S T A T U S R E P O R T S

To The

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

October 18, 1989
Institute of Paper Science and Technology
Atlanta, Georgia

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October 16, 1989

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 PAC meeting scheduled for October 18, 1989, in Atlanta. A meeting agenda can be found inside the booklet.

We look forward to seeing you on October 18. Best regards.

Sincerely yours,

RLE/at
Enclosure

Richard L. Ellis, Director
Engineering and Paper
Materials Division

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AGENDA

PAPER PROPERTIES AND USES
PROJECT ADVISORY COMMITTEE

October 18, 1989
The Institute of Paper Science and Technology
Radisson Inn and Convention Center
Atlanta, Georgia

WEDNESDAY -- OCTOBER 18

8:00 AM	OPENING REMARKS	Yeske
8:30	Division Organization & Staff	Ellis/Hall
	Project Advisory Committee	Betz
8:45	PROJECT REVIEWS	
	Strength Improvement and Failure Mechanisms	Waterhouse
	Fundamentals of Paper Surface Wettability	Etzler
	Process, Properties, Product Relationships	Hall/Lorenz
	On-Line Measurements of Paper Properties	Hall
	Internal Strength Enhancement	Stratton
	Utilization of Recycled Fiber	Stratton
11:45	LUNCH/TOUR IPST	
1:30	COMMITTEE MEETING	
4:00	END OF PAC MEETING	

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ON-LINE MEASUREMENT OF PAPER

STATUS REPORT

FOR

PROJECT 3332

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

October 18, 1989

PROJECT SUMMARY

PROJECT NO. 3332: ON-LINE MEASUREMENT OF PAPER PROPERTIES

PROJECT STAFF: M.S. Hall, K.W. Lorenz

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

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

See attached 1989-90 Project Form.

SUMMARY OF RESULTS LAST PERIOD: (October 1988 - March 1989)

(1) Software has been developed to handle the on-line fluid-filled wheel operation.

(2) Special homemade transducers have been constructed for the fluid-filled wheels.

(3) A procedure for sealing the immersion transducers has been adopted.

(4) Improvements in the mounting of the bender transducers have reduced their sensitivity to web tension.

(5) IPST PVDF, on-line, wheels have been built. These should stand up to the rigors of operation on a moving web.

(6) The diamond coating, now in process, will hopefully lead to extremely wear-resistant bender transducers.

SUMMARY OF RESULTS THIS 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 liquid-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.

PROJECT TITLE: ON-LINE MEASUREMENT OF PAPER

PROJECT STAFF: M. Hall

Date: 11/1/88

Budget: $50,000

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.

Status Report

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 in-plane and thickness directions of paper while the paper is moving at line speed on the paper machine. A further objective is to use these sensors to develop hardware and techniques to control the paper manufacturing process. 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. 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 currently 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. Work with both transducer approaches is continuing.

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

New IPST wheel-transducers have been designed and built which incorporate harder, more durable elastomer front-faces, and higher temperature, more sensitive PVDF piezoelectric films.

A 386-type computer is being used for our on-line work. The software necessary to support the on-line operation of the fluid-filled wheels is complete. 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 in order to integrate the in-plane and ZD measurements to provide simultaneous data collection on a moving web.

A portable test stand for mounting both fluid-filled wheels and the IPST wheels has been ordered and is being built. This test stand will have cross web positioning, wheel-to-web speed matching,

portability, and adjustable nip pressure. The purchase order was placed in mid-September and delivery is expected in January.

Concurrently we have placed an order with the same firm to construct an unwind/rewind system. 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 pilot-machine prepared webs in the laboratory. We are reviewing the price increase associated with increasing our maximum test speed to 3000 fpm.

ELASTOMER FRONT-FACED, PVDF WHEEL-TRANSDUCERS

The first set of wheel-transducers, constructed with piezoelectric polyvidnylidene fluoride (PVDF or Kynar), provided encouraging results. With proper calibration, time-of-flight and caliper could be determined at the highest speed available (about 180 ft/min). However, the design of these wheel could not be expected to withstand the high speeds and temperatures routinely encountered on-line. Therefore, with the objective of designing and building more rugged wheel-transducers, a second set was built.

The new wheel design incorporates harder, more durable rubber front-faces, and higher temperature, more sensitive PVDF piezoelectric films.

In addition, improvements were made in the uniformity of the piezoelectric element and the front-face. The wheel construction is essentially as before.

A PVDF film is clamped between a Kynar core and an undersized Kynar ring, and an elastomer is applied to the contact surface of the Kynar ring. Instead of the standard PVDF film, a new experimental PVDF copolymer film (VF2-VF3 from Pennwalt Corp.) was used. This provides for higher temperature performance, as the film can be subjected to 100°C without significant depolarization. It also has approximately a 50% higher coupling coefficient. Two wheels were fabricated, one with a stack of two 220 micron thick films, and one with a stack of four 110 micron films.

A major challenge in the development of these wheel transducers is the fabrication of the elastomer front-faces. One needs a durable interface that efficiently couples acoustic energy between the wheels and paper. We decided to use a hard polyurethane rubber, that is manufactured for repairing conveyor belts. The elastomer coupling was formed on the wheel.

This produced a seamless tire that was not subjected to nonuniform stresses during application and bonding. Much effort and skill

were required to overcome a number of difficulties in perfecting a method for forming the polyurethane into a tough, uniform coupling layer free of air bubbles and well-bonded to the transducer.

The resulting wheels proved to have approximately the same sensitivity as the first set of laboratory wheels. Mounting yokes that can be bolted to the frame being constructed for the fluid-filled wheels were designed and built. The bottom yoke will be rigid, and the top one will be mounted on a plate attached to a linear bearing. 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. This is similar to the mounting used for the rotary laboratory apparatus.

FLUID-FILLED WHEEL TRANSDUCERS

Work continues with 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 wheel. 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. 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.

A new 386-type computer has been set up for our on-line work. 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 be capable of analyzing the reference signal obtained without a sample in place. The three discrete pulses must be detected and separated. Then they are 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 is complete.

Other work has concerns transducer development. The commercial

transducers, originally used in the fluid-filled wheels, leaked fluid after prolonged encapsulation in the wheels. This degraded and, in some cases, completely destroyed their sensitivity. After discussions with the manufacturer, it was clear that the transducers were not designed for long-time immersion. We developed a way of sealing transducers if they are not designed for long-time immersion.

Temperature changes encountered with moving-web operation affect the three delay times. In order to monitor and correct for this, we have mounted a thermocouple in one of the wheels. This is part of a digital thermometer that is interfaced to the computer over an GPIB bus. We found it necessary to sheath the thermocouple in a nylon tube and ground the shield to the frame in order to eliminate a high frequency, periodic noise that could not be eliminated by signal averaging.

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

We have 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. This translates to easier maintenance when used on-line.

(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 spacing increase effectively eliminates any 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 tire.

One problem was discovered with the tire of the new wheels. The shape of the signals received, though strong, did not appear as clean as previous signals. We believe that a reflection from the rubber-liquid interface interferes with the signal of interest. Measuring the old tire and new tire thickness revealed that the new tires are thinner than the old. This means that, for the new tires, the reflective surface is nearer to the sample. This, in turn, means that the reflection would be causing more interference

in the new wheels than in the old. As a potential remedy for this, thicker tires have been ordered for the new wheels.

ON-LINE BIMORPH TRANSDUCERS

Two problems were encountered that required resolution for the use of bimorph transducers on-line to be practical. The first was to provide a durable contacting surface. The original plan of coating the transducer with carborundum or alumina loaded epoxy did not prove to be satisfactory. We were hopeful that a commercial process, which uses a plasma discharge to apply a diamond wear surface, might sufficiently protect the ceramic piezoelectric element. We prepared a series of different elements and submitted them for diamond application but were unsuccessful in having our samples coated.

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. Excellent wear resistance has been experienced to date. We are hopeful that this problem is solved.

The other major challenge is to mount the bimorphs in the roll housing, so that the three-transducer velocity measurement technique is not sensitive to web tension. We designed and had built a new roll for mounting the bimorph transducers. This one is made of aluminum and is lighter than the original steel roll. It has the groove pattern that we feel is best for achieving strong, narrow acoustic pulses. It is more amenable to experimentation with different mounting procedures. A custom-manufactured spring mounting fixture has been built to reduce variability in the loading of the transducers to the web. This has reduced tension sensitivity. We will continue to investigate possible improvements to reduce sensitivity to tension, especially for the MD longitudinal mode.

PROCESS, PRODUCT, PROPERTIES

STATUS REPORT

FOR

PROJECT 3467

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

October 18, 1989

PROJECT SUMMARY

PROJECT NO.: 3467 PROCESS, PROPERTIES, PRODUCT RELATIONSHIPS

PROJECT STAFF: M. Hall, M. Lorenz

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.

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

SUMMARY OF RESULTS LAST PERIOD: (October 1988 - March 1989)

(1) The ultrasonic technique for measuring shear coupling coefficients in polymeric sheets has been verified by experiments with laminates. A paper has been written.

(2) The use of ultrasonic reflection coefficients on paper has been investigated. This resulted in a new, one-sided, easily administered measurement. However, we found that the transmission coefficients could not be inferred from the reflection coefficients, and they, therefore, could not be used for rigorous loss coefficient calculations. A paper, whose subject is the transducers used in the reflection measurements, has been written.

(3) An investigation of the effects of tissue manufacturing variables on out-of-plane ultrasonic measurements was conducted. A paper has been written on the empirical relations between out-of-plan ultrasonic parameters and tissue softness.

(4) An aperture wheel has been installed on the transmission detector of the Board Optical Transmission Meter (BOTM) for easy selection of port sizes to cover the full range of measurable transmittances.

(5) After calibration, it was demonstrated that transmission measurements could be made on a 39.2 g/m^2 white bond paper in stacks containing 1 to 23 sheets. The calculated transmittances extended to a low of about 7 x 10-8. It was also demonstrated that the specific scattering coefficients of a range of papers from ordinary bond to a heavy unbleached kraft linerboard could be obtained easily.

