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*Institute of Paper Science and Technology  
Atlanta, Georgia*

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# SLIDE MATERIAL

to the

PAPER PHYSICS

PROJECT ADVISORY COMMITTEE

March 7-8, 2000

John Waterhouse, IPST PAC Liaison  
Pierre Brodeur, IPST PAC  
Doeung Choi, PAC Chairman  
David Knox, Vice-PAC Chairman  
Ross MacHattie, RAC Liaison  
Kari Ebeling, Alt. RAC Liaison

## TABLE OF CONTENTS

<u>Project</u>	<u>Name</u>	<u>PAGE</u>
F008	Fundamentals of Acoustic Radiation	01
F007	On-Line Measurement of Paper Properties	
F031	Non-contact Ultrasonic Stiffness Measurements	
F020	Fundamentals of Dimensional Stability	
F026	Fundamentals of Accelerated Creep	
F023	Fundamentals of Micromechanics of Fiber Networks	
F024	Improving the Refining of Chemical Pulps	
F025	Fundamentals of Interfiber Bonding	
F044	Liquid/Substrate Interactions	



# FUNDAMENTALS OF ACOUSTIC RADIATION PRESSURE

STATUS REPORT

FOR

PROJECT F008

Pierre Brodeur  
Joseph Gerhardstein  
Feler Bose  
Jimmy Jong  
Dolon Silimon

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# **Mill Demonstration of Ultrasonic Whitewater Clarification**

**F008: Fundamentals of Acoustic Radiation Pressure**

**Institute of Paper Science and Technology**

**SP Newsprint**

**Pierre Brodeur, Joe Gerhardstein, Jimmy Jong, Feler Bose, Dolon Silimon**

**Jim Ramp (SP Newsprint)**

**March 7, 2000**

## **Project Objective (FY99-00)**

- u To perform a mill demonstration of ultrasonic whitewater clarification (AST Clarification)**

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## Background Information

- u **Project F008 : To investigate fundamentals of acoustic radiation pressure effects on pulp suspensions and evaluate promising industrial applications.**
- u **Over time, parallel projects to F008 were funded:**
  - u ***DOE (Agenda 2020 Program): Acoustic Separation Technology***
  - u ***State of Georgia (TIP<sup>8</sup> Program): Dual Flocculation/Ultrasonic Clarification Method***

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## Background Information

- u **All resources are now combined to support the development of a 100 gpm pilot-scale Acoustic Separation Technology (AST) clarifier and perform a mill demonstration in collaboration with SP Newsprint in Dublin, GA.**

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## Background Information

- u **Mill demonstration is scheduled to begin in May 2000.**
- u **Combined Budget (FY99-00):**
  - u **IPST: \$141k**
  - u **SEP: \$50k**
  - u **State of Georgia: \$225k**
  - u **DOE: \$150k**
  - u **TOTAL: \$566k**

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## Rationale for New Technology

- u **Water consumption during paper manufacturing is by all accounts significant:**

*Approx. 200 tons of water are required to obtain 1 ton of paper*
- u **Hence, water clarification needs are very important, especially for whitewater, which is the filtrate from the forming fabrics of paper machines**
- u **Several methods are used to clarify whitewater (e.g., passive dissolved air systems or DAF, sand filtration)**
- u **There is a need for lower cost, in-line clarification systems**

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## Clarification Principles

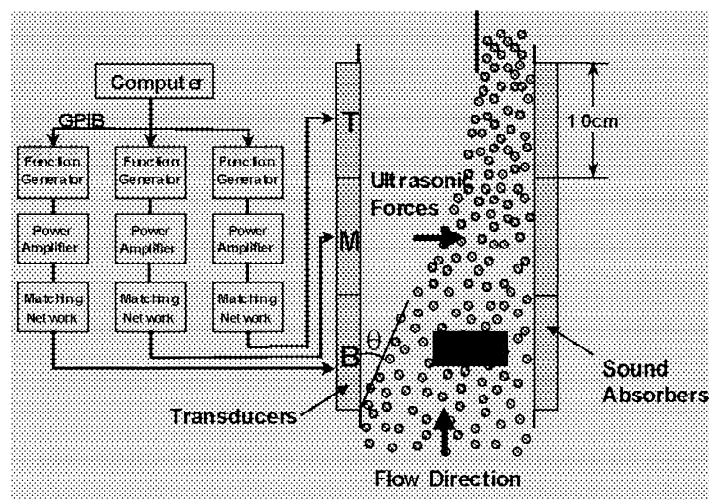
The method is a two-step process:

- 1- Use of chemical flocculants to increase the particle size in a whitewater stream, and hence, create flocs
- 2- Use of ultrasonic forces to deflect flocs in such a way as to obtain two output streams:
  - 1) clean water stream
  - 2) floc-concentrated stream

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## Floc Deflection by Ultrasonic Forces



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- ii **Optimization of chemical flocculation using different flocculants at different dosages**
- iii **Clarification experiments as a function of flow velocity, acoustic intensity, ultrasonic frequency, and transducer area**

