 1990)

(1) Improvement of mass density measurement system and related software.

(2) Incorporation of fast fourier transform and contour maps into software for formation measurements.

(3) Measurements of combined stress have been made at one level of refining and wet pressing.

(4) A presentation on out-of-plane combined stress measurements was made at the 1989 ASCE/ASME Mechanics Conference at the University of California at La Jolla.

SUMMARY OF RESULTS THIS PERIOD: (October 1989 - March 1990)

(1) The beta source, promethium 147, has been re-installed in the IPST Formation Tester and measurements of mass-density made on selected samples.

(2) The dependence of a number of physical properties including out-of-plane elastic properties on formation has been examined.

PROJECT TITLE: STRENGTH IMPROVEMENT AND FAILURE MECHANISMS

PROJECT STAFF: J. Waterhouse

Date: 3/5/90
Budget: $110,000
Period Ends: 6/30/90
Project No.: 3469

PROGRAM GOAL:

Identify critical parameters which describe converting and end-use performance and promote improvements in cost/performance ratios.

CURRENT OBJECTIVE:

To evaluate deformation behavior and its relationship to sheet composition and structure, and to establish practical methods for enhancing strength properties.

PROJECT RATIONALE:

Deformation and strength properties are important in predicting end use performance. An improved understanding of failure mechanisms and ways to improve certain strength properties are important to nearly all grades. The recognized importance of compressive strength in linerboard and corrugated medium to box performance provides impetus for research in this area. The approach is to meet the objectives through new papermaking and converting strategies.

RESULTS TO DATE:

We have shown that compressive strength of paper is highly related to a product of in-plane and out-of-plane elastic stiffnesses. The relationship holds for commercial and experimental sheets made under a variety of conditions. This development suggests it will be possible to monitor compressive strength in the mill using ultrasonic techniques.

Compressive strength is enhanced by high densification, which increases bonding, and high fiber axial compressive stiffness. Thus compressive strength increases with refining and wet pressing. Within a practical range, higher CD compressive strength can be achieved by decreased fiber

orientation, loose draws, and/or increased CD restraint during drying. Where limitations to increased refining and wet pressing exist, low levels of polymer addition could be used as a viable means to improve compressive strength. The effect of pulp type and additives on the stiffness compressive strength correlation has been investigated. A technique involving small wood coupons and mini handsheets has been developed to measure the compressive strength potential of wood fibers.

We have developed a torsion mode technique for measuring the out-of-plane shear stress-strain behavior, and studied the effect of ZD shear straining on compressive strength. Internal stress variations have been determined in the thickness direction together with the variation of in-plane and out-of-plane properties. Measurements of the relative losses in elastic and strength properties due to supercalendering have been made. A procedure for determining the residual stress distribution in paperboard has been developed using a layer removal method.

The improvement of compressive strength and reduction of forming losses has been investigated using synthetic binder and fiber addition as a model systems. The concept of activating a synthetic binder system to reduce forming loss during corrugating has been demonstrated.

We have made hardware and software improvements to the IPST Formation Tester. Software development includes scan line and image maps. A preliminary study of base paper coating interaction on formation and other properties has also been made. In conjunction with a formation study for API the formation characteristics of newsprint, tissue, offset bond, and corrugating medium formation samples have been measured using the IPST Formation Tester.

An out-of-plane biaxial device for measuring the deformation behavior of paper and board when subjected to combined stresses has been designed and fabricated.

PLANNED ACTIVITY FOR THE PERIOD:

Formation - Continue hardware and software development for relating formation to converting and end-use performance, including the use of textural and pattern recognition techniques. Develop strategies for measuring the formation of unbleached board.

Combined Stresses - Determine the effect of papermaking variables on the failure envelope of board when subjected to combined out-of-plane stresses. Explore techniques for measuring the strain behavior under the action of combined out-of-plane stresses.

Internal Stresses - Determine how z-direction structure, composition, and residual stresses affect curl behavior, especially off axis curl.

RELATED STUDENT RESEARCH:

T.W. Bither, Ph.D. - 1990; A. Gao, M.S. - 1990, R. Ebert, M.S. - 1990

PROJECT SUMMARY

PROJECT: Strength Improvement and Failure Mechanisms

PROJECT STAFF: J. Waterhouse

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An out-of-plane biaxial device for measuring the deformation behavior of paper and board when subjected to combined stresses has been designed and fabricated.

PLANNED ACTIVITY FOR THE PERIOD:

Formation - Continue hardware and software development for relating formation to converting and end-use performance, including the use of textural and pattern recognition techniques. Develop strategies for measuring the formation of unbleached board.

Combined Stresses - Determine the effect of papermaking variables on the failure envelope of board when subjected to combined out-of-plane stresses. Explore

techniques for measuring the strain behavior under the action of combined out-of-plane stresses.

Internal Stresses - Determine how z-direction structure, composition, and residual stresses affect curl behavior, especially off axis curl.

RELATED STUDENT RESEARCH:

T.W. Bither, Ph.D. - 1990; A. Gao, M.S. - 1990, R. Ebert, M.S. - 1990

STRENGTH IMPROVEMENT AND FAILURE MECHANISMS

Project 3469

INTRODUCTION

This project is comprised of four distinct but interconnected project areas namely: Compressive Strength Improvement, Formation, Combined Stresses, and Internal Stresses in Paper and Board. Following PAC committee recommendations, the interest and focus of our work has more recently been concentrated in the area of Formation. Our efforts this quarter have been mainly devoted to re-establishing our capability for making formation measurements, and only recently have we been able to re-install our beta source in the formation tester. Nevertheless we have been able to make both optical and mass density measurements on a number of furnishes as well as begin to examine the relationship of formation to end use properties.

In the area of compressive strength improvement, we have in recent work, examined the possibility of reducing forming losses by synthetic binder and fiber addition, with the idea of using the hot corrugating process to activate the binder system. A paper entitled "Effect of Synthetic Fiber and Binder Addition on the Strength Losses Associated with Corrugating Medium" has been accepted for presentation at the forthcoming 1990 Spring Meeting of the Materials Research Society to be held in San Francisco April 18-20, 1990.

1. COMPRESSIVE STRENGTH IMPROVEMENT

No significant new developments have occurred in this area since our last report. Work is now in progress to produce a wood grain report detailing our efforts to develop methods and strategies for the improvement of compressive strength. These are summarized in Table 1.

TABLE 1

Methods for the Improvement of Compressive Strength

RAW MATERIALS - Evaluation of Fiber Potential using wood veneers and mini handsheets, High Yield Hardwood Pulps, Inter and intra fiber reinforcement with natural and synthetic polymers

PAPERMAKING PROCESS - Refining, Fiber Orientation, Forming Consistency, High levels of Wet Pressing together with Restrained Drying, Wet Press Felt Type.

It is proposed that future work in this area might be concerned with the effects of recycling on the fiber's compressive strength potential.

FORMATION

The IPST Formation tester is shown in Figure 1. This instrument has the capability of making light transmission and reflectance measurements in the wavelength range of 400-700 nm. Beta particle absorption measurements are also made using a promethium 147 source. The aperture for light transmission and beta particle absorption measurements is 1mm x 1mm.

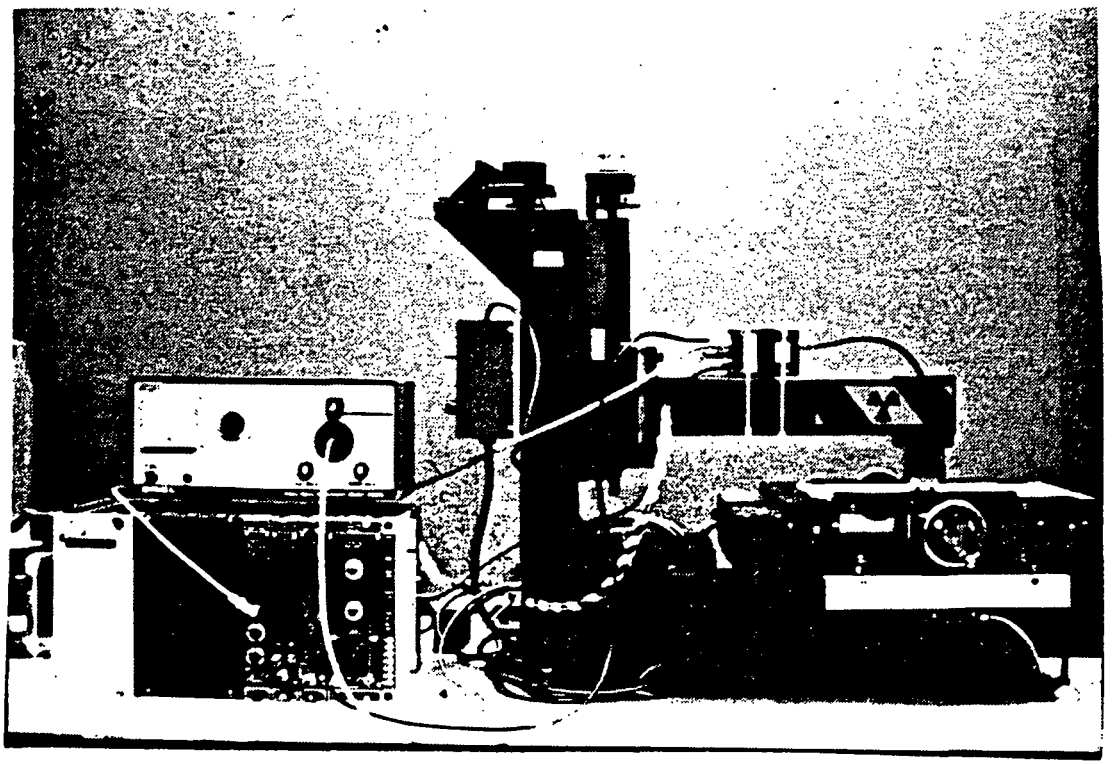

Figure 1.

Work during this last period has been mainly devoted to re-establishing our capabilities for making both optical and mass density measurements. We were recently able to re-install our beta source. We also hope we have now managed to eliminate noise and other transient effects with which we have been plagued.

Light transmission and beta absorption measurements have been made on selected newsprint, tissue and corrugating medium formation samples. We have also begun to examine the influence of formation on a number of paper properties.

Due to the stochastic nature of beta emitters there will be an inherent variability in the number of beta counts even in the absence of a sample.

This means that to be consistent we must change the time for beta particle counting when the average grammage of the sample is changed. Typical beta particle count variability is given below for an air gap setting of 20 mils and amplifier gain of 128.5:

TABLE 2
INHERENT VARIABILITY IN BETA PARTICLE COUNTING

Counting Time secs.	Average Beta Counts	Coeff. of Variation %
2	44988	0.48
5	108877	0.24

The decay of beta particle transmission is to a good first approximation exponential in form. The variation of the natural logarithm of transmitted beta particle counts with grammage for various substrates is shown in Figure 2. The substrates are a polyethylene film, tissue and newsprint

formation samples. We note that the correlation coefficient $r^2 = 0.995$, and differences in substrate composition appear to have a negligible effect.

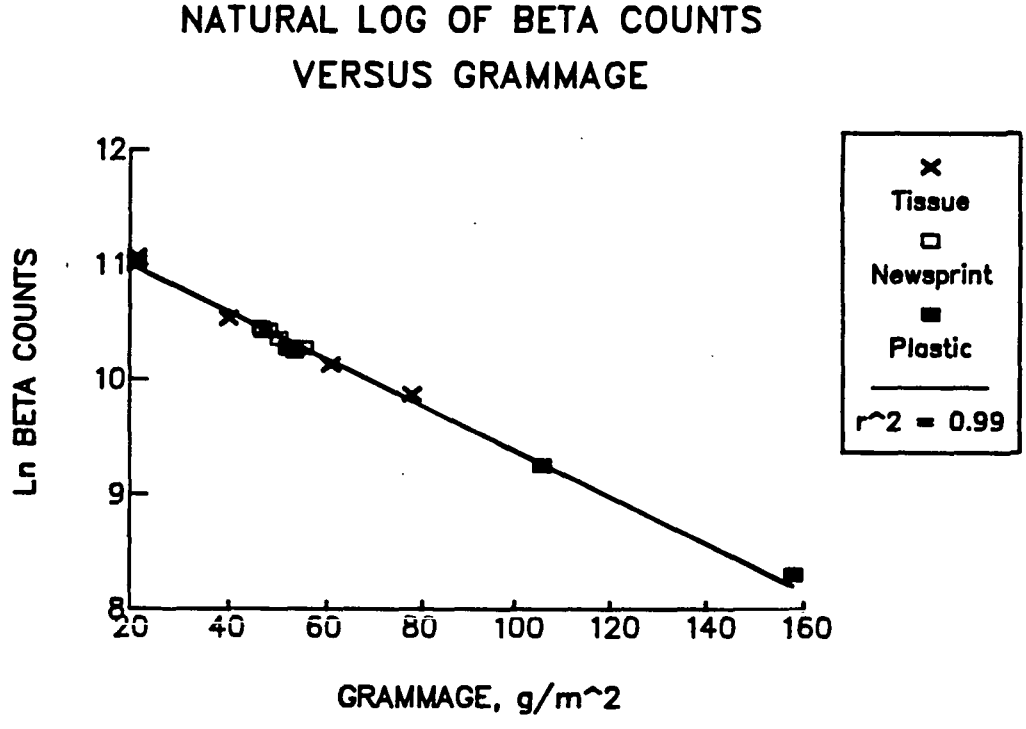

Figure 2.

The variation of the natural logarithm of transmitted beta particle counts with grammage for tissue samples made on the Formette Dynamique is shown in Figure 3 and a very good fit of the data is noted. The variation of the coefficient of variation (% c.v.) of both beta and light transmission with grammage for the tissue sheets is shown in Figure 4. We see that there is a significant drop in % c.v. for the light transmission measurements with increasing grammage whereas % c.v. for beta transmission shows an increase with grammage i.e light transmission indicates that formation is improving with an increase in grammage, while beta transmission indicates the converse may be true. However since the beta measurements were made at a constant counting time of two seconds, it is believed that the increase shown may be incorrect.

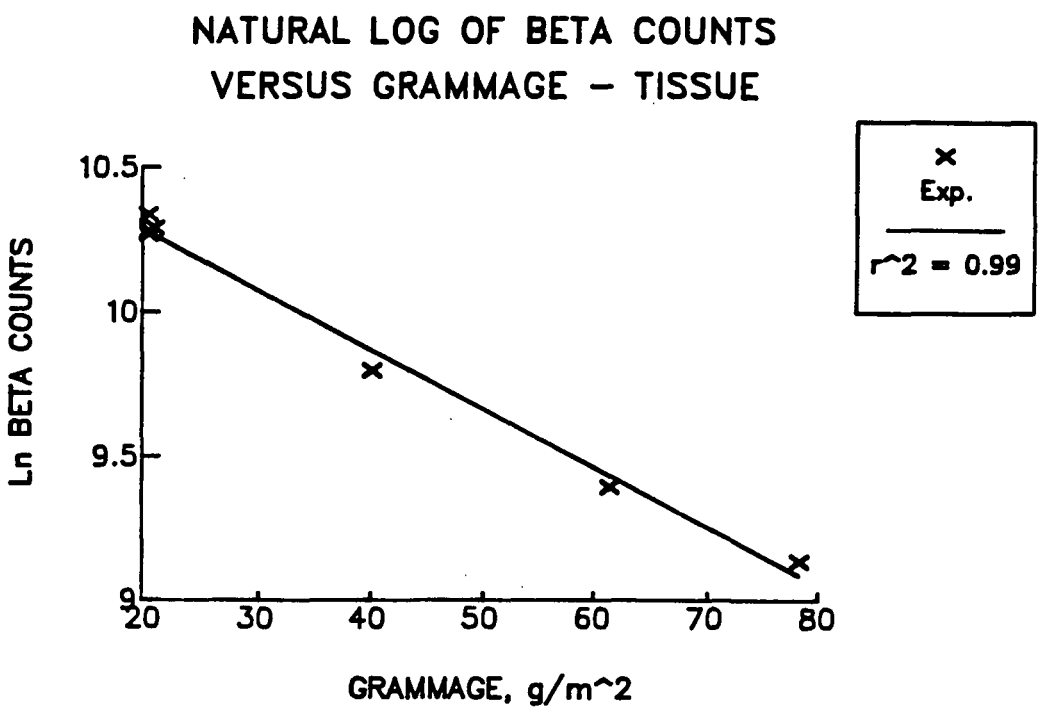

Figure 3.

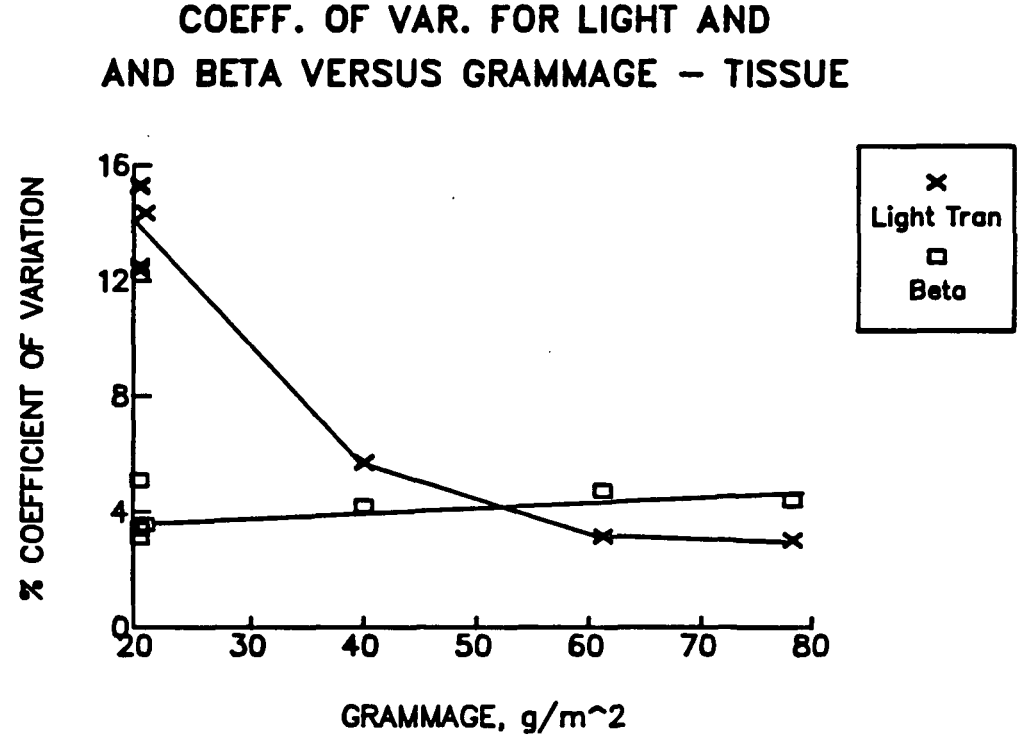

Figure 4.

Handsheets made on the Formette Dynamique generally have excellent formation when judged casually in transmitted light and therefore one would not expect very much variation in mass density. However, more careful subjective measurements (i.e. using a pair comparison technique) can reveal small but significant differences in the visual uniformity of Formette handsheets. We have proposed in a previous progress report that the reciprocal of the coefficient of variation of transmitted light might be used as a measure of visual uniformity % 1/c.v. The variation of % 1/c.v. (both light transmission and beta) with visual ranking for the Formette tissue handsheets are shown in Figure 5.

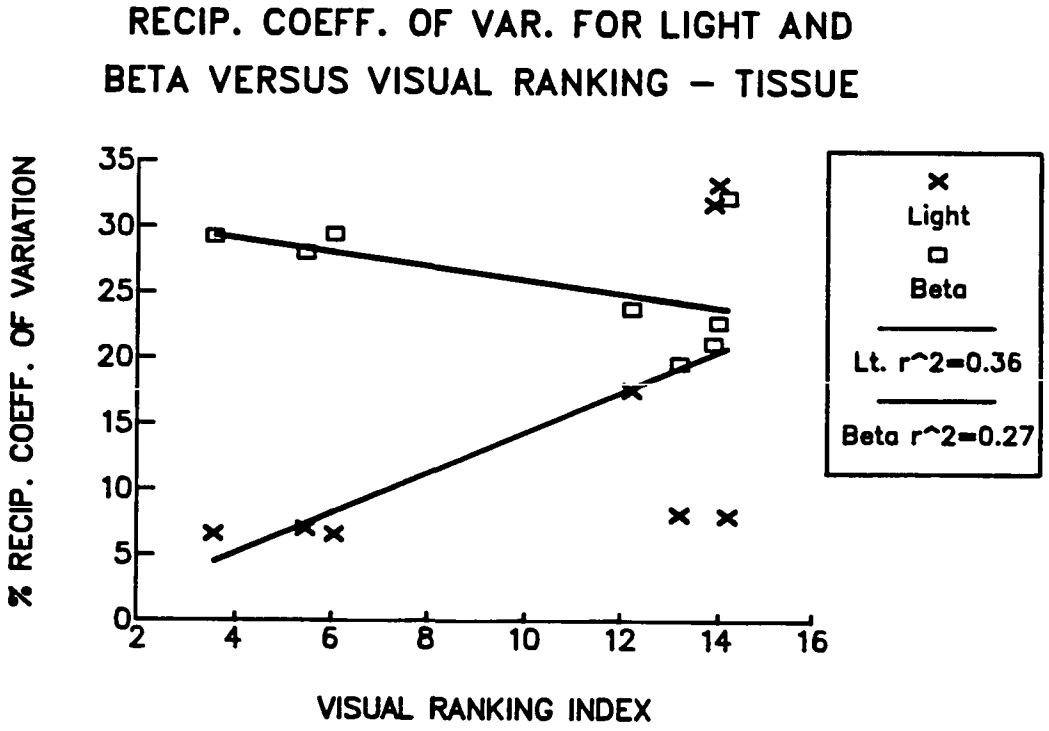

Figure 5.

Although the light transmission data does not yield a very good correlation ($r^2 = 0.36$) there is an increase in measured formation index % 1/c.v. with increase in visual ranking (the higher the visual ranking index the better the formation is judged to be). By comparison the beta results indicate a higher level of uniformity, and possibly a slight decrease with increasing visual uniformity. This is a case where mass density measurements may be misleading when used as an indication of visual uniformity. This has long been a controversial point with respect to formation measurements.

The variation of the natural logarithm of beta counts with grammage for a series of production made and Formette and Noble and Woods handsheet newsprint samples is shown in Figure 6. In this series a beta counting time of five seconds was used, and a very good correlation $r^2 = 0.97$ is obtained.

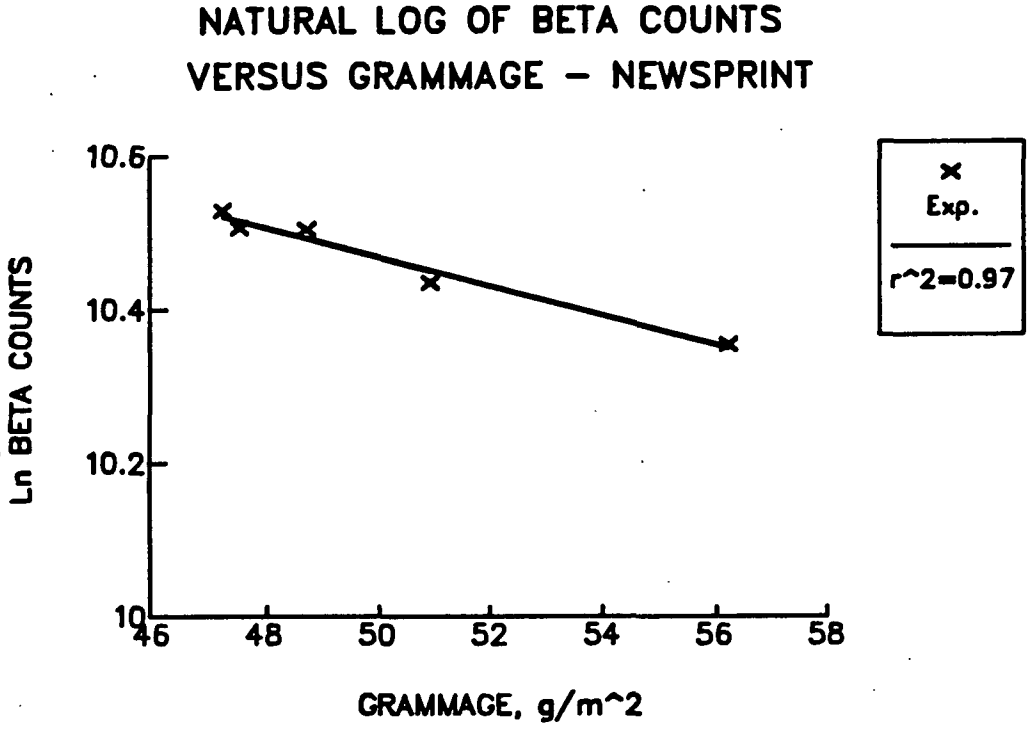

Figure 6.

The variation in transmitted light versus the variation in beta transmission is shown in Figure 7 and an excellent correlation is in evidence $r^2 = 0.99$. The transmitted and beta reciprocal coefficients of variation versus visual ranking are also plotted in Figure 8. In this case both indexes of formation show a very good correlation with visual ranking.

It should be emphasized that the range of formation for the newsprint samples is considerably greater than for the tissue samples discussed earlier.

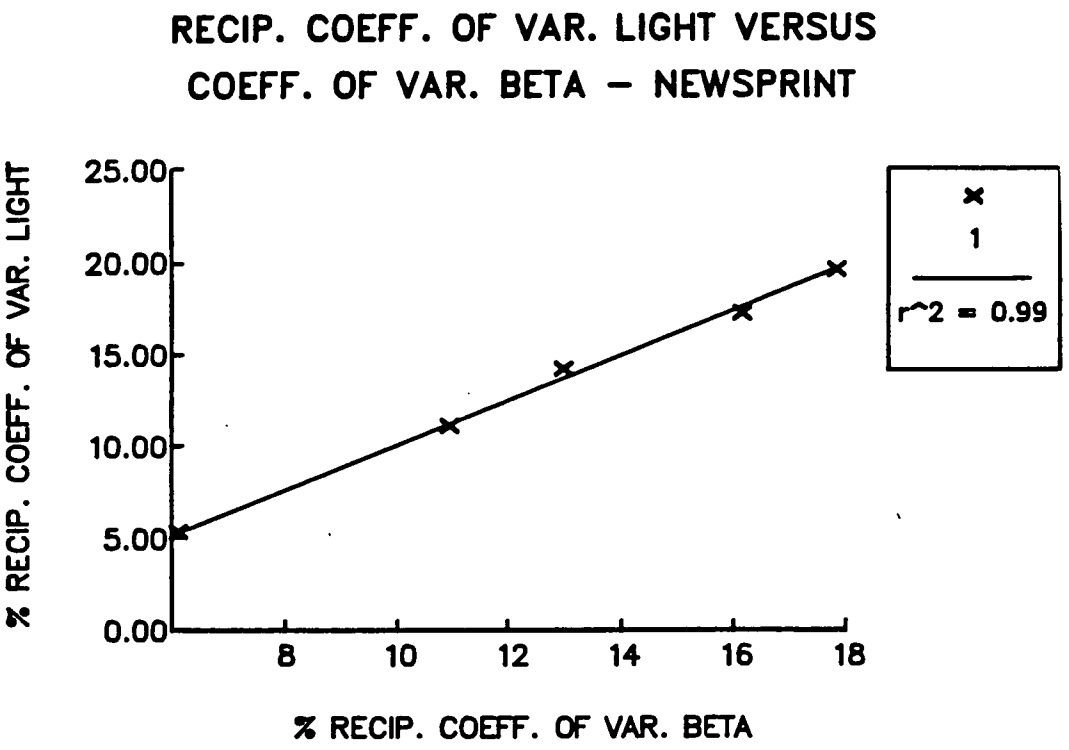

Figure 7.

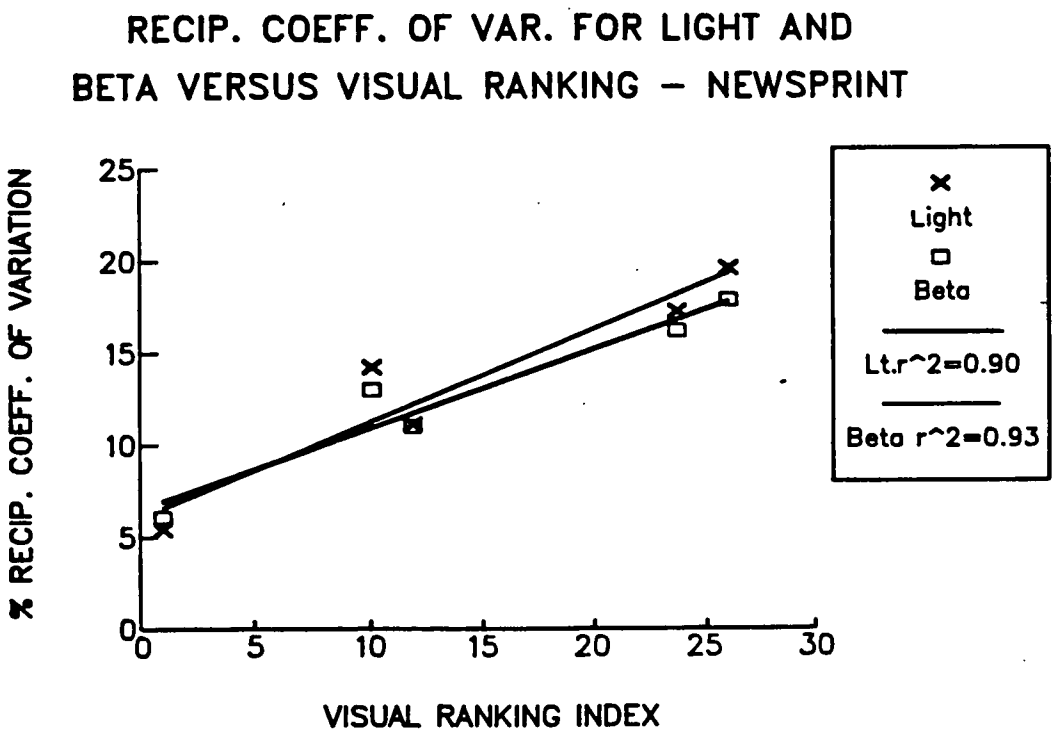

Figure 8.

