THE INSTITUTE OF PAPER CHEMISTRY, APPLETON, WISCONSIN

STATUS REPORTS

To The

PAPER PROPERTIES AND USES

PROJECT ADVISORY COMMITTEE

April 1-2, 1986
The Institute of Paper Chemistry
Continuing Education Center
Appleton, Wisconsin
ATTENDANCE
PROJECT ADVISORY COMMITTEE MEETING
PAPER MATERIALS DIVISION

April 1-2, 1986

COMMITTEE MEMBERS

Gary G. Homan (Chairman)
Westvaco Corporation

Alan F. Button
Champion International

Hanuman P. Didwania
Container Corporation of America

John L. Firkins
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IPC STAFF

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Norm Colson  Kurt Schrampfer  Mike Westphall
Jerry Dees  Carl Smith  Bill Whitsitt
Chuck Habeger  Wendall Smith  Jill Zeitler
Keith Hardacker  Bob Stratton

STUDENTS

Bernie Berger  Brian Berger
Proposal for Consideration

Background: Paper is currently being challenged by plastics and other materials in several markets. This competition between paper and other materials is based in large part upon technology advances and fundamental properties of paper versus these competing materials. IPC has the opportunity to provide leadership in basic research into some of these fundamental properties and provide information important to a broad segment of the membership. IPC resources are limited, and new projects must not result in significant dilution of effort or staff expertise built up in primary areas. However, some initial efforts on one or more of the suggested new areas or alternatives suggested by the committee could be undertaken and reviewed every six months at the PAC meetings to ensure that the projects enhanced overall IPC programs and support to members.

Specific Technical Project Options For Consideration

1. Surface and Printability - In many new paper applications, printability is critical. In turn, this printability is related to the surface characteristics of the paper. New technologies in papermaking provide the means of altering the paper surface either on-machine or in off-machine processing. However, the basic technical understanding of what is occurring at the surface of the paper, how processing steps modify the paper surface, and how these surface modifications impact printability are not well understood.

2. Adhesion to Paper Surfaces - In a wide variety of applications, paper is bonded to or coated with other materials. The chemistry and physics of paper surfaces and individual pulp fibers will impact adhesion. Studies of techniques to physically or chemically modify paper surfaces or individual cellulosic fiber surfaces could yield information of value in designing improved paper composite structures or coated products.

Committee members may wish to add other topics, or expand on the two listed. In either case, any new projects must explore technical fundamentals, and avoid specific product and process areas where member companies have proprietary technology.

Recommendation: Approve appointment of a subcommittee consisting of at least one member of the IPC staff and at least one member of the PAC. This subcommittee will review the project options listed and any others added by the committee with member company representatives and the RAC and prepare a research project proposal for committee action at the next PAC meeting.
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 April 1-2, 1986, in Appleton. A meeting agenda can be found inside the booklet.

If you are staying at the Continuing Education Center, the attached pick card gives the combination to the front door so that you may gain entrance if you arrive after the door is locked. Room schedules will be posted in the lobby. If you have not yet made your reservations, and wish to stay at the CEC, please advise Ms. Sheila Burton at (414)738-3259.

The meeting will adjourn at 11:30 a.m. on Wednesday, April 2 in time for you to catch the 12:15 flight to Chicago if you wish.

We look forward to seeing you on April 1. Best regards.

Sincerely yours,

Gary A. Baum
Director
Paper Materials Division

GAB/sb
Enclosures
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AGENDA

PAPER PROPERTIES AND USES
PROJECT ADVISORY COMMITTEE

April 1-2, 1986
The Institute of Paper Chemistry
Continuing Education Center
Appleton, WI

Tuesday -- April 1

8:30 a.m. Welcome/Introduction  Homan/Baum
8:45  PROJECT OVERVIEW  Baum
9:15  PROJECT REVIEWS  Stratton/Hardacker
      Internal Strength Enhancement
10:15  COFFEE BREAK
10:45  PROJECT REVIEWS  Whitsitt/Halcomb
      Board Properties and Performance
12:00 noon  LUNCH
1:00  TOUR OF PAPER MATERIALS DIVISION LABORATORIES
2:15  PROJECT REVIEWS  Waterhouse
      Strength Improvement and Failure Mechanisms
3:00  COFFEE BREAK
3:30  PROJECT REVIEWS  Baum/Habeger
      Process, Properties, Product Relationships
      On-Line Measurement of Paper Mechanical Properties
5:15  SOCIAL TIME
6:00  DINNER (CEC Dining Room)

Wednesday -- April 2

7:15 a.m.  BREAKFAST (CEC Dining Room)
8:00  DISCUSSION OF PROJECTS  Committee
Dr. Gary G. Homan (Chairman) -- 6/86
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*Date of retirement from committee.
Status Report
to the
PAPER PROPERTIES AND USES
PROJECT ADVISORY COMMITTEE

Project 3526
INTERNAL STRENGTH ENHANCEMENT

February 26, 1986
PROJECT SUMMARY

PROJECT NO. 3526: INTERNAL STRENGTH ENHANCEMENT

PROJECT STAFF: R. Stratton, J. Becher, K. Hardacker

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 1986-87 are on the attached 1986-87 Project Form.

SUMMARY OF RESULTS LAST PERIOD: (April 1985 - September 1985)

(1) Polymer combinations including polystyrene sulfonic acid (PSFA)/polyamide polyamine epichlorohydrin (PAE) and alginate/PAE provided significant improvements in strength properties over the controls in a 57% yield classified softwood unbleached kraft pulp but these combinations were generally less effective than the previously tested carboxymethyl cellulose (CMC)/PAE and polyacrylic acid (PAA)/PAE combinations.

(2) The effectiveness of pectins in an average-yield classified softwood unbleached kraft pulp was greatly enhanced by the addition of PAE to the extent that the resulting strength properties approached, equalled, or exceeded those of CMC/PAE or PAA/PAE depending upon the polymer ratio and addition level. However, cost would eliminate pectins from further consideration at this time.

(3) A study of fines and polymer combinations in a softwood TMP indicated that the readdition of untreated fines to the classified pulp produced tensile strength levels which approached or equalled those of the original whole pulp in dry and wet tensile and dry Et. The addition of polymer bonding to the classified (long) fiber fraction was generally more effective than addition to the fines fraction. Combining CMC/PAE-treated classified fibers and fines to form a whole treated pulp was, in most cases, more effective than adding the same amount of polymer to the original untreated whole pulp. In general, CMC/PAE was more effective than PAA/PAE in this 88% yield pulp.

(4) Diffuse reflectance FTIR analysis of handsheets treated with PAE and polymer combinations containing PAE indicates that ester formation and hence, covalent bonding occurred with several pulps varying in yield and wood source.
(5) Repulpability studies using handsheets from a 49% yield whole unbleached softwood kraft showed that the Thwing formation values of papers treated with CMC/PAE of PAA/PAE were equivalent to the blank controls and those treated with PAE alone after 5 or 7 minutes of intensive mechanical and chemical treatment.

(6) Initial attempts to use the new fiber load-elongation instrument (FLER II) to measure the strength of single fiber-fiber bonds were unsuccessful. New techniques are being developed.

(7) Techniques were further developed to measure the bending modulus of paper and board samples as a function of relative humidity using the vibrating reed method. A humidity control system was built which will allow measurements to be made in the range 0-95% RH at room temperature. Studies on the effect of air damping and strain amplitude showed that only minor corrections to the modulus were required.

(8) Fixtures and techniques are being developed for measuring the bond strength of fibers attached to the edges of microscope cover slips. A precision-ground differential lead screw has been ordered to replace the original cut-thread screw, which has found to have unacceptable pitch variations.

SUMMARY OF RESULTS THIS PERIOD: (October 1985 - April 1986)

(1) The duopolymer systems comprised of CMC/PAE and PAA/PAE were found to be effective bonding agents in a spruce chemimechanical pulp as had been found previously in softwood unbleached kraft and TMP pulps. Also, as previously noted, the duopolymer combinations were more effective (relative to the blank controls) in the classified pulp than in the whole pulp.

(2) A study of fines and polymer combinations in a softwood unbleached kraft pulp showed that re-addition of fines to the classified pulp failed to match the original whole pulp in dry and moist strength properties. This differs from the previously tested TMP in which case re-addition of fines produced dry breaking length, Et, and moist tensile properties which were roughly comparable to or greater than those of the whole pulp when measured at the same moisture content. Once again, maximum strength was generally obtained when the polymers were added to the long fiber fraction. Although, in this case, the duopolymer systems were more effective than PAE in both the classified and whole pulps.

(3) Available results indicate that PAE is as effective as CMC/PAE and/or PAA/PAE in improving moist compressive strength.

(4) In a continuing study of bonding mechanisms in duopolymer systems, several series of tests were carried out using diffuse reflectance FTIR analysis. The results revealed that rather substantial strength improvements can be achieved in the absence of covalent bonding; more specifically, in the absence of ester formation. However, the maximum strength levels attained under these conditions are notably lower than those produced by ester formation.