(6) The BOTM may be used to measure R_o with the specimen either bare or in the Teflon sandwich needed for transmittance measurement. This requires a specific calibration for the particular specimen type being measured. Best accuracy is obtained using R_o values from the TB-1 brightness tester.

(7) The design, construction, and calibration of the instrument have now been completed, and it is ready for use.

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

(1) The in-plane ultrasonic robot is 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.

PROJECT TITLE: PROCESS, PROPERTIES, PRODUCT RELATIONSHIPS

PROJECT STAFF: M. Hall

Date: 11/1/88

Budget: $175,000

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

STRENGTH IMPROVEMENT AND FAILURE MECHANISMS

STATUS REPORT

FOR

PROJECT 3469

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

October 18, 1989

PROJECT SUMMARY

PROJECT NO.: 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 1988 - March 1989)

(1) Improvement of circuitry for light transmission and reflectance measurement on the IPC Formation Tester.

(2) Failure stress measurements have been made on the recently fabricated out-of-plane biaxial tester.

(3) A limited set of handsheets, containing blends of NSSC pulp and synthetic fiber, have been saturated and dried under restraint after saturation to determine the effects of such restraint on forming losses.

(4) Differences in "wire side" and "felt side" fiber orientation on lean angle (polar plot diagram) and curl have been investigated.

(5) Student Related Work - Robert Aloisi has investigated the effects of felt type and formation on surface roughness using a profilometer.

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

(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.

PROJECT TITLE: STRENGTH IMPROVEMENT AND FAILURE MECHANISMS

PROJECT STAFF: J. Waterhouse

Date: 10/9/89

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 corrugating 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 system. 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

Status Report

STRENGTH IMPROVEMENT AND FAILURE MECHANISMS

Project 3469

INTRODUCTION

Project 3469 "Strength Improvement and Failure Mechanisms" embraces the following areas: Compressive Strength Improvement, Formation Measurements, Combined Stress Measurements and Internal Stresses in Paper and Board. Since our last report activity has been mainly focussed in the areas of Formation and Combined Stress Measurements. With regard to Formation we are now able to make satisfactory mass density measurements, and have further developed the software capabilities of the formation tester. Measurements of combined stress have also been made at one level of refining and wet pressing. A presentation on out-of-plane combined stress measurements was made at the 1989 ASCE/ASME Mechanics Conference at the University of California, La Jolla.

COMPRESSIVE STRENGTH IMPROVEMENT

Forming processes in converting can have an adverse effect on the strength properties of paper and board. A better understanding of these forming losses, and strategies to reduce them, should lead to a more effective utilization of the raw material and process improvements we have discussed in previous work. This understanding may further effect raw material selection and process requirements.

The forming of corrugating medium is one area where forming losses can have a significant impact on combined board compressive strength and flat crush. We have, therefore, been seeking ways to improve medium performance to reduce these losses. A complex stress situation is imposed on the medium during forming, however, it is believed that the large bending strains and "calendering" of the medium are primarily responsible for the large losses in MD and CD compressive strength. Another strength requirement aspect of corrugating is flute fracture. It is suspected that the medium requirements to avoid flute fracture could be in conflict with those required to minimize forming losses.

To better understand the forming process, and evolve a medium more tolerant of the forming process, we have been doing exploratory work with various model systems. One concept we have been evaluating is based on incorporating suitably positioned high modulus fibers into the medium, and then activating them during the forming process. Activation might be achieved by the addition of a polymer or bicomponent fiber system which will go through its softening temperature during flute formation.

A number of natural fiber, synthetic fiber, and polymer combinations have been investigated as shown in Tables 1 and 2. Orientated handsheets were made using the Formette Dynamique.

Table 1. Fiber furnishes.

Fiber Series 1

1. NSSC pulp
2. NSSC/Kevlar "pulp" blends

Fiber Series 2

1. NSSC pulp
1. NSSC pulp/Kevlar staple blends
2. NSSC pulp/Kevlar "pulp" blends
3. NSSC pulp/1/4 in. glass fiber blends
4. NSSC pulp/1/8 in. glass fiber blends

Table 2. Binder series 1.

A. Carboxylated SBR	T_g = 42°C
B. Carboxylated SBR*	T_g = 42°C
C. Styrene latex	
D. Styrene latex	T_g = 105°C
E. SBR latex (99% styrene)	
F. SBR latex (85% styrene)	T_g = 54°C

*Plasticized version of A, T_g Glass Transition Temperature as supplied by manufacturer.

Physical property measurements were made on these sheets before and after binder addition, and after heat treatment, and were reported in the last status report. Forming losses were evaluated using the Concora Fluter. After fluting compressive strength measurements were made in both the flank (leading and trailing) and tip (top and bottom) positions. Forming losses for the flank and tip positions were calculated as the loss in compressive strength with respect to the uncorrugated heat treated samples. As stated above the medium is subjected to a combined stress situation during corrugating. Each section of the flute will experience some level of bending, lateral compression, and shear stress. Bending is considered to be the major contributor to forming losses and is presumed to be responsible for the greater losses which are seen in the tip regions of the flute (see Table 3).

TABLE 3
AVERAGE TIP AND FLANK FORMING LOSSES FOR BINDER SERIES 1
FOR 100% NSSC AT TWO LEVELS OF WET PRESSING

Binder	Low W.P. $p = 0.540 g/cm^3$		High W.P $p = 0.738 g/cm^3$	
	t %	f %	t %	f %
A	46	34	35	16
B	43	32	35	16
C	40	34	35	6
D	40	35	27	12
E	47	32	34	16
F	46	28	34	12

Furthermore since flank losses are also reduced with wet pressing (see Table 3) indicates that the bending contribution is not insignificant in this region either. It can be argued that our investigation of forming loss reduction should be conducted at constant bending strain (i.e. at constant caliper for a given flute configuration). The variation of tip forming loss as a function of caliper for a variety of mediums is shown in Figure 1. We see that as caliper is reduced both by grammage reduction and increased wet pressing for the control mediums, there is a reduction in forming loss.

Also shown are some results for mediums containing synthetic fiber addition and polymer reinforcement, indicating that at constant caliper there is a significant reduction in forming loss. This significant finding should be more fully explored in future work.

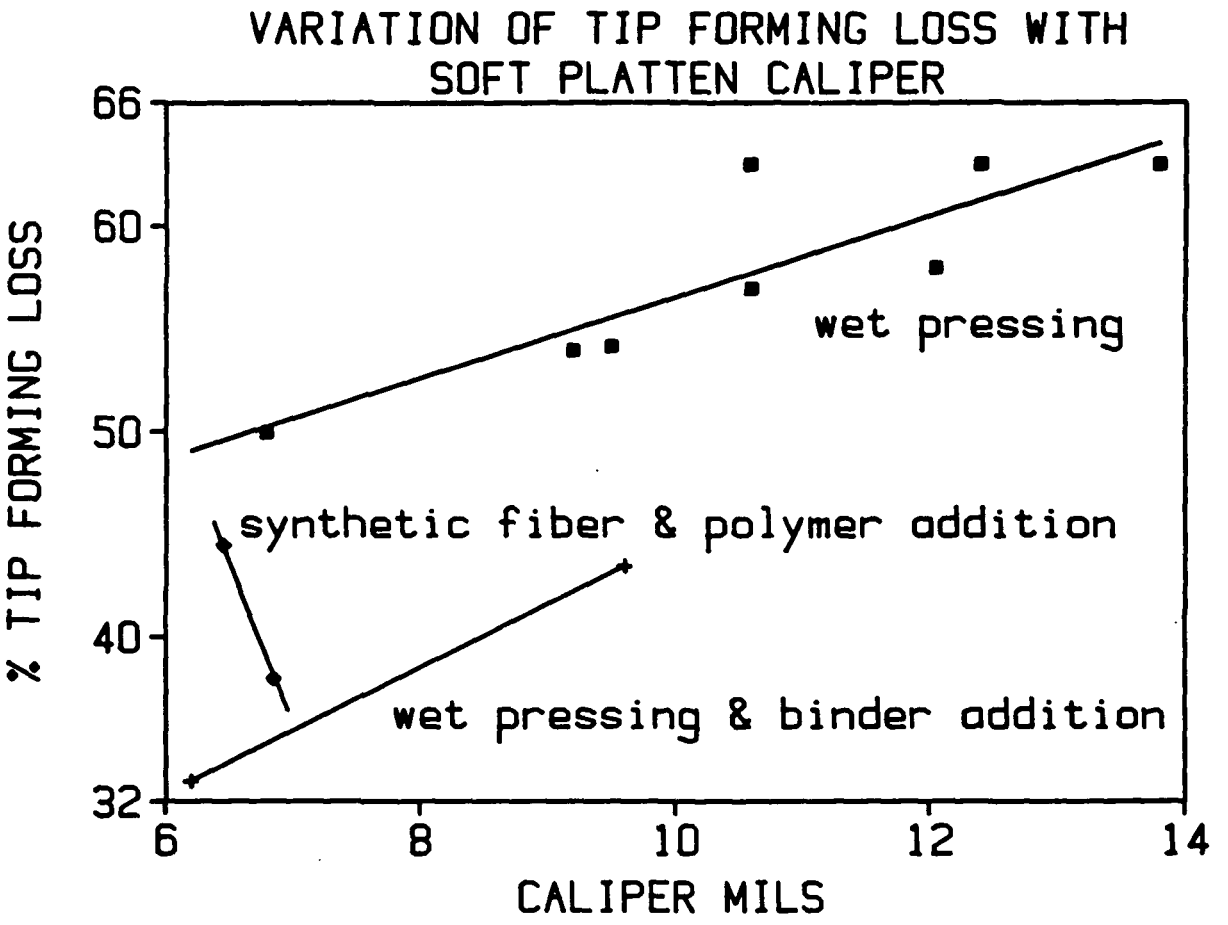


Figure 1

For webs containing only NSSC pulp, polymer addition and heat activation significantly improved both in-plane and out-of-plane moduli and compressive strength. Synthetic fiber addition, as expected, lowers both in-plane and out-of-plane elastic constants, and although there is a small increase in in-plane modulus with polymer addition and heat activation, the out-of-plane modulus is still lower than the control. This suggests that the fibers are not effectively bonded or activated. Therefore, it is proposed that in future work a more in depth investigation be conducted to determine more suitable synthetic fiber/binder combinations than we have had the opportunity to explore so far.