The Dependence of Other Physical Property Measurements on Formation

The dependence of a number of other physical property measurements including elastic properties, strength, caliper, porosity and surface roughness on formation have been examined. The results presented are for a set of offset sheets made on production, Formette and Noble and Woods formers. It should be emphasized that we have not yet had the opportunity to make beta transmission measurements on these sheets.

Longitudinal out-of-plane elastic constant measurements were made using 3/8" probes (it is proposed that we might attempt formation related measurements using even smaller probes e.g. 1 mm diameter). The variability in caliper with the IPST formation tester index % 1/c.v. is shown in Figure 9 as might be expected the variability in caliper is reduced as formation improves. A similar result is obtained when visual ranking is employed as the independent variable as shown in Figure 10. It is also noted that production made sheets and handsheets fall approximately on the same curve, particularly when the IPST formation tester index is used i.e. Figure 9.

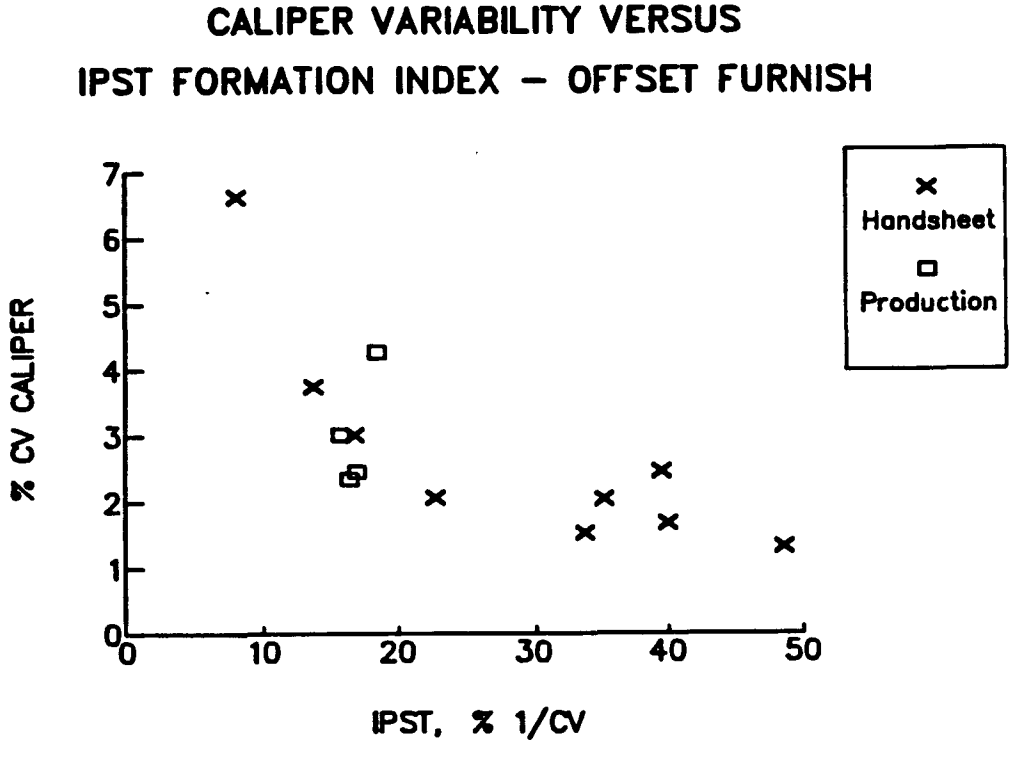

Figure 9.

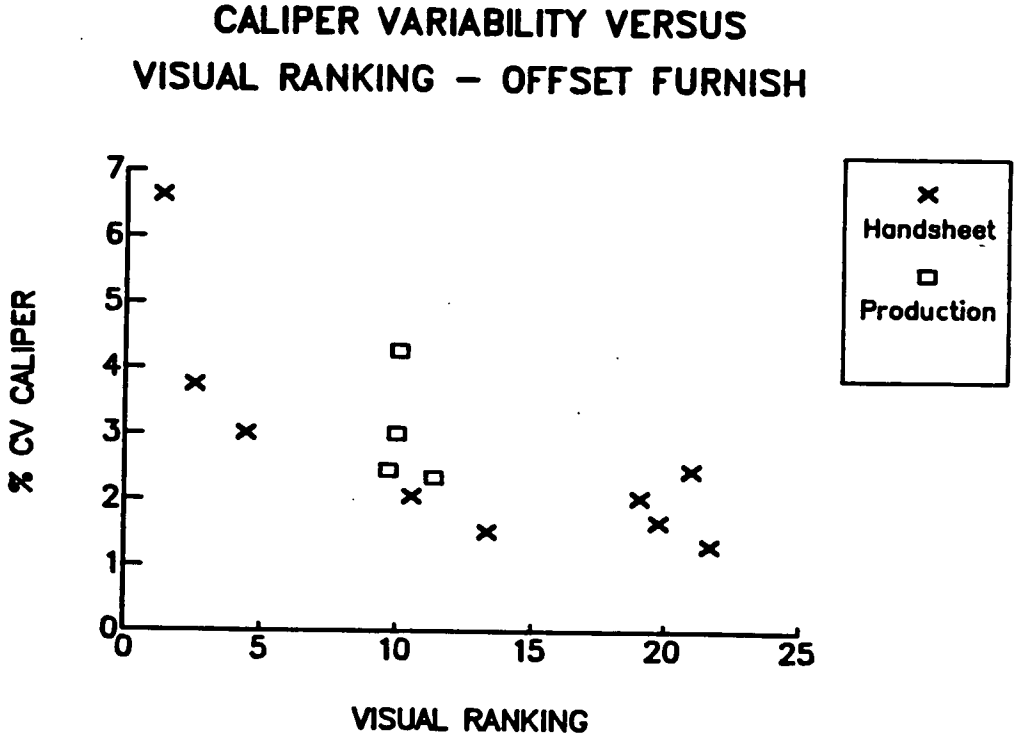

Figure 10.

When the out-of-plane longitudinal modulus is plotted versus IPST formation tester index (% 1/c.v.) it is relatively independent of changes in formation as shown in Figure 11. However, we do see higher modulus values for the production made sheets. Figure 12 indicates that differences in apparent density are not responsible for the difference between production and handsheet values. A similar result is obtained, if visual ranking, is again used in place of % 1/c.v. There is considerable variability in the modulus measurements as shown in Figure 13. There does not appear to be a clear trend with increasing formation index. If anything the variability seems to be worse as formation improves!

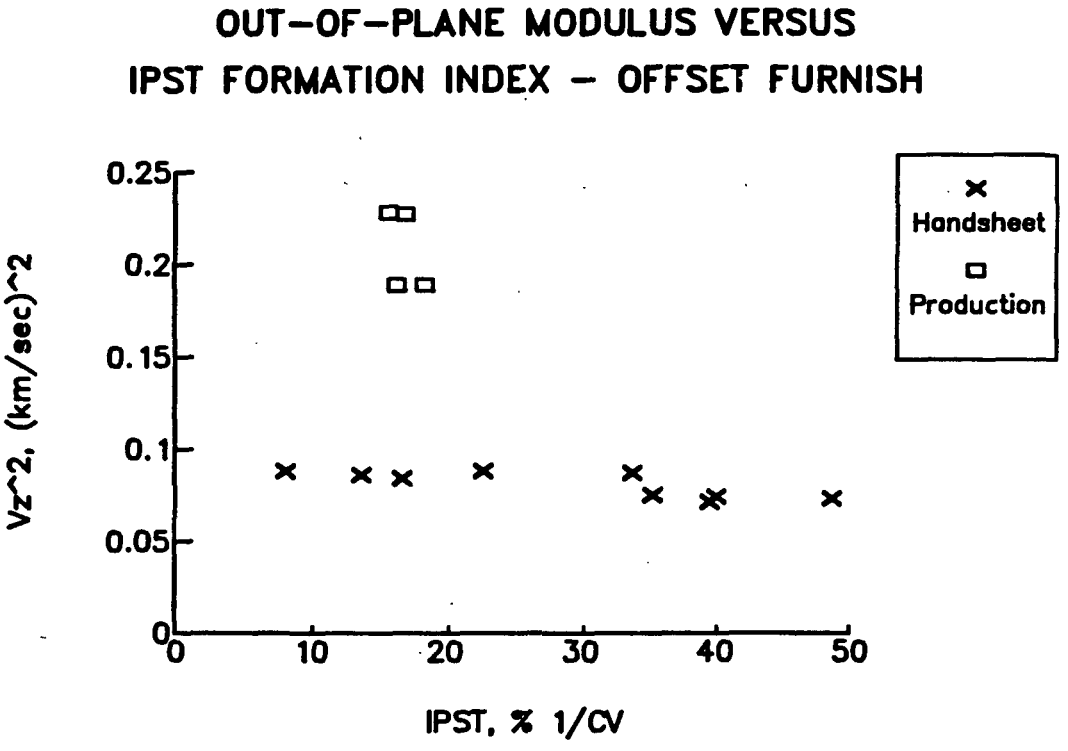

Figure 11.

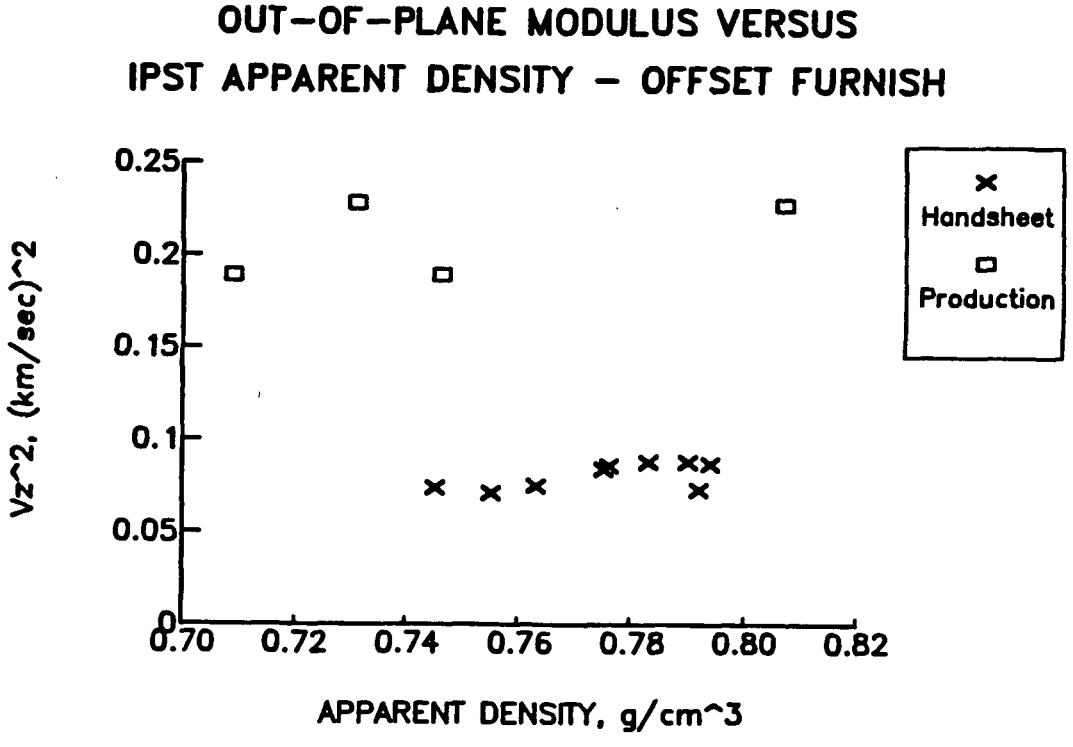

Figure 12.

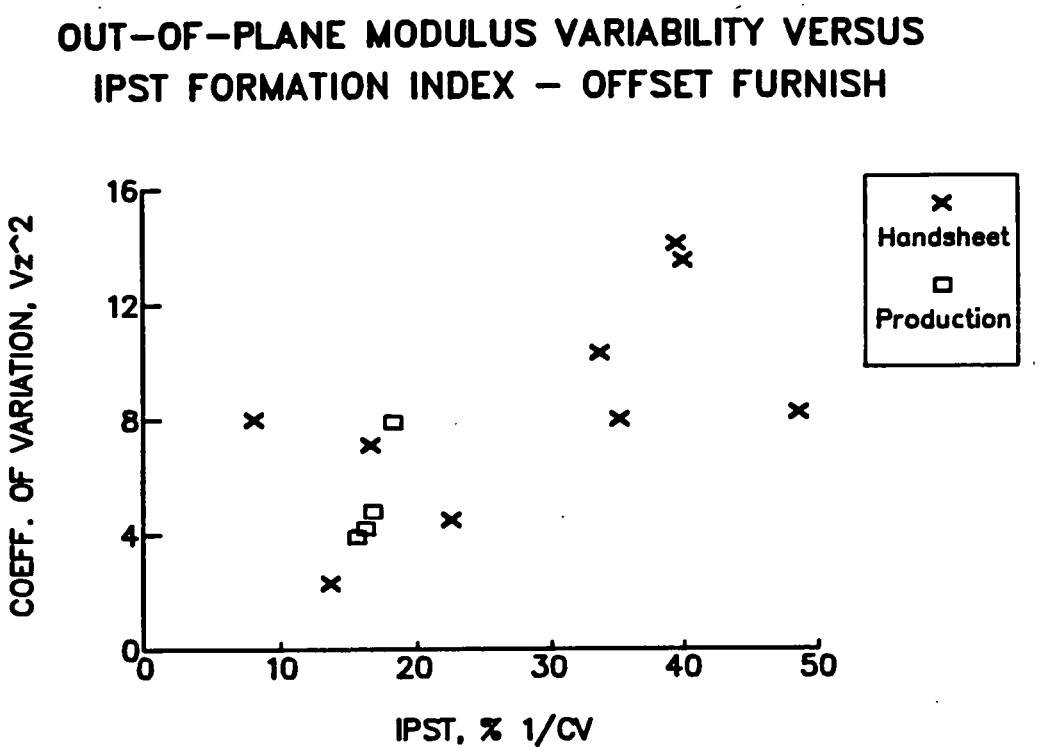

Figure 13.

COMBINED STRESS MEASUREMENTS

Corrugating, calendering, adhesive joints, paper tube performance are examples of converting and end use performance where combined stresses are thought to be important.

In previous work we have been concerned with how combined stresses affect paper properties. In particular the effects of corrugating and calendering on the elastic and strength properties have been investigated.

An out-of-plane biaxial device for measuring the failure envelope of paper and board when subjected to combined out-of-plane stresses has recently been designed and fabricated and is shown in Figure 14. We are in the process of determining the effects of refining and wet pressing on the out-of-plane failure envelope. A failure envelope for a linerboard made from unrefined stock and subjected to an intermediate level of wet pressing is shown in Figure 15. We are currently rechecking the properties of our samples which were made in Appleton before proceeding to complete our failure envelope measurements.

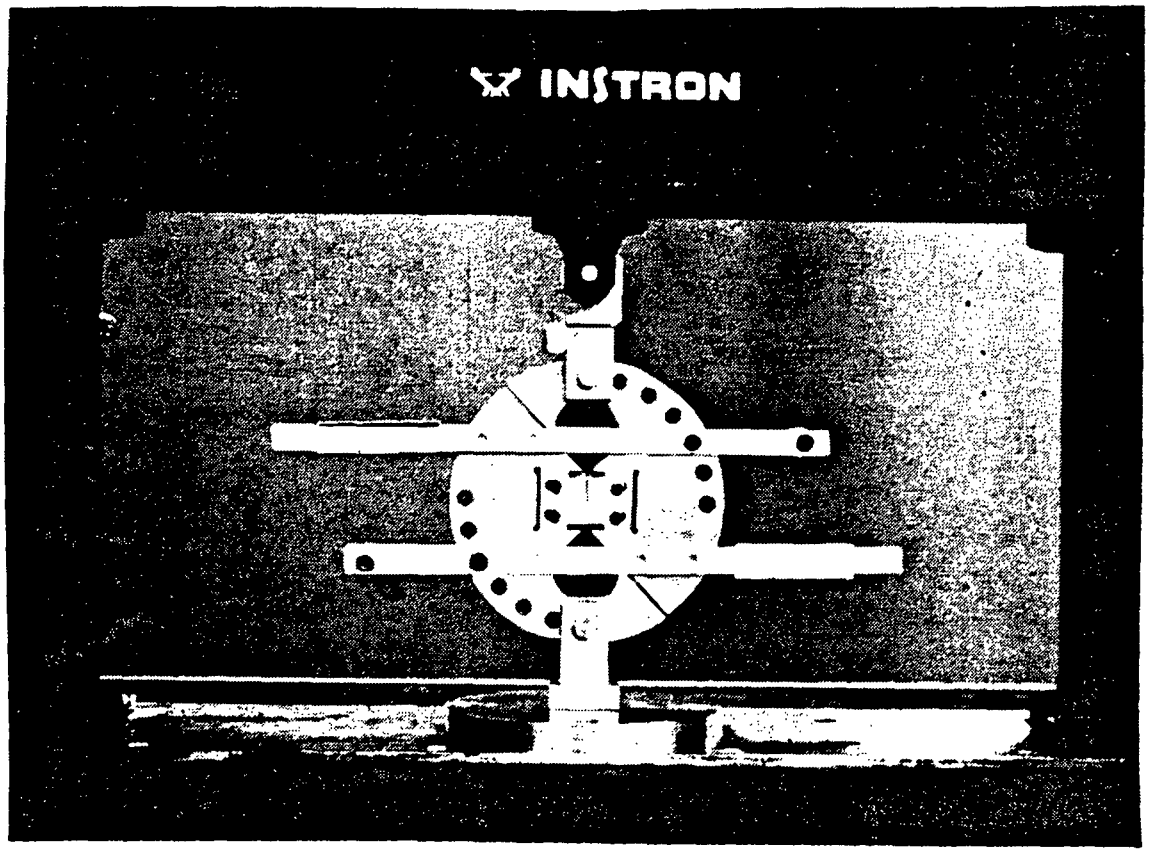

Figure 14.

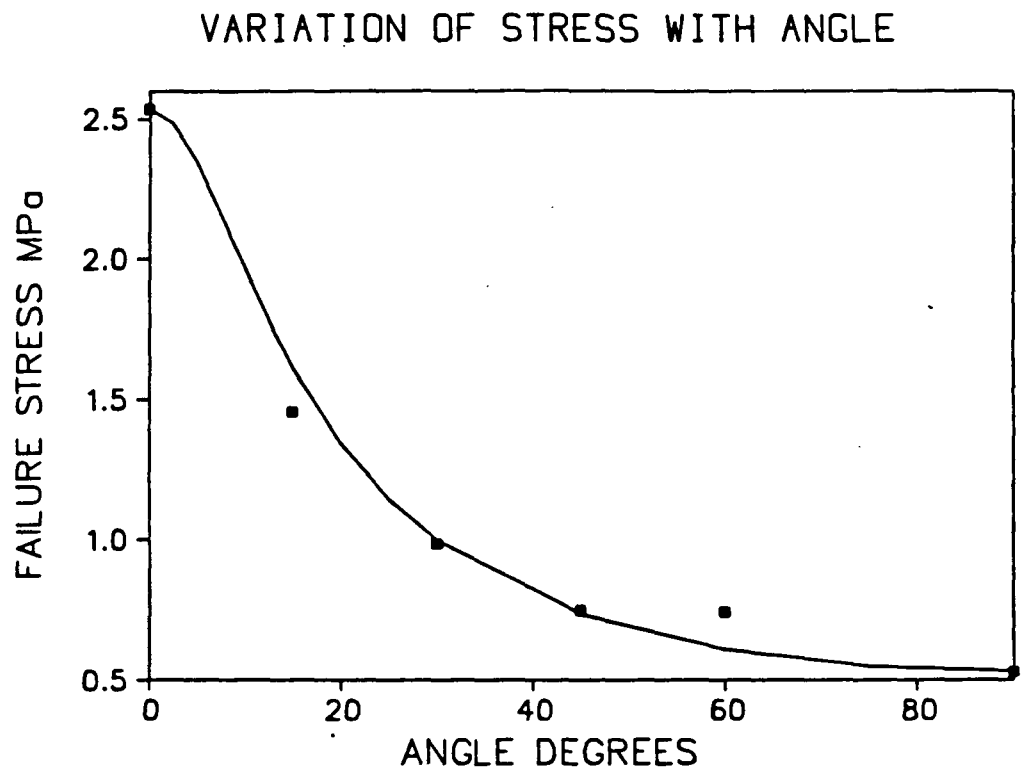

Figure 15.

INTERNAL STRESSES IN PAPER AND BOARD

Internal stresses are important in a number of paper and board performance areas. They are important in controlling, amongst other things, the elastic and strength properties, as well as dimensional stability and curl.

In previous work we have examined both sectioning and layer removal techniques for measuring property changes e.g. in-plane and out-of-plane elastic properties from layer to layer in the thickness direction of paper. From these measurements and curvature measurements we have been able to calculate the residual stress distribution. Furthermore, we have also measured changes in lean angle from layer to layer in the thickness direction using the ultrasonic robotic tester. This information is particularly valuable in understanding off-axis curl problems.

We do not currently have facilities for layer removal by surface grinding as developed by Wink at the Institute a number of years ago. However, we are investigating alternate techniques such as ion milling, excimer laser, and plasma etching (Dr Pierre Brodeur recently brought the plasma etching technique to my attention, which has been the subject of several papers from the University of Montreal). We of course would like a "clean" technique and one which would not alter the physical properties of the paper.

We are hoping to explore the excimer laser technique at Georgia Tech. Research Institute in the near future.

STUDENT RELATED WORK

Titles and comments on student related work are given below.

A190 Research Projects

1. "Non-destructive Measurement of Aging" - Andrew Gao

   The objective of this study is to examine non-destructive techniques for monitoring the aging of paper. We are specifically interested in possible changes in the viscoelastic

properties of aged paper, and if they can be measured using the ultrasonic rod resonance technique developed by Pankonin. As part of this investigation the natural aging of rag and high alpha grade fine papers initiated by Harry Lewis 60 years ago is also being studied.

2. "The Effect of Internal Stresses on the Modification of Paper Properties by Calendering." - Renee Ebert

In past work on calendering we have found that significant changes in both in-plane and out-of-plane elastic properties occur. The objective of this investigation is to determine the extent to which initial internal stress levels, and stress relaxation are responsible for these changes.

A490 Doctoral Research

1. "Strength Development through Refining and Wet Pressing" - Thomas Bither

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A490 Doctoral Research

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INTERNAL STRENGTH ENHANCEMENT

STATUS REPORT

FOR

PROJECT 3526

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

March 21, 1990

PROJECT SUMMARY

DATE: March 1, 1990

PROJECT: 3526 - INTERNAL STRENGTH ENHANCEMENT

PROJECT STAFF: R. Stratton

PROGRAM GOAL: Bring new attributes to fiber based products

PROJECT OBJECTIVE:

To improve internal strength and moisture tolerance in paper and paperboard. The short term goals are to establish those parameters fundamental to inter-fiber and intra-fiber bonding in conventional and ultra high yield pulps and to control these parameters if possible by chemical or mechanical treatments.

PROJECT RATIONALE, PREVIOUS ACTIVITY, AND PLANNED ACTIVITY FOR FISCAL 1989-90 are on the attached 1989-90 Project Form.

SUMMARY OF RESULTS LAST PERIOD: (March 1989 - October 1989)

(1) Measurements have been completed on all of the pulp samples.

(2) Analysis of this large mass of data is in progress and will be reported in a woodgrain report before the next PAC meeting.

(3) Offices, laboratories, and instruments were disassembled, packed, shipped to Atlanta, and unpacked. The laboratory is mostly set up although most of the instruments need to be placed into operation and calibrated.

SUMMARY OF RESULTS THIS PERIOD - (October 1989 - March 1990)

Analysis of results to date indicate:

1) The delamination tester provides a sensitive measure of the toughness of the fiber/fiber bonds.

2) The ability of the ultrasonic out-of-plane longitudinal velocity measurement to provide information about the bond strength between fibers is limited.

3) Most correlations between measured properties were independent of pulp yield. An exception is that between tensile strength and Z-toughness. The data are forced into separate groups depending on the pulp yield. This is mainly a result of the limiting factor of the individual fiber strength which decreases with increasing yield.

4) Tensile Energy Absorption (TEA) is a unique function of the tensile strength independent of pulp yield and treatment with strength aids.

5) STFI compressive strength can be increased by the use of strength aids to a limited extent. Factors other than bond strength curtail further increases.

6) STFI is directly correlated with the tensile strength independent of pulp yield and strength aid addition.

7) Pulp fines increase both tensile strength and Z-toughness. Fines in a system treated with a strength aid are much more effective in enhancing Z-toughness than in the untreated case.

8) The specific Z-toughness, a measure of the inherent "bondability" of a pulp was found to be independent of pulp yield but strongly increased when strength aid was present.

PROJECT TITLE: INTERNAL STRENGTH ENHANCEMENT

Date: 11/1/88

Budget: $160,000

PROJECT STAFF: R. Stratton

Period Ends: 6/30/90

PROGRAM GOAL: Bring new attributes to fiber based products

CURRENT OBJECTIVE:

To improve internal strength and moisture tolerance in paper and paperboard. The short term goals are to establish those fundamental parameters affecting inter-fiber and intra-fiber bounding in conventional and ultra high yield pulps and to control these parameters, if possible, by chemical or mechanical treatments.

PROJECT RATIONALE:

Major limitations of paper and board for many uses are low internal bond strength and poor moisture tolerance. Improved internal strength and enhanced moisture resistance would allow a number of present grades to be produced using less fiber and would also allow new end uses to be developed.

At present, commercial papers do not attain strength levels that realize the full potential of the wood fibers. Most paper mechanical properties are markedly degraded with increasing moisture content. We need to better understand the nature of fiber properties and fiber-to-fiber bonding and changes in them with increasing moisture content, if we are eventually to improve the moisture tolerance of paper.

RESULTS TO DATE:

The major areas of activity and results can be separated into two areas: handsheet studies of strength enhancement by chemicals; and bonding studies at the level of individual fibers. A number of polymers have been shown to be effective strength aids for a variety of

chemical and mechanical pulps. In particular, combinations of a cationic polymer followed by an anionic polymer have provided substantial improvements in dry (50% RH), moist (92% RH), and wet tensile strength. Although fines contribute to strength, polymer absorbed on long fiber is much more effective in improving strength than that absorbed on fines. Use of a rigid SBR latex provided marked improvement in compressive strength measured at high humidity. Techniques have been developed to prepare, handle, and measure individual fiber/fiber bonds. Significant improvements in bond strength were achieved when strength aids were used. For unrefined fibers the location of bond failure (as observed in the scanning electron microscope) was shifted from between the fibers to within the fiber wall by the addition of a strength aid. Unrefined latewood fibers were found to produce stronger bonds than earlywood fibers. Comparison of lightly refined to heavily refined fibers showed that the strength of individual bonds did not change with refining. When wet strength is not required, the use of strength aids acting by ionic interactions only allow the preparation of sheets that can be readily repulped.

Individual fiber bonds prepared using such additives were as strong as those made with covalently-bonding strength aids. Measurements of the loss of individual bond strength with increasing moisture content paralleled the similar losses in sheet strength and individual fiber axial strength.

Compressive strength and tensile strength are affected differently by additives. Internal addition of aids produces stronger sheets than does external addition showing the relative enhancement by the additive within the bonded area as opposed to around its periphery.

An instrument to measure the internal bond strength of sheets has been constructed. A series of pulps have been produced which will be used to evaluate the effects of yield, refining, fines content, and strength additives on bond strength.

Measurements on handsheets from a series of kraft pulps with yields from 47 to 80% have been completed. A partial analysis of the results has shown that the relative bonded area (RBA) of a sheet can be calculated from measurements of the scattering coefficient and the tensile modulus at different levels of refining and wet pressing. Bond strength, ultrasonic out-of-plane acoustic impedance, tensile index and TEA index are directly proportional to RBA.