(5) New bonding and handling techniques were developed to permit the use of the FLER II for measurement of single fiber/fiber bond strengths.
Better defined bond area measurements via Page's vertical polarized light method were obtained by bonding an undyed fiber to a fiber dyed black.

Measurements of bond strength, bonded area, and locus of failure for an unrefined, loblolly pine springwood, kraft pulp revealed a broad distribution of the quantitative results. Poor correlation between bond failure load and bonded area, in agreement with previous workers, may suggest that Page's technique is not a valid measure of bonded area. SEM micrographs showed permanent deformation in the bonded area but little fiber wall tearing or disruption.

The measurements were repeated on the same pulp which had been treated with the PAE/CMC additive combination found effective for conventional kraft pulps in the earlier handsheet studies. Increases in average load at failure, bonded area, and specific bond strength (load/area) of 150, 20, and 80%, respectively were found. In contrast to the untreated fibers, examination of the formerly bonded areas by SEM now showed extensive tearing and picking of the fiber walls.

There has been little activity in development of the FLER II in the last six months. Nevertheless, a new differential lead screw has been obtained and installed. Operation of the instrument is now satisfactory.

The techniques are being developed for testing fiber/fiber bond strength by mounting a fiber on the edge of a microscope glass cover slip, placing two such mounted fibers in the FLER II at right angles to each other, wetting them, applying a small compacting load, then tensile testing the bond after it has dried. Early tests have been partially successful.

A small, 90-degree, glass prism has been mounted at the "fixed" specimen clamp of FLER I. One face forms a surface for fiber lateral compaction; another permits direct viewing of the fiber as it is being compacted. Compacting is done with a narrow flat ground on the edge of a razor blade mounted in the "movable" clamp. The technique is being further developed.

It will be desirable to photograph the fibers as they are being tested in the several possible modes. A video camera has been considered, but its resolution would not be adequate as it would have to be used. Consequently, we are looking at suitable photographic equipment.

During the next period we intend to bring the bond testing and the compaction techniques to working order and begin the investigation of fiber characteristics.
PROJECT TITLE: Fundamentals of Internal Strength Enhancement  

PROJECT STAFF: R. Stratton/J. Becher/K. Hardacker  

PRIMARY AREA OF INDUSTRY NEED: Properties related to end use  

PROGRAM AREA: Moisture tolerant, superior strength paper and board  

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:  

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

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

RESULTS TO DATE:  

PART ONE: Improved bonding via chemical additives.  

Results presented in previous reports indicated that cationic/anionic duopolymer additives (primarily CMC/PAE and CMC/PAA) were very effective in improving the strength properties of several softwood unbleached kraft pulps as well as a softwood TMP. In addition to high levels of dry, moist, and wet tensile properties, these combinations significantly improved tensile energy absorption (TEA), extensional stiffness (Et), and stretch. The addition of these bonding agents to the softwood TMP revealed that superior results were generally obtained when added to the long (classified fiber) fraction. A subsequent study of fines and bonding agents in an average yield softwood unbleached kraft pulp showed that readdition of fines to the classified pulp failed to match the original whole pulp in any of the measured dry or moist tensile properties. This differs somewhat from the TMP where readditions of fines produced dry tensile and Et values roughly comparable to the whole pulp. As was found in the case of the TMP, maximum strength in the kraft pulp was generally achieved when CMC/PAE was added to the long fiber fraction.
CMC/PAE and PAA/PAE also proved to be effective bonding agents for a spruce chemimechanical pulp. One or both of these combinations has proved effective in all pulps tested thus far. This favorable result is somewhat tempered by the fact that their efficiency is generally lower in whole pulps than in classified pulps.

Diffuse reflectance FTIR analysis has indicated that covalent bonding occurs when the duopolymer systems are applied to cellulose, but it has not been established if the bonding occurs between the added polymers or between the polymers and cellulose. This work is being extended to include polymer systems where covalent bonding is not possible but where other forms of bonding may occur.

PART TWO: Fundamentals of bonding.

A literature search has been conducted. An instrument to measure axial or transverse fiber mechanical properties and fiber-fiber bond strength has been designed and constructed and is currently being readied for data gathering.

Techniques were developed to study the details of the fracture of the bond between two single fibers. They consisted of:

a) forming the fiber/fiber bond,
b) measuring the bond area using vertical polarized illumination,
c) determining the bond strength, and
d) determining the locus of failure of the bond using the scanning electron microscope.

Results on loblolly pine earlywood fibers revealed a normal distribution of bond areas and a bimodal distribution of bond breaking loads. Examination of the formerly bonded areas with the SEM showed permanent deformation where the fibers had been pressed together but little rupture (tearing) of the external fiber surfaces.

A vibrating reed instrument has been developed to measure the bending modulus of paper and board samples. A range of temperature from ambient to 200°C and a range of relative humidities from 0 to 95% at room temperature can be covered.

PLANNED ACTIVITY FOR THE PERIOD:

PART ONE:

(1) The study of bonding agents will continue. While several anionic/cationic polymers combinations have been found to be quite effective, other materials will be given consideration based on chemical structure and known properties.

(2) The utilization of duopolymer bonding agents will be expanded to include one or more bleached pulps.

(3) Means will be sought to improve the efficiency of polymer bonding agents in whole (fines-containing) pulps.
(4) The study of bonding mechanisms through chemical analysis will be continued in an effort to differentiate between polymer-to-polymer bonds and polymer-to-fiber bonds.

PART TWO:

(1) We plan to measure single fiber properties as functions of moisture content, refining, yield, and pulping method. The measurements will include both axial and transverse properties. The initial work will be with softwoods.

(2) Failure of single fiber/fiber bonds will be continued, with correlations expected among bonded area, bond strength, and locus of failure.

(3) Effective chemical additives identified in Part One will be used in forming single fiber/fiber bonds, whose failure will then be examined as above.

(4) Studies on the effects of pulp yield and refining on mode of bond failure will begin.
Status Report
to the
PAPER PROPERTIES AND USES
PROJECT ADVISORY COMMITTEE

Project 3571
BOARD PROPERTIES AND PERFORMANCE

February 26, 1986
PROJECT SUMMARY

PROJECT NO. 3571: BOARD PROPERTIES AND PERFORMANCE  
February 26, 1986

PROJECT STAFF: W. J. Whitsitt, R. A. Halcomb

PROGRAM GOAL:

Develop relationships between 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:

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

* To improve the performance/cost ratios of combined board (including medium).

* The short term goals are directed to (1) using structural models to assess the impact of papermaking factors on combined board and box performance and (2) improving medium end-use and converting performance properties.

PROJECT RATIONALE, PREVIOUS ACTIVITY AND PLANNED ACTIVITY FOR FISCAL 1986-87 are on the Project Form that follows.

SUMMARY OF RESULTS LAST PERIOD: (March 1985 - September 1985)

Section 1 - Corrugating Medium Runnability and Improvement

(1) We have proposed a model which potentially relates critical corrugating speeds for high-lows, fracture and strength degradation to medium properties, nip geometry and operational factors.

(2) Our model indicates speeds which result in fracture will increase as the medium friction coefficient and thickness decrease, and tensile strength and stretch increase. These trends appear reasonable. Our initial comparisons of observed and predicted fracture speeds are encouraging.

(3) It appears that high-low flute formation should be related to the stress levels predicted from the model as a function of corrugator speed. The higher the stress level, the greater the high-lows. Thus it appears high-lows are influenced by the same medium properties listed above. However it may be necessary to take other medium properties into account.

(4) In addition to the medium properties the model indicates that fracture speeds will increase as the wrap angle and brake tension decrease and the flute tip radius increases.

(5) A computer program for analyzing flute profile and nip geometry effects has been developed. We are using this to provide input for our modeling and to consider better profiles for heavy weight mediums.
Section 2 - ECT Results

(1) To clarify the relative effects of linerboard compressive strength and flexural stiffness experimental linerboards have been made which have differing ratios of these properties. For this purpose we have varied density, directionality and an internal strength additive at three basis weight levels.

(2) Densification increased compressive strength but flexural stiffness decreased.

(3) Increasing MD/CD orientation decreased CD compressive strength but the geometric mean stiffness remained constant.

(4) A starch additive was also used. It slightly increased both compressive strength and flexural stiffness.

(5) These changes in papermaking factors affected the desired variations in linerboard strength/flexural stiffness ratios. We have combined the linerboards with a 26-lb medium and are proceeding to evaluate their ECT strengths.

SUMMARY OF RESULTS THIS PERIOD: (October 1985 - March 1986)

Section 1 - ECT/Box Compression

(1) Experimental linerboards were made to test the effects of changing linerboard compressive strength-to-flexural stiffness ratios on combined board and box performance. These linerboards were made into C-flute combined boards on the Institute's pilot corrugator. In other work we have experimentally varied medium properties.