FORMATION

The recently fabricated IPST Formation Tester has the capability of making light transmission and reflectance measurements in the wavelength range of 400 - 700 nm, as well as beta particle absorption measurements. An aperture of 1mm x 1mm is used for both light transmission and beta measurements. The respective circuits are shown in Figure 2.

Formation samples of various sizes can be measured and are held in place with magnetic strips.

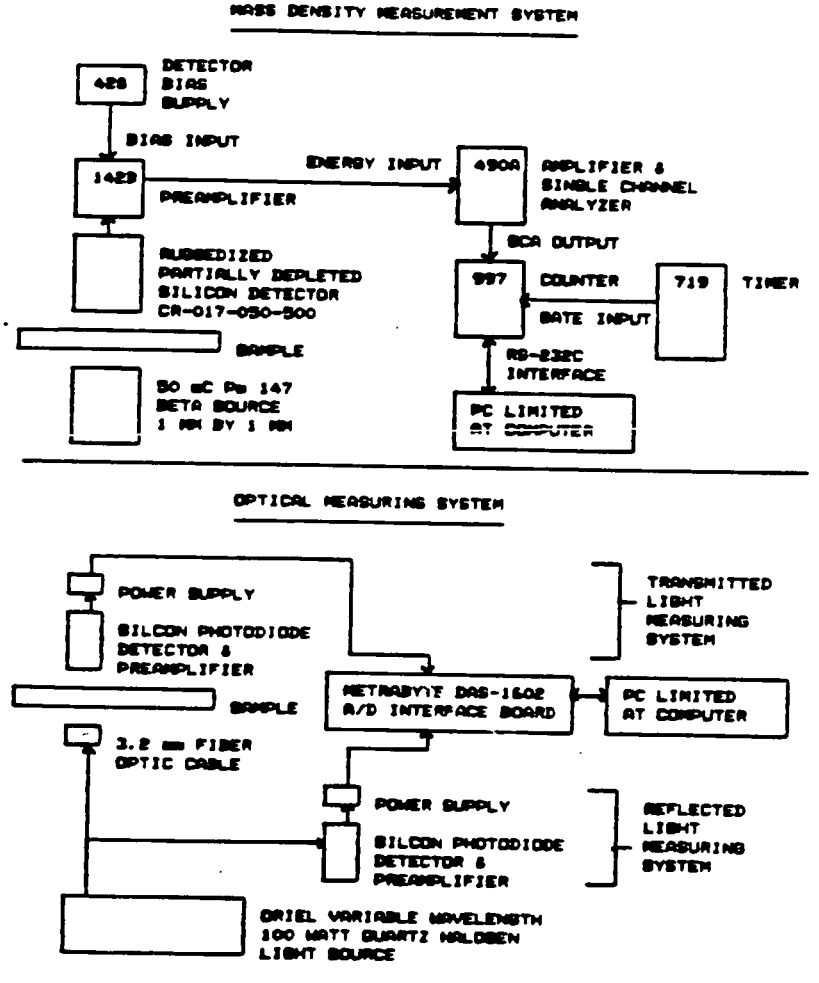


Figure 2 Schematic layout of mass density and optical measuring system for IPC formation tester.

The difficulty stated in our last status report of insufficient beta particle counts per second has now been unexpectedly resolved by simply increasing the gain control on the single channel

analyzer (see Fig. 2) thanks to Mike Kleiber! This improvement has resulted in a performance closer to expectations. Computer interfacing with the output of the beta particle circuitry has also been completed.

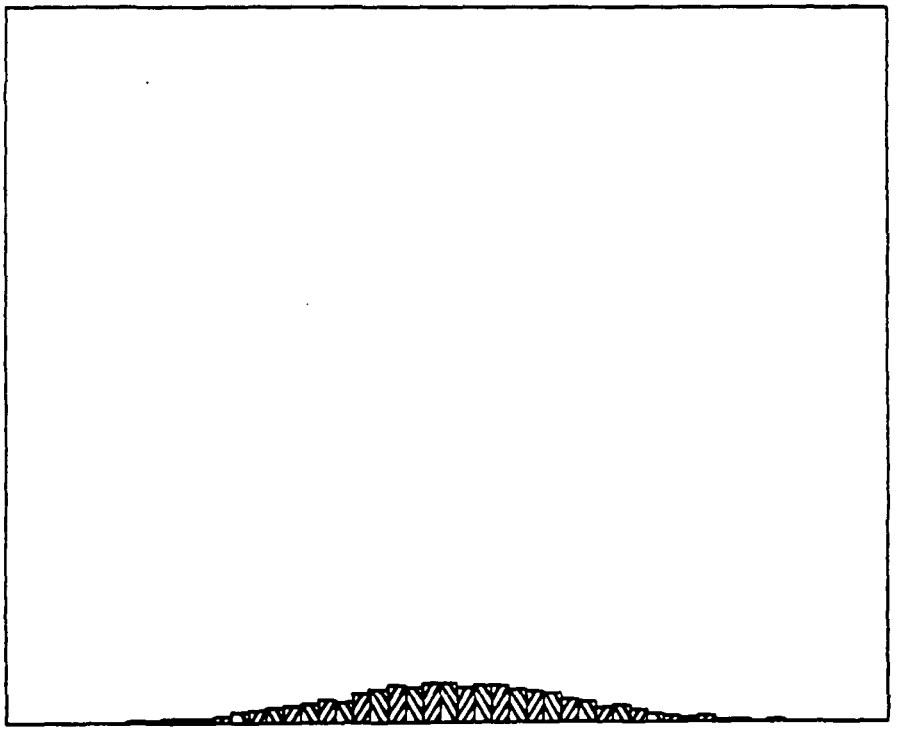
INSTITUTE OF PAPER CHEMISTRY
FORMATION TEST RESULTS

Date: OCTOBER 6, 1989
Time: 11:08:34

Operator : Kleiber
Sample ID : Engineering grid paper
Project : 3469

Head air gap setting (mils) :
Light source wavelength (nm) : WHITE
Number of data points averaged : 100

TRANSMITTED LIGHT



Total Number of Data Points : 6400
Area Measured (mm) (80 X 80)

Average (volts) : 0.3728
Standard Deviation : 0.0284
Percent of Variance : 7.61

Row - Percent of Variance : 6.62
Col - Percent of Variance : 7.01

Figure 3

INSTITUTE OF PAPER CHEMISTRY
FORMATION TEST RESULTS

Date: OCTOBER 6, 1989
Time: 11:08:34

Operator : Kleiber
Sample ID : Engineering grid paper
Project : 3469

TRANSMITTED LIGHT FFT (Rows)

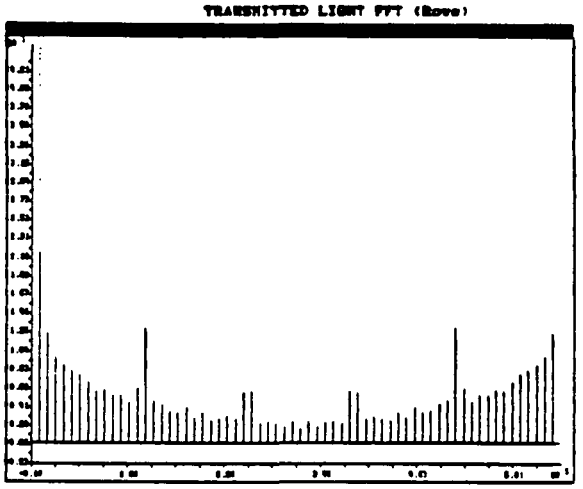


Figure 4

INSTITUTE OF PAPER CHEMISTRY
FORMATION TEST RESULTS

Date: OCTOBER 6, 1989
Time: 11:08:34

Operator : Kleiber
Sample ID : Engineering grid paper
Project : 3469

TRANSMITTED LIGHT FFT (Columns)

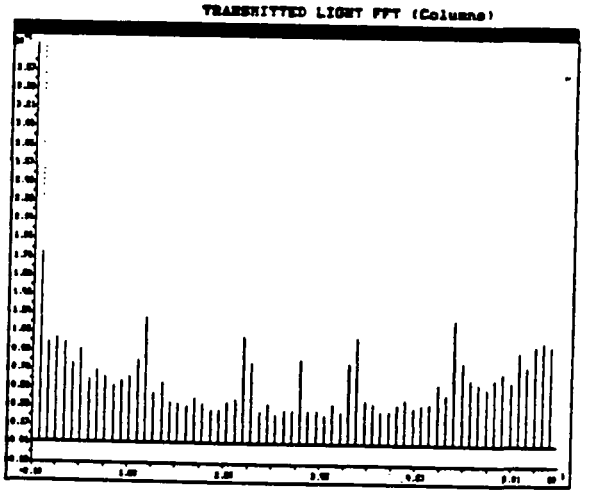


Figure 5

INSTITUTE OF PAPER CHEMISTRY
FORMATION TEST RESULTS

Date: OCTOBER 6, 1989
Time: 11:08:34

Operator : Kleiber
Sample ID : Engineering grid paper
Project : 3469

Scale Factor = 0.009514
Maximum Value = 26.0865
Minimum Value = -26.4697

TRANSMITTED LIGHT

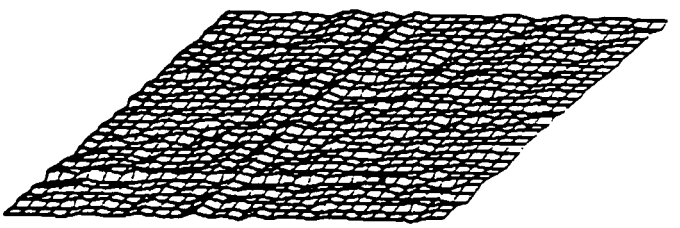


Figure 6

Significant software development to make the IPST Formation Tester user friendly has also occurred. Our current philosophy is not too limit ourselves to particular measures of formation, but rather to have a menu together with recommendations, for which measures are appropriate to a given situation. Software development includes: changing from continuous to discreet steps when beta source output is selected, calculation of row and column variance, incorporation of fast fourier transforms, and contour maps. The stored data can also be accessed by Quatro or Lotus 123 for further analysis. Examples of output data are shown in Figures 3,4,5 and 6.