PLANNED ACTIVITY FOR THE PERIOD:

The new bond strength instrument as well as standard tests and ultrasonic measurements (in and out-of-plane) will be used to better understand the effects of the following parameters on inter-fiber bonding: oriented sheets -- both laboratory made (Formette Dynamique) and machine made; calendering and supercalendering, moisture (RH) and moisture cycling, temperature, additives both between the fibers and within the fiber walls.

To better characterize the mechanisms contributing to the measured delamination force in the new instrument, the possibility of changes in bonding outside the delamination zone will be tested.

Other additives systems will be explored as either internal or external treatments to enhance tensile and compressive strength under normal and high relative humidity conditions.

Two woodgrain reports will be issued on the results of the a) single fiber/fiber bond studies and b) handsheet studies of the effects of pulp yield, refining, wet pressing, and chemical additives on sheet properties.

RELATED STUDENT RESEARCH:

M.A. Friese, Ph.D. - 1991; M.T. Goulet, Ph.D. - 1989; C.O. Luettgen, Ph.D. - 1990; C.E. Miller, Ph.D. - 1989; D. L. Horstmann, M.S. - 1989; M.H. Lang, M.S. - 1990; M.W. Sachs, M.S. - 1989; S.J. Wallace, M.S. - 1990; T.M. Braga, M.S. - 1991

STATUS REPORT

INTERNAL STRENGTH ENHANCEMENT

PROJECT 3526

The objective of this research is to obtain a better understanding of bonding, both natural and chemically aided, between cellulose fibers and its relation to sheet properties. The rationale is that this knowledge would guide us in the design of better paper and board products and better strength additives. The early parts of this project focused on a) the investigation of the strength properties of individual fiber/fiber bonds and b) the evaluation of a number of presently used and potentially useful strength aids. The latter showed that the combination of cationic polyamide-polyamine-epichorohydrin (PAE) followed by anionic carboxymethycellulose (CMC) provided improved strength (+ See below) (in shear) of the fiber/fiber bond, the (optical contact) bonded area, and the locus and extent of damage of the failed bond. The results showed the striking increase in individual bond strength when the fibers were pretreated (wet end addition) with strength additives. Although interesting and useful, this work was very labor and time intensive. Therefore, an alternative method for evaluating bond strength was implemented. This technique, based on the work of Skowronski and Bichard (1), uses handsheets for the tests which also allows other typical sheet properties to be measured on the same sheet for correlation.

The Delamination Tester

The wheel which is mounted in an Instron load-elongation tester is shown schematically in Figure 1.

The paper sample is mounted between two pieces of transparent tape as shown in Figure 2. One flap is then attached to the wheel while the other is held in the jaws of the Instron. This arrangement insures that the delaminating force is always perpendicular to the paper

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+ both wet and dry. The single fiber work gave results for the breaking strength

sample. The wheel is provided with an air bearing to eliminate friction. The resulting load-elongation curve is shown schematically in Figure 3. The load rises rapidly and reaches a steady state. The force is not constant but oscillates about an average value due to formation and local strength variations. The average force is obtained by dividing the area, W, in the steady state region by the corresponding delamination length.

The stress distribution in the region of the delamination ("peeling") as a function of peeling angle has been analyzed by Kaelble (2) In general the geometry leads to mixed mode failure. That is, there are contributions from both z-direction separation and in-plane shear. The relative amounts of each depend on the corresponding moduli and the thickness of the sample as well as the peel angle. For the 90° peel angle and the relatively thin samples (60 g/m 2) used here, the shear component should be negligible. Delamination forces that were independent of basis weight in the range 40 to 60 g/m2 support the assumption of primarily Mode I failure.

A more serious objection to the test is that the measured value (an energy) includes irreversible work of deformation of the surrounding fiber network adjacent to the delamination zone. However, the order of magnitude difference in moduli (and strength) between the out-of-plane and in-plane directions suggests that deformation in the latter will be negligible. It is possible that (partial) delamination may occur in adjacent layers. If the resultant surface separation is below optical resolution (<300 nm), the calculated specific z-toughness (see below) will be too large. Again, the relative weakness of the sheet in the Z-direction would tend to confine the failure to the primary delamination layer where the stresses are maximal. In any case the problem of assigning the work done to a particular region is common to all measurements beyond the elastic limit of paper or other materials.

It has been shown (3,4) that the delamination force (i.e. force per unit sample width) is equivalent to the work of peeling per unit area or the energy (+ See below). Skowronski and Bichard (1) recognize this but choose to call the quantity "bond strength" with units of J/m^2. To minimize confusion with tensile, compressive, and bond shear strengths (each having units of N/m^2 or papermakers units of Nm/g), the measured quantity will be referred to in this report as Z-toughness (G_z) following Schulz - Eklund, Fellers, and Olofsson (5).

+ absorbed in deformation and fracture per unit area of new (geometric) surface.

By measuring the scattering coefficient of the paper sheet sandwiched between the transparent tapes before and after delamination, the increase in scattering ΔS can be determined. The change in optical contact area per unit of geometric area ΔOCA is then

$$\Delta OCA = (\Delta S)(G)$$

where G is the basis weight or grammage. The bond breaking energy per unit of bond-broken optical contact area will be called the specific Z-toughness (SG_z) and is calculated as

$$SG_z = (G_z)/\Delta OCA$$

This quantity should reflect the inherent "strength" of the bonds in the paper. Note that the optical contact area is much less than the true surface area (although directly proportional to the latter) (6). Thus SG_z cannot be directly compared with the energy to break particular bonds, eg., hydrogen bonds.

Scope of Present Work

This study is concerned with the relation between pulp yield and the Z-toughness and other mechanical properties. The independent variables were levels of refining and wet pressing pressure. In addition, sheets were made either without (U) or with (T) strength aid treatment. The strength aid chosen was 1% PAE and 0.4% CMC added sequentially at the wet end.

Starting with a single supply of loblolly pine chips, we made kraft cooks to 80.7, 60.4, 51.2, and 47.5% yield. The corresponding kappa numbers were 167, 116, 42.2 and 34.7 (Figure 4). Each of those pulps were refined to 600, 350, and 200 mL CSF (Figure 5). Handsheets were made from each following standard procedures on a Noble and Wood sheet mold. Sheets were couched between blotters and pressed at either 0, 20, 50, 100, or

200 psi for five minutes. The sheets were drum dried at 105°C for seven minutes. This range of conditions provided samples over the usual commercial range of freeness and density. Portions of some of these pulps were classified to remove the fines. The fines were collected and could be added back to the classified pulp to study the effect of fines level.

In addition to Z-toughness the usual mechanical properties were measured. In general sheet strength may be said to be a function of four parameters (Figure 6).

1) fiber properties (strength and length)
2) bond strength
3) bonded area
4) fiber and bond distribution

We will characterize the first three of these by zero-span tensile strength, Z-toughness, and relative bonded area (RBA), respectively. The fourth factor which is governed by formation and fiber orientation will be assumed invariant here. The RBA can be calculated from a comparison of the surface areas of completely unbonded sheets and bonded sheets. (Figure 7). If we assume the surface area is directly proportional to the light scattering coefficient, RBA is given as

$$RBA = (S_o - S)/S_o \qquad \text{(Figure 8)}$$

We use Luner, Karna, and Donofrio's (7) method to evaluate S_o, the scattering coefficient of the completely unbonded sheet. This involves a plot of S against the sheet modulus and a linear extrapolation to zero modulus. (Figure 9).

The vast amount of information generated is still in the process of being analyzed and will be the subject of a wood-grain report to be issued shortly. The following represents some of the more interesting findings to date.

Measures of Bond Strength

It has been found that for paper a linear relation holds between the in-plane ultrasonic modulus measured in a given direction and the corresponding tensile strength (8). This correlation is the basis for commercial instrumentation to measure "strength" on-line. It has also been postulated that measurement of out-of-plane moduli (longitudinal or shear) should be related to the degree of bonding in the sheet. We carried out a comparison of the out-of-plane ultrasonic results with those of Z-toughness on the pulps in this study.

The modulus is approximately given by $v^2\rho$, where v is the velocity of the ultrasonic wave (longitudinal or shear) and ρ is the density of the sheet. The measured quantities are pulse transit time, basis weight, and caliper (sheet thickness). The first two can be measured quite accurately, but the thickness can have much error associated with sheet roughness, compressibility, and non-uniformity. A more accurately measured parameter is the mechanical impedance Z_M which is given by $v\rho = G/t$ where G is the basis weight and t is the transit time. We will use Z_{ML} and Z_{MS} to denote the mechanical impedance in the z-direction of the longitudinal and shear modes, respectively.

A comparison of the Z_{ML} with Z-toughness is shown in Figure 10 for all the whole pulps. The open and closed data points indicate untreated and (PAE/CMC) treated sheets, respectively. It is seen that the data fall into two general groupings based on their treatment. A given acoustical impedance can correspond to two different levels of bond "strength" as measured by Z-toughness. So long as one sticks with a given treatment, there is a monotonic increase of Z_{ML} with G_z. The response is more clearly revealed in Figure 11 where Z_{ML} is seen to be an approximately linear function of the sheet density independent of pulp yield refining, wet pressing, and chemical treatment. That is, decreasing pulp yield and freeness, increasing wet pressing, or adding a strength aid all increase Z_{ML}, but the various parameters have a similar effect on density.

The mechanical impedance in the shear mode is much more difficult to measure because of problems of coupling the ultrasonic transducer to the sample. The results on the two pulps of lowest yield are shown in Figure 12. In spite of the increased scatter in the data, we can see a trend of increasing Z_{MS} with density up to a value for the latter of about 0.5 g/cm^3. At still higher densities Z_{MS} appears to level off or perhaps even decrease with density.

Plots of Z_{MS} against Z-toughness show similar trends: that is, increasing values of Z_{MS} at low G_z followed by a plateau or decrease at higher G_z. The reason for this behavior is not clear. In view of the greater experimental difficulties with the shear mode measurement, it is recommended that the emphasis in instrumentation development be placed on the longitudinal mode.

Correlation of Z-toughness with Other Properties

If Z-toughness is truly a measure of how strongly bonded the sheet is, there should be a direct correlation between G_z and the relative bonded area. A representative graph is shown in Figure 13. The plot is linear and G_z goes to zero at zero RBA within the experimental scatter. Similar plots were made for the other pulps.

The slopes showed no regular dependence on pulp yield. The averaged values and standard deviation for the three pulps of lowest yield (47,51,60%) are shown in Figure 14. As expected, whole pulps and chemically treated pulps are stronger at a given RBA than classified and untreated pulps. Note, though, that the treated, classified pulps are stronger than the untreated, whole pulps. These results provide strong support for the use of Z-toughness to characterize the "bond strength" of paper sheets.

It is generally thought that the relative bonded area for a given pulp should be a linear function of the density. From the previous results we would expect G_z to similarly vary with density. The results for the 47% yield classified pulp are shown in Fig. 15. Indeed, the correlations are linear and the results show that much larger G_z can be obtained at a given density when the fibers are chemically treated. The results for all four yields are shown in Figure 16. Broadly, the data fall into two families depending upon whether the strength aid is used. The additive has a much larger effect on the Z-toughness than does the yield, even at 80% yield. This suggests that the use of chemical additives with high yield pulps may permit the latter's use if other properties are in the proper range for a given grade of paper.

There is one correlation where the effect of pulp yield is strong. The tensile index is plotted against Z-toughness in Figure 17 for the indicated yields. The data at each yield include that for both whole and classified pulps and both untreated and treated with PAE/CMC.

The data for the 51% yield fall between those for the 47 and 60% yields as would be expected from the trend, but have been omitted here for clarity of presentation. It is seen that even with the use of a strength aid (i.e.,. the data at the highest Gz for a given yield), the tensile index for the 80% yield is much lower than the values for the lower yields. This is true even when low yield samples of much lower Z-toughness are used for comparison. The reason for this is the strength of the individual fibers. As discussed by Page (9) and by Kallmes and Perez (10), the strength of a paper sheet is a function of both the strength of the individual fibers and the bond strength holding them together. Either one or the other of these factors may be limiting for a particular sample. As the bond strength is increased, however, it is the fiber strength that finally governs the maximum tensile strength achievable. For the present pulps the zero span tensile strengths in order of increasing yields are 129, 133, 109, and 86 Nm/g. Those represent average values for all the samples of a given yield. No effect on the zero span strength was found for different levels of refining or wet pressing or when a strength additive was used. This agree with prior knowledge. The apparent plateau levels in Figure 17 are about 50-60% of the corresponding zero span strengths. This agrees with the findings of Kallmes (11), who further suggests that sheet failure is induced by fiber failure when the ratio of tensile strength to zero span strength exceeds 4/9. A valid question for further reflection might be: since we can achieve high bond strengths with any yield pulp, how can we attain a better zero span strength at high yields?

Correlation of Tensile Index with Extensional Stiffness

The data for all untreated pulps (different yields, classified and whole) are plotted in Figure 18. It is seen that the tensile index follows a smooth curve as a function of Et index with no systematic deviations. The curvature implies that the tensile strength increases more rapidly than does the extensional stiffness. The curve drawn in Figure 18 represents a smooth curve drawn through similar data obtained on the same pulps but having been treated with PAE/CMC. The individual data points have been omitted here for clarity, the experimental scatter being roughly the same for both data sets. As a given value of the extensional stiffness the chemically treated pulps have a tensile strength about 30% higher than the untreated pulps.

Correlation Between Tensile Energy Absorption and Tensile Strength

The tensile energy absorption (TEA) of a paper sheet is defined as the area under the stress-strain (or load-elongation) curve when the test is carried to failure. This property is shown plotted against tensile strength in Figure 19. This is the only correlation we have found that is independent of pulp yield, chemical treatment, and the presence of pulp fines. Increasing the tensile strength by increasing refining or wet pressing produces a similar increase in TEA such that the curve remains invariant. When the range of fiber strengths and flexibilities and of bond strengths are considered, this result is truly remarkable. It apparently reveals an inherent nature of the stress-strain properties of a sheet of paper.

Correlation of STFI Compressive Strength With Other Properties

The STFI compressive strength (abbreviated in this report to STFI) is an important determinant of corrugated carton performance. This study shows the impact of pulp yield and chemical treatment on this property. The variation of STFI with Z-toughness is presented in Figure 20 for the whole pulps at the three lowest yields with no strength additive. A plateau value of about 25 Nm/g, independent of pulp yield, is apparently being reached at the highest levels of refining and wet pressing.

When the pulps are treated with PAE/CMC, the curve shifts to higher Z-toughness values and reaches a higher plateau of about 30 Nm/g as shown in Figure 21. Apparently, STFI can be improved about 20% by increasing the bonding between the fibers via a strength additive.

The compressive strength is also directly correlated with the tensile strength as shown in Figure 22 for the same pulps. The effect of the strength additive is to extend the upper and of the curve about 20%. Does this correlation mean that, if we could increase the tensile strength by improving the individual fiber strength (zero span strength), we would expect to also achieve a higher STFI?

Improvement of the performance of corrugated containers at high relative humidity is of vital interest to the boardmaker. All of the data discussed to this point were taken at standard TAPPI conditions: 50% RH. The STFI compressive strength was also measured

at 90% RH and is shown in Figure 23 plotted against z-toughness (measured at 50% RH). The results are similar to those at 50% RH (Figure 21). The use of the strength additive (closed data points) improves the STFI by roughly the same factor as at 50% RH. The plateau at high values of the bond strength suggests that methods other than improved bonding must be sought to increase the STFI compressive strength beyond present levels.

Effect of Fines Content

It is well-known that pulp fines improve sheet strength, although the detailed mechanism is not clear. To investigate this dependence in more detail, we prepared a series of pulps composed of the classified pulp from the 47% yield which had been beaten to 350 mL CSF plus added pulp fines at levels of 7.5,15, or 22.5%. The original beaten pulp (before classification) had contained 7.5% fines.

Sheets were made from these synthetic pulps as usual but were wet-pressed only at 50 psi. The whitewater was collected and analyzed for fines content to determine fines retention. Retention was high in all cases as would be expected in a low turbulence sheet mold. The retention was independent of the fines level in the headbox and averaged 67% for the untreated pulps and 74% for those treated with PAE/CMC. The standard deviation in each case was 3%. The slightly higher retention for the treated pulps is as expected. The cationic PAE is known to act as a retention aid. The slight difference in fines retention will be ignored in the present analysis and the results will be given in terms of the fines content in the headbox.

The effect of fines on sheet density is shown in Figure 24. The close agreement between the treated and untreated pulps suggests that our ignoring the slight difference in retention between the two mentioned above is acceptable.

The dependence of tensile strength on fines content is shown in Figure 25. Both treated and untreated cases appear to be reaching plateau levels above a fines content of about 20%. There is an approximately constant increment of strength produced by the chemical additive independent of fines content. The fact that two different plateaus are being approached for the same pulp (that is the having same zero span tensile strength) suggests

that the mechanisms of strength enhancement by fines and by strength aids are significantly different - not a revolutionary thought.

The role played by fines in bonding is further illustrated in Figure 26 for Z-toughness. There is a linear increase in bonding with fines content for both treated and untreated cases. The enhancement of Z-toughness when the system is treated with PAE/CMC is remarkable. Apparently, fines are much more effective as bonding agents when treated with strength aids.

The results for STFI compressive strength and TEA as a function of fines content parallel those described above for tensile strength. This is not unexpected in light of the results in Figures 19 and 22.

Finally, the effect of fines on the extensional stiffness is given in Figure 27. Here, increasing fines has a much greater effect on Et than does the strength additive. Is this just an effect of increasing density? How exactly do the fines enhance the stiffness?

Specific Z-Toughness

As mentioned previously, the specific Z-toughness can be evaluated as the ratio of the Z-toughness to the change in optical contact area. Representative data are shown in Figure 28 for the 47% yield pulp. Note that the linear regression lines do not pass through zero. To calculate a value for SG_Z the slopes were used. That is,

$$SG_Z = \Delta G_Z/\Delta(\Delta OCA)$$

The results are presented in Figure 29 for all the pulps. There is no apparent trend with pulp yield so simple averages of the untreated and treated samples were taken. The value for the untreated pulp is of similar magnitude to that determined for a bleached kraft sheet (1) by the same technique.

The much larger (2.7X) values of SG_z for the treated sample indicates the much stronger bonding between the fibers when the strength aid is present. It is interesting that there is no trend with pulp yield for the untreated samples. Intuitively, one would think that the strength of the bonding would decrease with increasing lignin content. More work will be required to clarify this.

Future Work

Measurements of Z-direction tensile strength will be performed on the various samples discussed in this report. The correlation of those with the Z-toughness results should reveal more clearly what the latter is indicating.

In-plane ultrasonic measurements will be completed for these samples.

In collaboration with John Waterhouse formation (optical and mass density) measurements will be performed on selected samples.

The data analysis will be completed and a woodgrain report will be issued covering this work.

Other additives systems will be explored as either internal or external treatments to enhance tensile and compressive strength under normal and high relative humidity conditions.

The delamination tester as well as standard tests and ultrasonic measurement (in and out-of-plane) will be used to better understand the effects of the following parameters on inter-fiber bonding: oriented sheets -- both laboratory made (Formette Dynamique) and machine made; calendering and supercalendering, moisture (RH) and moisture cycling, temperature, additives both between the fibers and within the fiber walls.

To better characterize the mechanisms contributing to the measured delamination force in the new instrument, the possibility of changes in bonding outside the delamination zone will be tested.

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PROJECT SUMMARY

PROJECT: 3526 - INTERNAL STRENGTH ENHANCEMENT

PROJECT STAFF: R. Stratton

PROGRAM GOAL: Bring new attributes to fiber based products

PROJECT OBJECTIVE:

To improve internal strength and moisture tolerance in paper and paperboard. The short term goals are to establish those parameters fundamental to inter-fiber and intra-fiber bonding in conventional and ultra high yield pulps and to control these parameters if possible by chemical or mechanical treatments.

PROJECT RATIONALE, PREVIOUS ACTIVITY, AND PLANNED ACTIVITY FOR FISCAL 1989-90 are on the attached 1989-90 Project Form.

SUMMARY OF RESULTS LAST PERIOD: (March 1989 - October 1989)

(1) Measurements have been completed on all of the pulp samples.

(2) Analysis of this large mass of data is in progress and will be reported in a woodgrain report before the next PAC meeting.

(3) Offices, laboratories, and instruments were disassembled, packed, shipped to Atlanta, and unpacked. The laboratory is mostly set up although most of the instruments need to be placed into operation and calibrated.

SUMMARY OF RESULTS THIS PERIOD - (October 1989 - March 1990)

Analysis of results to date indicate:

sample. The wheel is provided with an air bearing to eliminate friction. The resulting load-elongation curve is shown schematically in Figure 3. The load rises rapidly and reaches a steady state. The force is not constant but oscillates about an average value due to formation and local strength variations. The average force is obtained by dividing the area, W, in the steady state region by the corresponding delamination length.

The stress distribution in the region of the delamination ("peeling") as a function of peeling angle has been analyzed by Kaelble (2) In general the geometry leads to mixed mode failure. That is, there are contributions from both z-direction separation and in-plane shear. The relative amounts of each depend on the corresponding moduli and the thickness of the sample as well as the peel angle. For the 90° peel angle and the relatively thin samples (60 g/m 2) used here, the shear component should be negligible. Delamination forces that were independent of basis weight in the range 40 to 60 g/m2 support the assumption of primarily Mode I failure.

A more serious objection to the test is that the measured value (an energy) includes irreversible work of deformation of the surrounding fiber network adjacent to the delamination zone. However, the order of magnitude difference in moduli (and strength) between the out-of-plane and in-plane directions suggests that deformation in the latter will be negligible. It is possible that (partial) delamination may occur in adjacent layers. If the resultant surface separation is below optical resolution (<300 nm), the calculated specific z-toughness (see below) will be too large. Again, the relative weakness of the sheet in the Z-direction would tend to confine the failure to the primary delamination layer where the stresses are maximal. In any case the problem of assigning the work done to a particular region is common to all measurements beyond the elastic limit of paper or other materials.

It has been shown (3,4) that the delamination force (i.e. force per unit sample width) is equivalent to the work of peeling per unit area or the energy (+ See below). Skowronski and Bichard (1) recognize this but choose to call the quantity "bond strength" with units of J/m^2. To minimize confusion with tensile, compressive, and bond shear strengths (each having units of N/m^2 or papermakers units of Nm/g), the measured quantity will be referred to in this report as Z-toughness (G_z) following Schulz - Eklund, Fellers, and Olofsson (5).

+ absorbed in deformation and fracture per unit area of new (geometric) surface.

By measuring the scattering coefficient of the paper sheet sandwiched between the transparent tapes before and after delamination, the increase in scattering ΔS can be determined. The change in optical contact area per unit of geometric area ΔOCA is then

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at 90% RH and is shown in Figure 23 plotted against z-toughness (measured at 50% RH). The results are similar to those at 50% RH (Figure 21). The use of the strength additive (closed data points) improves the STFI by roughly the same factor as at 50% RH. The plateau at high values of the bond strength suggests that methods other than improved bonding must be sought to increase the STFI compressive strength beyond present levels.

Effect of Fines Content

It is well-known that pulp fines improve sheet strength, although the detailed mechanism is not clear. To investigate this dependence in more detail, we prepared a series of pulps composed of the classified pulp from the 47% yield which had been beaten to 350 mL CSF plus added pulp fines at levels of 7.5,15, or 22.5%. The original beaten pulp (before classification) had contained 7.5% fines.

Sheets were made from these synthetic pulps as usual but were wet-pressed only at 50 psi. The whitewater was collected and analyzed for fines content to determine fines retention. Retention was high in all cases as would be expected in a low turbulence sheet mold. The retention was independent of the fines level in the headbox and averaged 67% for the untreated pulps and 74% for those treated with PAE/CMC. The standard deviation in each case was 3%. The slightly higher retention for the treated pulps is as expected. The cationic PAE is known to act as a retention aid. The slight difference in fines retention will be ignored in the present analysis and the results will be given in terms of the fines content in the headbox.

The effect of fines on sheet density is shown in Figure 24. The close agreement between the treated and untreated pulps suggests that our ignoring the slight difference in retention between the two mentioned above is acceptable.

The dependence of tensile strength on fines content is shown in Figure 25. Both treated and untreated cases appear to be reaching plateau levels above a fines content of about 20%. There is an approximately constant increment of strength produced by the chemical additive independent of fines content. The fact that two different plateaus are being approached for the same pulp (that is the having same zero span tensile strength) suggests

that the mechanisms of strength enhancement by fines and by strength aids are significantly different - not a revolutionary thought.

The role played by fines in bonding is further illustrated in Figure 26 for Z-toughness. There is a linear increase in bonding with fines content for both treated and untreated cases. The enhancement of Z-toughness when the system is treated with PAE/CMC is remarkable. Apparently, fines are much more effective as bonding agents when treated with strength aids.

The results for STFI compressive strength and TEA as a function of fines content parallel those described above for tensile strength. This is not unexpected in light of the results in Figures 19 and 22.

Finally, the effect of fines on the extensional stiffness is given in Figure 27. Here, increasing fines has a much greater effect on Et than does the strength additive. Is this just an effect of increasing density? How exactly do the fines enhance the stiffness?

Specific Z-Toughness

As mentioned previously, the specific Z-toughness can be evaluated as the ratio of the Z-toughness to the change in optical contact area. Representative data are shown in Figure 28 for the 47% yield pulp. Note that the linear regression lines do not pass through zero. To calculate a value for SG_z the slopes were used. That is,

$$SG_Z = \Delta G_Z/\Delta(\Delta OCA)$$

The results are presented in Figure 29 for all the pulps. There is no apparent trend with pulp yield so simple averages of the untreated and treated samples were taken. The value for the untreated pulp is of similar magnitude to that determined for a bleached kraft sheet (1) by the same technique.

The much larger (2.7X) values of SG_z for the treated sample indicates the much stronger bonding between the fibers when the strength aid is present. It is interesting that there is no trend with pulp yield for the untreated samples. Intuitively, one would think that the strength of the bonding would decrease with increasing lignin content. More work will be required to clarify this.

Future Work

Measurements of Z-direction tensile strength will be performed on the various samples discussed in this report. The correlation of those with the Z-toughness results should reveal more clearly what the latter is indicating.

In-plane ultrasonic measurements will be completed for these samples.

In collaboration with John Waterhouse formation (optical and mass density) measurements will be performed on selected samples.

The data analysis will be completed and a woodgrain report will be issued covering this work.

Other additives systems will be explored as either internal or external treatments to enhance tensile and compressive strength under normal and high relative humidity conditions.

The delamination tester as well as standard tests and ultrasonic measurement (in and out-of-plane) will be used to better understand the effects of the following parameters on inter-fiber bonding: oriented sheets -- both laboratory made (Formette Dynamique) and machine made; calendering and supercalendering, moisture (RH) and moisture cycling, temperature, additives both between the fibers and within the fiber walls.

To better characterize the mechanisms contributing to the measured delamination force in the new instrument, the possibility of changes in bonding outside the delamination zone will be tested.

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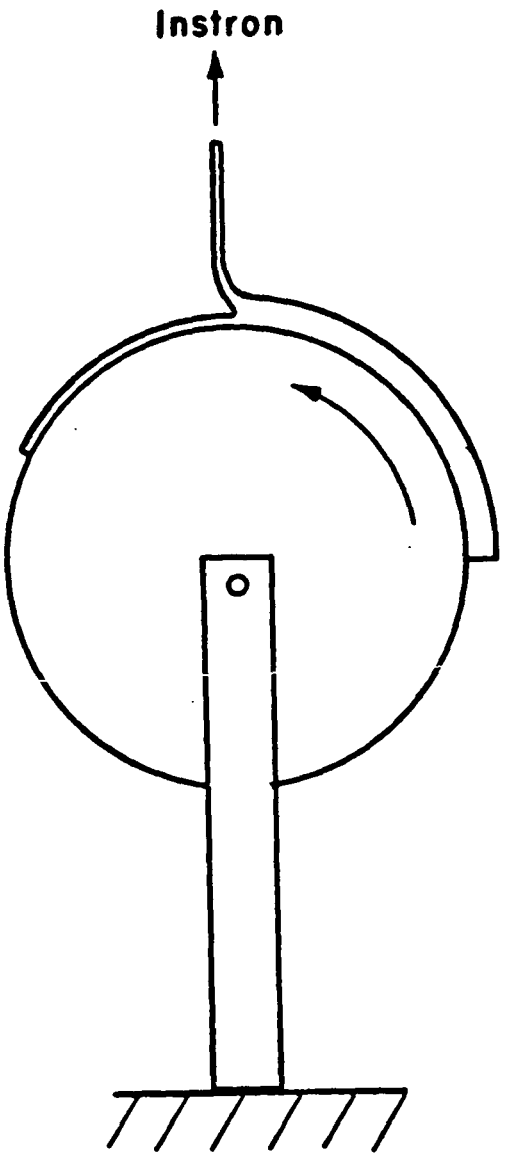


Figure 1. Schematic representation of the delamination tester.