(2) Our results indicate that ECT is primarily dependent on the compressive strengths of the liners and medium. The flexural stiffness of the liners plays a minor role.

(3) ECT can be satisfactorily related to the elastic stiffnesses of the linerboard and medium.

(4) We have also related the elastic stiffnesses of the components to the flexural stiffnesses of the combined board, taking into account flute geometry.

(5) Box compression predictions indicate that top load compressive strength increases as the linerboard and medium are densified by wet pressing. Making a squarer linerboard also increases box compressive strength.

Section 2 - Process Research

(1) Fracture speeds predicted with our runnability model decrease with increasing basis weight of medium. This is the same trend as is obtained experimentally.
(2) The stress ratios calculated from the model are correlated with the occurrence of high-lows for mediums of different basis weights.

(3) Work is underway to make mediums with various combinations of fiber orientation, refining, pressing, and weight. In terms of end-use the results will show how to balance flat crush and ECT demands. They will also provide information on some of the parameters entering into our runnability model.

(4) The strength degradation of the medium as speed is increased is under study. Preliminary results show that the tensile strength of the formed medium decreases with increasing speed. The reductions in strength are related to the applied stresses and changes in draw factor.
Project 3571

PROJECT TITLE: Board Properties and Performance
PROJECT STAFF: W. Whitsitt/R. Halcomb
PRIMARY AREA OF INDUSTRY NEED: Properties related to end uses.
PROGRAM AREA: Performance and Properties of Paper and Board

PROGRAM GOAL:

Develop relationships between 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:

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

• To improve the performance/cost ratios of combined board (including medium).

• The short term goals are directed to (1) using structural models to assess the impact of papermaking factors on combined board and box performance and (2) improving 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 structurally sound models. Such structural models identify the critical board properties needed for end use performance. They can then be used to select papermaking approaches to maintain or improve box performance at less cost. An important step is to incorporate the elastic stiffnesses of the board into such models if possible. This will allow us to use our developing knowledge on how papermaking factors affect the elastic stiffnesses to make board improvements.

RESULTS TO DATE:

Rayleigh-Ritz methods have been used to analyze container failure under several types of load. Finite element techniques have been used to model the bending behavior of container board. Analysis of present ECT vs. component local buckling models indicates they fail to predict ECT performance when the liner or medium density is changed. Therefore new models have been developed which show that combined board 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. These results have been experimentally validated and are being extended to box compression. In the case of medium we have shown that the compressive strength is lowered by high bending and shear stresses imposed during forming. These losses in strength lower flat crush and ECT. The losses are inversely related to the density and Z-direction elastic stiffness of the medium. Densification via wet pressing is one way to improve end-use performance of medium.
Our current 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.

PLANNED ACTIVITY FOR THE PERIOD:

The relationships being developed will show how the elastic stiffnesses and compressive strengths of the components will affect combined board ECT and box compression strength. The analysis will help us assess the relative importance of compressive strength and the bending stiffnesses of the liners and medium in determining box performance. We will confirm and validate the relationships using components made under various papermaking conditions as well as commercial boards.

We plan to use finite element techniques to model flat crush load-deformation characteristics in relation to medium properties and flute geometry. This work will better define how crushing of board during conversion and end-use degrades box performance. The same technique will be considered as a way to improve our understanding of the bending stresses during fluting.

Our research on medium shows that densification via wet pressing improves strength retention during fluting and gives higher ECT and flat crush in the combined board. We will continue and extend this research to consider other ways to improve formability and performance. This will include work on sheet directionality, pressing, and refining.

As an outgrowth of this and related work for FKBG, we will investigate ways to show what properties of the linerboard and medium are required for high-speed runnability on the corrugator. Runnability refers to the critical speeds associated with strength retention, the development of high-lows and flute fracture.

We are also considering initiating work in fracture mechanics as related to flute fracture and the application of power spectral density techniques to flute uniformity.

POTENTIAL FUTURE ACTIVITIES:

Application of similar techniques to end-use failures involving flexure, shear and combined tension, flexure and shear.
Status Report
BOARD PROPERTIES AND PERFORMANCE
Project 3571

The objectives of this program are to: (1) develop relationships between container performance, combined board and component properties, and (2) determine ways to improve the cost/performance ratios of medium and linerboard. To fulfill these objectives we must consider both end-use performance and processing runnability on the corrugator. Therefore our current work is divided into two parts, namely, (1) ECT and box compression performance and (2) process performance research.

ECT and Box Compression Relationships

An important part of our past work has been directed to developing relationships between combined board ECT and the properties of the linerboard and medium which will be valid under most papermaking conditions. A specific goal is to incorporate the elastic stiffnesses of the components in such relationships. This will allow us to use nondestructive ultrasonic techniques to characterize board and box performance. It also enables us to use our developing knowledge on how papermaking factors affect elastic stiffnesses to assess ways to improve performance.

Initially we tried to adapt the FPL ECT model to incorporate either STFI compressive strength values and/or the component elastic stiffnesses. While these attempts achieved moderate success, they required the introduction of many empirical constants. In the course of this work it was found that the model did not properly predict the effects of some papermaking changes.

As an alternative approach we decided to model ECT in the same way as the Institute top load box compression formula. This is termed the miniature
plate approach. Conceptually ECT is set equal to the sum of the maximum strengths of the individual liner and medium plate elements. Following this approach the contributions of the liners and medium are formulated as the product of two terms:

Liner: \((\text{compressive strength})^b \times (\text{mean flexural stiffness of liner})^{1-b}\)

Medium: \((\text{compressive strength})^c \times (\text{mean flexural stiffness of medium})^{1-c}\)

The constants \(b\) and \(c\) must be experimentally determined and their magnitudes will reflect the relative importance of the two properties. For compressive strength we employ either short span compressive strength values or the in-plane and out-of-plane elastic stiffnesses.

As discussed in the last status report we obtained good predictive accuracies using this approach. The magnitudes of the exponents indicated that ECT is primarily dependent on the compressive strength characteristics of the liners and medium. The flexural stiffness of the medium has a negligible effect; the flexural stiffness of the liners appears to have only a small effect on combined board ECT.

To validate these results experimental linerboards were made wherein we varied the ratio of CD compressive strength to flexural stiffness. This was accomplished by varying the density via wet pressing, directionality and incorporating additives. The effects of these factors on the compressive and flexural properties of linerboard were summarized in the October status report. In general, densification increased linerboard compressive strength but decreased flexural stiffness. Decreasing the directionality had little effect on the geometric mean flexural stiffness of the liners but increased compressive strength. These were the expected changes.
The combined board results for the constructions showed that the edgewise compressive strength is, by far, the most important factor affecting ECT. Thus the best way to improve ECT is to increase the compressive strengths of the liners and medium.

Since the last meeting we have expanded our database to include constructions having varying medium qualities combined with a commercial 42-lb liner or experimental liners of different weights and densities. This supplements the above data which were based on constructions having varying liner qualities combined with a commercial medium.

The ECT model equations for the complete array are essentially similar to those obtained previously. We are now able to relate ECT to either the short span compressive strengths or the elastic stiffnesses of the liners and medium.

Combined Board Flexural Stiffness

We are now expanding our work to show how changes in linerboard and medium manufacture can be expected to affect box compression performance. For this purpose it is necessary to relate both ECT and combined board flexural stiffness to component properties.

Combined board flexural stiffness is primarily related to the elastic stiffnesses of the liners and combined board caliper. The medium makes a small contribution in the cross direction. Because the ultrasonically determined elastic stiffnesses are greater in magnitude than stiffnesses determined in mechanical test machines, it was necessary to develop an adjustment factor. Comparison of E\(t\) values obtained from compression load-deformation curves on a mechanical tester with ultrasonic values exhibited good correlation. The
results indicated that multiplying the ultrasonic values by 0.74 would bring them into good agreement with the mechanical test results.

Comparisons were then made between measured combined board flexural stiffnesses in both directions and values estimated from the elastic stiffnesses of the components and flute geometry. Good agreement between observed and estimated combined board flexural stiffnesses was obtained.

Inspection of the combined board flexural stiffness values showed that they increased as the density of the linerboard facings was increased by wet pressing. Increasing the directionality of the liners increased the MD and decreased the CD flexural stiffness as expected. In this case the geometric mean stiffness which enters into the McKee box compression formula remains essentially constant.

We are now using our ECT and flexural stiffness relationships to estimate how various papermaking changes in the manufacture of linerboard and medium would affect box compressive strength. For this purpose the McKee box compression formula is being used. Using the relationships developed above, our results indicate that:

1. Top load box compressive strength increases as the density of the liners is increased by wet pressing.
2. Making a more square linerboard sheet increases box compression because the CD compressive strength of the liners is increased.
3. Increasing the density of the medium increases box compression strength.
4. Chemical additives also increase box compression strength if present in sufficient amounts to increase the compressive strength and elastic stiffnesses of the linerboard. Corresponding trials have not been made on medium but the same effect would be expected.
This work is still in progress. It may be desirable to analyze the box compression results to determine how to optimize box compression strength in terms of liner and medium fiber usage.