COMBINED STRESS MEASUREMENTS

Combined out-of-plane stresses arise in a number of converting and end uses of paper and board such as corrugating, calendering, adhesive joints and paper tube performance.

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

The preferred method of sample mounting is the use of photographic mounting tissue as used for making out-of-plane shear measurements (Waterhouse J.F., TAPPI 67 (6) June 1984).

The properties of formette handsheets made to investigate the effects of refining and wet pressing on the out-of-plane biaxial failure envelope are shown in Table 4.

TABLE 4
FORMETTE HANDSHEET PROPERTIES

Freeness C.S.F.	Grammage g/m^3	Caliper μm	Density g/m^3	E/p $(k/sec)^2$	R $(k/sec)^2$	Ez/p lbs/in^2	Z.D.T.
667	313	642	0.487	6.51	2.26	0.144	-
640	310	454	0.682	7.46	2.11	0.226	-
-	312	411	0.759	7.79	2.13	0.264	-
433	299	557	0.537	7.13	1.75	0.163	-
436	288	395	0.730	8.29	1.71	0.245	-
-	295	371	0.796	8.50	1.71	0.265	-

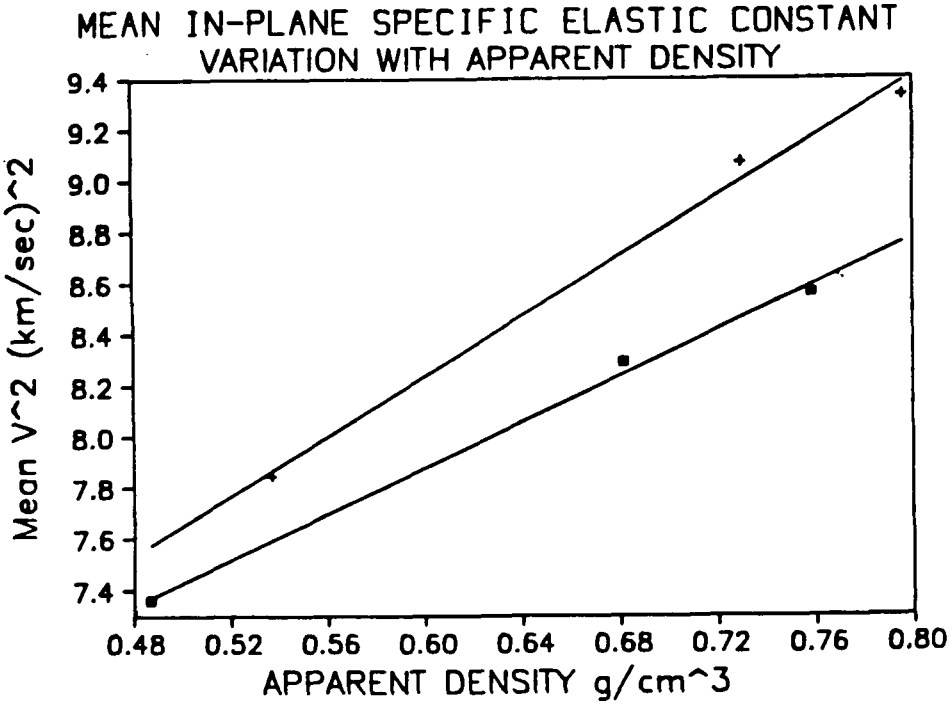


Figure 7

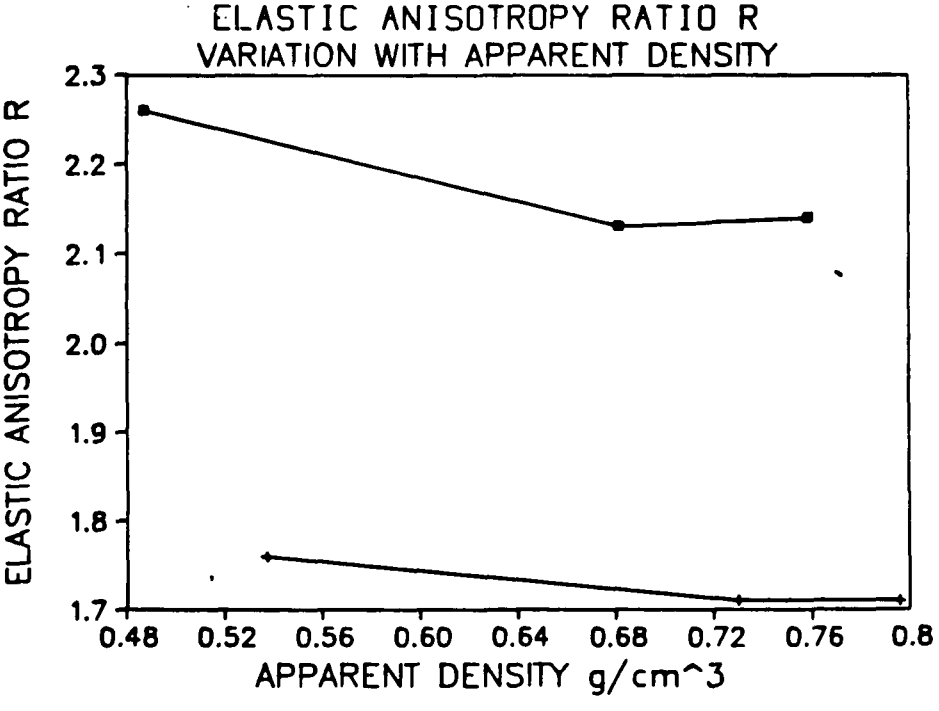


Figure 8

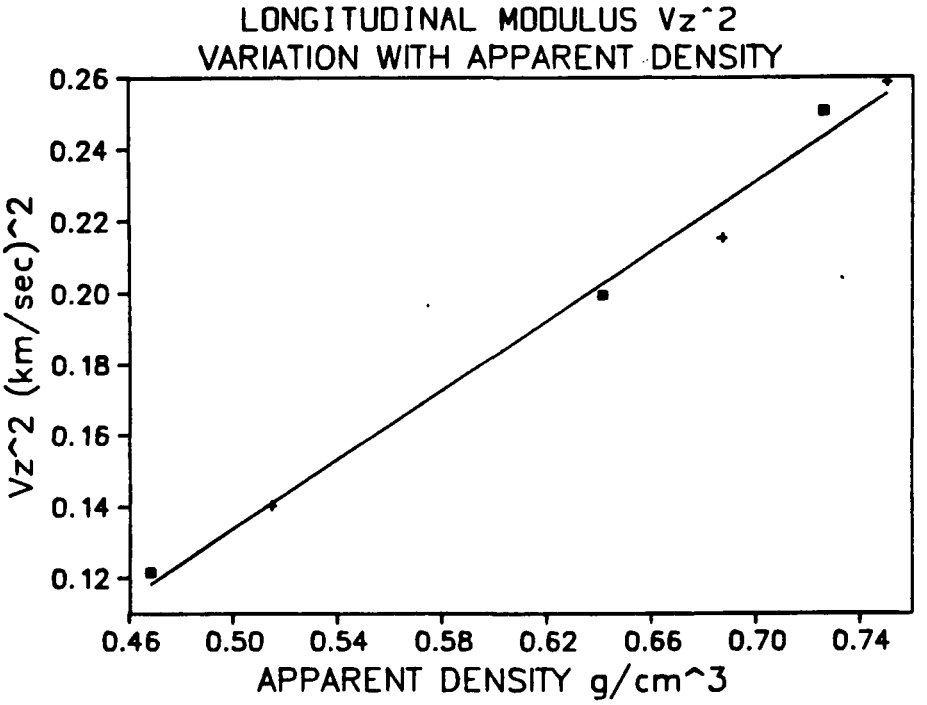


Figure 9

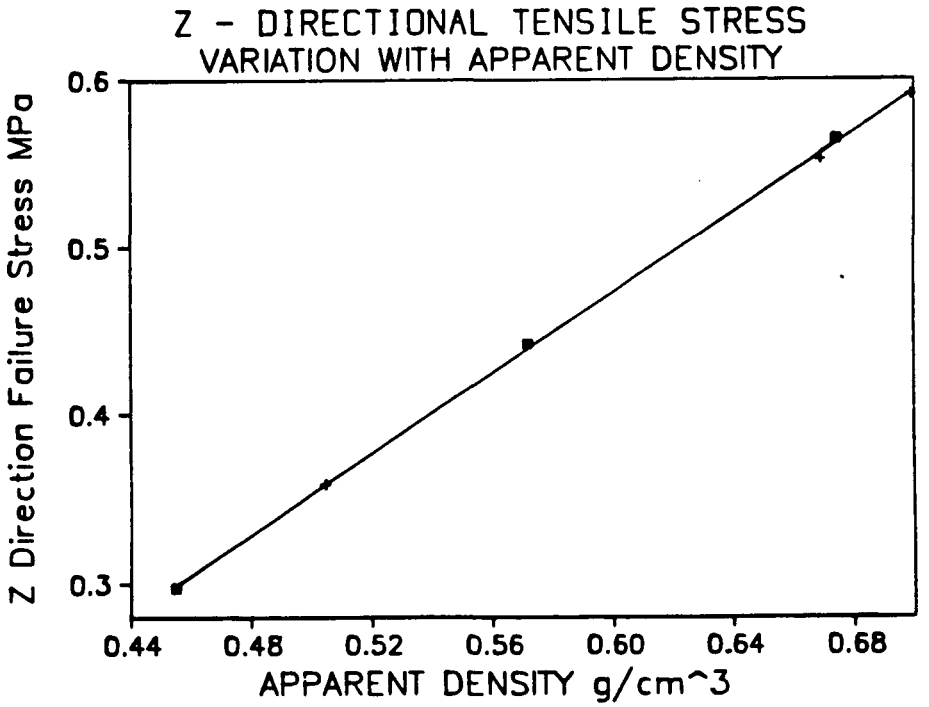


Figure 10

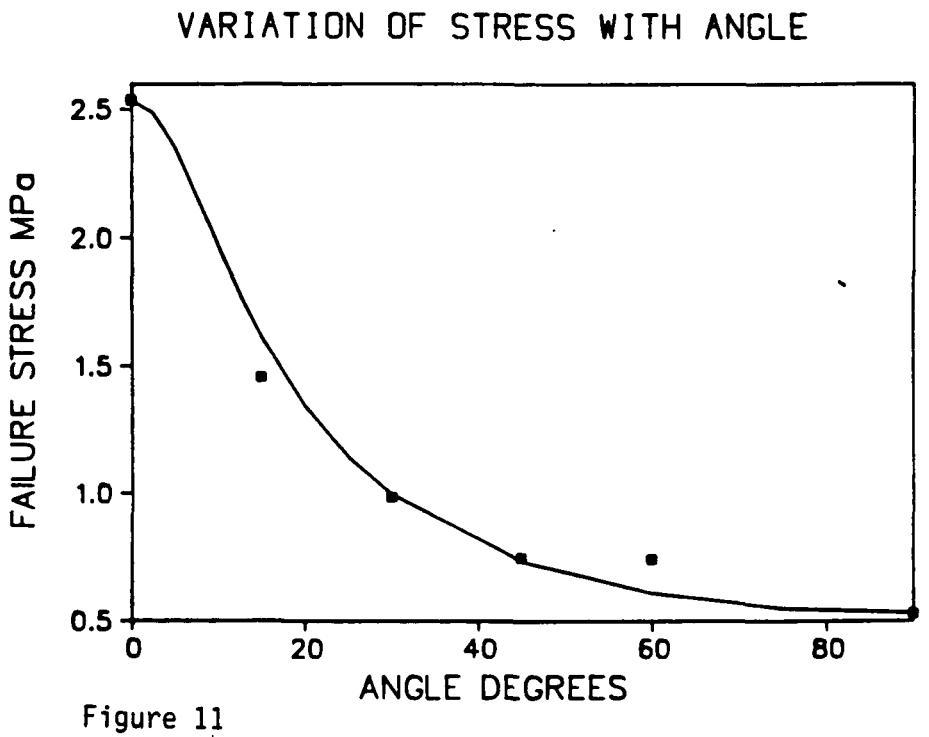


Figure 11

The variation of geometric mean in-plane and out-of-plane specific elastic constants with apparent density is shown in Figures 7 and 9. We note that in contrast to the in-plane data the out-of-plane data collapses on to a common line for wet pressing and refining. As found in previous work the elastic anisotropy decreases with densification as shown in Figure 8.

Normal or Z direction failure stress measurements are shown in Figure 10. These were measured at 0° using the out-of-plane biaxial device. Consistent with the out-of-plane elastic constant measurements Figure 9 we see that the data collapses onto a common line. The variation of failure stress with angle for the unrefined and intermediate level of wet pressing case is shown in Figure 11. The solid line is a modification of the tensorial form used by Lui, J. Y., and Floeter, L. H. ("Shear Strength in Principal Plane of Wood" Journal of Engineering Mechanics, Vol. 110, NO. 6, June 1984). In this work they measured the shear strength of wood coupons cut at different angles, using the device originally employed by Arcan, Hashin, and Voloshin. The solid line in Figure 14 would be an ellipse if a plot of normal stress against shear stress is made.

INTERNAL STRESSES IN PAPER AND BOARD

No significant progress has been made in this area since our last status report. We nevertheless would hope to continue our work in the area of internal and residual stresses particularly as related to curl problems i.e. both on-axis and off-axis curl. In student

related work the effect of internal stresses on the modification of paper properties by calendering is also to be studied.

STUDENT RELATED WORK

Titles and brief comments on student related work are given below.

A190 Research Projects

1. "A Comparison of Hard Nip and Soft Nip Calendering" - Eero Palogankas

   Eero Palogankas was a special student from Finland who completed his A190 project in June 1989. Highlights of this work will be presented at the forthcoming PAC Meeting.

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

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

A490 Doctoral Research

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

INTERNAL STRENGTH ENHANCEMENT

STATUS REPORT

FOR

PROJECT 3526

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

October 18, 1989

PROJECT SUMMARY

PROJECT NO. 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: (October 1988 - March 1989)

(1) Measurements of the whole and the classified pulps at the four levels of yield have been completed. Analysis of this data is partially completed.

(2) Measurements on the effects of pulp fines are partially completed and the data are being analyzed.

(3) Extrapolation of results of scattering coefficient versus tensile strength, elongational modulus, m of bond strength was used to evaluate the scattering at zero strength, i.e. the unbonded sheet. Only the modulus plot gave a single curve with small experimental scatter. From these results the relative bonded area (RBA) of the various sheets could be calculated.

(4) Several properties (bond strength, ultrasonic out-of-plane longitudinal velocity, tensile index, and TEA index) are directly proportional to the RBA.

(5) Two correlations (out-of-plane longitudinal velocity versus RBA and density versus RBA) clearly show a dependence on pulp yield. For the other properties the dependence on yield is weak or absent.

(6) Most of the property correlations showed a dependence on whether or not the stock was treated with a strength aid. However, correlations between bond strength and tensile index and between TEA index and tensile index are independent of

treatment.

(7) The correlation between TEA index and tensile index are independent of pulp yield and strength aid treatment.

(8) At densities (or bond strengths) below a critical value delamination of the sheet produced a negligible increase in scatter coefficient (increase in unbonded area). The critical value for the bond strength increases when a strength aid is used.

(9) The specific bond strength (or the ratio of the bond strength to the decrease in optical contact area) is a decreasing function of RBA or density.

SUMMARY OF RESULTS THIS 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.

PROJECT TITLE: INTERNAL STRENGTH ENHANCEMENT

PROJECT STAFF: R. Stratton

Date: 10/9/89

Budget: $160,000

Period Ends: 6/30/90

Project No.: 3526