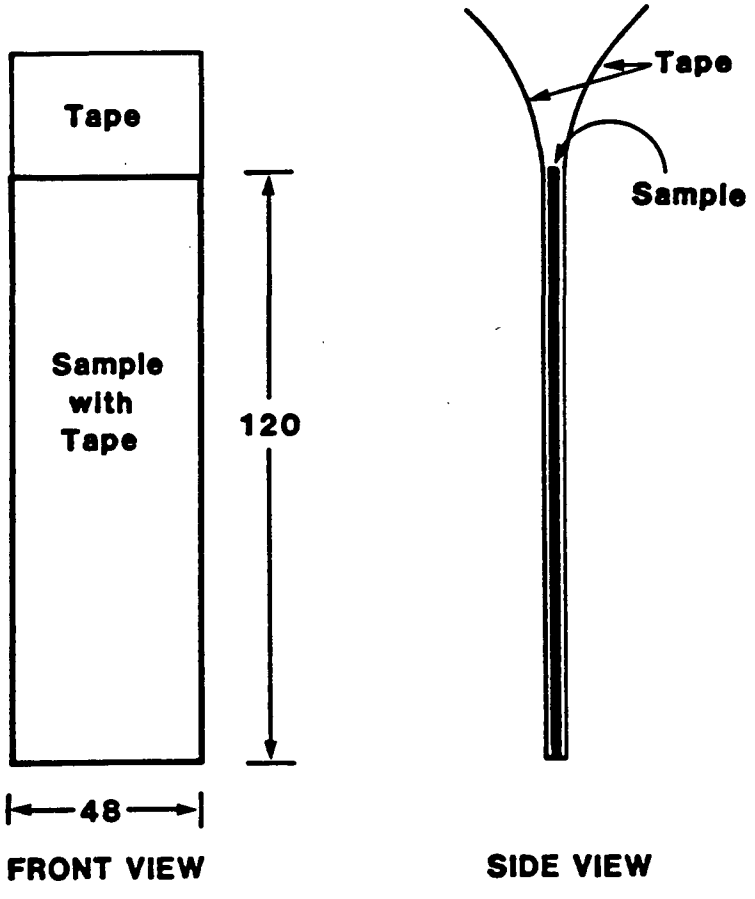


Figure 2. Preparation of sample for testing.

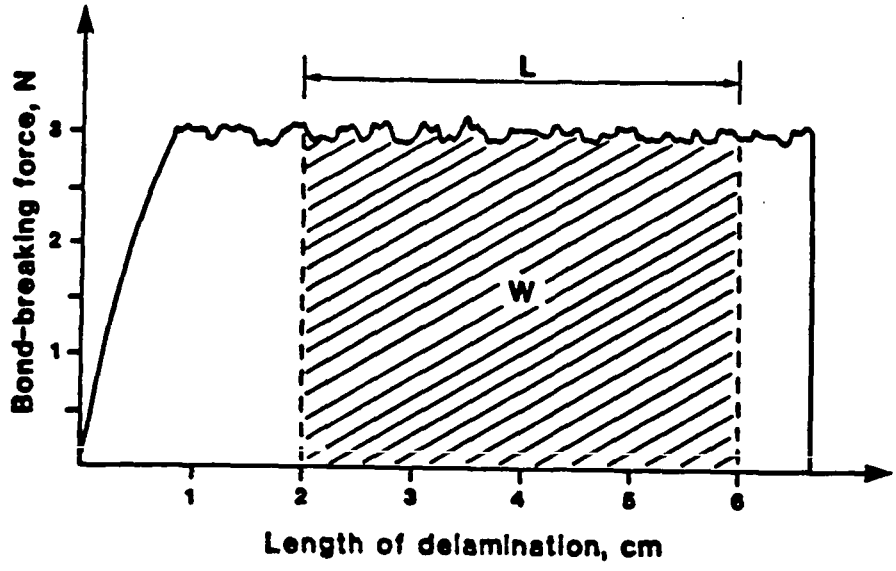


Figure 3. Schematic representation of the load-elongation curve produced during delamination.

PULP PROPERTIES

YIELD, %	KAPPA NO.
47.5	34.7
51.2	42.2
60.4	116.0
80.7	167.0

Figure 4. Pulp properties.

PULPS				
YIELD, %	FREENESS, mL CSF			
47	685	600	350	200
52	740	600	340	200
60	750	600	350	200
80	780	600	340	200

Figure 5. Pulp freeness.

FACTORS DETERMINING SHEET STRENGTH

1) FIBER PROPERTIES - STRENGTH AND LENGTH

2) BOND STRENGTH

3) BONDED AREA

4) FIBER AND BOND DISTRIBUTION

Figure 6. Factors determining sheet strength.

RELATIVE BONDED AREA

$RBA = (A_u - A_s)/A_u$

A_u = AREA OF COMPLETELY UNBONDED SHEET

A_s = AREA OF SAMPLE SHEET

ASSUMPTION: $A = kS$

Figure 7. Relative bonded area

RELATIVE BONDED AREA

$RBA = (S_o - S)/S_o$

S_o = Scattering from completely unbonded sheet

S = Scattering of sample sheet

Figure 8. Relative bonded area

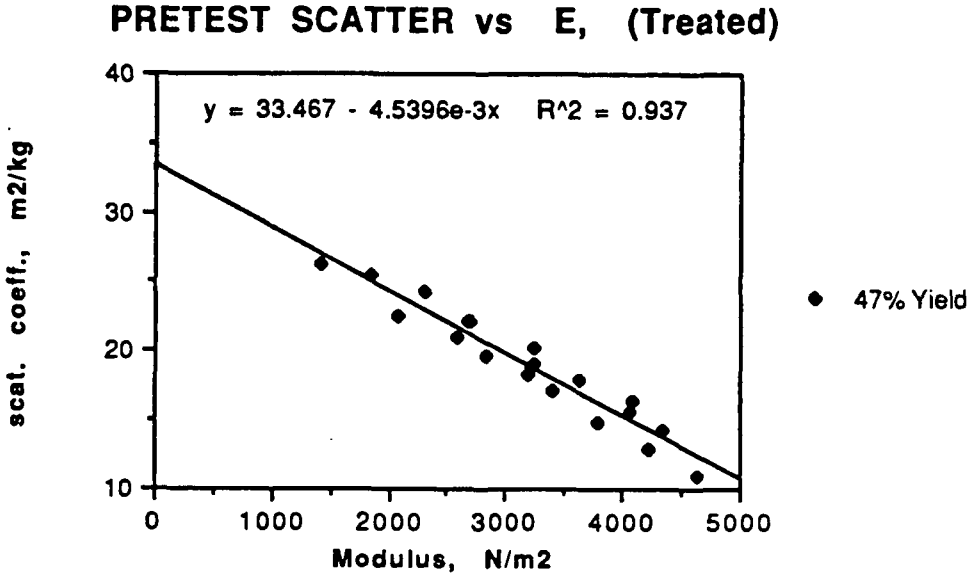


Figure 9. Plot to determine the scattering coefficient of unbonded sheet.

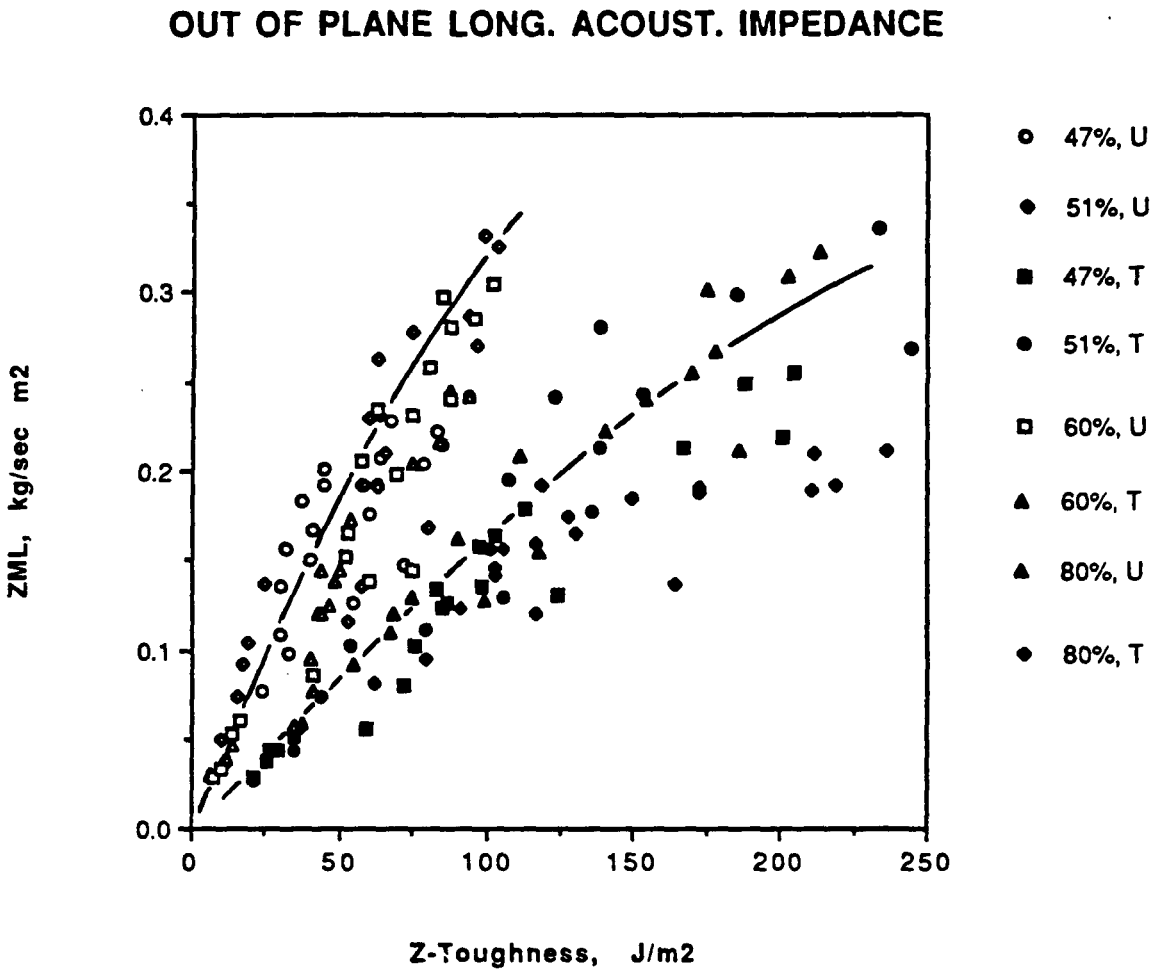


Figure 10. Out-of-plane longitudinal acoustic impedance plotted against Z-toughness for treated (closed symbols) and untreated (open) sheets.

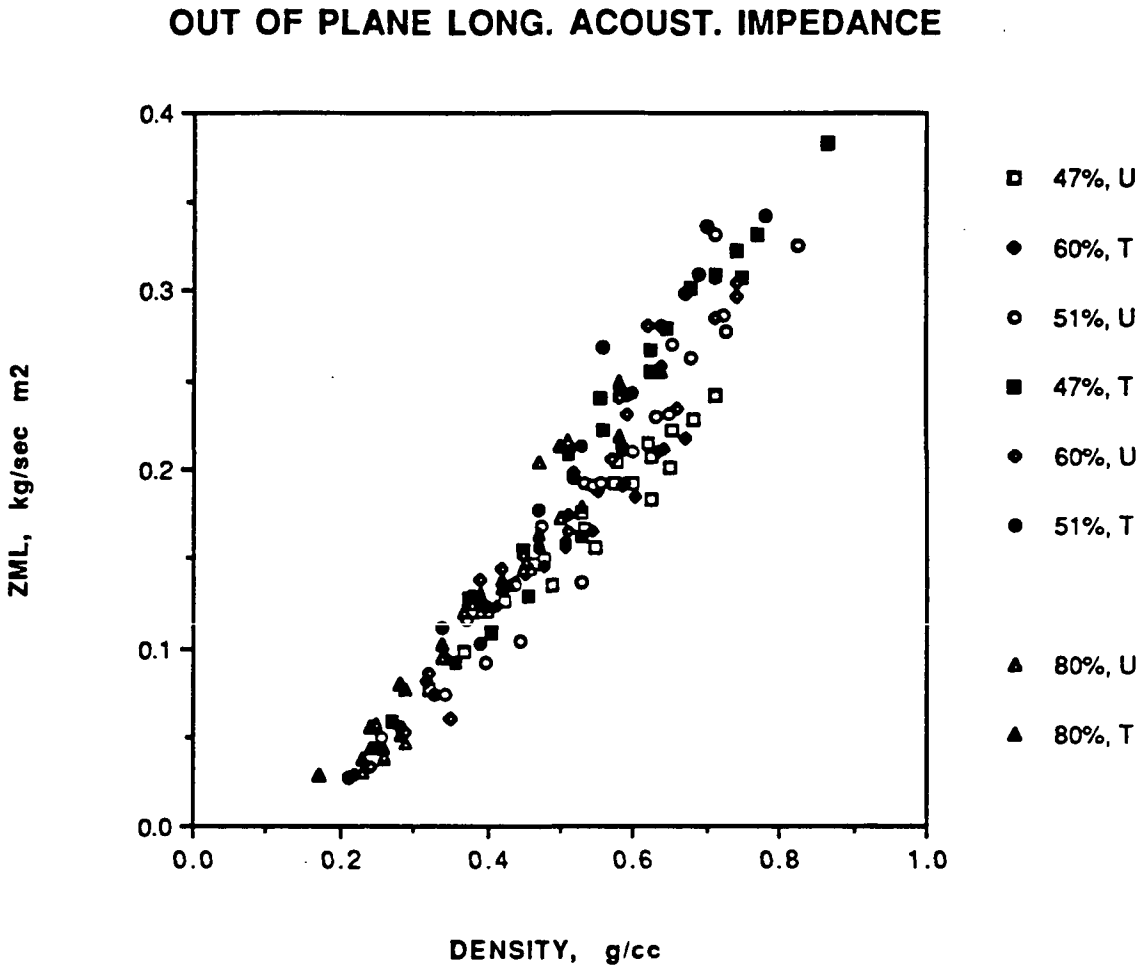


Figure 11. Out-of-plane longitudinal acoustic impedance plotted against density for treated (closed) and untreated (open) sheets.

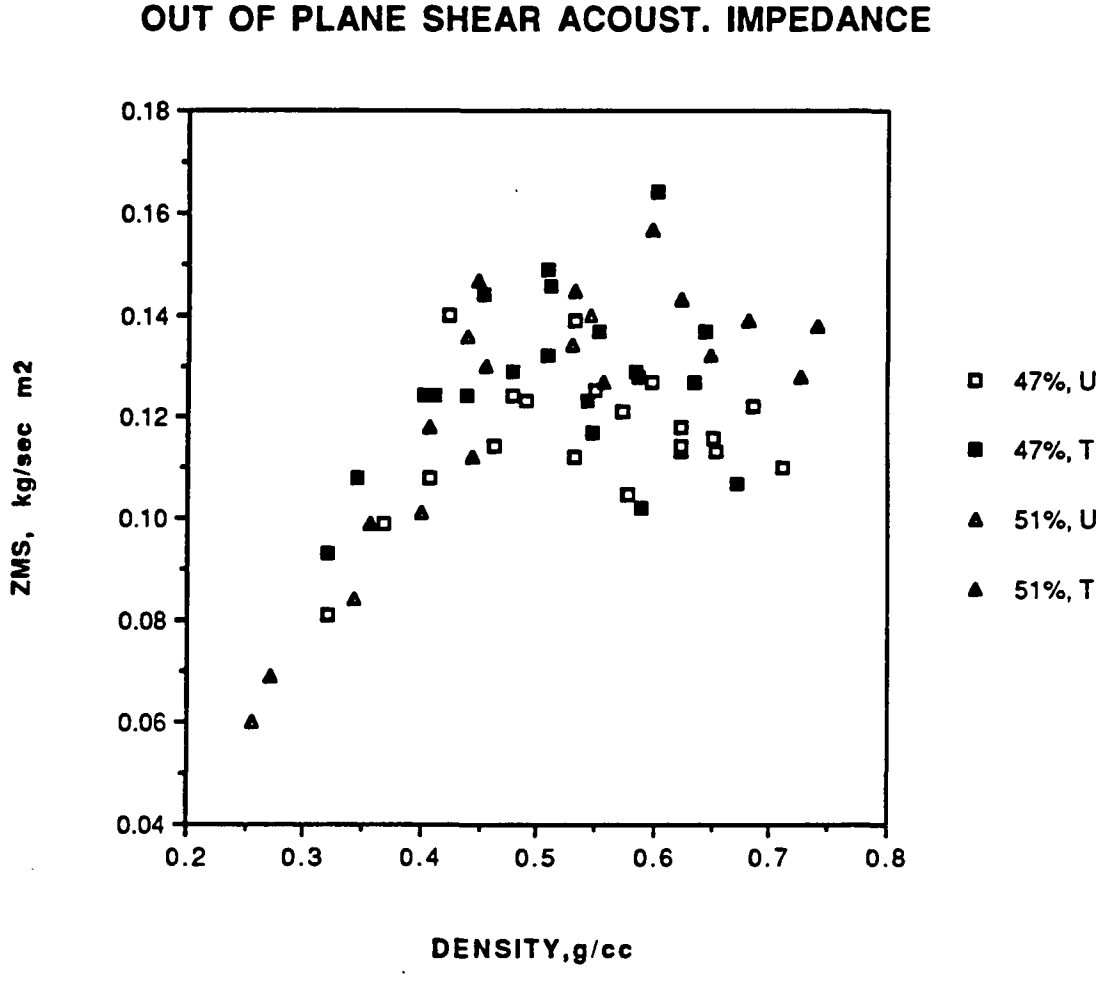


Figure 12. Out-of-plane shear acoustical impedance plotted against density for treated (closed) and untreated (open) sheets.

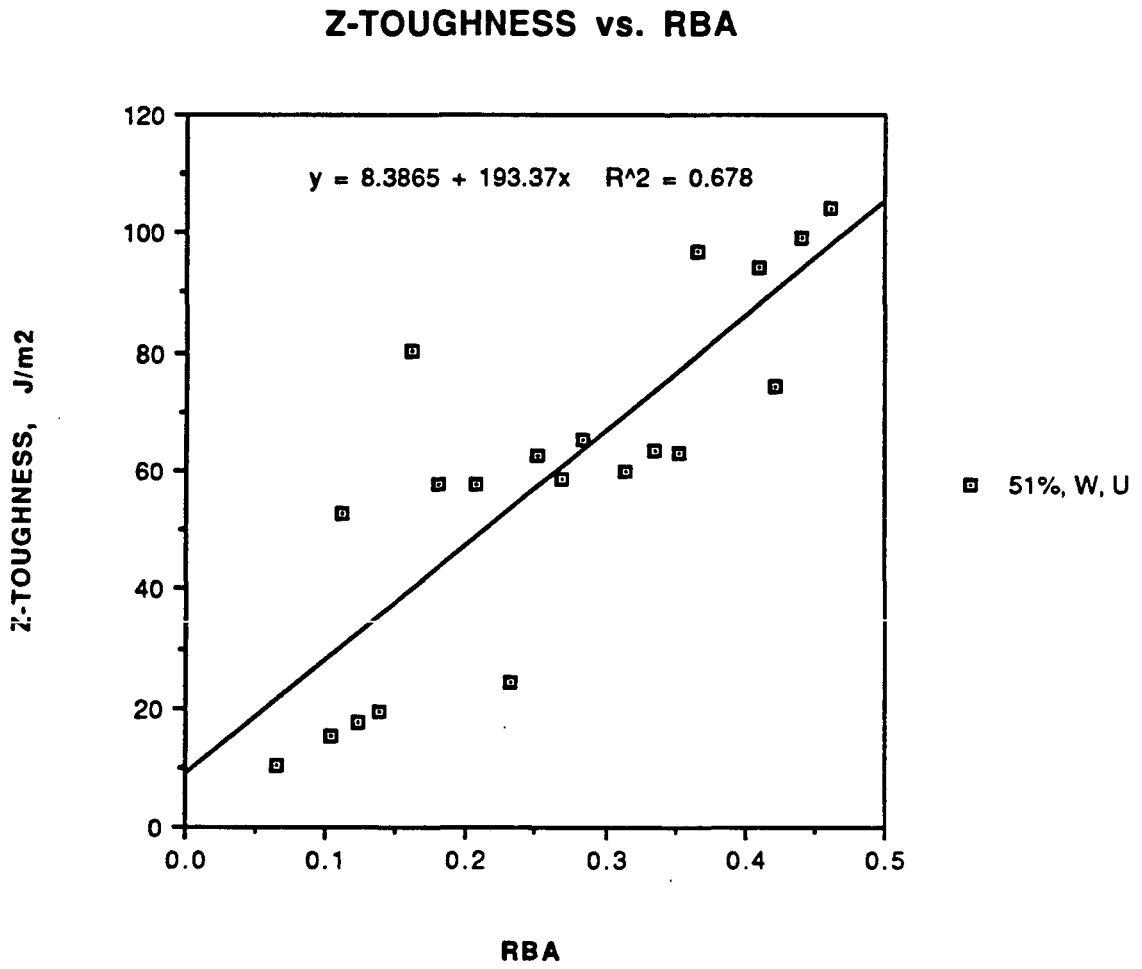


Figure 13. Z-toughness plotted against relative bonded area for one pulp at different levels of refining and wet pressing.

DEPENDENCE OF Z-TOUGHNESS ON RBA

		dG_z/d RBA, J/m^2	
PULP	TREATMENT	AVERAGE	S.D.
Whole	No	170	20
Whole	Yes	530	40
Class.	No	50	10
Class.	Yes	230	30

Figure 14. Dependence of Z-toughness on relative bonded area.

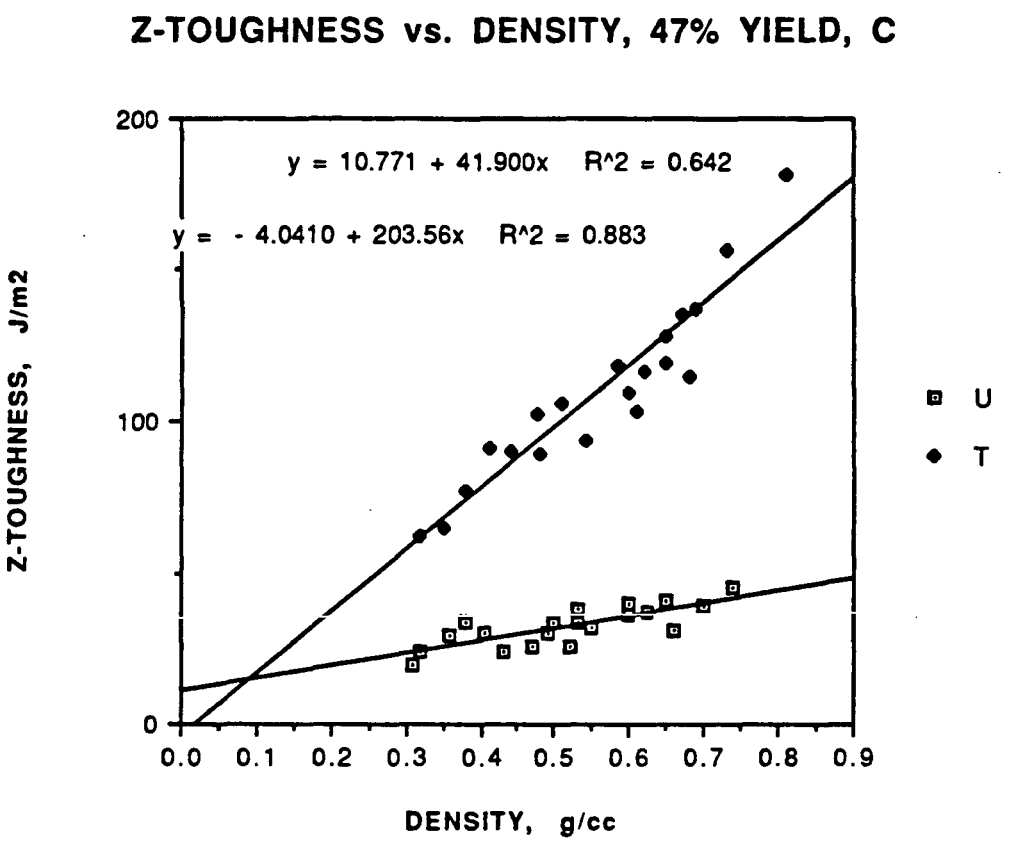


Figure 15. Z-toughness plotted against density for treated (closed) and untreated (open) sheets.

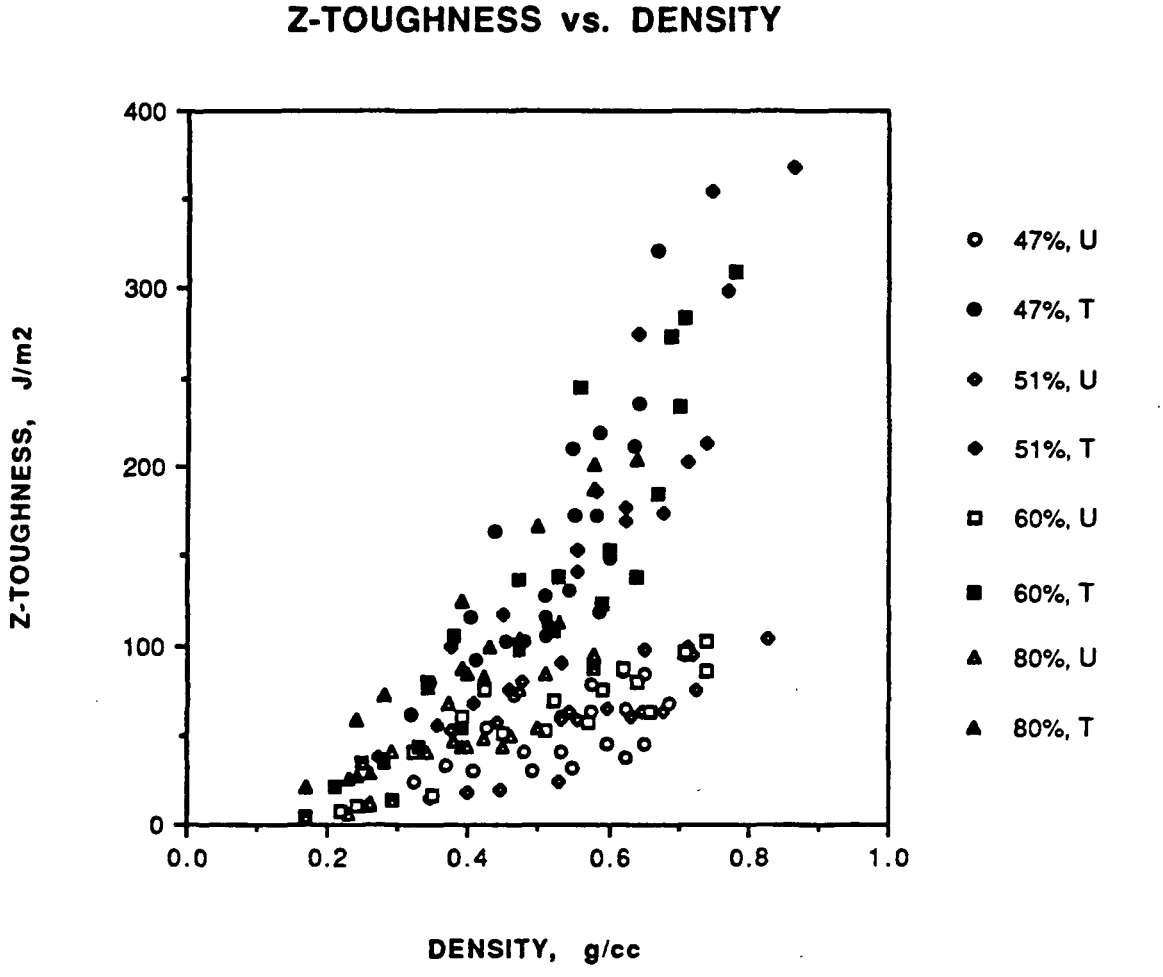


Figure 16. Z-toughness plotted against density for treated (closed) and untreated (open) sheets.