Flat Crush Modeling

As noted in the last status report we are planning to apply finite element analysis techniques in a number of structural applications. One of the first applications will involve the flat crush load-deformation curve. The analysis will help identify the medium properties needed to enable combined board to resist crushing stresses during manufacture and use.

After surveying available software it was decided to purchase MSC/pal from The MacNeal-Schwendler Corporation. This should enable us to start work on the flat crush application. As our needs expand other alternatives can be considered.

Process Research

To supplement our research on high speed runnability for the FKBG we are developing models which will explain how critical corrugating speeds are dependent on medium properties, nip geometry, and operational factors. The models are based on physical analysis of the corrugating process but are empirical at this time. We believe the model has application to high-low flute formation, flute fracture, and to strength losses during fluting. A discussion of the model concepts is contained in the last status report.

Our runnability testing for FKBG on current commercial mediums will allow us to test the model and will suggest where modifications are needed. Analysis of average results for 26, 33, and 40-lb mediums indicates that the fracture speeds decrease with basis weight in the expected way. These estimates
appear to properly capture the basis weight trend. However, it may be necessary to make some allowance for the decreased flank clearance in the nip for the thicker mediums.

The stress ratios calculated from the model are correlated with the occurrence of high-lows. For example for a given speed 26-lb mediums exhibit lower stress ratios and high-lows than 33-lb medium on the average. Thus mediums with combinations of friction, tensile, stretch and thickness which yield lower stress ratios at a given speed should exhibit lower high-lows.

We have considered introducing another factor to allow for shear effects as the medium is bent to the flute contour. Exploratory calculations were made for various mediums, but there was no clear indication that the shear correction would materially improve runnability predictions. We need to carry out a more detailed analysis of the stresses induced during bending.

Work is underway to determine how quickly the medium tensile strength is reduced as corrugating speed is increased. As speeds increase the medium is exposed to higher stresses during forming. These result in greater high-lows and flute fracture. Several 26, 33 and 40 lb mediums are being evaluated. Long-span tensile tests are being carried out on medium formed at speeds to 1000 fpm. Preliminary results on one medium show that the tensile strength reductions are related to the applied stresses or change in draw factor. Near the fracture speed the tensile strength of the medium is reduced to nearly zero.

Medium Improvement

Medium requirements include both MD and CD strengths for end-use and MD strength for runnability. As medium strength is increased it should be
possible to square up the sheet to improve CD strength while maintaining reasonable levels of flat crush and runnability. Work is underway to make experimental mediums with various combinations of fiber orientation, refining, wet pressing, and weight. In terms of end-use the results should show how the flat crush and ECT demands can be balanced. They will also provide information on some of the runnability model parameters.

**Exploratory Research**

At the last meeting committee members discussed two potential areas for future study. First, questions were raised relative to the occurrence of periodicities in high-low flute formation. We are reviewing past work in this area, much of which we carried out for the FKBG. With the completion of our new high-low monitor it will be possible to analyze high-lows more efficiently than in the past. Initially it is planned to determine whether high-lows exhibit periodicities which could be related to machine element phenomena using power spectral analyses. This work will be carried out at a range of speeds. In addition, a commercial sample has been procured to study cross machine variations.

The second area involves possible application of fracture mechanics to runnability modeling. Presently we are reviewing the literature to determine how this approach could best be used.
Status Report
to the
PAPER PROPERTIES AND USES
PROJECT ADVISORY COMMITTEE

Project 3469
COMPRESSIVE STRENGTH

February 26, 1986
PROJECT SUMMARY

PROJECT NO. 3469: STRENGTH IMPROVEMENT AND FAILURE MECHANISMS

STAFF: J. Waterhouse, W. Whitsitt

February 26, 1986

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.

PROJECT RATIONALE, PREVIOUS ACTIVITY and PLANNED ACTIVITY FOR FISCAL 1986-87 are on the attached 1986-87 Project Form.

SUMMARY OF RESULTS LAST PERIOD: (April 1985 - September 1985)

(1) Exploratory work has been done to determine the compressive strength potential of small wood coupons (16 mm x 16 mm) using ultrasonic characterization techniques.

(2) The effects of PAE, PAE/pearl corn starch additions on compressive strength and related properties has been determined.

(3) Formette linerboard handsheets have been made for investigating the effects of basis weight, fiber orientation and cationic starch addition on combined board strength and model evaluation (Project 3571).

(4) The surface addition of certain polymers to wet and dry Formette handsheets has been investigated. The polymers included pearl corn starch, cationic high M.W. starch and a polyvinyl acetate latex.

(5) The anisotropy of Formette handsheets dried under complete restraint is reduced by processes which improve bonding, e.g. refining, wet pressing, polymer addition, while the converse is true for processes which reduce bonding, e.g. calendering.

(6) A review paper on "Converting and Paper Properties" has been prepared and presented at the Tappi Plastics, Polymers and Laminations meeting in Chicago September 8.

(7) A paper on "Z-direction variation of internal stress and properties in paper" has been prepared and will be presented at the Japanese Tappi/CPPA Tech. Sect. Paper Technology Meeting in Tokyo, Japan on October 17, 1985.

(8) In student related work Laurine Charles is investigating the effects of supercalendering on the strength related properties of paper and board.
SUMMARY OF RESULTS THIS PERIOD: (October 1985 - March 1986)

(1) The compressive strengths of small pulped wood samples were measured and compared with compressive strengths measured on paper samples made from the separated wood fibers. The values for the handsheets (of the same nominal density as the wood specimens) were typically one-half the values obtained for the wood samples.

(2) Work has begun in characterizing small wood coupons, differing in lignin content, using non-destructive measurements. The purpose is to elucidate, if possible, the maximum strength and/or stiffness potential of pulped unseparated fibers.

(3) In student related work L. Charles has determined the effects of supercalendering on strength and other physical properties of uncoated Formette handsheets made with various levels of wet pressing and fiber orientation.

(4) In student related work T. Bither has started work on determining the factors responsible for differences in strength development which occur by refining and wet pressing.

(5) In student related work M. Kemps has started work on the measurement of internal stresses in paper and board using the layer removal technique.
PROJECT TITLE: Strength Improvement and Failure Mechanisms

PROJECT STAFF: J. Waterhouse/W. Whitsitt

PRIMARY AREA OF INDUSTRY NEED: Properties related to end use

PROGRAM AREA: Improved converting processes and converted products

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.

PROJECT RATIONALE:

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 and likely changes in shipping regulations provide impetus for research on compressive strength. Research to date suggests that there are ways to approach the objective through new papermaking strategies.

RESULTS TO DATE:

We have shown that compressive strength is highly related to a product of in-plane and out-of-plane elastic stiffnesses of paper. 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 compressive stiffness. Our results on oriented sheets indicate that compressive strength increases with refining, and further increases can be obtained by wet pressing to increase density. Within a practical range, higher CD compressive strength can be achieved by decreased fiber orientation 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.

We have developed torsion mode for measuring the out-of-plane shear stress-strain behavior, and studied 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.
PLANNED ACTIVITY FOR THE PERIOD:

We will continue investigations of the compressive behavior of board as a function of composition, structure, and process variables. For the coming period this will include the effects of HW/SW furnish blends on compressive strength and a study of the compressive strength potential of pulped wood chips.

STUDENT RELATED RESEARCH:

Compressive Strength

We are continuing to seek practical ways to improve compressive strength by elucidating the basic mechanisms involved and utilizing them to pursue realistic improvements in the papermaking process and to aid in raw material selection. To this end some of the potential areas for compressive strength improvement we have investigated to date include:

1. Raw materials
   - furnish improvement by species selection
   - high yield pulps with chemical treatment
   - polymer addition

2. Papermaking process variables
   - formation and fiber orientation
   - wet pressing and drying restraint

We expect work to continue in the above areas as new opportunities present themselves.

We are currently concentrating our effort in the raw materials areas. Specifically we are interested in determining the compressive strength potential of certain species as a function of yield and the extent to which this can be realized through the papermaking process.

The compressive strength correlations and mathematical model developed by Habeger and Whitsitt* is continuing to guide our efforts in improving

compressive strength. A simplified version of the final equation from their mathematical model is given as follows:

\[ \sigma_{C/\rho} \propto (C_{11}/\rho)^{2/3} (C_{55}/\rho)^{1/6} (C_{33}/\rho)^{1/6} / RW \]  

where \( \sigma_{C/\rho} \), \( C_{11}/\rho \), \( C_{55}/\rho \), \( C_{33}/\rho \) are specific compressive strength, and the in-plane and out-of-plane specific elastic constants respectively. \( RW \) is the roughness-weakness factor and is given by:

\[ RW = \left( \frac{\text{initial curvature of lamina}}{\text{lamina thickness}} \right) \frac{\text{shear modulus}}{\text{shear strength}} \]  

In our work to date the roughness-weakness factor has been assumed to be constant, and the correlation appears to be unaffected by fiber orientation refining, wet pressing and drying restraint for a given pulp.