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 fiver 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 lati8x 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 require 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 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, m refining, fines content, and strength additives on bond strength.

Measurements on handsheets from a series of draft 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 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.

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

BOARD PROPERTIES AND PERFORMANCE

STATUS REPORT

FOR

PROJECT 3571

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

October 18, 1989

PROJECT TITLE: BOARD PROPERTIES AND PERFORMANCE

Date: 11/1/88

Budget: $180,000

PROJECT STAFF: Staff

Period Ends: 6/30/90

Project No.: 3571

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.

CURRENT OBJECTIVE:

To develop relationships between container performance, combined board properties, and component properties.

To improve the performance/cost ratios 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 and 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. An important step is to incorporate at the elastic stiffnesses of the board into such models, if possible. This will enable us to use our knowledge on how papermaking factors affect the elastic stiffnesses to make board improvements.

RESULTS TO DATE:

Our Rayleigh-Ritz analysis of box failure under several types of load indicated that compressive strength (ECT) is the limiting property governing performance. Further analyses of the ECT behavior of corrugated board showed that present local buckling models fail to properly predict ECT strength when the strength of the liners or medium is changed by certain papermaking operations, e.g. wet pressing. Therefore, new models have been developed which show that ECT is primarily dependent on the compressive strength and/or elastic stiffnesses of the liners and medium. The bending stiffness of the liners appears to have only a minor effect on ECT.

The importance of linerboard bending stiffness has been a point of concern to our industry and these results indicate that it is much less important than compressive strength. We extended this work to combined board flexural stiffness and box compression.

Finite element techniques are being used to study the effects of the high stresses imposed on the medium during fluting on the subsequent flat crush strength of the board. Initial results demonstrate that the leading and trailing sides of the flute are stressed differently which, if too extreme, can cause learning flutes and poor flat crush.

Our forming models indicate that satisfactory high speed runnability on the corrugator is dependent on at least four medium properties as well as nip geometry and medium web tension. Better runnability is obtained as MD tensile and stretch are increased and the coefficient of friction of medium and medium thickness are decreased.

Our research has shown that fluting reduces the compressive strength of medium and hence reduces flat crush and ECT. Densification via wet pressing is one way to reduce these losses. Current work shows that the tensile characteristics of medium are also reduced by forming. it appears that these reductions can be predicted from our runnability model.

We have also shown that strength losses during fluting are also explained by the runnability model. As the stresses applied to the medium become high, the compressive and tensile strength of the fluted medium decreases. For example, the tensile strength of the fluted medium approaches zero when the applied stress approaches the MD tensile strength of the medium.

In work underway, we have shown that combined board twist warp is developed when the ultrasonic polar angles of the single- and double-faced liners are aligned in opposite directions in the combined board. Better CD paper machine stiffness and strength profiles would help the box plant avoid twist warp.

PLANNED ACTIVITY FOR THE PERIOD:

Our short term objectives are directed to (1) improving liner and medium end-use and converting performance properties and (2) using structural models to assess the impact of papermaking factors on combined board and box performance. The structural models we are developing show how the elastic stiffnesses and compressive strengths of the components will affect combined board and box compressive strength.

During 1989/90 research is planned in the following areas:

1. Combined Board Warp
2. Liner and Medium Improvement
3. Runnability Modeling
4. Flat Crush and Flute Formation Modeling
5. Commercial Box Abuse Effects

A current study on combined warp is directed to showing how combining liners with unlike elastic stiffness polar angles can greatly affect warp and hence, affect box plant productivity. When liners with polar angles deviating in opposite directions are combined on the corrugator, the resulting board will exhibit large amounts of twist warp as their moisture content changes. The magnitudes of the effects are being determined as a function of polar angle and moisture content. This work will support allied work for the FKBG on causes of such deviations.

Past work has shown that surface treatments with PAE type agents increase compressive strength under normal and high RH environments. However, the agents tend to reduce water receptivity and hence, reduce adhesion strength in the corrugating operation. We are screening agents which can increase liner and medium strength but which allow high corrugating speeds with adequate pin adhesion strength.