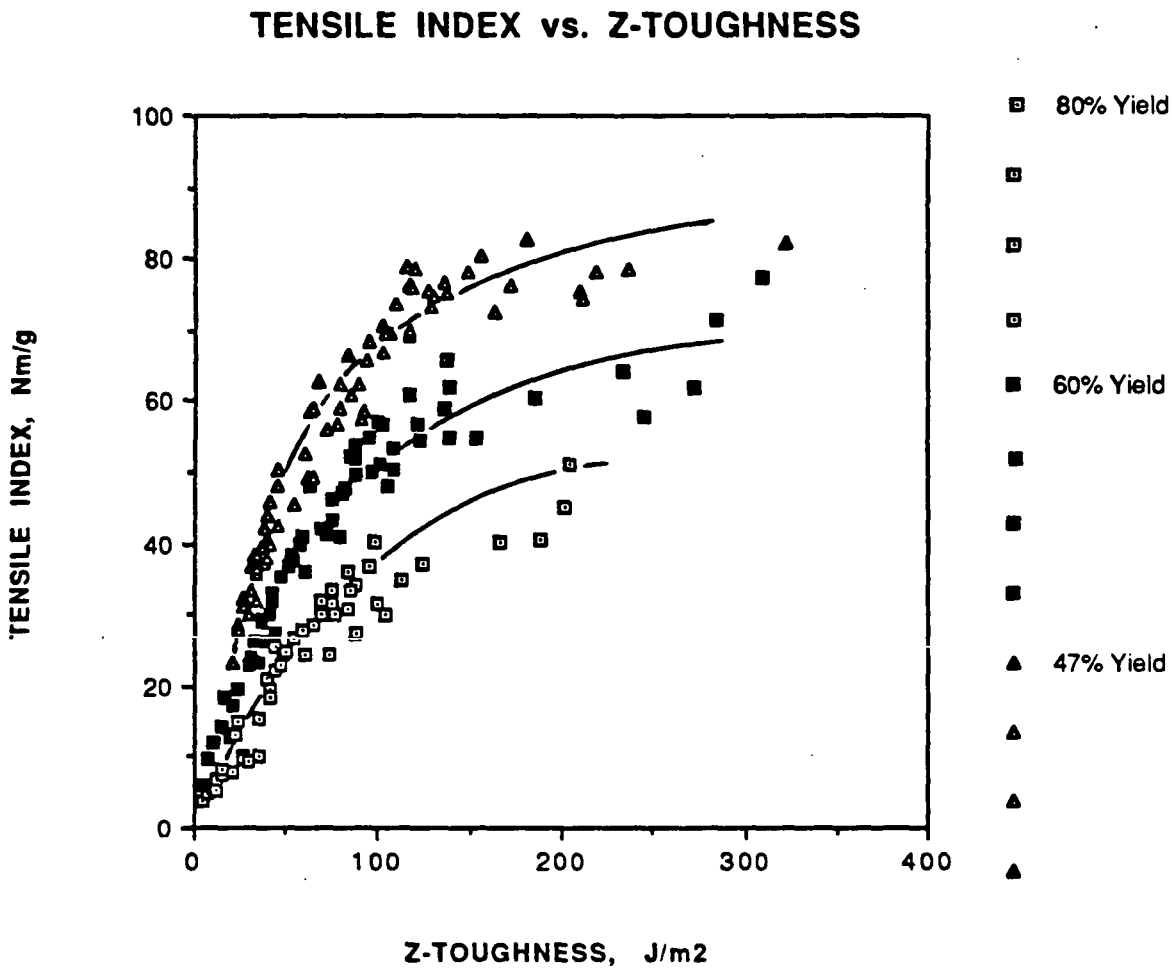


Figure 17. Tensile index plotted against Z-toughness for three different yields.

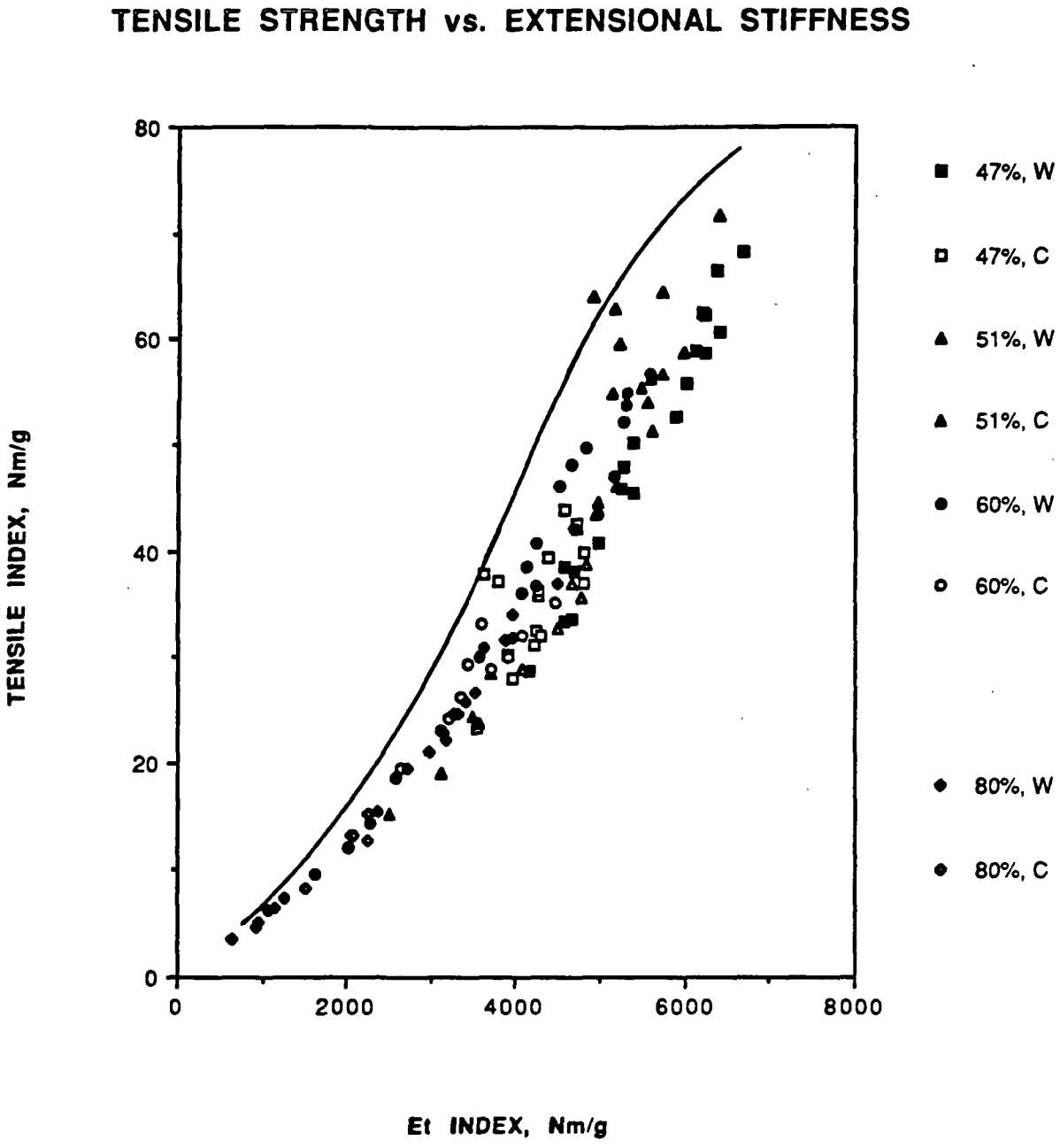


Figure 18. Tensile index plotted against extensional stiffness index for untreated sheets from whole and classified pulps. The curve shows the position for similar data for treated pulps.

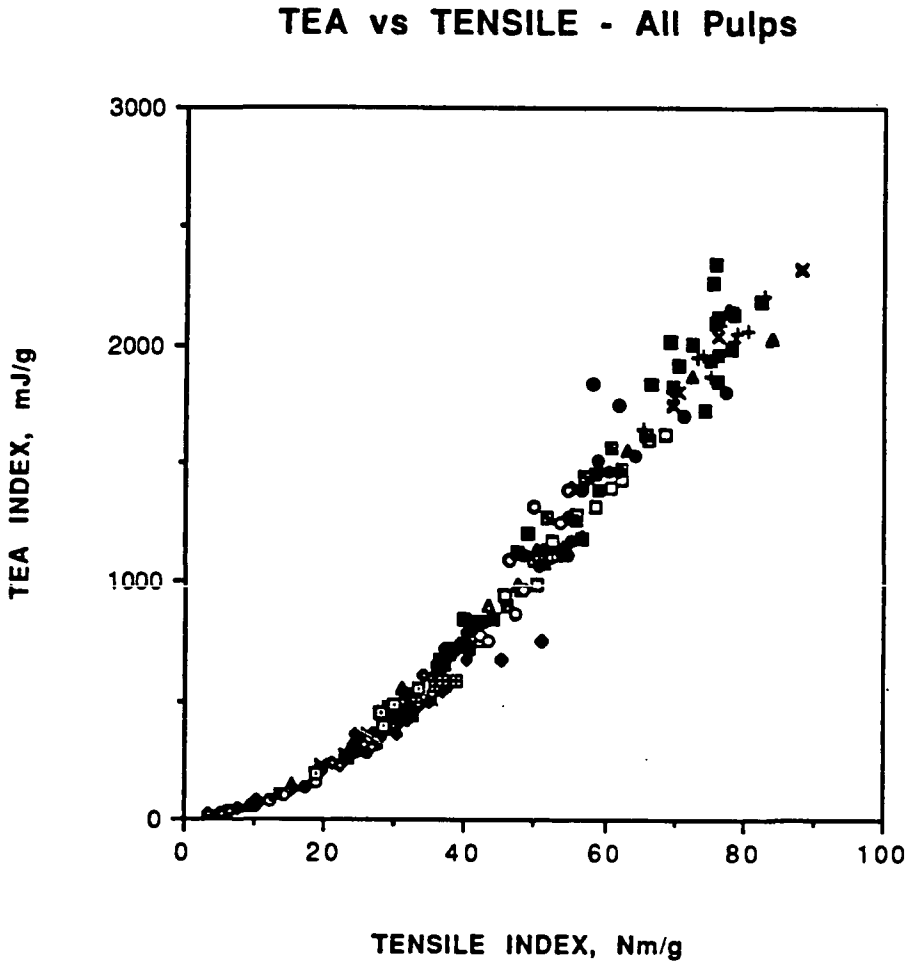


Figure 19. Tensile energy absorption index plotted against tensile index for all sheets.

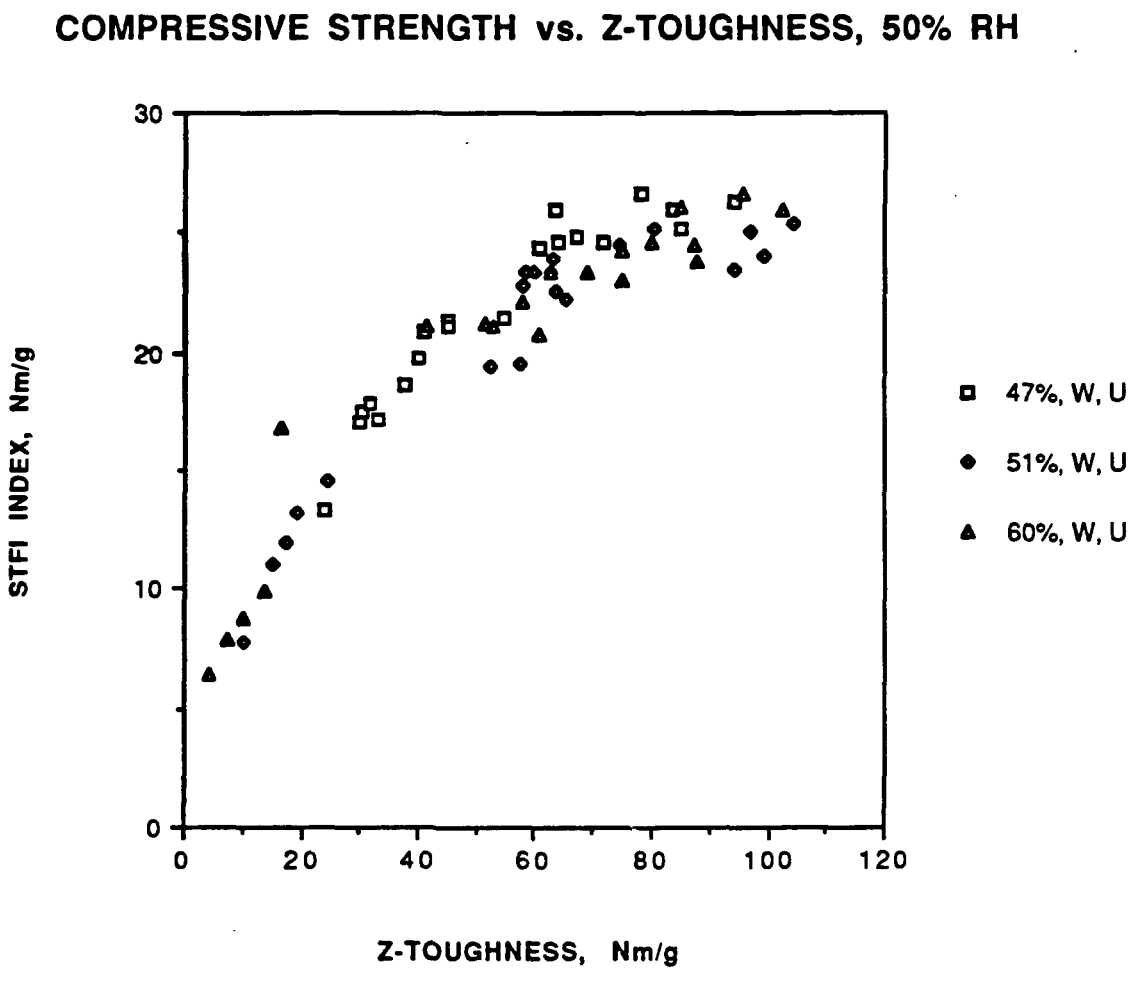


Figure 20. STFI compressive strength index plotted against Z-toughness for untreated, whole pulps.

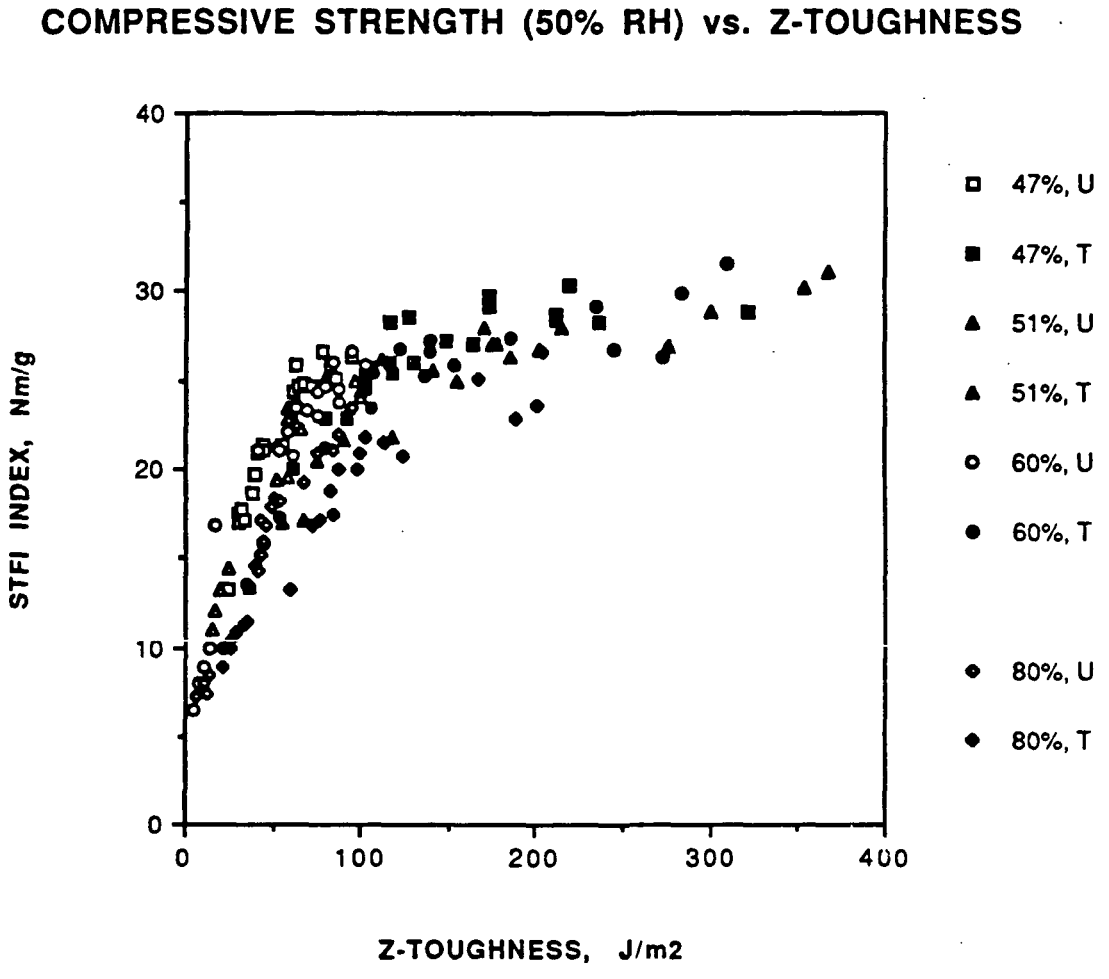


Figure 21. STFI compressive strength index plotted against Z-toughness for treated (closed) and untreated (open) sheets.

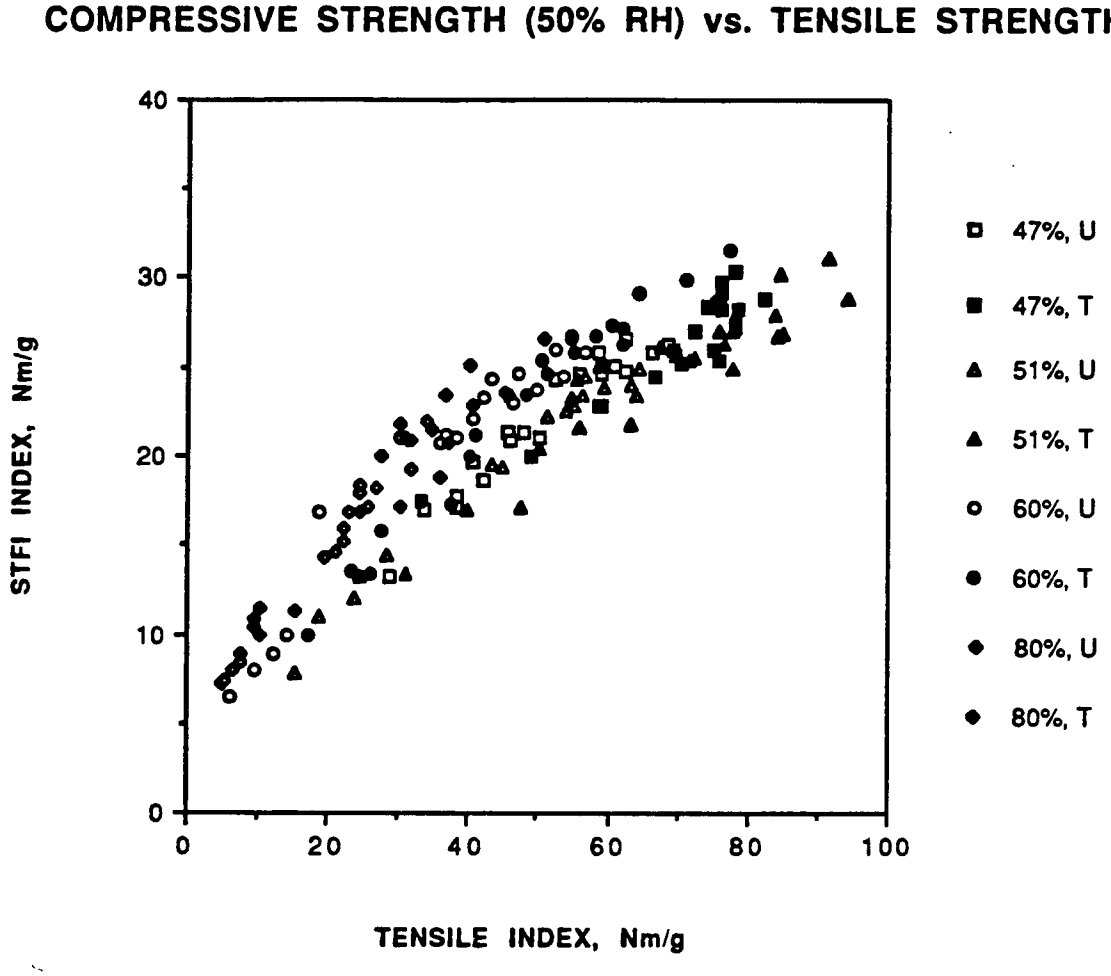


Figure 22. STFI compressive strength plotted against tensile strength for treated (closed) and untreated (open) sheets.

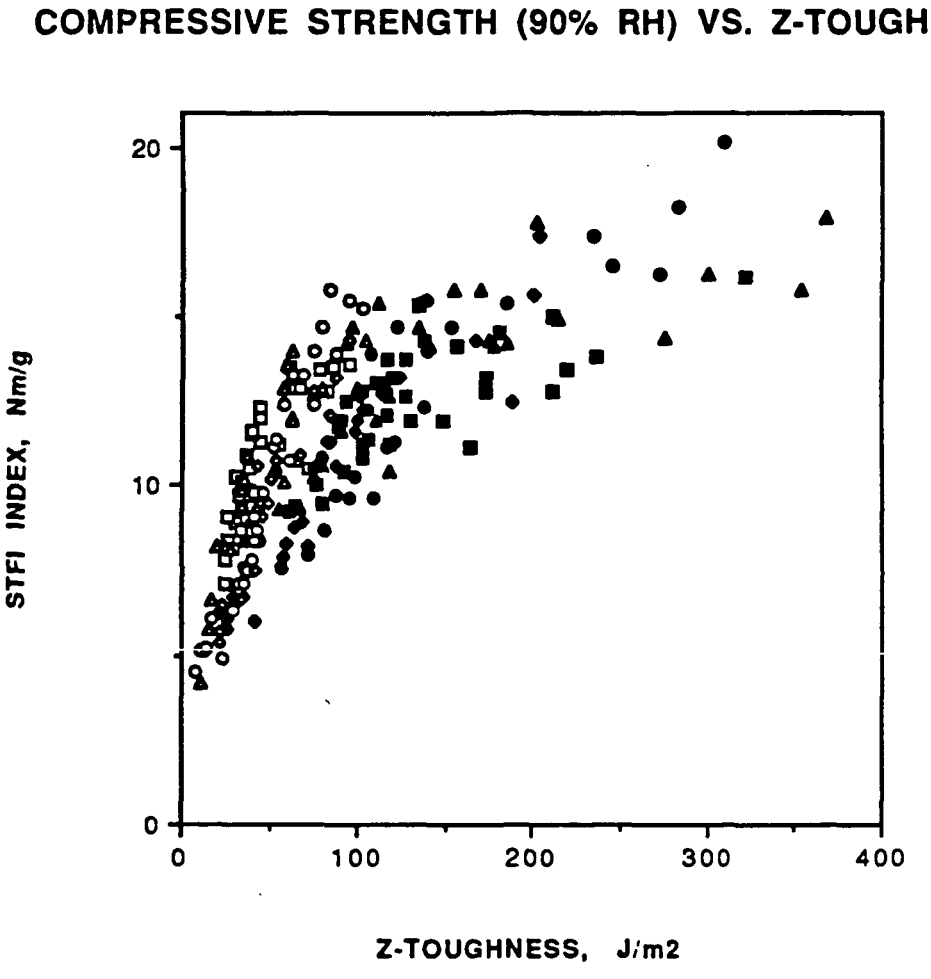


Figure 23. STFI compressive strength measured at high relative humidity plotted against Z-toughness for treated (closed) and untreated (open) sheets.

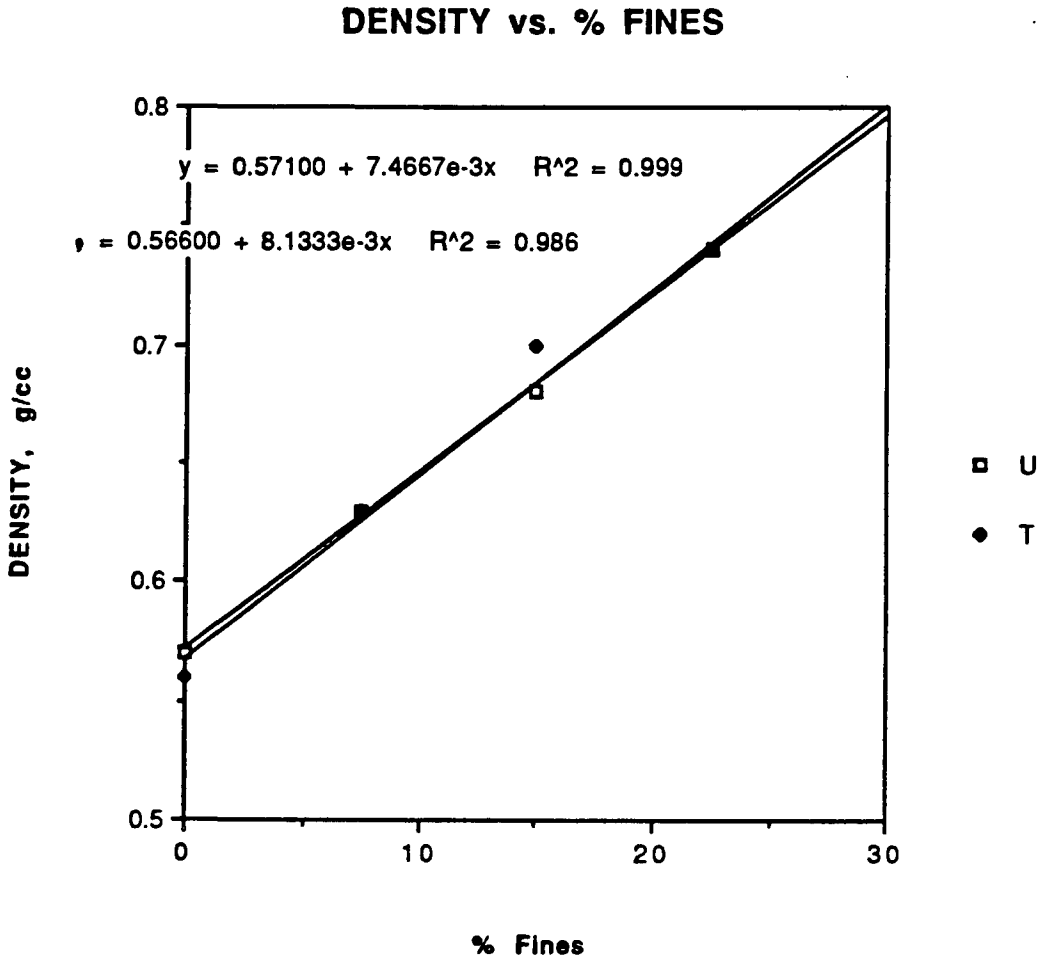


Figure 24. Density plotted against percent added fines for treated (closed) and untreated (open) sheets.

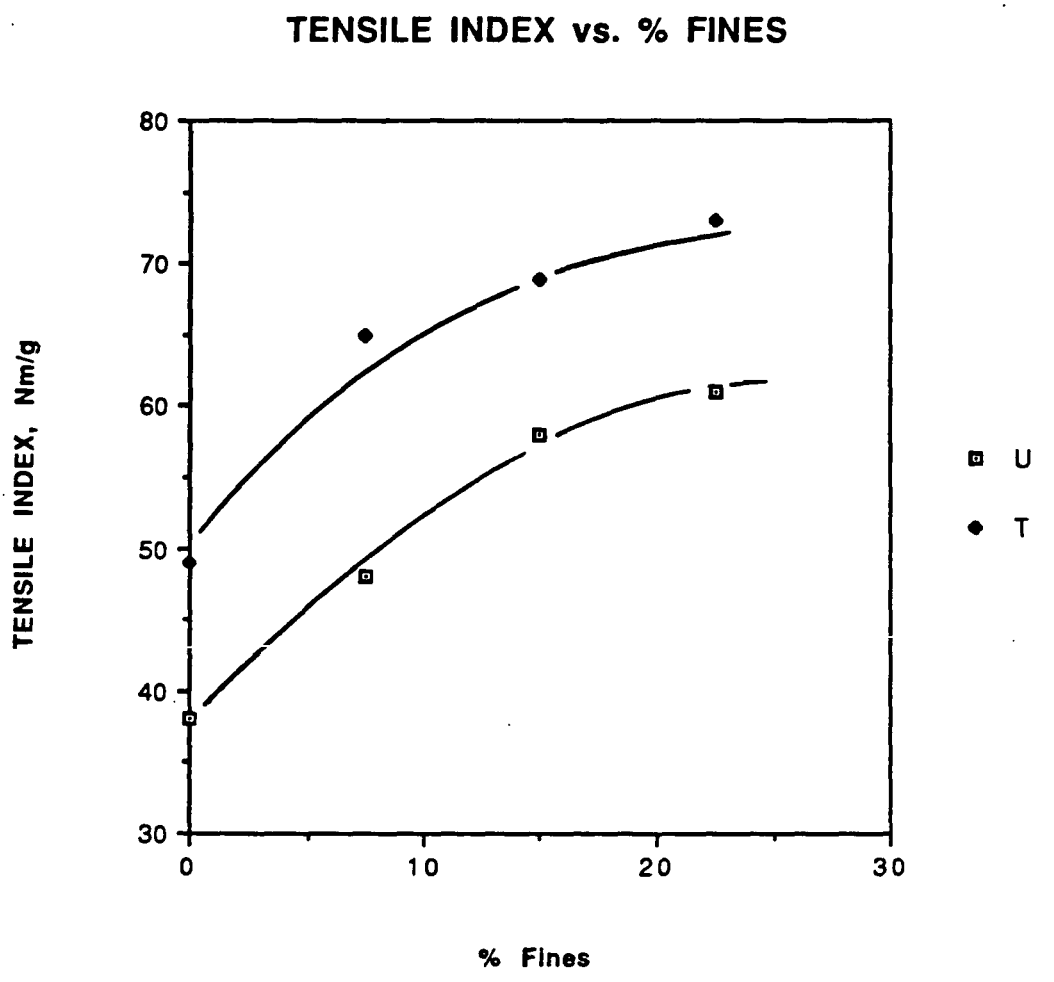


Figure 25. Tensile index plotted against percent added fines for treated (closed) and untreated (open) sheets.