A further simplification of Eq. (1) is to write it in terms of in-plane and out-of-plane longitudinal specific moduli. This eliminates the need for measuring the out-of-plane specific shear modulus:

\[ \sigma_{C/\rho} \propto \bar{E}/\rho^{3/4} E_z/\rho^{1/4} \]  

\( \bar{E}/\rho \) is the geometric mean in plane specific modulus and \( E_z/\rho \) is the out-of-plane longitudinal specific modulus.

The correlation given by Eq. (3) above is illustrated in Fig. 1 for a number of pulps. A summary of the regression equations together with their correlation coefficients are given in Table 1.

A change in the correlation with species type and pulping process is noted. The results suggest hardwoods offer high potential for compressive
Table 1. Summary of regression equations (see Fig. 1).

<table>
<thead>
<tr>
<th>Pulp Type</th>
<th>Slope</th>
<th>Intercept</th>
<th>$r^2$</th>
</tr>
</thead>
<tbody>
<tr>
<td>unbl. southern pine</td>
<td>7.845</td>
<td>0.1667</td>
<td>0.974</td>
</tr>
<tr>
<td>bl. southern pine</td>
<td>7.982</td>
<td>-2.984</td>
<td>0.975</td>
</tr>
<tr>
<td>sulphonated red oak</td>
<td>9.507</td>
<td>-4.612</td>
<td>0.961</td>
</tr>
<tr>
<td>NSSC medium</td>
<td>10.81</td>
<td>-6.437</td>
<td>0.992</td>
</tr>
<tr>
<td>unbl. southern pine/ cationic starch/PAE</td>
<td>-2.62</td>
<td>10.0</td>
<td>0.947</td>
</tr>
</tbody>
</table>

![Graph showing regression equations](image)

**Figure 1.** Effect of pulp type on compressive strength correlation.

strength improvement. It has not yet been determined whether the differences in the correlations may be attributed to changes in the roughness - weakness factor. In Fig. 1 and Table 1 it is seen that the addition of additives such as
starch and PAE also have a significant impact on the compressive strength correlation. This effect has been further verified in more recent work, as shown in Fig. 2. A series of liners were produced at three levels of wet pressing, and two levels of fiber orientation. The correlation line for this data is shown by the solid line in Fig. 2. The data points represent the addition of starch and PAE (Kymene 557H), together with the regression line (broken) from Fig. 1, and Table 1. In the earlier results it was found at a PAE addition level of 0.8% that the in-plane specific modulus was only increased by 7.2%, while the specific tensile strength was improved by 40%. This effect is not untypical of polymer reinforcement behavior where the initial modulus is relatively unaffected but there can be substantial increases in tensile strength and other failure properties. Therefore, although it has yet to be proven, it seems reasonable that the improvement in compressive strength, and hence the change in the correlation with polymer addition is due to a reduction in the roughness-weakness factor given by Eq. (2). That is, it is speculated that the shear strength/shear modulus ratio is increased by polymer addition.

It has also been noted in earlier work that where limitations exist to increased refining and wet pressing, polymer reinforcement may be a viable alternative. A further benefit with polymer addition is that improvements in compressive strength may be achieved without reducing flexural stiffness (EI) which occurs with densification by wet pressing.

Species and Yield

We now wish to concentrate our efforts in determining the compressive strength potential of certain species as a function of yield. The intention is to determine this potential by making both in-plane and out-of-plane ultrasonic and compressive strength measurements on small wood coupons, e.g. 1.6 cm X 1.6 cm
at various yield levels. In addition we would like to take a portion of these wood coupons, separate the fibers and form small handsheets (3/4" dia.) and similarly characterize them. These procedures are diagrammed in Fig. 3.

<table>
<thead>
<tr>
<th>Material</th>
<th>Process</th>
<th>Characterization</th>
</tr>
</thead>
<tbody>
<tr>
<td>wood coupon</td>
<td>delignification</td>
<td>in-plane and</td>
</tr>
<tr>
<td></td>
<td></td>
<td>out-of-plane NDT,</td>
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<tr>
<td></td>
<td></td>
<td>compressive</td>
</tr>
<tr>
<td></td>
<td></td>
<td>strength, moisture</td>
</tr>
<tr>
<td></td>
<td></td>
<td>content, etc.</td>
</tr>
<tr>
<td></td>
<td>consolidation</td>
<td></td>
</tr>
<tr>
<td></td>
<td>fiber separation</td>
<td></td>
</tr>
<tr>
<td></td>
<td>mini handsheets 3/4&quot; dia.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>consolidation</td>
<td></td>
</tr>
</tbody>
</table>

Figure 2. Effect of strength additions on compressive strength correlation.

Figure 3. Flow diagram for determining strength potentials.
The feasibility of NDT of wood coupons was demonstrated at our last PAC meeting. We have now prepared a series of wood coupons from two sections of a spruce log for which there is a substantial data base available (Chemical Sciences Division). The coupons have also been subjected to two levels of delignification using the acid-chlorite method.

A technique for making small handsheets 3/4" dia. which might be employed in the above work has been developed by B. Allender, a special student. His research project is concerned with the effects of fiber morphology on the development of internal fibrillation. Water retention values are being measured using an ultracentrifuge and the pulp mat preparation for these measurements appears to be suitable for making small handsheets. Out-of-plane ultrasonic characterization of these small sheets compares favorably with similar measurements made on standard handsheets, i.e. British handsheet mold.

We have recently compared compressive strength measurements on small pulped wood specimens and on handsheets made from the separated fibers. Zero span measurements were also carried out on the wood specimens. The specimens were from an earlier FKBG project (2695-20) concerned with the effects of fiber properties (particularly bending stiffness, EI) on paper compressive strength. Seven pulps were investigated including: Virginia pine earlywood and latewood; Douglas-fir earlywood and latewood; loblolly pine earlywood and latewood; and gum. All of the starting wood chips were pulped using the kraft process to yields near 48%, and then stored in water.

The wood specimens used for testing were separated from the wet pulped chips and had nominal dimensions of 0.01 x 0.1 x 0.7 in². They were dried under a slight pressure before testing. Typical dried densities were around
0.9 gm/cm³. A large number of the small wood specimens were prepared and tested, but many of the results were discarded due to excessive fiber crushing in the STFI or zero span devices, clamp slippage, or damage caused by handling.

The handsheets had been prepared during the earlier work. Sheets were selected that had about the same density as the dried wood specimens (approximately 0.9 gm/cm³). Figure 4 shows the specific STFI compressive strength data versus the specific compressive strength of wood specimens as measured. The handsheet values are typically one-half the values obtained for the wood specimens. If the results are adjusted by correcting for the amount of material actually tested (estimated) and then replotted, one obtains the results in Fig. 5. Now it is seen that some of the results are quite close to the one-to-one correspondence line. This implies that the compressive strength potential of the fibers (as measured in the wood specimens) is being utilized in the paper. Figures 6 and 7 show the wood specimen and handsheet compressive strengths, respectively, plotted against the wood specimen zero span strength. In each case there appears to be a relationship. Figure 6 shows that the compressive strength of the wood specimens is about one-fourth that of the zero span strength. A similar result is found for paper tested in compression and tension.

Failure Mechanisms

During converting and end use, paper may be subjected to a variety of deformation modes which can change its geometry and physical properties. We need to understand these changes and their dependence on the initial paper properties. In future work we will study the deformation behavior of paper when subjected to combined out-of-plane stresses, i.e. shear and normal stresses. This work will be complementary to our current study of a specific converting area, namely supercalendering.
Figure 4. Uncorrected handsheet compressive strength vs. uncorrected wood chips compressive strength.

Figure 5. Handsheet STFI-CS vs. wood chip compressive strengths. Some of the results for Virginia pine, Douglas-fir and gum lie near the perfect compression line.
Figure 6. STFI compressive strength vs. zero span strength for the wood specimens.

Figure 7. STFI compressive strength for the handsheets vs. zero span strength for the wood specimens.
In our previous work on the out-of-plane shear deformation of paper, an appreciation was gained of the variation of internal stress and other physical properties in the Z-direction. Work is continuing in this area in both staff and student work.

**Effect of Supercalendering on Strength Related Properties of Paper**

In student related work Laurine Charles has completed a study of the effects of supercalendering on strength and other physical properties of paper. When paper is supercalendered it may be subjected to as many as nine nips. In this study we were specifically interested in how the action of a single nip modifies paper properties. The normal and shear stresses which a web is subjected to during supercalendering are shown in Fig. 8.