The MD tensile and stretch of medium are two of the properties included in our runnability model. Both will be affected by the high temperatures and steam showers used in the corrugating operation. Furthermore, the lignin and hemicellulose content of the medium can be expected to affect their high temperature behavior and hence, their corrugating runnability. We have constructed an apparatus which permits us to determine the effects of high temperatures similar to those in the fluting process on the tensile load-elongation characteristics of medium. To explore the effects of furnish on performance, mediums are being made from furnishes with varying chemical content and mixtures thereof. The results will be analyzed to compare the effects of furnish composition on the tensile characteristics and runnability. These results will be used to improve our runnability model and to develop ways to improve runnability and end-use performance.

Additional runnability modeling of strain rate effects is also underway.

A part of planned research is directed to modeling flute formation and flat crush performance using finite element techniques. Initial finite element trials indicated we needed more information

on the stiffness and strength of the medium after fluting, i.e., the strength and stiffness losses in the medium needed to be taken into account. Experiment work is underway to better define the medium damage during fluting. This information will be incorporated in the finite element models.

A study of the effects of combined board crush under varying RH levels is underway. This information will be incorporated in current box models.

FUNDAMENTALS OF PAPER SURFACE WETTABILITY

STATUS REPORT

FOR

PROJECT 3646

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

October 18, 1989

PROJECT SUMMARY

PROJECT NO.: 3646 Fundamentals of Paper Surface Wettability

PROJECT STAFF: F. Etzler

PROGRAM GOAL:

Develop and understanding of the influence of paper structure and properties of paper and board on liquid absorption and penetration.

SUMMARY OF RESULTS LAST PERIOD (October 1988 - March 1989)]

Initial calculations of Voronoi polyhedra for SPC/E water have been completed. These results suggest that the volume distribution of water molecules is bimodal. This calculation represents the first microscopic evidence supporting Roentgen's hypothesis (1892) suggesting that water exists in "bulky" and "dense" configurations. Furthermore the calculations suggest that the model for vicinal water is microscopically robust.

The Seteram DSC has been installed and initial calibrations completed (Appleton).

Initial experiments on synthesis of chemically modified surfaces have been started.

Experiments on Kraft linerboard samples have shown that wettability is unrelated to lignin or benzene/alcohol extractives content.

SUMMARY OF RESULTS THIS PERIOD

A constant pressure simulation of water at various temperatures has been performed at University of North Carolina. We await for tapes to arrive in order to continue Voronoi analysis.

The utilization of statistical geometry to understand liquid structure continues.

A device for introducing water to porous materials under vacuum has been constructed. Such a device appears to be necessary in order to make precision heat capacity measurements.

Heat Capacity measurements for water in 242 A diameter silica pores have been measured over the temperature interval of 10 - 60 C. The results suggest that vicinal water structure persists to high temperatures and that structural transitions occur near 15, 30, 45 C. Etzler and Drost-Hansen previously reported independent

evidence suggesting the existence of such structural transitions and their importance to physiology.

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.

Status Report

FUNDAMENTALS OF PAPER SURFACE WETTABILITY

Project 3646

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 paper making 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 systems; 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 performed on water in silica and clays. Silicas and clays are nearly ideal substrates as 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 1-20nm, 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 3-6nm. 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, 1967) has calculated the model independent distribution of single particle enthalpies for water and a number of other liquids. It was found that the enthalpy distribution for water is unusual in that the distribution is bimodal. This type of distribution contrasts with the nearly Maxwell-Boltzmann type distributions found for more simple liquids.

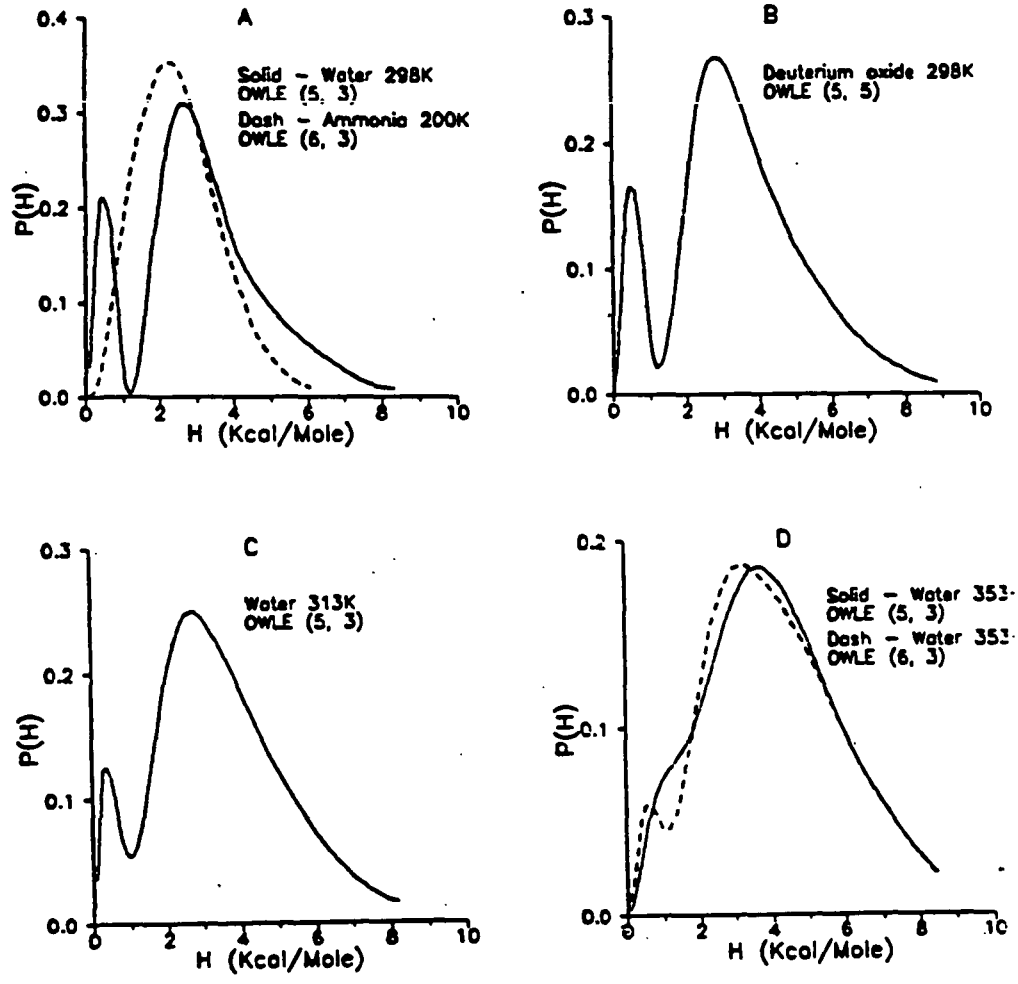


Figure 1. Stey's distribution functions. Probability, P(H), vs. enthalpy, H. (A) Water at 298 K and NH_3 at 200 K. (B) D_2O at 298 K. (C) H_2O at 313 K. (D) H_2O at 353 K.

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, $\sigma\ h^2$, 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. $C_p(1)$ it 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 298 K 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 Eqn. 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 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

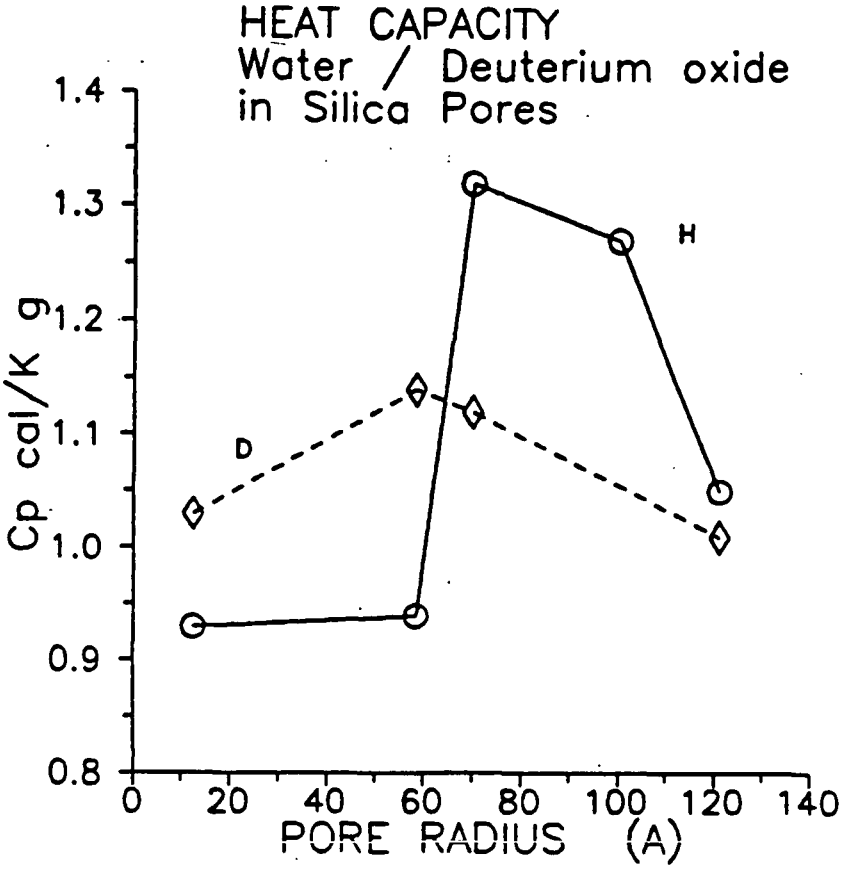


Figure 2. Heat capacities of water in silica pores as a function of pore radius at 298 K: o squares H_2O; diamonds, D_2O. Radius in Angstroms (10 A = 1 nm).