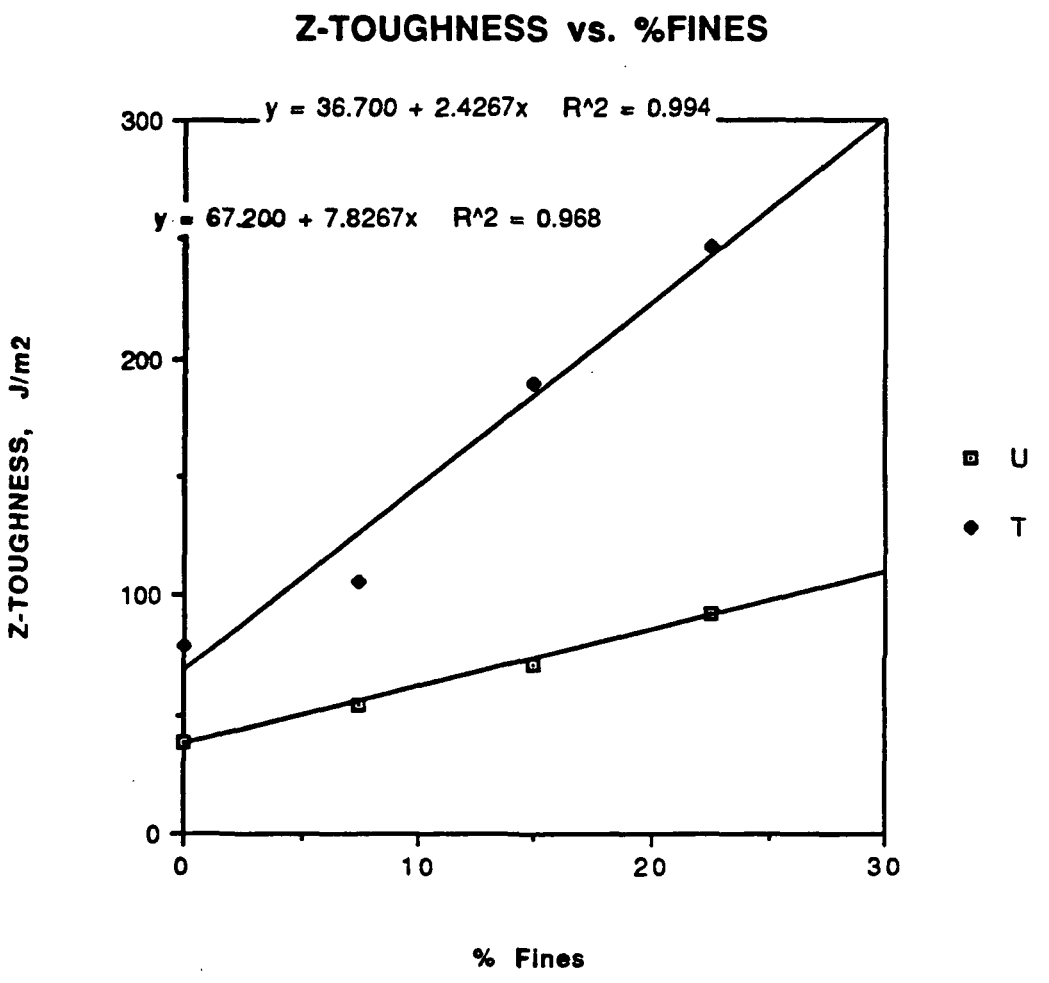


Figure 26. Z-toughness plotted against percent added fines for treated (closed) and untreated (open) sheets.

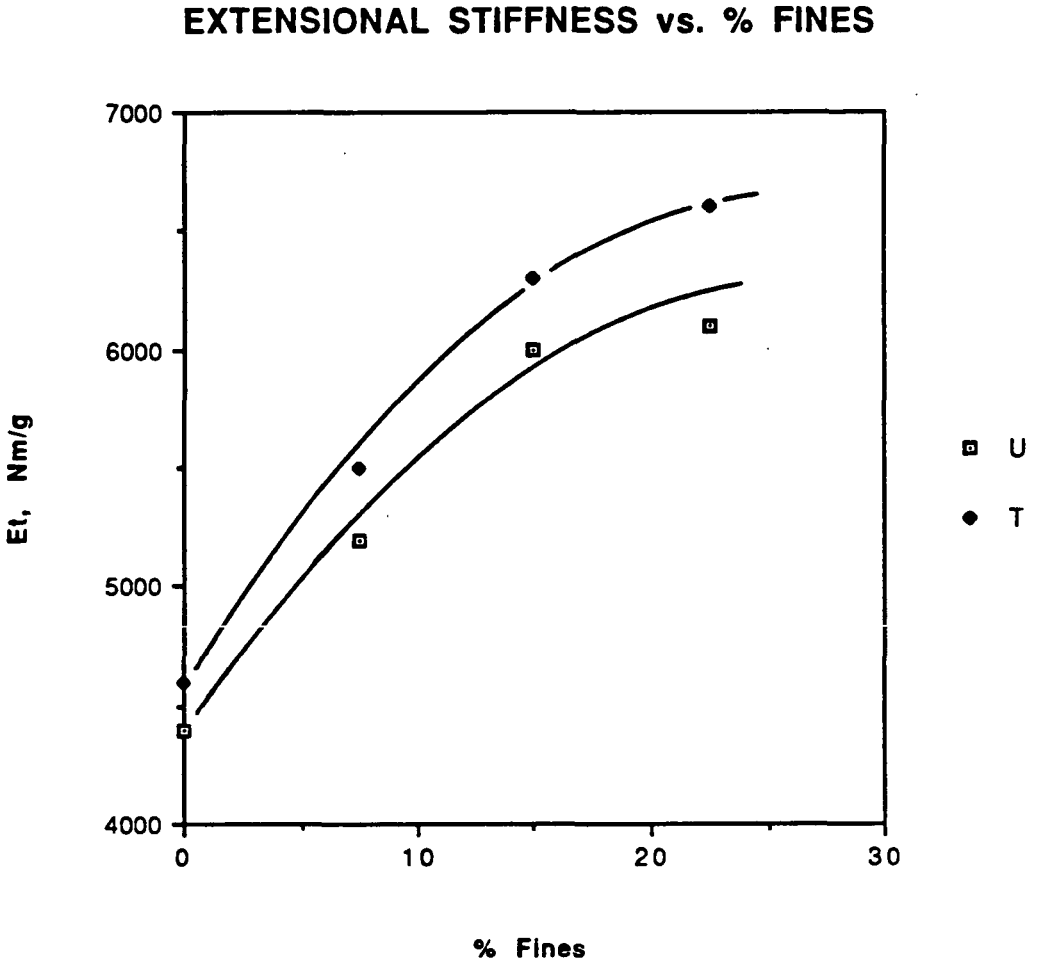


Figure 27. Extensional stiffness index plotted against percent added fines for treated (closed) and untreated (open) sheets.

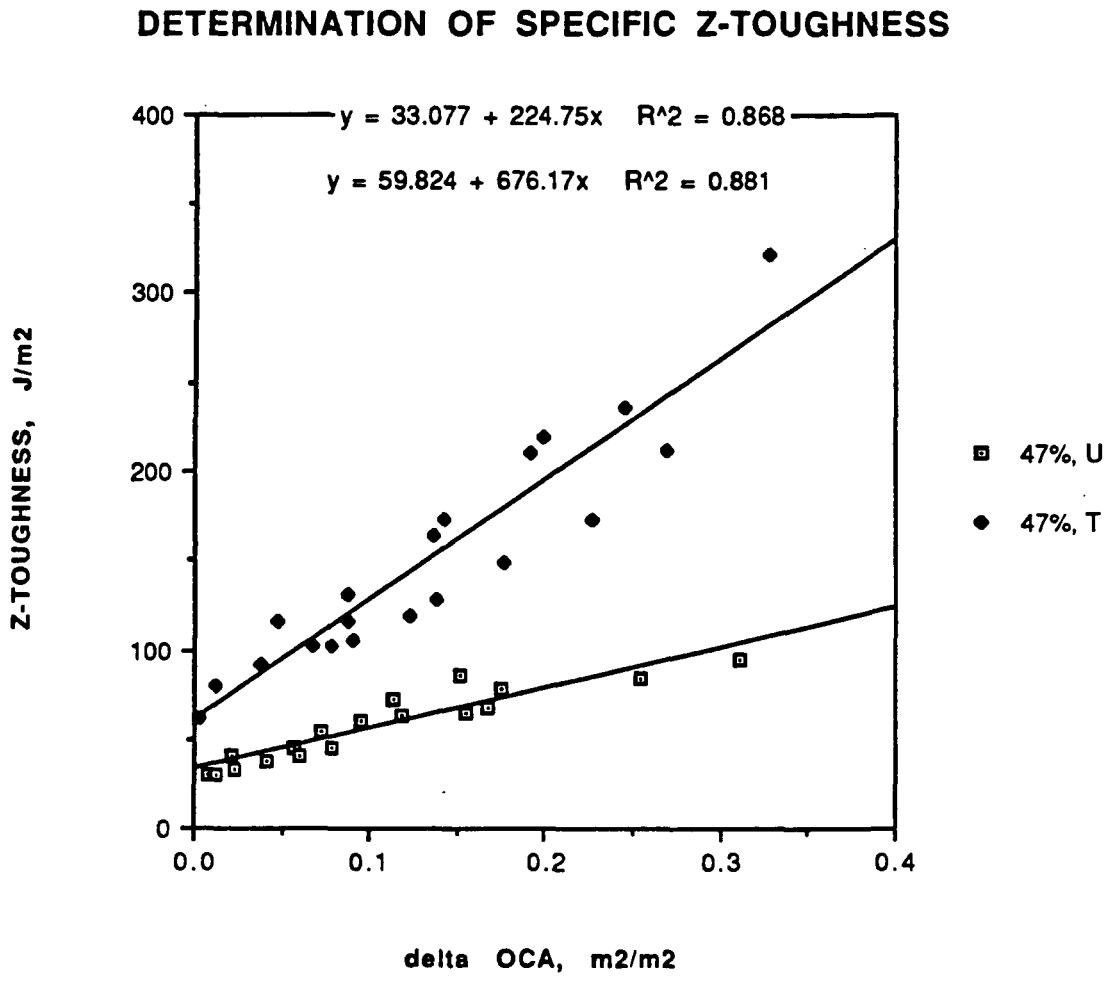


Figure 28. Plot of Z-toughness and the change in optical contact area per unit geometric area for treated (closed) and untreated (open) sheets.

SPECIFIC Z-TOUGHNESS

	SG_z, J/m^2	
YIELD	UNTREATED	TREATED
47	230	670
51	330	800
60	280	990
80	300	590
AVERAGE:	280	760

Figure 29. Summary of specific Z-toughness results.

BOARD PROPERTIES AND PERFORMANCE

STATUS REPORT

FOR

PROJECT 3571

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

March 21, 1990

PROJECT SUMMARY

DATE: March 2, 19990

PROJECT: 3571 - BOARD PROPERTIES AND PERFORMANCE

PROJECT STAFF: R.L. Ellis/C. Smith/M. Kangas/M. Lorenz

PRIMARY AREA OF INDUSTRY NEEDS: Properties related to end use.

PROGRAM AREA:

Performance of properties of paper and paperboard.

PROGRAM GOAL:

Develop relationships between critical paper and board property parameters and determine how they are achieved in terms of raw materials, sheet structure and processing conditions.

PROGRAM OBJECTIVES:

To develop relationships between container performance, combined board properties, and component properties. To improve the performance/cost ratio of combined board, linerboard, and medium. The short term goals are to (1) use structural models to assess the impact of papermaking factors on combined board and box performance and (2) improve medium end use in converting performance properties.

PROJECT RATIONALE:

There are many aspects of container and component performance which have not been adequately related to board properties through sound structural models. Such models would identify the critical board properties needed for end use performance. They would be used to select papermaking approaches to maintain or improve box performance at less cost. The important step is to incorporate the elastic stiffness of the board into performance models. This will enable us to use our knowledge of how papermaking factors affect the elastic properties of board. A knowledge of the treatability characteristics of both linerboard and medium on the single-facer would enable a manufacturer to choose the critical factors to control during board production. Finally, an improvement of our understanding of the stresses developed during flute formation will allow us to improve the performance of corrugating medium.

SUMMARY OF RESULTS:

Experimental work has shown that the angular distribution of elastic modulus is important in understanding the warp characteristics of combined board. If the angular distribution of the single-face and double-back liner differ by more than 10 degrees substantial twist warp occurs in the combined board. During the past period, we have investigated methods to analyze the CD profile of polar angles. The approach is based on fitting a polynomial to the CD profile and abstracting from the polynomial certain measures of polar angle which can be used to compare different paper machines. To date we have found that a linear relationship is a satisfactory representation of the data. The variation about the linear regression, the slope of the linear regression, and the translation of the linear regression

from the center line of the machine are the three measures used to compare different machines. Our analysis shows that machines generally have low random variation. The analysis indicates a "spreading" flow pattern from the headbox dominates the development of polar angles. Two machines show some translation which suggest misalignment of the headbox and the wire or a spread headbox flow.

Our studies to investigate the impact of dry strength additives on combined board strength is complete. We found that the dry strength additive improve the overall strength of corrugated medium and that this strength improvement is reflected in the combined board properties of edge crush and flat crush. We found that the strength improvement is equivalent for different processing methods including refining, wet pressing, and internal dry strength additives.

PLANNED ACTIVITIES FOR FISCAL 1990-1991

During 1990 we will expand our study of the impact of process variables on polar angle for linerboard and medium. During 1990 we will study the impact on galls in the wood press and drying restraint on elastic modulus. We expect to find that the axis of symmetry for these affects is different from those of fiber orientation. Analysis of the shape of polar angles diagram will allow us to asses the impact. Assessing control of the impact of wet straining and drying restraint. During 1990 we will study the impact on elastic modulus of wet press draws and dryer restraints. We will also study the impact of high temperatures and moisture on medium performance. The experimental results will be utilized to improve our model of the corrugated process and should allow for improvement in single-face runnability.

We will begin anew the use of finite element methods to study flute damage in the corrugating operation.

Finally, during 1990 we will initiate studies into the impact of cyclic humidity on the components of corrugated boxes. The primary emphasis will be placed on studying the role of the adhesives and the interaction between the adhesive and the board.

STATUS REPORT

BOARD PROPERTIES AND PERFORMANCE

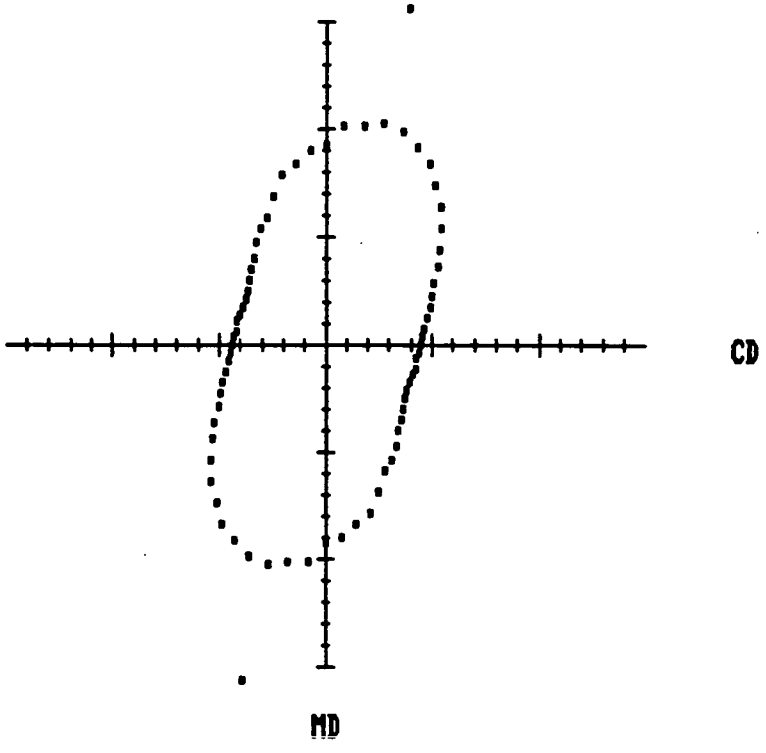
Project 3571

The objective of this project are to : (1) Develop relationships between container properties, combined board and component properties, and (2) to determine ways to improve the cost/performance ratios of medium and linerboard. To fulfill this objectives both end use performance and runnability on the corrugator are being considered. Our current work is divided into several parts namely: (1) Combined board warp vs. liner directionality effects, (2) liner and medium improvement, (3) runnability modelling

COMBINED BOARD WARP - LINER ORIENTATION EFFECTS

The research conducted by the Paper Physics Group has allowed us to rapidly measure the elastic stiffness of linerboard as a function of angle from the machine direction. The results are plotted as a polar diagram and give information concerning headbox flows, jet to wire differential speeds, conditions in open draws, and drying restraint. The angle of the polar diagram, shown in Figure 1 is related to the average fiber orientation machine. The details of the shape of the diagram are related to wet straining and drying restraint. Figure 2 is an example of how the polar angle profiles varies by position across the machine. Our work in the past period has been directed at developing a qualitative representation of this profile and comparing different machine based on the measures of the representation.

ANGLE TO PRINCIPAL AXIS OF MOMENT OF INERTIA = 14.3° MD IS ALONG Radius
AREA = 157.6 (km^4/sec^4) AVERAGE RADIUS = 7.08 (km^2/sec^2)
MD-CD STIFFNESS RATIO = 2.07



PLOT OF VEL SQR VS ANGLE AS SEEN FROM FELT SIDE
(Graph Scale = 1 km^2/sec^2/div)

Figure 1.

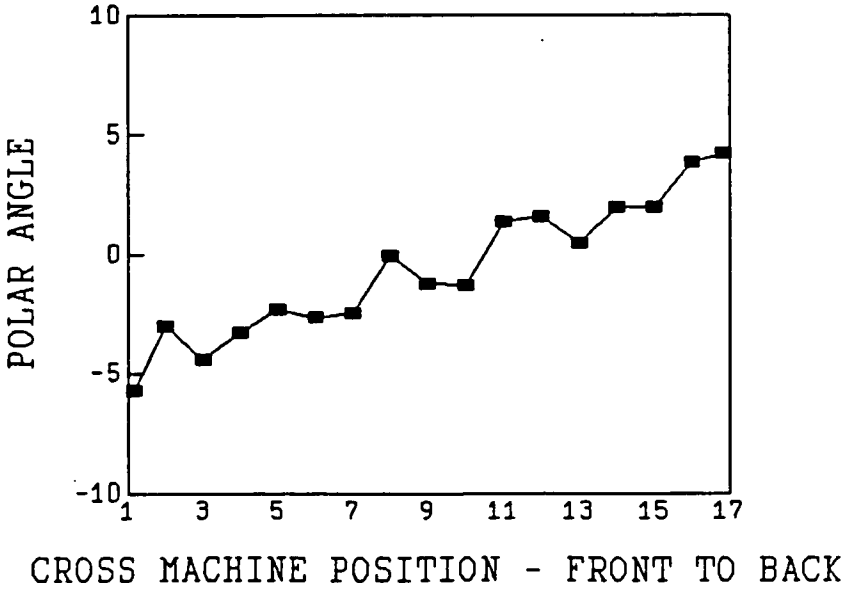


Figure 2.

Figure 3 is a schematic representation of the polar angle profile showing only random variability in the cross machine direction. Such a pattern could be characterized by simply calculating the standard deviation around the horizontal axis. This would be similar to calculating the standard deviation of the CD profile of moisture and basis weight. Figure 4 is a schematic representation of one of the patterns we have observed. In this pattern the CD profile is translated by a positive shift in polar angle. As shown in Figure 5 this profile could be generated by a difference in the bulk flow from the headbox as compared to the wire. Such a flow could be generated by a physical misalignment of the headbox to the wire or by a asymmetric pressure drop in the headbox header. The consequences of such a flow pattern are shown schematically in Figure 6. For the pattern shown we would measure a positive polar angle for every CD position.

Another pattern we have observed is a rotation around the center line of the machine. Such a rotation is shown schematically in Figure 7. For this pattern the random slice-to-slice variability will still exist but the entire CD profile is rotated. The angle could serve as an index for machine comparisons. Figure 8 is a schematic representation of a flow pattern that could cause the rotation. In this situation we have divergent flow from the headbox relative to the constant direction of the wire. In fact we expect this type of flow to occur on most headboxes. The placement of the machine deckles will determine the degree to which "flow spreading" can occur. Flow spreading such as that shown in Figure 8 would appear in polar measurements shown in Figure 9. On the front of the machine we would measure negative polar angles relative to the machine direction. In the center of the machine there would be a zero polar angle and the back of the machine would show a positive polar angle.

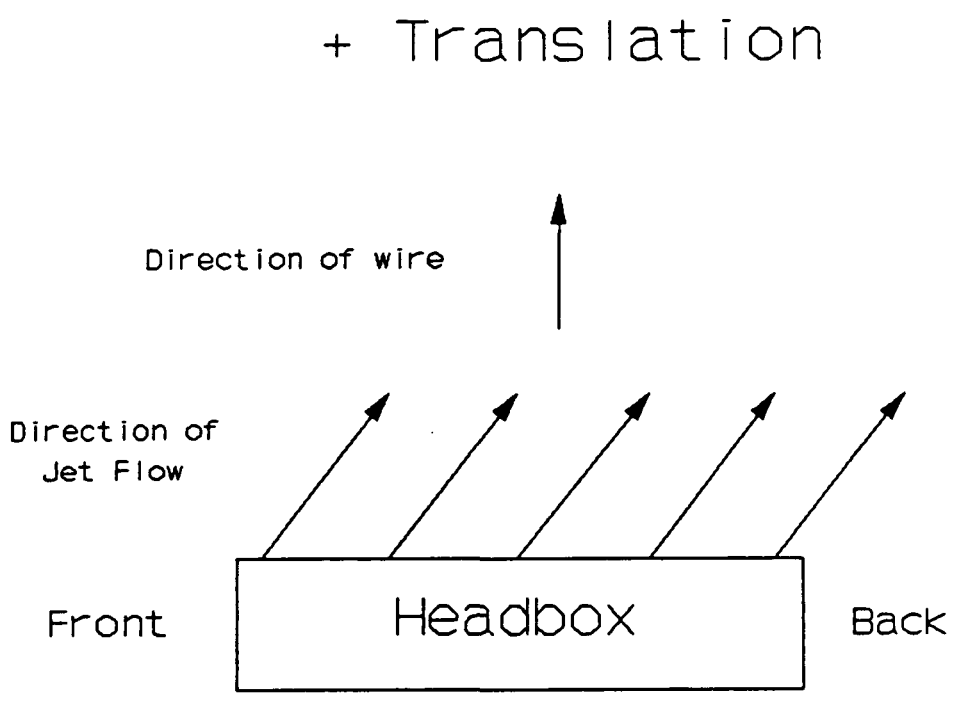


Figure 5.

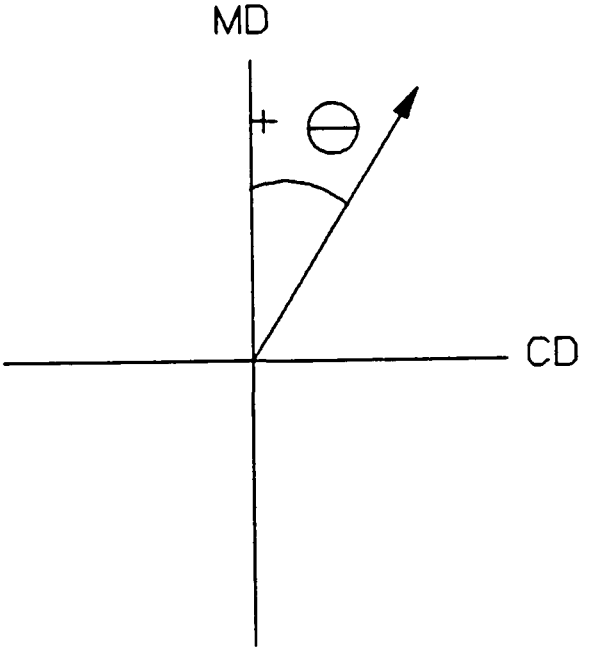


Figure 6.

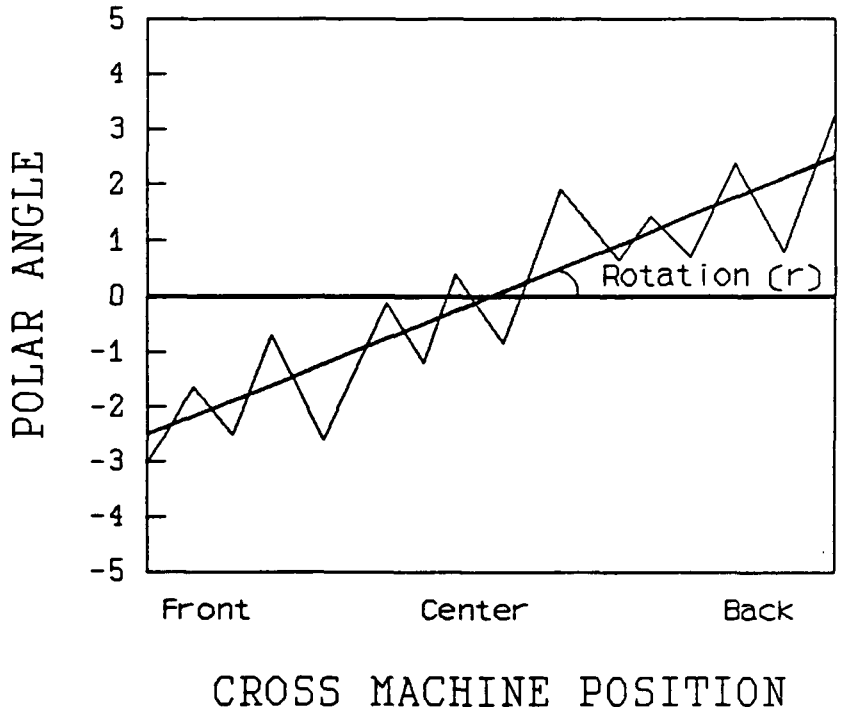


Figure 7.

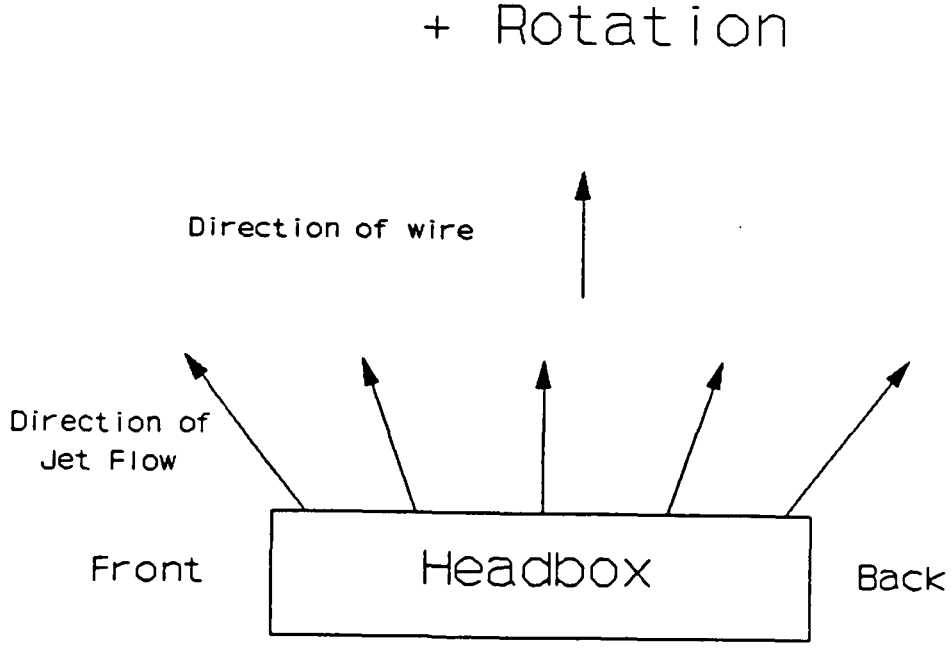


Figure 8.

All of the machines we have studied to date show a pattern in which the polar angle vs. machine position fits a straight line. Analysis of this pattern allows us to generate three measures for a machine.

1. Translation, t

2. Rotation, r

3. Variation, σ

The pattern analysis is performed by fitting a straight line of the form $y = mx + b$ to the data. Where y is the polar angle and x is the cross machine distance. The specific equation is :

POLAR ANGLE = M(cross machine position) + b

The three measures of this pattern are:

1. TRANSLATION = M (center position) + b

2. ROTATION = arctan(M)

3. VARIATION = standard deviation around the regression

Figure 10 shows a typical pattern that exhibits all three measures of variability. Analysis of the straight line shows that the translation is equal to 5.7 degrees of polar angle and that the rotation parameter is 34 degrees of arc. Figure 11 is another pattern showing only rotation of the profile about the center line of the machine. The translational parameter while statistically significant was only about minus 1 degrees. The arc of rotation was 37 degrees.