Figure 8. Pressure \( Z_z \) and shear \( Z_y \) through a supercalendar nip (after Peel and Hudson). Filled roll 21 in. diameter, steel roll 31 in. diameter, loaded to 990 pli, nip width 0.24 in.
In our experiments the temperature of a supercalender iron roll was held constant at 165°F and four levels of nip loading (100, 1000, 1500, and 2000 pli at 300 ft/min) were used. The uncoated handsheets subjected to supercalendering were made on the Formette Dynamique using a market bleached kraft pulp having a freeness of 600 CSF. The papermaking variables examined included three levels of fiber orientation and three levels of wet pressing.

Some of the results are shown in Figs. 9-15. We see in Figs. 9 and 11 that whereas both in-plane and out-of-plane elastic constants are increased by wet pressing, supercalendering results in a significant reduction, the rate of which appears to increase with increased wet pressing.

Figure 9. Effects of wet pressing and supercalendering on mean in-plane specific modulus.
Figure 10. Effects of wet pressing and supercalendering on in-plane anisotropy.

Figure 11. Effects of wet pressing and supercalendering on out-of-plane longitudinal modulus.
Figure 12. The effects of wet pressing and supercalendering on compressive strength.

Figure 13. Effect of supercalendering on compressive strength correlation.
Figure 14. Effect of wet pressing and supercalendering on mean specific tensile strength.

Figure 15. Effect of wet pressing and supercalendering on the variation of scattering coefficient.
The in-plane anisotropy of the sheet, as shown in Fig. 10 decreases as the sheet is densified by wet pressing. This effect has been noted in previous work where increased bonding by refining, wet pressing or additives reduces sheet anisotropy. However we note that supercalendering increases anisotropy. Whether this is an indication of a reduced state of bonding within the sheet has not yet been established. Scattering coefficient reduction which is often used as an indication of increased bonding is consistent with increased wet pressing as shown in Fig. 15 while the net effect of changes induced by supercalendering is also a reduction in scattering coefficient.

It would be expected from the changes in elastic properties that there would also be dramatic loss in strength properties particularly at the higher levels of wet pressing. The effects of supercalendering on compressive and tensile strength are illustrated in Figs. 12 and 14 and show, in contrast to our expectations, lower losses in properties particularly at the highest level of wet pressing. Also shown in Fig. 13 is the relationship between compressive strength and the elastic properties, i.e. using Eq. (3) discussed earlier. Clearly the compressive strength correlation obtained by wet pressing is significantly altered by supercalendering.
Status Report
to the
PAPER PROPERTIES AND USES
PROJECT ADVISORY COMMITTEE

Project 3467
PROCESS, PROPERTIES, PRODUCT RELATIONSHIPS

February 26, 1986
PROJECT SUMMARY

PROJECT NO. 3467: PROCESS, PROPERTIES, PRODUCT RELATIONSHIPS

PROJECT STAFF: G. A. Baum, C. C. Habeger

February 26, 1986

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:

(1) To improve our capability of characterizing paper and board materials,
(2) to relate measured parameters to end-use performance (especially in the case of Z-direction measurements), and
(3) to relate measured parameters to machine and process variables.

PROJECT RATIONALE, PREVIOUS ACTIVITY, AND PLANNED ACTIVITY FOR FISCAL 1986-87 are on the attached 1986-87 Project Form.

SUMMARY OF RESULTS LAST PERIOD: (April 1985 - September 1985)

1. Work is progressing to improve the out-of-plane measurements of elastic properties. New transducers have been designed, fabricated, and tested which are superior to previous designs. Equipment has been acquired to automate data acquisition and handling.

2. The robotic or automatic device for measuring in-plane elastic properties has been modified to provide a much more positive action in the turntable assembly. The unit continues to function satisfactorily.

3. An in-plane ultrasonic measurement system has been installed in a Blue-M oven in which the temperature and humidity can be controlled. An automatic balance is mounted on the top of the oven so that a sample may be weighed continuously as oven conditions change (except during ultrasonic measurements). This unit is currently being tested.

4. The microwave device for measuring fiber orientation in paper has been tested on a number of grades with good results. Comparisons with other methods of estimating fiber orientation are underway.

5. A simple three dimensional network model has been developed which appears to be useful in explaining some observed effects. Much additional work is needed in this area, however.

SUMMARY OF RESULTS THIS PERIOD: (October 1985 - March 1986)

1. The anisotropy of an in-plane elastic property vs. angle from the MD is being studied. The area and general geometry of the polar graphs is being investigated relative to process variables such as refining, wet pressing, and yield.

2. For machine made papers the envelope of elastic properties vs. angle from the MD is usually elliptical. The angular displacement of the major axis from the MD is indicative of flows from the paper machine headbox. These vary from point to point in the cross machine direction.

3. Elastic properties have been examined at intermediate to high moisture contents (up to 65%). Generally the water in the sheet dominates the results at moisture contents over 40 to 50%.

4. The new broadband plastic (PVDF) ZD transducers have been perfected and are in use in the longitudinal ZD apparatus. Automation of the laboratory ZD measurement equipment is underway.

5. Work on the effects of refining and yield on the ZD properties is continuing. Oriented sheets have been made from the laboratory pulps described earlier.

6. The effects of non-uniform drying restraints on local sheet properties have been investigated. The results are more complicated than anticipated. Analysis is underway.

7. Surface roughness measurements have been made using a stylus type instrument. The results were compared with standard air leak methods for estimating smoothness. The effects of some papermaking variable on roughness were also examined.

8. A new automatic in-plane elastic property measurement system is under development. Improvements in both hardware and software are planned, including a computer interface to the IBM family of 8088 machines.
PROJECT TITLE: Process, Properties, Product Relationships

PROJECT STAFF: G. Baum, C. Habeger, J. Waterhouse

PRIMARY AREA OF INDUSTRY NEED: Properties related to end uses

PROGRAM AREA: Performance and Properties of Paper and Board

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:

(1) To improve our capability of characterizing paper and board materials, 
(2) to relate measured parameters to end-use performance (especially in the case of Z-direction measurements), and 
(3) to relate measured parameters to machine and process variables.

PROJECT RATIONALE:

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

RESULTS TO DATE:

Ultrasonic techniques for measuring in-plane and out-of-plane elastic properties of paper have been developed. Instruments for measuring these properties have been designed, constructed and tested. These include separate instruments for out-of-plane shear and out-of-plane Youngs modulus, and a robotic tester for measuring the four in-plane elastic properties. A soft platen caliper gage which gives values comparable or superior to existing caliper gages has been designed and constructed. The effects of fiber orientation, wet straining, and wet pressing on elastic properties have been extensively studied using softwood kraft furnishes. The in-plane and out-of-plane elastic parameters have been related to end use tests and converting operations in a number of cases. A microwave technique for determining fiber orientation has been developed.

PLANNED ACTIVITY FOR THE PERIOD:

1. In-plane and out-of-plane elastic constants will be measured on a representative group of samples differing in composition and structure (yield and refining) in different ambient environments. These data will be compared with use-oriented test results, where possible.

2. A device to measure specific scattering coefficients in heavy board materials has been designed and is undergoing construction. This will be used to test boards differing in composition and structure.
3. Work on automation of the ZD velocity measurements is underway. Improvements in the existing apparatus are anticipated.

4. The effort to establish relationships between properties and end-use performance will continue.

5. A fundamental study of formation is planned. This effort will be complementary to an existing contract research program with the API Instrumentation Research Program.

6. A licensing agreement has been prepared concerning the laboratory ultrasonic equipment. Negotiations are underway with several instrument manufacturers.

STUDENT RELATED RESEARCH:

Anisotropy of Elastic Properties

The in-plane robotic tester measures the four in-plane elastic properties of paper and also has the capability to measure them at specific angles to the MD. The areas and general shape of the resultant polar graphs for a particular elastic property have been studied relative to process variables, including refining, wet pressing, and yield. The samples used in the investigation were those prepared and tested by Brian Berger, which have been described in the last Status Report, dated October 22-23, 1985.

Figure 1 shows a polar-like graph of specific stiffness for three levels of Kappa number at constant refining and wet pressing. Only two quadrants are shown. The figure is not a true polar graph because the MD and CD scales are not the same. As expected, as the yield decreases the stiffness increases, but the increase is greatest in the cross machine direction. (The figure is somewhat misleading because of the different scales.) Figure 2 depicts the same data as in Fig. 1 except that specific stiffness is plotted against angle as measured from the MD.

Figure 3 shows the effect of wet pressing on specific stiffness. Increased wet pressing increases the specific stiffness uniformly at all angles. This is in contrast to the results in Fig. 4 which depict the effects of changes in refining level. As in the case of yield, the percentage changes in the cross machine direction are greater than along the MD. These results cannot be explained in terms of drying restraints since all of the specimens were tried under full restraint in both MD and CD. It may be that the increased fiber flexibility due to refining affects the level of fiber orientation in the sheet,
Figure 1. Polar plot of specific stiffness for varying Kappa numbers, all at a refining level of 35 minutes and a wet pressing pressure of 100 psi. Kappa number: □ - 46.5; x - 31.7, ▽ - 16.4.