298 K.

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

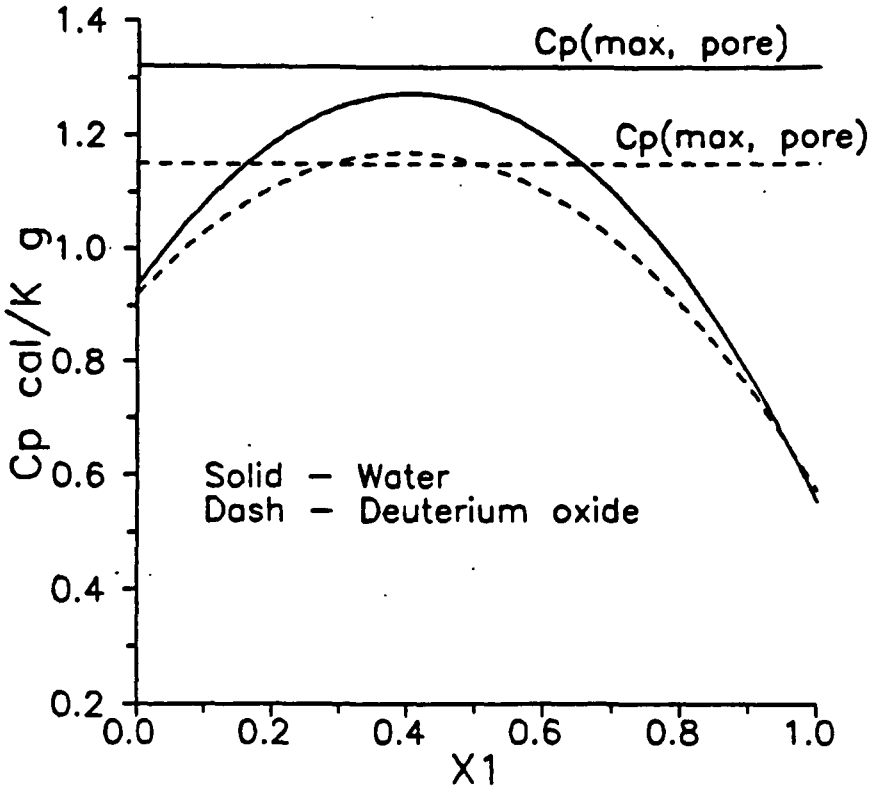


Figure 3. Hypothetical heat capacity of water and deuterium oxide as a function of x(1) at 298 K.

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 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

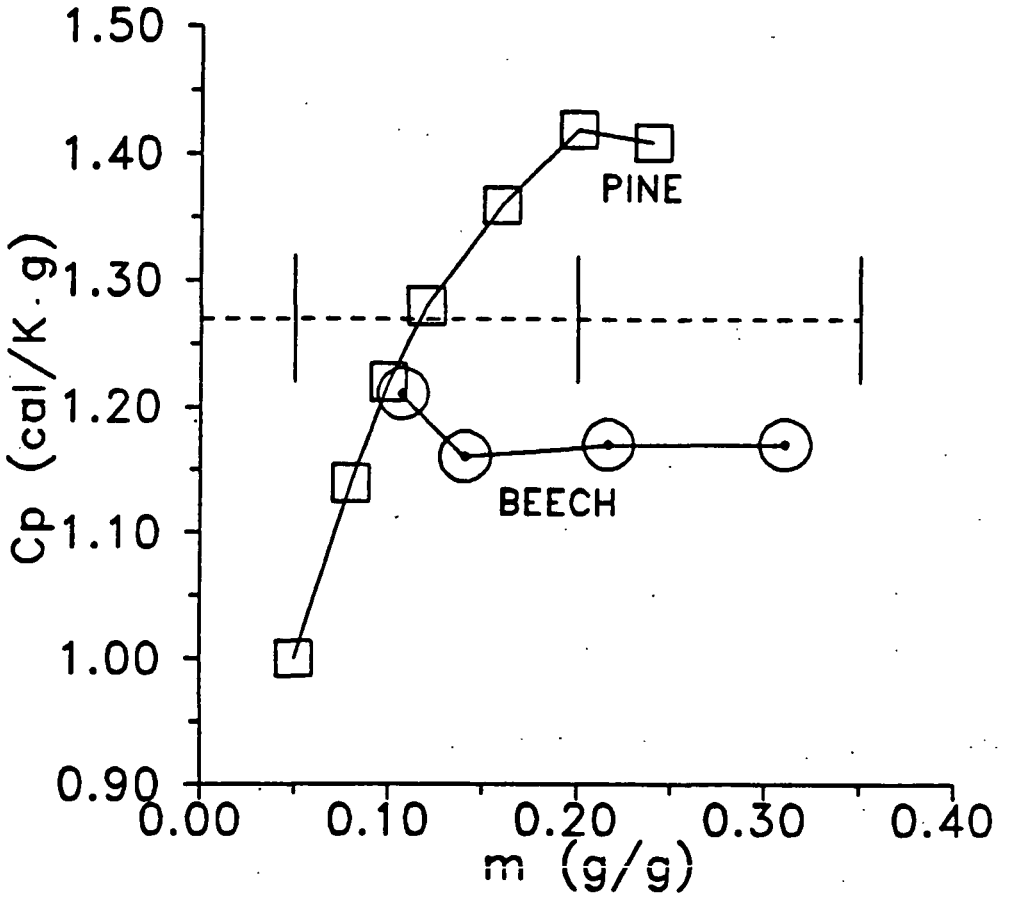


Figure 4. Apparent heat capacity of water in woods. Squares - pine; circles - beech; dashed line - maximum heat capacity as calculated from author's model.

adjacent to either surface is more structured than the bulk.

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

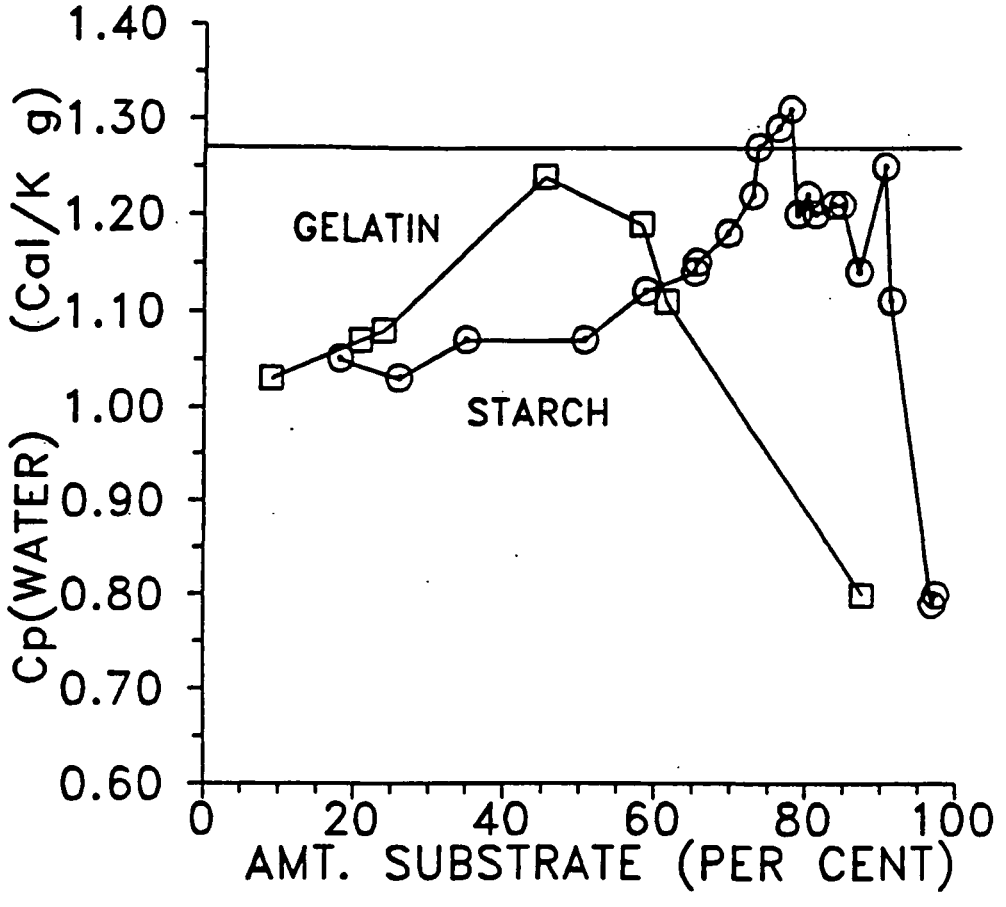


Figure 5. Apparent heat capacity of water versus per cent of substrate material in mixture. Squares - gelatin; circles - starch. Horizontal line - maximum heat capacity calculated from author's model.

of the intermolecular potential energy function. Knowledge of this function is not necessary, however, for the calculation of single particle 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 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 liquids such as argon on 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