Table 1 lists the translation, rotation, and variability of all the machines studied to date. Table 2 summarizes the results. The range of the random variation, σ, was between 0.72 to 2 degrees polar angle. These are very small polar angles and we would not expect variation of this magnitude to impact on the performance of corrugated board with respect to twist warp. We found that the range of translation was from zero to +8.5 degrees and from zero to -5.2 degrees. In each case only one machine was greater than 5 degrees. These results suggest that translation will not be an important overall contributor to twist warp. The rotation of the entire profile ranges from zero to +37 degrees or from zero to -30 degrees. Seven machines showed a positive rotation of greater that 20 degrees. This positive rotation implies flow spreading leaving the headbox. Two machines showed a negative rotation and we speculate that this negative rotation is due to heavy deckle waves. We have selected a number of machines to study in more detail and these are listed in Table3. Machines 01-01 and 06-01 were chosen because they are machines with large translational parameters. Machine 05-01 will be studied in more detailed because of its unique feature when running 69 lb linerboard. We see that all translation and rotation disappears from the pattern, however, this machine has the largest residual standard deviation when running heavy weight.

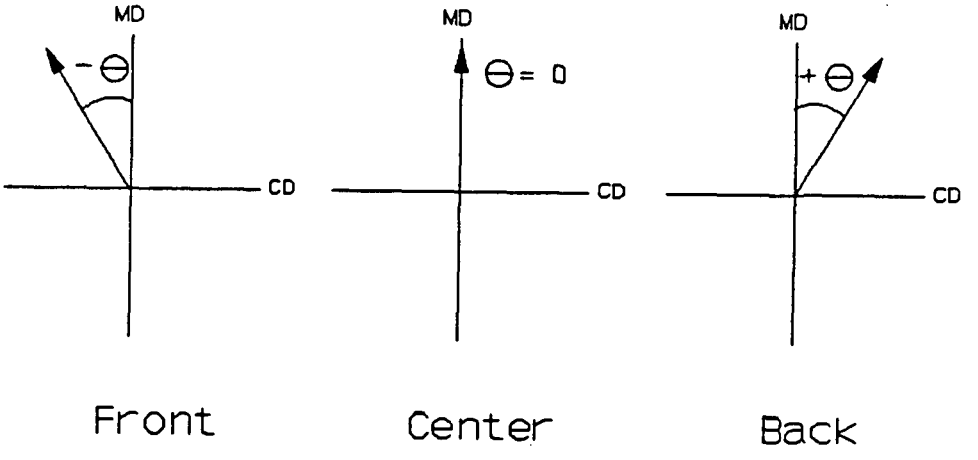


Figure 9.

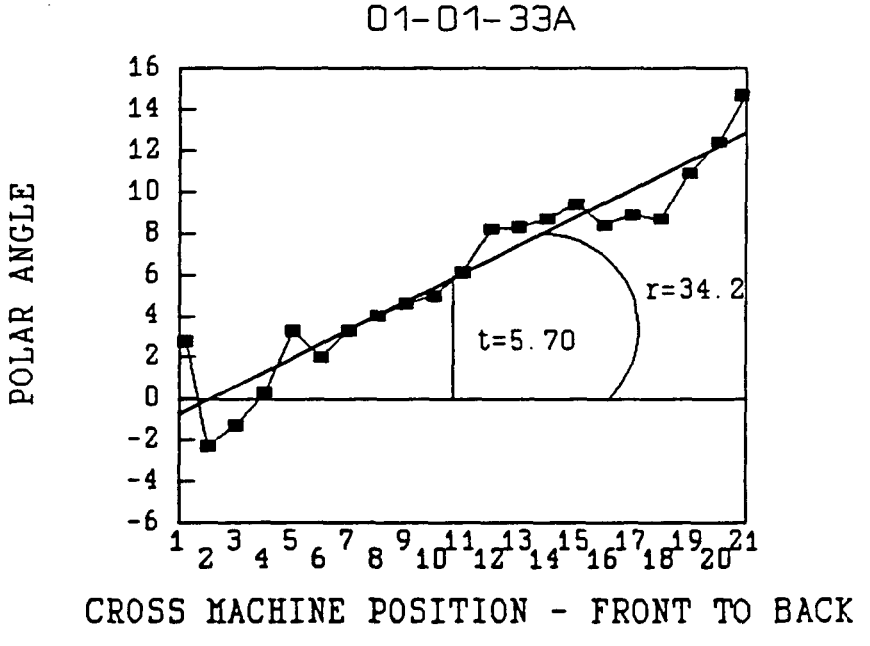


Figure 10.

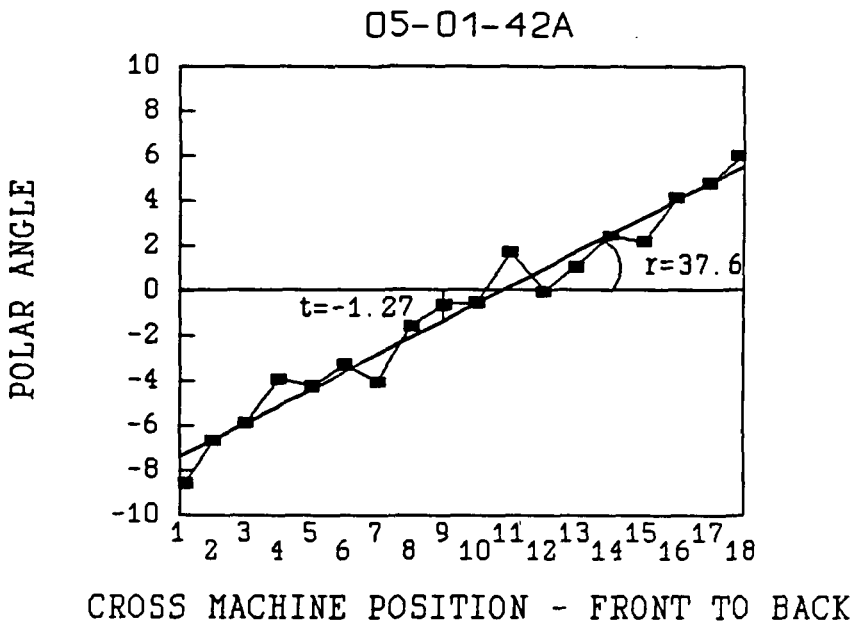

05-01-42A
POLAR ANGLE
10
8
6
4
2
0
-2
-4
-6
-8
-10
1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18
t=-1.27
r=37.6
CROSS MACHINE POSITION - FRONT TO BACK

Figure 11.

Table 1. Statistically significant values for translation, rotation, and variation of samples tested.

Sample	Translation t	Rotation r	Variation sigma
01-01-26A	8.19	33.4	1.21
01-01-33A	5.70	34.2	1.20
02-01-26A	-0.93	10.2	0.88
02-01-33A	-0.58	22.8	0.72
03-01-26A	2.38	N.S.	1.35
05-01-33A	-0.52	33.4	1.30
05-01-42A	-1.27	37.6	0.90
05-01-69A	N.S.	N.S.	2.00
06-01-42A	-5.31	17.2	1.41
06-01-69A	-4.82	20.8	1.49
08-01-33A	2.50	N.S.	1.21
08-01-42A	1.89	-21.3	1.17
08-02-42A	0.44	11.3	1.15
08-02-69A	-1.56	N.S.	1.27
09-01-42C	0.59	11.9	1.36
10-01-26A	-0.66	-30.1	1.19
10-02-33A	-1.37	31.0	0.96
10-02-42A	-0.22	27.5	0.99
11-01-69A	-0.90	28.8	0.92
16-01-26A	1.09	25.6	0.97
16-01-33A	1.89	N.S.	0.99
18-01-42A	N.S.	N.S.	1.62
18-02-42A	0.29	18.8	1.58

N.S. - Not statistically significant

SUMMARY OF RESULTS

	RANGE	NO. OF MACHINES
TRANSLATION		
	+ 0 TO + 8.2	1 (>5)
	- 0 TO - 5.3	1 (>5)
ROTATION		
	+ 0 TO +37.6	7 (>20)
	- 0 TO -30.1	2 (>20)
VARIATION		
	0.72 TO 2.00	

Table 2.

TABLE 3

SAMPLE	TRANSLATION t	ROTATION r	VARIATION σ
01-01-26A	8.19	33.4	1.21
01-01-33A	5.70	34.2	1.20
05-01-33A	-0.52	33.4	1.30
05-01-42A	-1.27	37.6	0.90
05-01-69A	N.S.	N.S.	2.00
06-01-42A	-5.31	17.2	1.41
06-01-69A	-4.82	20.8	1.49
16-01-26A	1.09	25.6	0.97
16-01-33A	1.89	N.S.	0.99

FUNDAMENTALS OF PAPER SURFACE WETTABILITY

STATUS REPORT

FOR

PROJECT 3646

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

March 21, 1990

PROJECT SUMMARY

Project Number: 3646 Fundamentals of Paper Surface Wettability

Project Staff: F. Etzler

Program Goal:

Develop an understanding of the interaction of liquids with water structure and paper materials in a fundamental way and the influence of this interaction on paper properties.

SUMMARY OF RESULTS LAST PERIOD (March 1989 - October 1989)
Work on utilization of statistical geometry to understand liquid structure continues.

A device for introducing water to porous materials under vacuum has been constructed.

Heat capacity measurements in 242A diameter silica pores have been measured. The results suggest that vicinal water structure persist to high temperatures and that structural transitions occur near 15, 30, 45^{o}C as has been suggested earlier by Etzler and Drost-Hansen.

A review of the literature suggests that dynamic surface tension effects detergent solutions. Effects on detergent solutions are important to the penetration of these solutions.

SUMMARY OF RESULTS THIS PERIOD

Seteram DSC has been installed and calibrated in Atlanta.

Testing for fresh method of loading of water into silica pores is in progress.

Manuscript on heat capacity of water in 242 A diameter silica pores has been submitted for publication.

Manuscript on Melting of Ice in Silica Pores has appeared in print Langmuir 5, 1439 (1989)].

Review paper on water in polymeric materials has been submitted for publication in "Water Relations in Foods" H. Levine and L.Slade eds. Cellulosic materials are of interest to both the paper industry and food industry.

Analysis of Voronoi Polyhedra in SPC/E Water continue to provide exciting results concerning the bimodal character of water. Our analysis suggests many new methods for data analysis of the results obtained from molecular dynamics studies and many provide further insights into the liquid state.

Several manuscripts relating to our theoretical effects are in preparation for publication.

Image analysis of first printing trial at Jefferson Smurfit Corporation have been performed. The results are consistent with earlier conclusions.

A second printing trial employing inks of various static surface tensions has been scheduled for February 20, 1990 in Chicago.

FUTURE WORK

It is planned to:

1. Continue measurements of the heat capacity of water in silicas of various pore diameters and in cellulosic materials.

2. Continue exploration into the distribution of Voronoi volumes and its relation to liquid structure. It is hoped that such an analysis will help to increase understanding of interfacial liquids.

3. Investigate possible experiments which may lead to an understanding of how the physico-chemical details a surface may influence the structure of water.

4. Investigate the feasibility of making direct force measurements between cellulosic materials. It is hoped that such measurements would yield information regarding the effects of vicinal water on interparticle forces. (This may relate to paper properties)

5. Investigate the suitability of thermoporometry for use on cellulosic materials. Thermoporometry is a technique for determining pore sizes for materials immersed in liquids such as water. This technique will be important for detailed investigations of water in cellulosic

PROJECT TITLE: FUNDAMENTALS OF PAPER SURFACE WETTABILITY

Date: 11/1/88

Budget: $110,000

PROJECT STAFF: F. Etzler

Period Ends: 6/30/90

Project No.: 3646

PROGRAM GOAL:

Develop an understanding of interactions between liquid and paper and their dependence on sheet structure and composition.

CURRENT OBJECTIVE:

To provide a fundamental understanding of the structure and properties of interfacial water and their relation to the properties of paper and board.

PROJECT RATIONALE:

Many converting and end uses of paper and board are associated with the application of a liquid to a surface. These include the processes of printing, coating, production of combined corrugated board, surface sizing and the exposure of the product in use, to a variety of liquids. In many cases (printing, coating, etc.) the phenomena of interest occur on short time scales. To improve various processes and point the way to new products, it is important to understand the influence of sheet structure and surface properties on the interactions between liquids and cellulosic materials.

RESULTS TO DATE:

The properties of water near a variety of surfaces have been found to differ from the bulk. For instance, in 7nm radius silica pores the heat capacity of water is found to be about 2-3% lower than that of the bulk. The properties of interfacial (vicinal) water may be understood in terms of statistical thermodynamic model proposed earlier [F.M. Etzler, JCIS, 92, 43 (1983), F.M. Etzler, Langmuir, 4, 878 (1988)]. The model suggest that propinquity to a surface increases the hydrogen

bond probability between water molecules. Experimental results suggest that the structural modification extends approximately 5 nm from the surface. Careful comparison of the measured properties of water adjacent to silica, clay and cellulosic materials suggests that the properties of vicinal water are nearly independent of the surface. Studies on model systems thus are likely to have direct impact on the understanding of water near materials of interest to the paper industry.

Data collected on the drop penetration time of isopropanol-water mixtures into kraft linerboard suggests surface wettability plays a major roll in printability.

PLANNED ACTIVITY FOR THE PERIOD:

It is planned to:

1. Begin calorimetric measurements of the heat capacities of water in model substrates and in cellulosic materials in order to understand better the nature of pore water.

2. Continue to explore the microscopic predictions of the model for vicinal water. Principally the prediction concerning the distribution of molecular volumes in vicinal water and bulk water will be studied using computer simulated water.

3. Investigate possible experiments which may lead to an understanding of how the physicochemical details of a surface may influence the structure of vicinal water.

4. Investigate the feasibility of making direct force measurements between cellulosic particles. It is hoped that such measurements would yield information regarding the effects of vicinal water on interparticle forces. (This may relate to paper strength, etc.)

5. Investigate the suitability of thermoporometry for use on cellulosic materials. Thermoporometry is a technique for determining pore sizes for materials immersed in a liquid such as water. The technique will be important for detailed investigations of water in cellulosic materials. The analysis is performed with a differential scanning calorimeter.

FUNDAMENTALS OF PAPER SURFACE WETTABILITY
Project 3646

Introduction

The objective of this project is to understand the interaction of liquids with paper materials in a fundamental way. It appears important to assess the current state-of-the art and suggest future research paths. The long-term objective of the project is to achieve a fundamental understanding of the role of liquid-paper interactions in papermaking and in determining paper properties. The current work has been directed to: 1) reviewing the literature regarding the present understanding of water-cellulose interactions; 2) comparing results from model systems with that known for water-cellulose system; 3) refining Etzler's statistical thermodynamic model for vicinal water, and 4) exploring the effect of paper surface chemistry on liquid penetration (particularly as related to flexographic printing).

The Structure of Water Near Surfaces

The nature of the liquid state, at the molecular level, continues to be a forefront topic of research in physics and chemistry. Despite the efforts of a considerable number of able researchers, much regarding the nature of the liquid state remains to be learned. Fortunately, much progress has been made in the last decade; this progress suggests that considerable advances will be made in the coming years. The nature of liquids near surfaces is, at present, receiving considerable attention by both experimentalists and theoreticians. A fundamental understanding of the processes important in determining the nature interfacial (vicinal) liquids has not been achieved. Indeed, considerable ignorance of the state of vicinal liquids exists. Many fundamental experiments are necessary in order for progress to be made.

As the state of water near cellulosic surfaces is of considerable importance to paper manufacture, it is important to discuss the current understanding of water near solid surfaces. A comparison of vicinal water in model substrates and in cellulosic substrates is also of importance.

It is known that the properties of water near surfaces are modified by propinquity to solid surfaces. To date the most comprehensive studies of interfacial water properties have been

water may be placed in pores of known size and geometry. It has been shown, for instance, that water in pores with a radii of 1 - 20 nm, exhibits a larger heat capacity, lower density and high viscosity. [See for instance, Etzler, F.M. Langmuir, 4, 878 (1988)]. From studies of water in clays and from density measurements of water in silica gel, it appears that water is structurally modified to distances of 3 - 6 nm. It also appears that the structural modification decays in approximately exponential manner with distance from the liquid-solid interface.

From work on a number of systems, it appears that the properties of vicinal water to a good first approximation are independent of the physicochemical details for the surface. For example, it has been shown that the heat capacity of water near a wide variety of surfaces is larger than the bulk. This aspect, however, deserves further attention.

The heat capacity of vicinal water appears to be a particularly useful quantity for understanding the nature of water in cellulosic systems; thus, this quantity is considered further. Stey (Ph.D. Thesis, University of Pittsburgh, 196?) has calculated the model independent distribution of single particle enthalpies for water and a number of other liquids. It was found that the enthalphy distribution for water is unusual in that the distribution is bimodal. This type of distribution contrasts with the nearly Maxwell-Boltzmann type distribution found for more simple liquids.

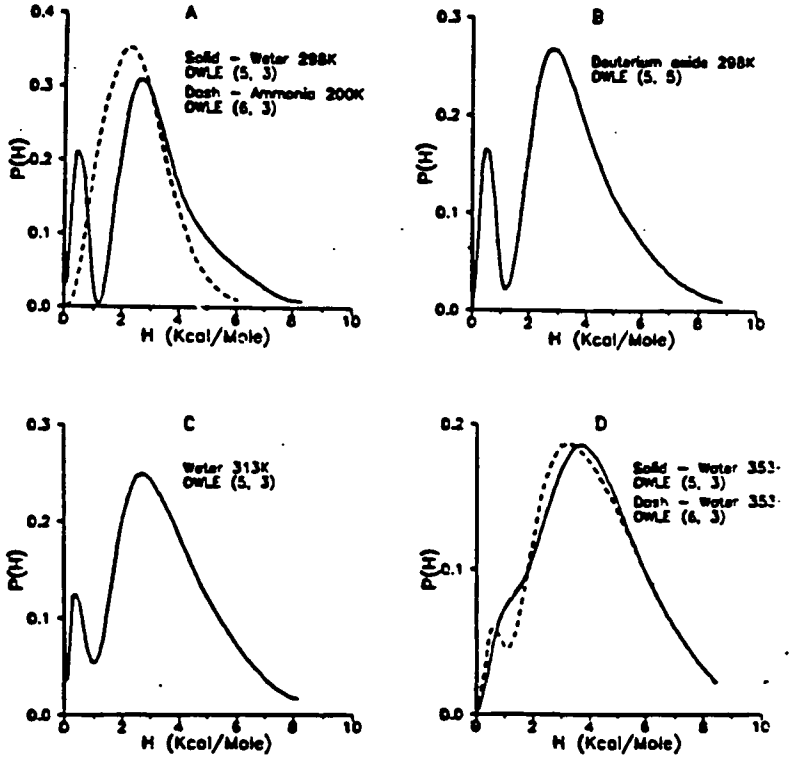


Figure 1. Stey's distribution functions. Probability, P(H), vs. enthalpy, H. (A) Water at 298 K and NH_3 at 200 K. (B) D_2O at 298 K. (C) H_2O at 313 K. (D) H_2O at 353 K.

Some of Stey's results are seen in Figure 1. It appears that the molecules represented by the low enthalpy peak in Stey's distribution are 4-hydrogen bonded water molecules while the high enthalpy peak represents molecules with 0, 1, 2, or 3 hydrogen bonds. The number of peaks in the distribution cannot be predicted from the number of hydrogen bonding states. It is generally presumed that the liquid state distributions would be nearly of the Maxwell-Boltzmann type.

The heat capacity, C_p is related to the variance, σ_h^2, of single particle enthalpies, through the well-known statistical thermodynamic relation:

$$C_p = (\sigma_h^2)/RT^2 \qquad (1)$$

For a bimodally distributed liquid the heat capacity may be considered as follows:

$$C_p = x(1)\, C_p\,(1) + x(2)C_p(2) + x(1)x(2)\,\frac{\Delta H^2}{RT^2} \qquad (2)$$

Here x(1) refers to the fraction of 4-hydrogen bonded water molecules. If it is assumed that hydrogen bonding is non-cooperative than x(1) equals the fourth power of the hydrogen bond probability between adjacent water molecules. $C_p(1)$ is taken to be the heat capacity of ice and $C_p(2)$ is estimated using a variety of experimental data, including, for instance, the activation energy of the rotational correlation time. At 298K $C_p(2)$ = 16 cal/K mole. ΔH is the mean enthalpy of transfer between the two peaks in Stey's distribution or 2.55 kcal/mole. $C_p(1)$, $C_p(2)$ and ΔH for deuterium oxide may also be estimated. At 298K approximately 6-10% of the water molecules in bulk water are 4 hydrogen bonded.

The heat capacity of water and deuterium oxide in silica pores of various diameters has been measured. The results are shown in Figure 2. A significant feature of the graph is the presence of the maxima near 7 nm pore radius. Figure 3 shows C_p as a function of x(1) as calculated from Equation 2. Significantly, Figure 3 suggests that vicinal water differs from the bulk in that hydrogen bond probability between adjacent molecules is enhanced by propinquity to solid surfaces and that the magnitude of the experimentally observed maxima may be calculated on the basis of Stey's earlier calculations. Density measurements on water in silica pores are in agreement with the heat capacity measurements and suggest that

hydrogen bond probability between adjacent water and molecules decays to the bulk value in an approximately exponential manner. Significant structuring extends 3-6 nm. The density of water in 7 nm radius silica pores is 2 - 3% lower than the bulk at 298K.

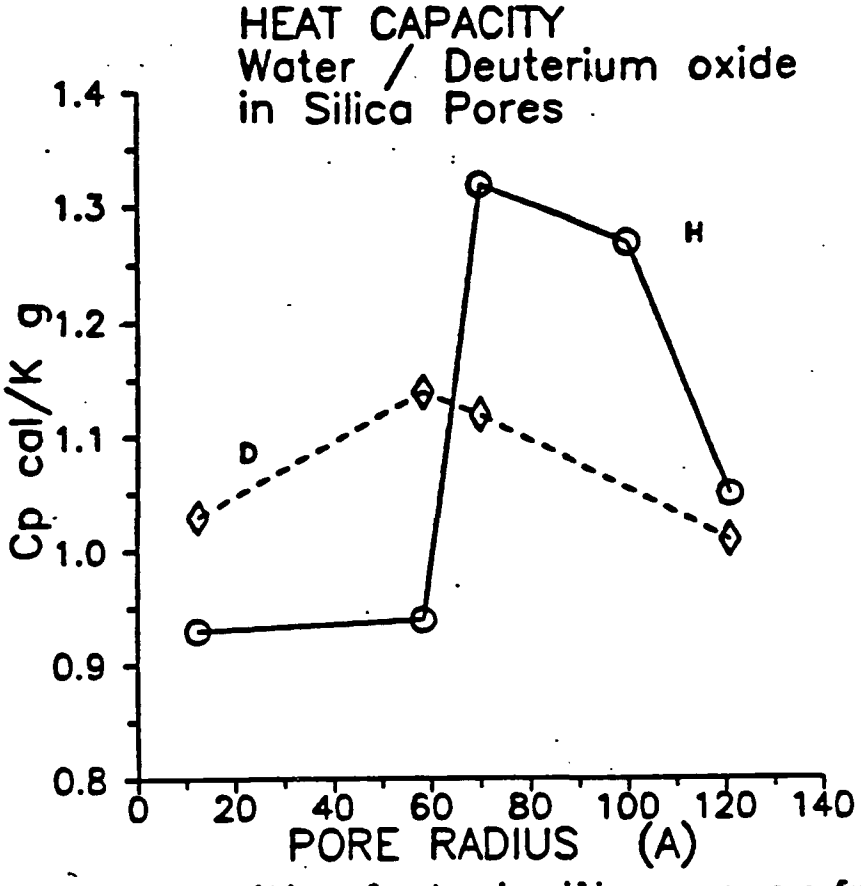


Figure 2. Heat capacities of water in silica pores as a function of pore radius at 298 K: squares H_2O; diamonds, D_2O. Radius in Angstroms (10 A = 1 nm).

Water in Cellulosic Materials

Measurement of the properties of water associated with polymeric substances is difficult as it is often impossible to separate polymer properties from water properties. Nonetheless several attempts have been made to measure the properties of water associated with cellulosic and other polymers. Early attempts to measure the density of water in wood suggested that the water had a density much larger than bulk water (Indeed much greater than the density of Ice VIII at 25kbar!). Other measurements suggested that the expansivity of water associated with cellulosic materials is less structured than the bulk. This

conclusion is in conflict with experimental evidence collected for water in clay and silica pores.

The use of thermal expansion as an indicator of vicinal water structure appears to be premature. It is not yet clear from statistical thermodynamics what the effect of enhanced hydrogen bonding between water molecules in pores would have on the magnitude of the thermal expansion coefficient. Reexamination of the apparent specific volume data for liquids associated with wood collected earlier by Weatherwax and Tarkow suggests that the high apparent density of water is due primarily to the opening of new pores (This is also an earlier conclusion of Weatherwax and Tarkow.) and that the density of water in the pores is 0.98 or 2% lower than the bulk if ethanol is treated as an unmodified pore liquid (Pore density equal to bulk density). This assumption is consistent with density measurements of water and alcohols in silica pores.

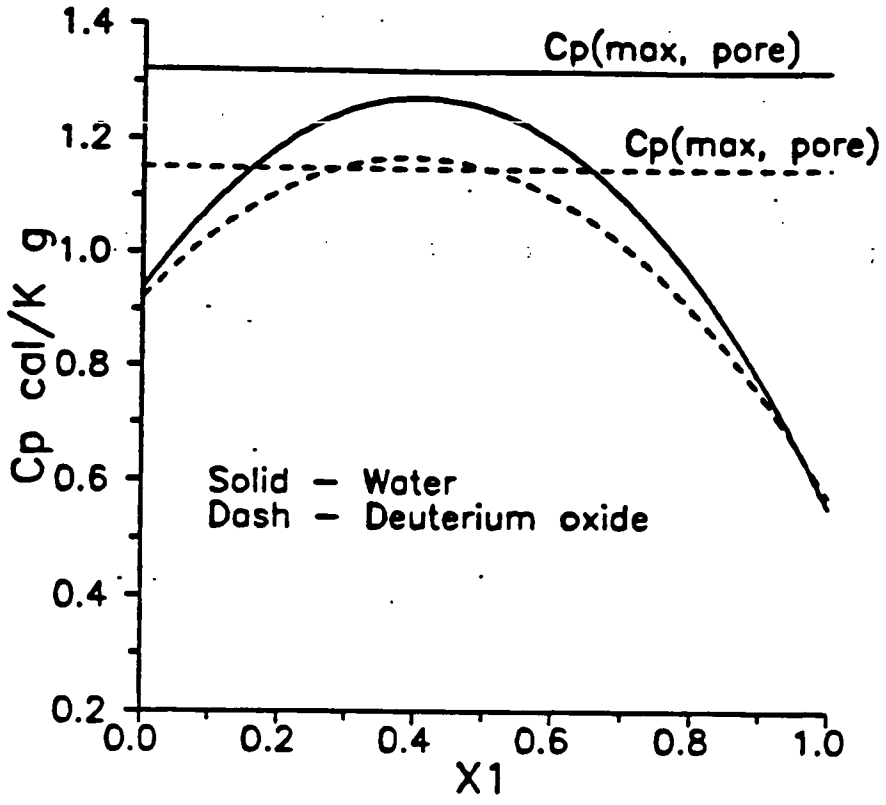


Figure 3. Hypothetical heat capacity of water and deuterium oxide as a function of x(1) at 298 K.

Figure 4 shows the apparent heat capacity of water in two kinds of wood. Figure 5 shows the apparent heat capacity of water in gelatin and starch suspensions. Both results are consistent with heat capacity measurements made in silica gel. In short, it appears that the properties of water associated with cellulosic materials and in silica gel are nearly identical and that water adjacent to either surface is more structured than the bulk.