Figure 2. Specific stiffness ($V^2$) vs. angle with different Kappa numbers. Same data as in Fig. 1. Wet pressing pressure 100 psi, refining level 35 min. Kappa number: □ - 16.4; x - 31.7; ▽ - 46.5.
causing fewer of them (or parts of them) to align in the MD. Figure 4 also shows that the results for the longest refining time are very similar to the 35 minute results, at all angles from the MD.

![Wet Pressing Diagram](image)

Figure 3. Specific stiffness ($V^2$) as angle for different wet pressing pressures, all at a Kappa number of 16.4 and a refining level of 35 min. Wet pressed at: □ - 25 psi; x - 50 psi, ▼ - 100 psi.

This work is continuing with a set of handsheets prepared from a commercial newsprint furnish. The effects of both wet pressing and refining level on specific stiffness are being studied. The results should be available by the time of the April meeting.

**Anisotropy and Headbox Flows**

For machine made papers the envelope of the specific stiffness vs. angle plot is usually elliptical (e.g. Fig. 1). We have discovered that the angular displacement of the major axis of the ellipse from the MD is indicative of local headbox flows not parallel to the MD. Such displacements may vary from...
Figure 4. Specific stiffness ($V^2$) vs. angle for different refining levels, all at a kappa number of 16.4 and a wet pressing pressure of 100 psi. Beating time: $\bigcirc$ - 0 min; $\times$ - 10 min; $\uparrow$ - 35 min; # - 50 min.

Point to point in the cross machine direction. Figure 5 is a polar graph of specific stiffness vs. angle which shows the major axis about 12 degrees to the left of the MD. This was an actual commercial sample that was submitted to our laboratories for testing because of certain converting problems. Figure 6 shows how the angular displacement may vary in the cross machine direction; four different CD strips were tested. The average value obtained from the four specimens in Fig. 6 are shown in Fig. 7. It is not known at present whether the sinusoidal shape is typical. If the interpretation above is correct, Fig. 7 suggests that there is a transverse component of flow on the front half of the web to the left of the MD which changes to the right of the MD just beyond the center, and then again switches to left of MD. It's not difficult to see how problems could arise in converting this web if it were cut into narrow rolls.
Figure 5. Polar graph of specific stiffness vs. angle.

Figure 6. Angular displacement across the paper machine for four scans.
Elastic Properties at High Moisture Levels

The elastic properties $C_{11}$, $C_{33}$, $C_{44}$, and $C_{66}$ were measured as functions of moisture content up to moistures exceeding 60%. The samples, differing in refining and wet pressing levels, were saturated with water and then placed between layers of Saran to minimize moisture loss during the measurements. After a given measurement one layer of Saran was removed and the sample allowed to air dry to a new, lower, moisture level. To minimize moisture loss during the actual measurements the out-of-plane elastic parameters were determined first because these could be done with the Saran in place, whereas the in-plane measurements required the Saran be removed. These measurements were made in regions of the specimen different from the out-of-plane measurements because the latter left depressions in the wet sample. The reported moisture contents are based on an average of the specimen wet weights before and after each experiment. While the moisture lost during a set of measurements was typically small,
less than 4%, there is some uncertainty as to the value of the moisture content in the sample at the location of any particular test. It is well known that the distribution of water in a sheet at high moisture levels can be very non-uniform.

Figures 8 and 9 show the two in-plane parameters, $C_{11}/\rho$ and $C_{66}/\rho$, respectively, versus moisture content. At high moistures both seem to approach a specific stiffness value less than one $(\text{km/sec})^2$, which would correspond to an ultrasound velocity less than one km/sec. This is lower than the velocity of sound in pure water (1.49 km/sec). The triangle, x, and square symbols in Fig. 9 represent low, medium, and high levels of refining, respectively. The effects of refining on $C_{66}/\rho$ are observed only at the lowest moisture levels.

Figure 10 shows that the relationship between $C_{66}$ and $C_{11}$ (found for paper) exists even when the measurements are made over a broad range of

![Figure 8. Specific stiffness $(C_{11}/\rho)$ vs. moisture content.](image-url)
moistures. The slope of the line here is 0.383, in excellent agreement with the published value 0.388. The out-of-plane specific stiffnesses vs. moisture content are shown in Figs. 11 and 12; both show the same sharp decrease in stiffness with increasing moisture followed by a plateau region at the highest levels of moisture. The data for $C_{33}/\rho$ seems to be separated somewhat by the three levels of refining (referred to above). The upward curvature in the data at the highest moisture levels may be an artifact of the difficulty in measuring an accurate caliper in the very wet samples. This is illustrated in Fig. 13 which shows sheet density plotted against moisture content. The nine curves represent three levels of wet pressing at each of the three refining levels. In general, the highest curves are at high refining and pressing levels, while the lowest curves are at low refining and low pressing levels. The observed decreasing density with increasing moisture at low moisture contents may be explained in
terms of the hygroexpansivity of the paper in the ZD, i.e. the water breaks bonds causing the paper to swell in the ZD. At higher moistures the density should reach some value between that for water (1 gm/cm$^3$) and cellulose (1.5 gm/cm$^3$). The data, however, do not appear to be reaching an equilibrium value. It is quite possible that the measurement of caliper becomes progressively in error (lower) at the higher moisture levels. The results in Fig. 12, however, refute this argument since no such upward curvature at high moistures is apparent.

Figure 10. Specific shear stiffness vs. specific stiffness.

The data obey the relationship $C_{66} = a(C_{11}C_{22})^{1/2}$, which for a handsheet becomes $C_{66} = aC_{11}$.

Broadband Transducer Development

We presented details of the construction of our new z-direction longitudinal transducers in the October 22, 1985 PAC report. The active element is a plastic, piezoelectric film made from polyvinylidene fluoride (PVDF). It has a lower mechanical impedance than standard ceramic piezoelectrics and is a more efficient coupler of energy into paper. It also has a lower quality factor and
Figure 11. Specific out-of-plane elastic stiffness vs. moisture content at three levels of refining (▽ - low refining, x - medium refining, □ - high refining).

Figure 12. Specific out-of-plane shear stiffness, \( \frac{C_{44}}{\rho} \), vs. moisture content.
Figures 13. IPC density (gm/cm$^3$) versus moisture content. $\Delta$, $\ast$, represent low refining and low, medium, and high levels of wet pressing, respectively; $\varpi$, $\Diamond$, $\lozenge$ are medium refining and low, medium, and high levels of wet pressing, respectively; and $\Box$, $X$, $\triangledown$ are high refining and low, medium, and high levels of wet pressing, respectively.

This provides an opportunity to design very broadbanded transducers. We need broadbanded transducers in our time-of-flight velocity measurements because paper is dispersive to ZD sound propagation. That is, the phase velocity and attenuation are frequency dependent; therefore, the form of a pulsed signal broadens as it propagates through paper. The value of a time-of-flight velocity derived by comparing points of equal phase depends on the definition of arrival time in the received pulse. One way around this is to compare the arrival times of complete pulses using a cross correlation technique. This requires a broadbanded transducer which, being nonresonant, can generate short pulses. These are detected before multiple reflections in the sample and transducer interfaces can interfere with the tail of the signal.
Our PVDF transducers have succeeded in giving pulse widths of about 1 μsec. This is much smaller than the widths created by ceramic transducers which need to be very resonant in order to couple sufficient energy into the sheet. Since the last report we have developed brass retaining rings for the neoprene used on the surfaces of the transducers. These result in caliper readings comparable to our soft platen caliper gage, which uses thinner rubber. Two spare transducers also have been constructed.

Analytical work which relates the time-of-flight velocity, determined by entire pulse cross correlation, to material properties is on-going. It is shown that in our experiment the maximum in the cross correlation can be used to find the phase velocity (in the case of constant velocity and frequency dependent attenuation) and the group velocity (when phase velocity varies linearly with frequency). We intend to employ fast Fourier transform routines to find the frequency dependent transfer function for the sample. From this, frequency dependent phase velocity and attenuation might be determined. Our previous attempts to determine attenuation have been frustrated by our inability to characterize the coupling between the sample and the transducers. There is some hope that Fourier analysis of the single pulse might circumvent this problem.

Measurement Automation

The realization of the out-of-plane cross correlation and Fourier analysis apparatus awaits completion of our digital signal analysis system. This is comprised of a Hewlett Packard model 1980A digital oscilloscope and an IBM XT. The scope can do analog to digital conversions at 100 MHz on repetitive signals. This is sufficient resolution for our transit times of about a micro-second. The computer-oscilloscope interface is an IEEE bus, which also transfers the output of an LVDT position transducer to the computer for caliper
determination. Presently, the software to drive this system is being developed; we are progressing and should have initial trials by the first of April.