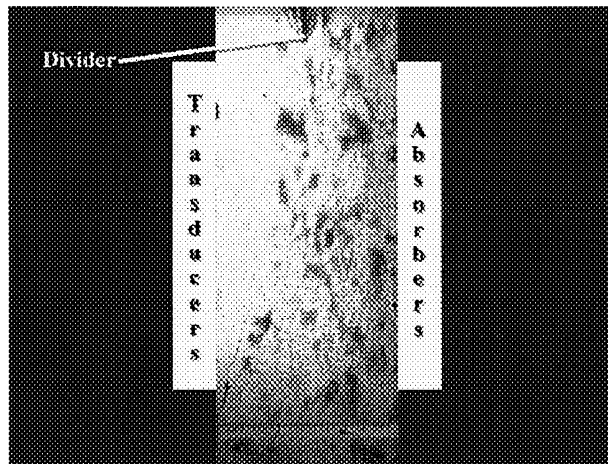


## Typical Real-time Observations

**Whitewater Feed  
Consistency:**  
0.03%

**Operating Frequency:**  
1.5 MHz

**Flow Velocity:**  
0.2 m/s



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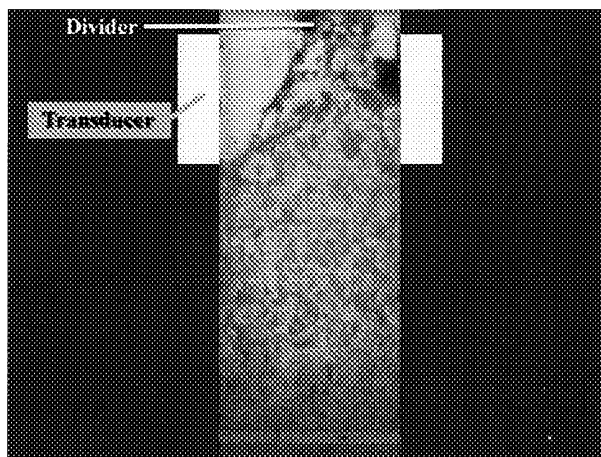


## Typical Real-time Observations

**Whitewater Feed  
Consistency:**  
0.04%

**Operating Frequency:**  
150 kHz

**Flow Velocity:**  
0.1 m/s



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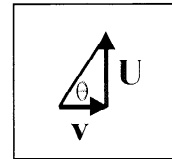


## Floc Deflection Angle $\theta$ vs. Flow Velocity $U$ and Floc Migration Velocity $v$

$$\tan \theta = v/U$$

or

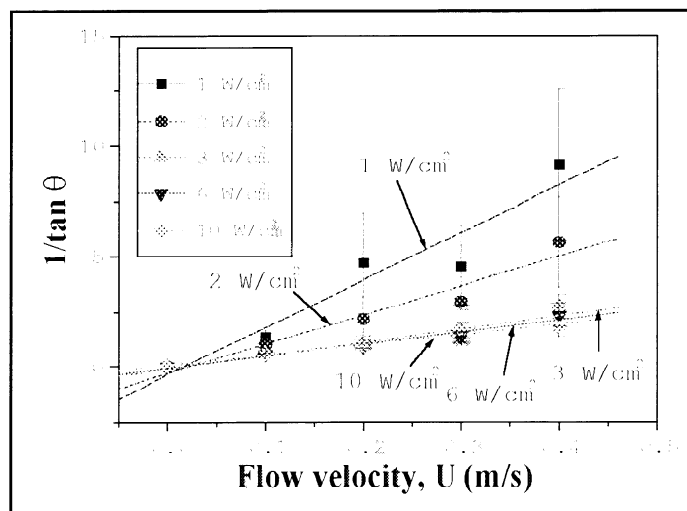
$$1/\tan \theta = (1/v) U$$



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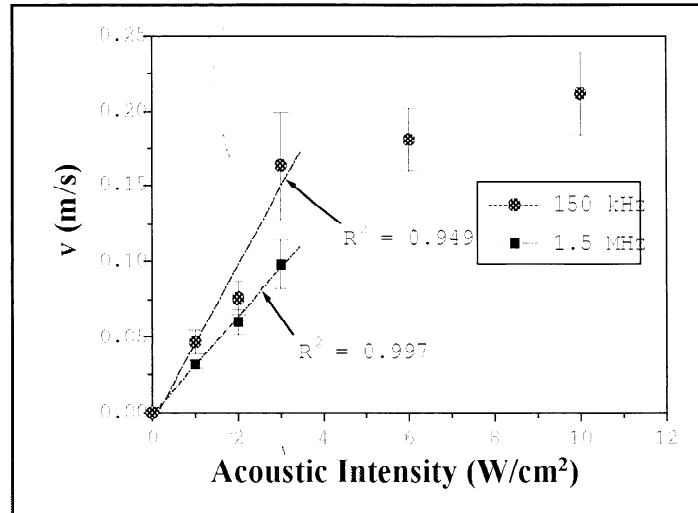
## $1/\tan\theta$ vs. Flow Velocity for Different Acoustic Intensities at 150 kHz