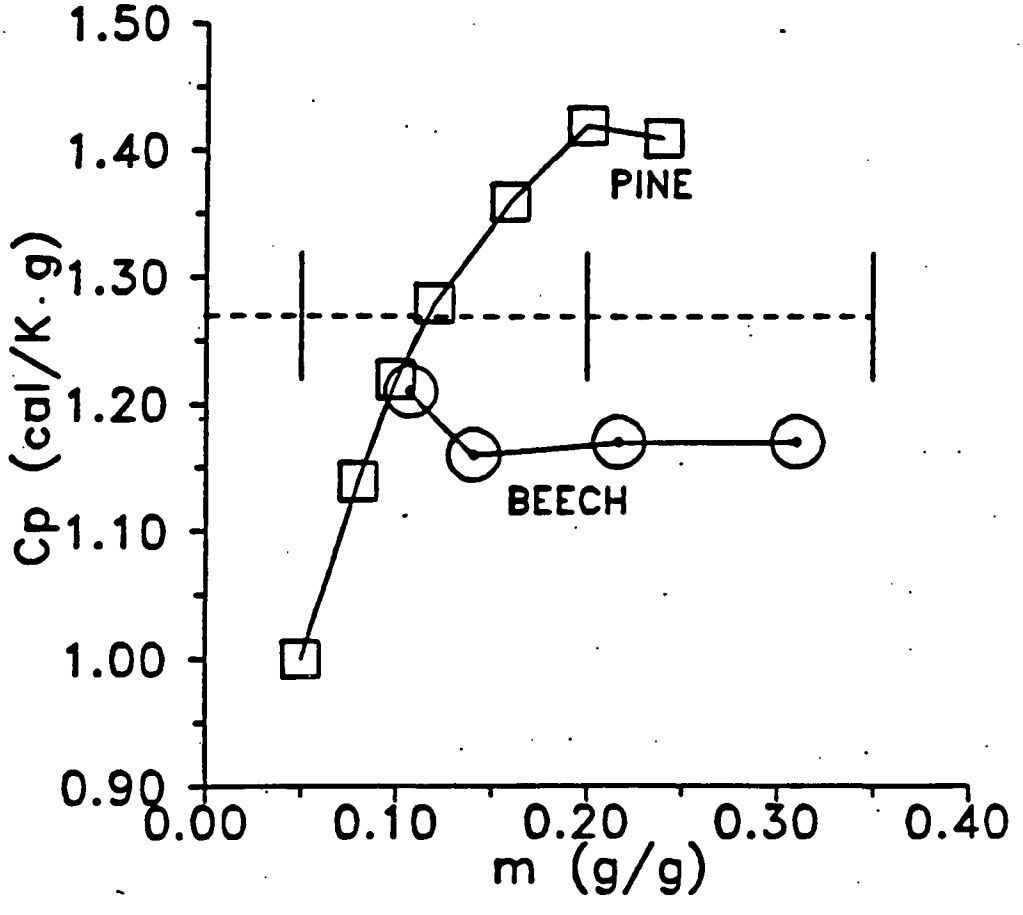


Figure 4. Apparent heat capacity of water in woods. Squares - pine; circles - beech; dashed line - maximum heat capacity as calculated from author's model.

Voronoi Polyhedra and the Structure of Vicinal Water

The results of Stey's calculations have been used successfully to correlate a number of thermodynamic properties of water near surfaces. Thus, it appears that Stey's calculated distribution can be used to make correct predictions of and correlations between macroscopic properties. Stey's calculation also makes predictions regarding the microscopic or molecular behavior of water. From thermodynamics Enthalpy, H = E + PV, where P is pressure, V is volume and E is energy. The single particle enthalpies discussed by Stey can thus be broken into two parts -- a volume part (Note: P = Constant) and an energy part. At present it is not clear whether energy or volume is the major factor in determining the form of Stey's enthalpy distribution.

It is not possible to calculate the distribution of molecular volumes in liquid via Stey's arguments without detailed knowledge of the intermolecular potential energy function. Knowledge of this function is not necessary, however, for the calculation of single particle

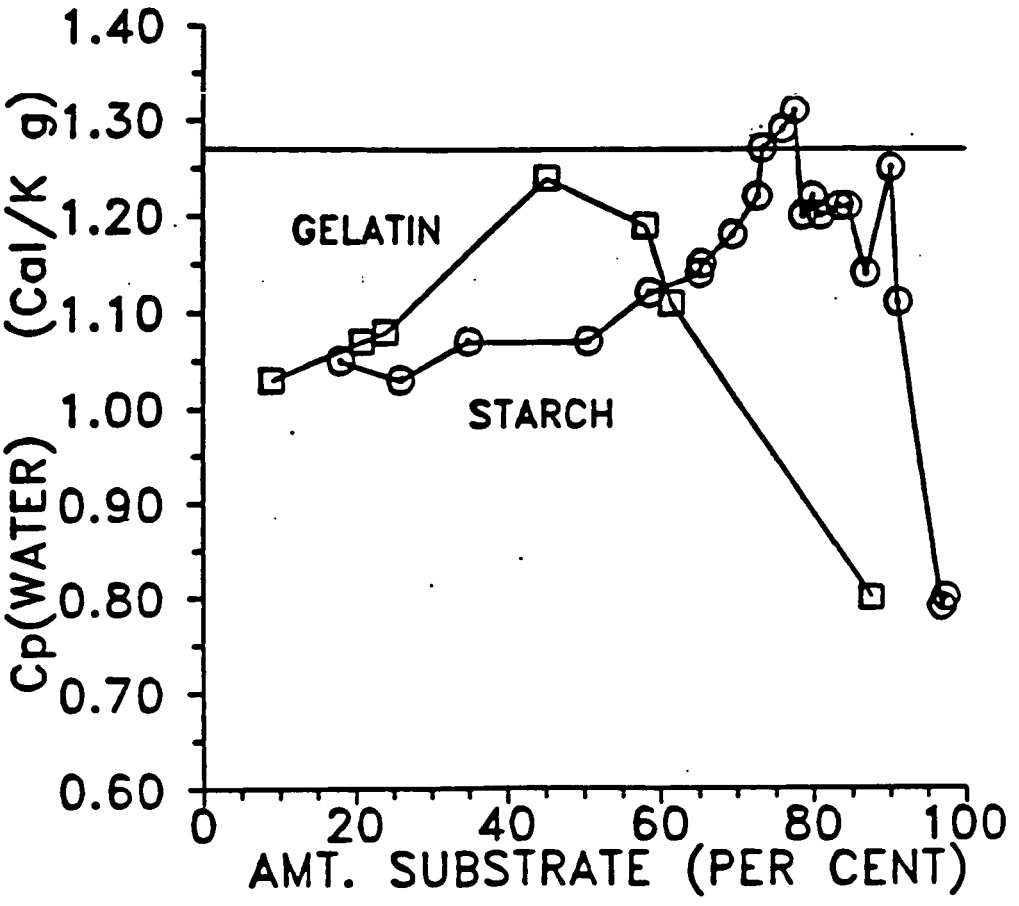


Figure 5. Apparent heat capacity of water versus per cent of substrate material in mixture. Squares - gelatin; circles - starch. Horizontal line - maximum heat capacity calculated from author's model.

enthalpies. It is possible, however, to estimate the distribution of molecular volumes from molecular dynamics calculations. The distribution of molecular volumes for liquid water is currently under investigation.

The volume of a molecule in a system can be regarded as the volume of the Voronoi polyhedra whose center is the molecular center of mass. The Voronoi polyhedra represents all points in space which are closer to a given molecular center of mass (or other reference point) than to any molecular center of mass. The temperature dependence of the isothermal compressibility for liquid water suggests that this distribution may have unusual features.

Our initial calculations concerning the properties of Voronoi polyhedra in computer-simulated water (SPC/E water) have been completed. Data on 30,000 polyhedra (3 sets of 10,000) have been collected. Figure 6 is a three-dimensional plot showing the probability of finding a polyhedron with a given number of faces and particular volume. The graph is unusual in a number of respects when compared to a simple liquid such as argon or Lennard-Jonesium. Two notable features are 1) the existence of two maxima in the distribution and 2) the high probability of 16-faced polyhedra. The abundance of 16-faced polyhedra is consistent with the existence of high-volume low energy (i.e. "ice-like") states is liquid water. The number of faces can be increased by lengthening the first neighbor distances while leaving the second neighbor distances essentially unchanged. The existence of two maxima give microscopic evidence which supports the earlier hypothesis of Roentgen and Frank suggesting simultaneous existence of "Bulky" and "Dense" regions in liquid water. The present calculation is important in that it presents the first microscopic evidence in support of Roentgen's hypothesis, first advanced nearly 100 years ago. It remains an important task to understand more clearly the interrelations between energy and volume which yield the bimodal enthalpy distribution calculated earlier by Stey.

Further analysis of the Voronoi polyhedra in SPC/E water, as seen in Figure 7 suggests that the two domains in the volume distribution are separated in energy as well as volume. A number of polyhedra types are found far more frequently in one or the other of the peaks.

These results suggest that the two peaks in volume distribution represent geometrically distinct domains.

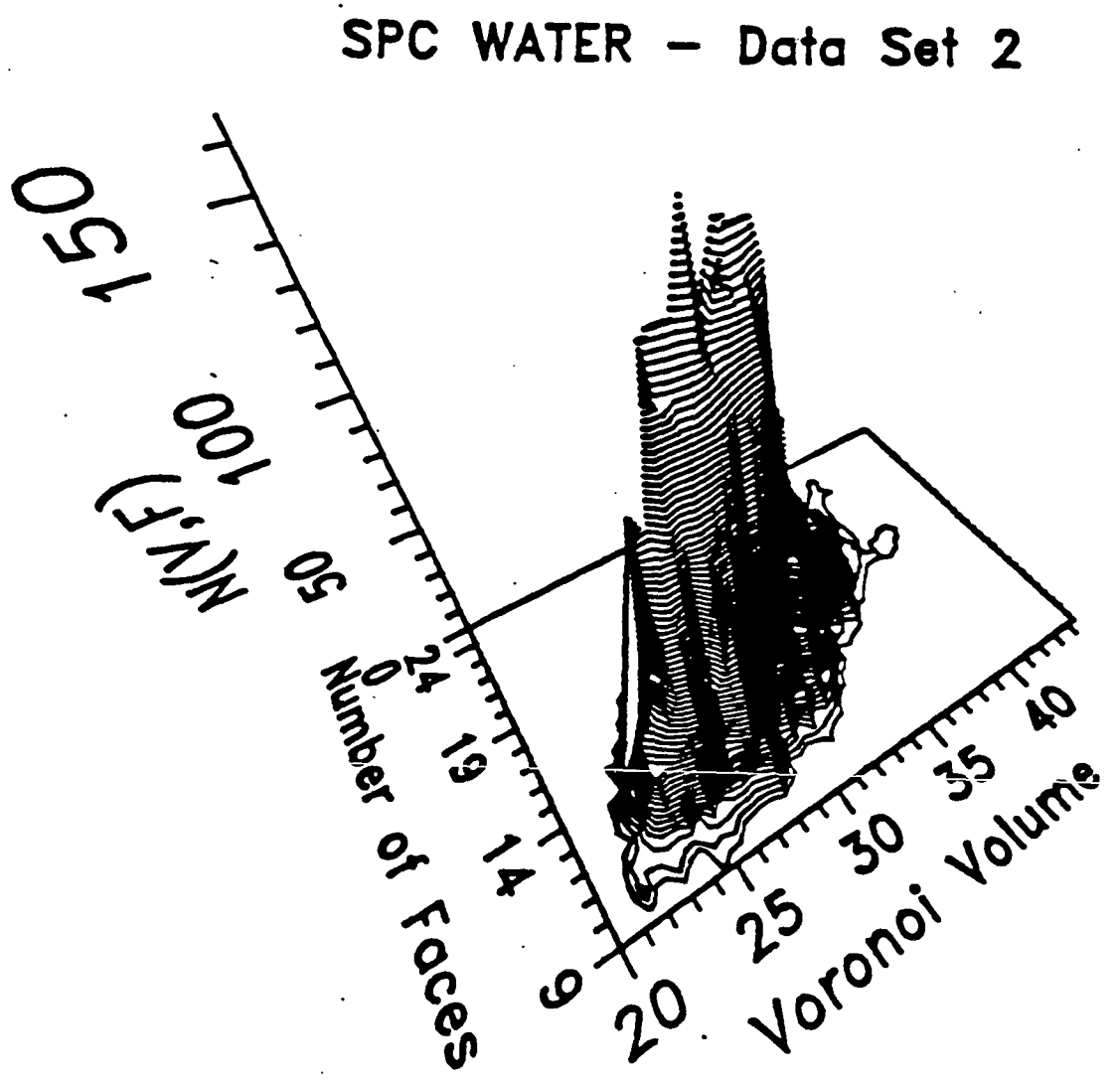


Figure 6. A three-dimensional plot showing the probability of finding a polyhedron with a given number of faces and particular volume.

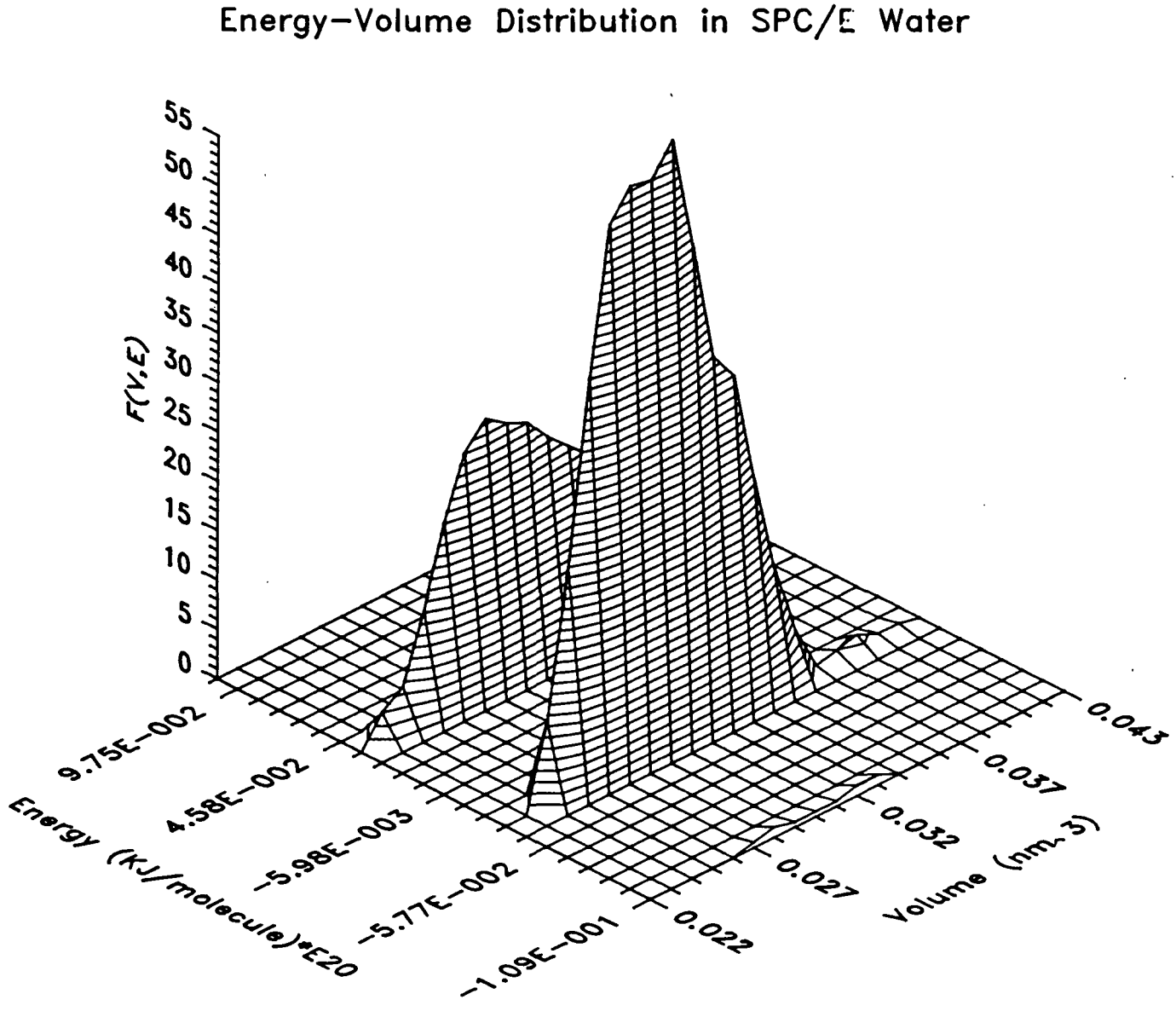

Energy–Volume Distribution in SPC/E Water
F(V,E)
55
50
45
40
35
30
25
20
15
10
5
0
9.75E-002
4.58E-002
-5.98E-003
-5.77E-002
-1.09E-001
Energy (KJ/molecule)*E20
0.022
0.027
0.032
0.037
0.043
Volume (nm^3)

Figure 7.

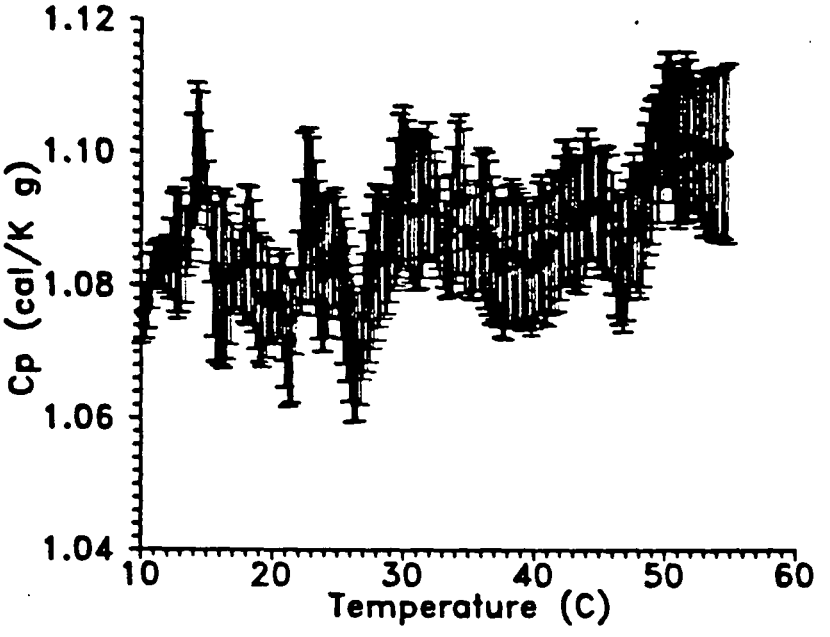


Figure 8. Heat capacity of water in 242 A diameter silica pores.

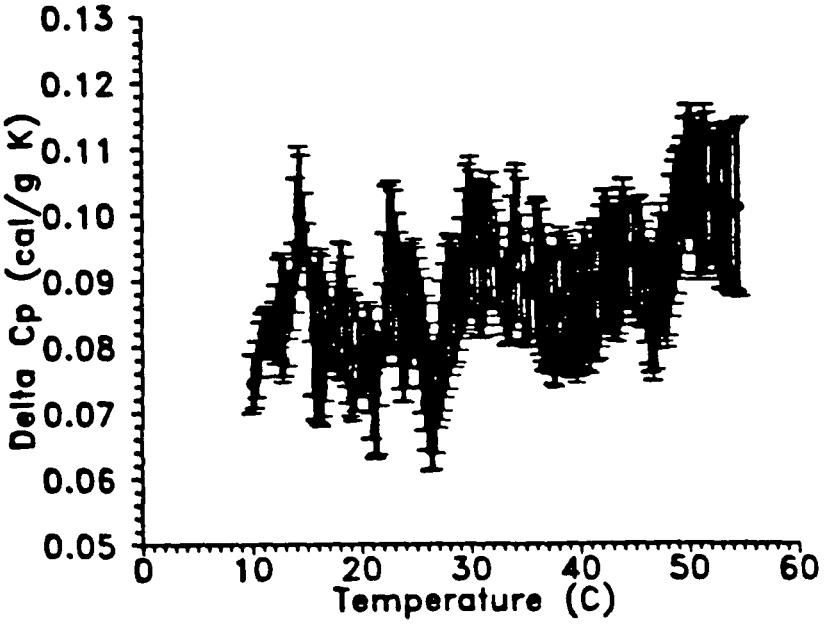


Figure 9. C_p (pore) - C_p (bulk) for water in 242 A diameter silica pores.

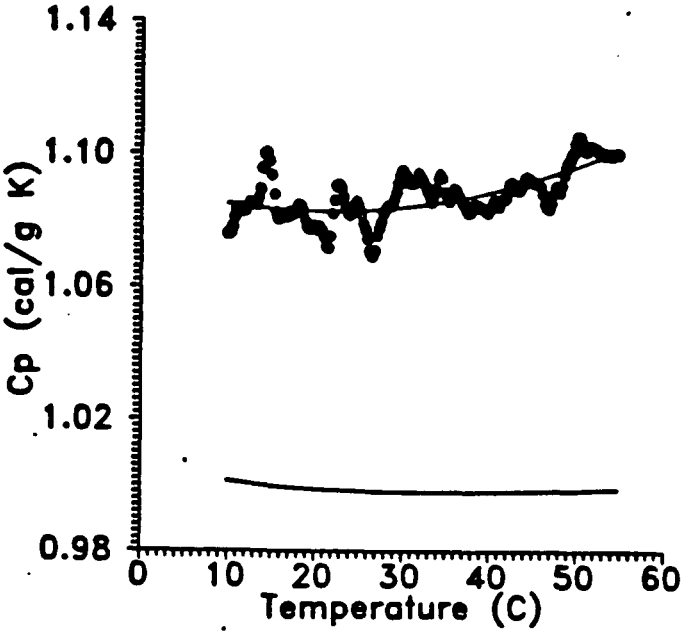


Figure 10. A comparison of C_p (pore) and C_p (bulk) versus temperature.

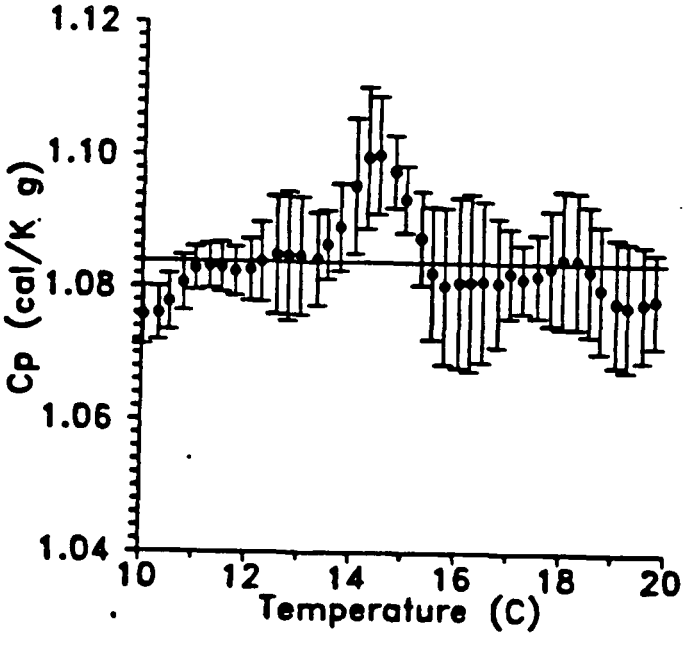


Figure 11. Detail over the temperature range from 10-20 C. Data as in Figure 7.

Methods for extracting information from the Voronoi analysis are being explored. This effort is taking us into unexplored territory.

Synthesis of Chemically Modified Surfaces

An understanding of the interaction of water with chemically modified surfaces will be undoubtedly important for achieving an understanding of a number of processes which occur at the liquid-paper surface.

Currently, the synthesis of silicas on which the normal surface hydroxyls are replaced by other chemical groups is underway. For instance it has been possible to place propyl amine groups on the surface. It is hoped that it will be possible to prepare chemically modified cellulosic surfaces.

The Temperature Dependence of the Heat Capacity of Vicinal Water

The Seteram DSC has been used to measure the heat capacity of water in 242 A diameter silica pores from 10-60°C. The results of calculations are found in Figures 8-11. The data indicate that significant structuring still occurs at rather high temperatures. Indeed ΔC_p increases slightly with temperature which suggests that bulk water structure breaks down more rapidly than vicinal water structure. Derjaguin has generally presumed that vicinal water structure breaks down by 70°C; clearly this is not the case.

More interestingly the data suggest the presence of "heat capacity spikes" near the Drost-Hansen temperatures. Drost-Hansen and later Etzler have suggested that physicological anomalies which occur near 15, 30, 45°C are the result of abrupt vicinal water structure changes. It is also known that ion selectivity by water confined to pores and that the viscosity of water between quartz plates both show unusual behavior consistent with structural transitions at the Drost-Hansen temperatures. The heat capacity spikes represent the first hard thermodynamic evidence for such vicinal water structure transitions.

Measurements of the heat capacity of water in smaller pores are planned and are expected to show more clearly the "heat capacity spikes."

An apparatus to facilitate the filling of small pores with liquids has been constructed.

Relation Between Independent Force and Vicinal Liquid Structure

The precise relation between vicinal liquid structure and interparticle force remains largely unexplored. Israelachvili et al. have, however, studied the forces between mica plates immersed in water and other liquids. This other group has discovered at least two forces not incorporated into DLVO Theory. These forces are the solvation force and the oscillatory force. The oscillatory force appear as very large oscillations in the interparticle force with a period equal to the molecular diameter near molecularity smooth surfaces.

The solvation force appears as an attractive force which is in addition to the van der Waals force incorporated into the DLVO Theory.

The solvation force is related to vicinal structuring and is typical 10-1000 times the van der Waals forces. Vicinal structure then may be responsible for the dominate attractive force between particles. [See Israelachvili et al. Macromolecules, 22, 4227 (1989)].

Penetration of Liquids into Kraft Linerboards

The penetration of liquids into kraft linerboards has been measured. The penetration of liquids into a porous substrate is governed by the Washburn equation:

$$\frac{dh}{dt} = [r/4\gamma h]\gamma_{LV} \cos\theta \qquad (3)$$

Here η is the liquid viscosity, θ is the contact angle and γ_{LV} is the liquid vapor surface tension and (dh/dt) is rate of penetration. According to the Washburn equation if a surface is not wet (contact angle > 90°) then the rate of penetration is negative which in turn implies that the liquid will not penetrate. If surface is completely wet (contact angle = 0) than the Washburn equation becomes:

$$\frac{dh}{dt} = [r/4\eta h]\gamma_{LV} \qquad (4)$$

thus penetration is governed primarily by viscosity and not surface energetics when $\theta = 0$. Drop penetration time measurements made on a large number of kraft linerboard samples suggest that paper surface energy may play a significant role in flexographic printing. Linerboard samples which are not wet by typical inks have been found to exhibit poor print quality.

ESCA (Electron Spectroscopy for Chemical Analysis) studies of various linerboard samples have been perfomed. Some correlations with wettability were found. Specifically, it was found that wettability correlated either with the amount of surface hydrocarbon materials or with the amount surface silicon. The silicon content which correlates with hydrophobicity presumably results from the presence of silicone compounds.

We have also noted that bleached linerboard show Cl at the surface and are generally more hydrophillic while linerboards which show S are more hydrophobic.

It appears that ESCA may be a useful tool for understanding the nature of paper surfaces. The details of the surface chemistry of the pulp from which paper is made, undoubtedly determine the properties of the resulting paper.

Printing trials using inks of various static surfaces tension are scheduled for February 20, 1990.

FUTURE WORK

It is planned:

1. Continue measurements of the heat capacity of water in silicas of various pore diameters and in cellulosic materials.

2. Continue exploration into the distribution of Voronoi volumes and its relation to liquid structure. It is hoped that such an analysis will help to increase understanding of interfacial liquids.

3. Investigate possible experiments which may lead to an understanding of how the physico-chemical details on a surface may influence the structure of water.

4. Investigate the feasibility of making direct force measurements between cellulosic materials. It is hoped that such measurements would yield information regarding the effects of vicinal water on interparticle forces. (This may relate to paper strength etc.)

5. Investigate the suitability of thermoporometry for use on cellulosic materials. Thermoporometry is a technique for determining pcre sizes for materials immersed in liquids such as water. This technique will be important for detailed investigations of water in cellulosic materials. This analysis will be performed on a differential scanning calorimeter.

6. Continue investigations into the use of ESCA as a tool for learning about the surface chemistry of paper.

7. Begin water vapor adsorption studies on paper and cellulose. In particular the relation of molecular events to paper mechanical such as cyclic humidity creep will be investigated.

UTILIZATION OF RECYCLED FIBER

STATUS REPORT

FOR

PROJECT 3681

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

March 21, 1990

PROJECT SUMMARY

PROJECT: UTILIZATION OF RECYCLED FIBER

PROJECT STAFF: Staff

PROJECT GOAL:

To develop the technological understanding necessary for a significant expansion in the use of recycled fiber from secondary sources.

CURRENT OBJECTIVE:

Examine the practical limitations on the expanded use of secondary fibers in various paper grades.

PROJECT RATIONALE:

The paper industry may be required through legislative or regulatory action to expand its utilization of recycled fiber in papermaking grades where such utilization is not now economically practiced. Some of the secondary fiber material may include recycled grades that are now considered to be of inferior quality, such as mixed grades and newsprint. The inferior quality of these raw materials is likely to be offset by their low cost and availability.

The impact of increased use of lower quality secondary fiber on the important properties of certain product grades such as writing papers, coating stock, linerboard, and the practical limitations on the use of secondary fibers of various types in such grades, have not been systematically explored. The purposes of this study are to characterize the degradation of important properties attendant on the use of non-traditional secondary fibers in non-tradition paper grades, determine the practical limits on the use of secondary fibers in various paper grades, and to identify processing alternatives that will permit the expanded

use of such fibers in various paper grades, and to identify processing alternatives that will permit the expanded use of such fibers without degradation of critical product properties.

PLANNED ACTIVITY FOR THE PERIOD:

A "white paper" will be prepared summarizing the critical issues arising from regulatory requirements for the use of secondary fiber resources. Paper grades likely to be targeted for increased utilization of secondary fibers will be identified for further study. Sources of various grades of secondary fibers prepared via state-of-the-art commercial practice will be identified. Laboratory paper samples will be prepared using furnishes involving amounts and types of secondary fibers on critical paper properties -- strength, tear, brightness, brightness stability, coating receptivity, etc. -- will be determined.