Robotic System

Our fully automated, in-plane apparatus continues to operate satisfactorily and provide routine test results for many contract and in-house projects. In the past six months we have changed the rotating sample platform from a friction drive to a direct belt drive. Software has been written to extend operation of the system. Three private firms have expressed interest in manufacturing the system and are at various stages in negotiating an agreement.

In view of the commercial interest and of our need for expanded test capabilities, we intend to construct a second system. We will purchase a 10 MHz LeCroy transient signal recorder to replace the custom designed, variable gain, analog to digital converter in the first model. We hope that the use of off-the-shelf instrumentation will ease some of the difficulties of technology transfer. Also, we will redesign the transducer translation mechanism, which is now controlled by air cylinders. We intend to change a stepping motor drive increasing reliability and positioning versatility.

Transducer Development

We have built a number of experimental in-plane transducers in the last half year. In hopes of repeating our success in the ZD work, we are making an in-plane transducer with PVDF. These are "bender" transducers made of thin (~25 μm) plastic sheets in epoxied stacks on the order of thirty layers thick. So far we have not achieved sufficient sensitivity or bandwidth; however, we still have a few ideas to work with.
We also have been working to make our conventional ceramic bender more durable. We added a light, brass "shoe" to the transducer tip. Without changing the acoustic response, this makes the transducers more rugged in the in-plane tester and gives us the opportunity to use benders in an on-line apparatus.

Student Research

Two interesting pieces of student work have recently been completed. Results were not available for inclusion in this report, however. Westerveldt studied the effects of non-uniform drying restraints on local sheet properties, including $C_{11}$ (or $C_{22}$) and $C_{33}$. It is anticipated that these will be presented and discussed at the PAC meeting. Waterman investigated the use of a stylus device for measuring surface roughness parameters. He correlated these measures with standard air leak values and investigated the effects of papermaking variables on roughness. These results will also be discussed at the meeting. Berger continues his work concerning the effects of refining and yield on ZD elastic properties.
THE INSTITUTE OF PAPER CHEMISTRY
Appleton, Wisconsin

Status Report
to the
PAPER PROPERTIES AND USES
PROJECT ADVISORY COMMITTEE

Project 3332
ON-LINE MEASUREMENT OF PAPER MECHANICAL PROPERTIES

February 26, 1986
PROJECT SUMMARY

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

PROJECT STAFF: C. C. Habeger, G. A. Baum                 February 26, 1986

PROGRAM GOAL: Develop ways to measure and control manufacturing processes.

PROJECT OBJECTIVE:

To develop the capability to measure elastic parameters on a moving paper web. Current emphasis is on out-of-plane measurements.

PROJECT RATIONALE, PREVIOUS ACTIVITY, AND PLANNED ACTIVITY FOR FISCAL 1986-87 are on the attached 1986-87 Project Form.

SUMMARY OF RESULTS LAST PERIOD (April 1985 - September 1985)

1. The comprehensive report covering the work for FKBG (the Owens-Illinois Valdosta sensor) has been published, dated May 15, 1985.

2. A paper summarizing the results obtained in Valdosta was written for publication in Tappi. This is IPC Technical Paper No. 157, entitled "On-line measurement of paper mechanical properties", a copy of which is attached as Appendix I.

3. The DOE proposal concerned with in-plane and out-of-plane measurements is expected to be funded at the start of the next government fiscal year (October).

4. The automation of the measurements of out-of-plane elastic properties described in the Project 3467 report translates directly into this project, concerned at present with on-line measurements of ZD properties.

5. The improved transducer design, also described in the Project 3467 report, is also the key to the success of the on-line measurement of ZD properties.

6. The ultrasonic laboratory measurement system in the oven, also described earlier, will be useful in this project to determine appropriate moisture and temperature corrections to the on-machine measurements.

SUMMARY OF RESULTS THIS PERIOD: (October 1985 - March 1986)

1. New broadband plastic (PVDF) ZD transducers for use in wheels are being designed for use in an out-of-plane measurement on a moving paper web.

2. Implementation of a high speed signal processing system for use with on-machine ZD measurements is underway using a LeCroy transient recorder and related equipment. The LeCroy system will be integrated with an IBM-XT.

3. The above will also be incorporated into a laboratory system which will make ZD measurements at low web speeds.
4. The study of transient effects in the mechanical properties vs. changing moisture content continues on both theoretical and experimental levels.

5. The DOE project concerned with an in-plane and out-of-plane sensor for on-machine measurements and subsequent machine control is expected to be funded by April 1, 1986.
PROJECT TITLE: On-Line Measurement of Paper Mechanical Properties

Date: 1/28/86

Budget: $100,000

Period ends: 6/30/87

Project No.: 3332

PROJECT STAFF: C. Habeger/G. Baum

PRIMARY AREA OF INDUSTRY NEED: Properties related to end uses

PROGRAM AREA: Control of manufacturing processes

PROGRAM GOAL: Develop ways to measure and control manufacturing processes

PROJECT OBJECTIVE:

To develop the capability to measure elastic parameters on a moving paper web. Current emphasis is on out-of-plane measurements.

PROJECT RATIONALE:

The ability to measure mechanical properties on the paper machine is valuable from several standpoints. It provides a potential means for control of process variables. It also provides a non-destructive way to assess product quality on a continuous basis.

RESULTS TO DATE:

Developed theory of ultrasound propagation in paper, and developed devices for measuring paper and board in-plane elastic parameters on-machine. Successfully tested devices in mill environments. Constructed and tested a version useful for light weight grades which is also self-calibrating. Developed cross correlation technique for use with in-plane velocity measurements, and initiated work relating to on-line measurements of z-direction properties. Developed a high-frequency, low impedance out-of-plane transducer using a plastic film piezoelectric material which is superior to commercial ceramic transducers. Developed superior in-plane "bender" transducer. Developed equipment for measuring moisture and temperature effects on paper elastic properties.

PLANNED ACTIVITY FOR THE PERIOD:

We intend to continue studies to explore the possibility of making out-of-plane ultrasonic measurements on a moving paper web. We will build high frequency, broad banded, and low impedance transducers that are mounted in wheels. We plan to look at both ceramic and plastic piezoelectric transducer constructions. Hardware and software for a high speed data acquisition system will be designed and built. On-line caliper measurements techniques will be investigated to be used with the ZD measurement system.

RELATED ACTIVITIES:

The proposal submitted to the Department of Energy to investigate possible control strategies on the paper machine and to develop a sensor to measure out-of-plane properties has been approved. Work should be underway by March, 1986.

On-line ZD Transducers

We are designing wheel transducers using PVDF active elements, for eventual on-line use. We plan to try two configurations: (1) our present laboratory design with necessary modifications to allow mounting in a rotating wheel (2) a circular transducer which would be active over the entire wheel surface. The first technique is safe, since we have already solved many of the technical problems; while the second would allow continuous signal transfer and preclude the need for mechanical synchronization of the wheels and for timing the trigger of the transmitter. One concern in using PVDF on-line is that its sensitivity degrades with temperature. However, we feel that we can live with this since our laboratory system, which applies voltage an order of magnitude less than the maximum, has good sensitivity. A more serious potential problem is the durability of the soft neoprene coupling layer in on-line operation.

On-line ZD Instrumentation

If we are forced to use the wheels with localized active regions, it will be necessary to do the full analog-to-digital conversion on single signals without repetition. To achieve this, we have purchased a LeCroy transient recorder with a 100 MHz analog-to-digital conversion. This will be interfaced to an IBM XT over an IEEE bus, and much of the software being developed for the digital oscilloscope will be converted to the on-line system. This process will begin after check-out and refinement of the ZD laboratory software.

Robotic ZD Instrument

As a precursor to the on-line ZD gage, we are planning a laboratory wheel transducer system that would measure caliper and ZD velocity auto-
matically. A sample would be placed in the nip of the two wheel transducer. One wheel would be driven by a stepping motor, causing the sample to feed between the transducers. Caliper and velocity would automatically be scanned over an entire length of the sample. This will provide an efficient laboratory instrument and force us to deal with the problems of on-line soft caliper and ZD velocity measurements in a cleaner context.

In-Plane Environmental Chamber

The construction of our temperature and humidity controlled in-plane test apparatus is complete. The digital balance, and thermometers are interfaced with an Apple computer which also controls a stepping motor to move the transducers and operate the oven fan. Revisions and improvements in the software to control weight, temperature, and velocity readings are in process. We hope to characterize a set of samples for in-plane velocities as functions of temperature and moisture in the near future. This apparatus will be routinely used to establish moisture-temperature correction curves for grades tested on-line.

We are also using the oven in student research to study the moisture transient effect on ultrasonic velocities. Preliminary results indicate that there is not a large difference at temperatures encountered on-line. A theoretical application of the principles of non-equilibrium thermodynamics to the moisture transient effect is continuing with some successes.