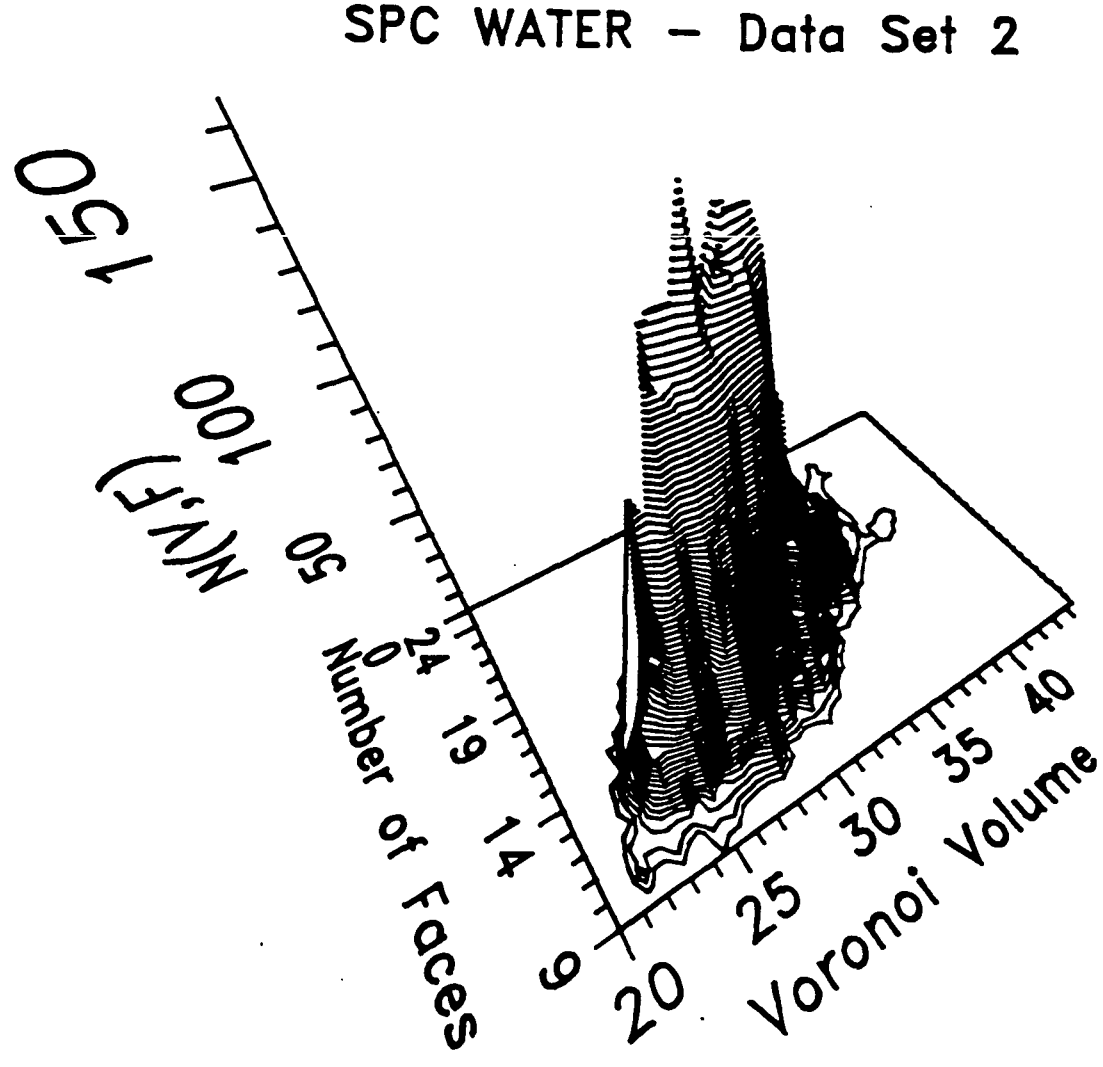


Figure 6. A three-dimensional plot showing the probability of finding a polyhedron with a given number of faces and particular volume.

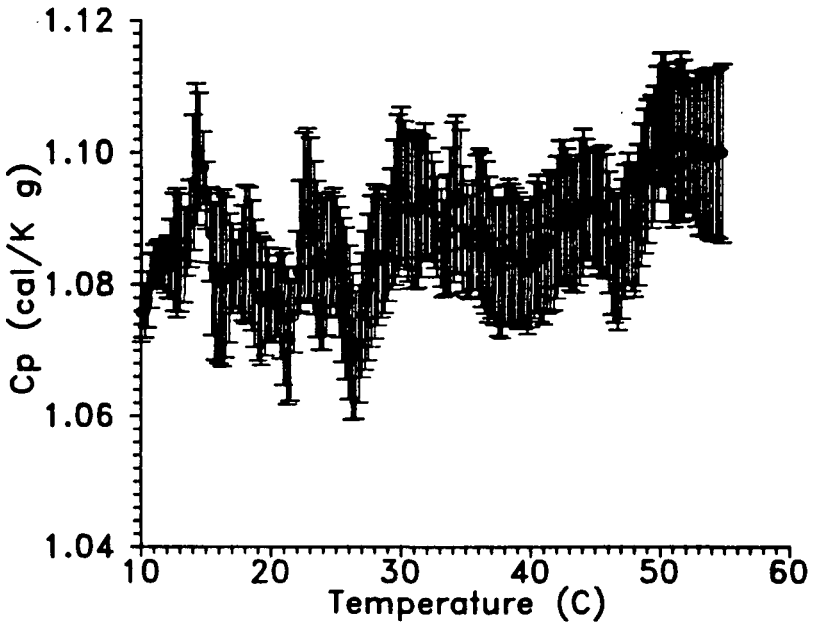


Figure 7. Heat capacity of water in 242 A diameter silica pores.

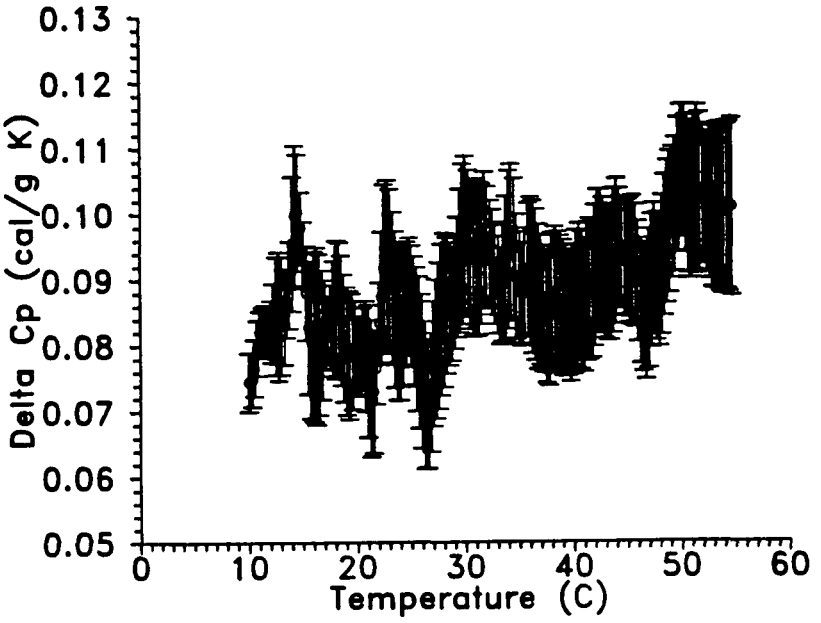


Figure 8. C_p (pore) - C_p (bulk) for water in 242 A diameter silica pores.

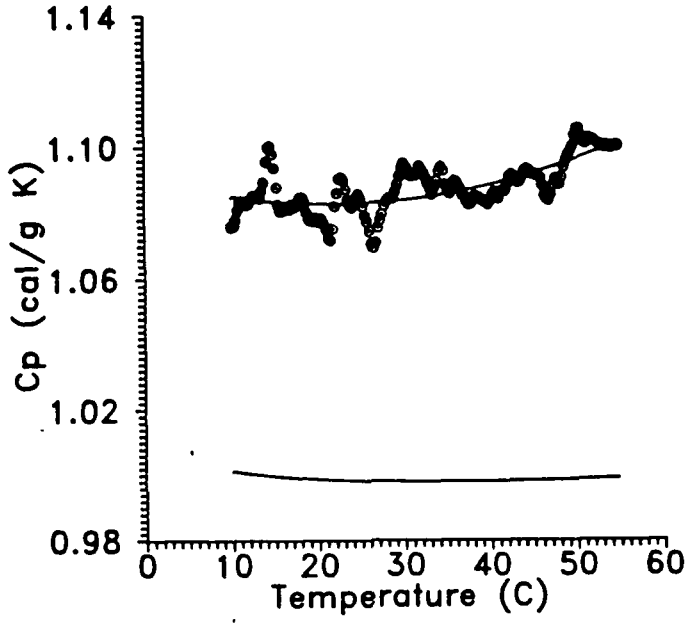


Figure 9. A comparison of C_p (pore) and C_p (bulk) versus temperature.

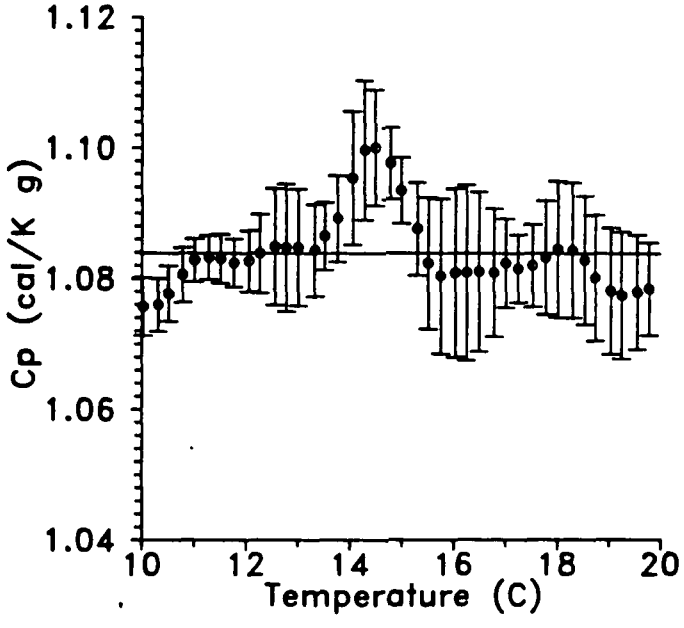


Figure 10. Detail over the temperature range from 10-20 C. Data as in Figure 7.

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.

Recently constant pressure simulations have been performed for water at various temperatures including those in the supercooled region. We await the arrival of the data tapes from the University of North Carolina. The temperature dependent simulation data is expected to give a better picture of the bimodal volume distribution in bulk water.

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 7-10. The data indicate that significant structuring still occurs at rather high temperatures. Indeed ΔCp 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 physiological 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.

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:

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

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) then 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 performed. 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 linerboards show Cl at the surface and are generally more hydrophillic while linerbaords 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.

A review of the literature suggests that dynamic surface tension effects in detergent solutions are important to the penetration of these solutions. Higher molecular weight detergent solutions exhibit more profound dynamic effects. Further study of dynamic surface tension and its effect on liquid penetration are planned.

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 and 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 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 used 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 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 properties such as cyclic humidity creep will be investigated.

UTILIZATION OF RECYCLED FIBER

STATUS REPORT

FOR

A NEW PROJECT

TO THE

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

October 18, 1989

PROJECT TITLE: UTILIZATION OF RECYCLED FIBER

Date: 1/9/89

Budget: $80,000

Period Ends: 6/30/90

Project No.: New

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 imitations 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 carious 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.