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## Flow Migration Velocity vs. Acoustic Intensity at 150 kHz and 1.5 MHz



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## Clean Stream Removal Efficiency and Clarification Efficiency

$$\% \text{Clean Stream Removal Eff.} = 100\% \left( 1 - \frac{C_{\text{clean}}}{C_{\text{feed}}} \right)$$

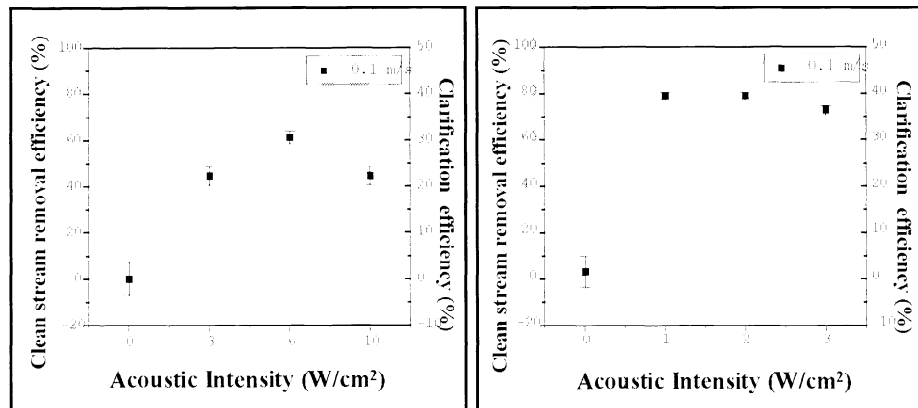
$$\% \text{Clarification Eff.} = 100\% \left( \frac{1_r}{1} \right) \left[ \frac{C_{\text{feed}} - C_{\text{clean}}}{C_{\text{feed}} \left( 1 - \frac{C_{\text{feed}}}{100\%} \right)} \right]$$

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## Clean Stream Removal Efficiency and Clarification Efficiency



150 kHz

1.5 MHz

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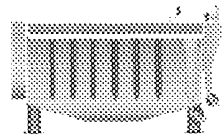
## Observations

- u **Best results using flocculant system PEO/PFR**
- u **Best deflection effect at 150 kHz**
- u **Best clarification efficiency at 1.5 MHz**
- u **Discrepancy between 150 kHz and 1.5 MHz results explained by the presence of undesirable secondary flows at 150 kHz**

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## Projected Economic Benefits of a 6000 GPM Ultrasonic Clarifier



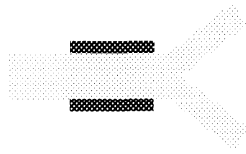
DAF

Capital Cost  
(\$k)

1,073

Operating  
Cost (\$k/yr)

353



Ultras. Clar.

352

221

Ultras. Clar.  
Benefit

721

132

(Based upon 1999 part costs)

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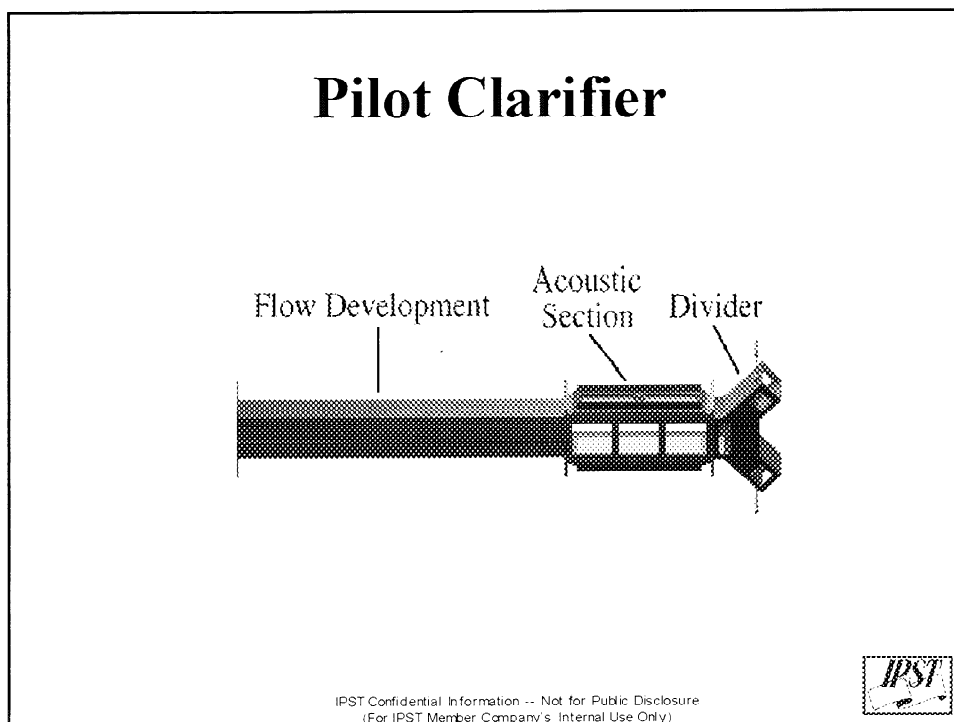
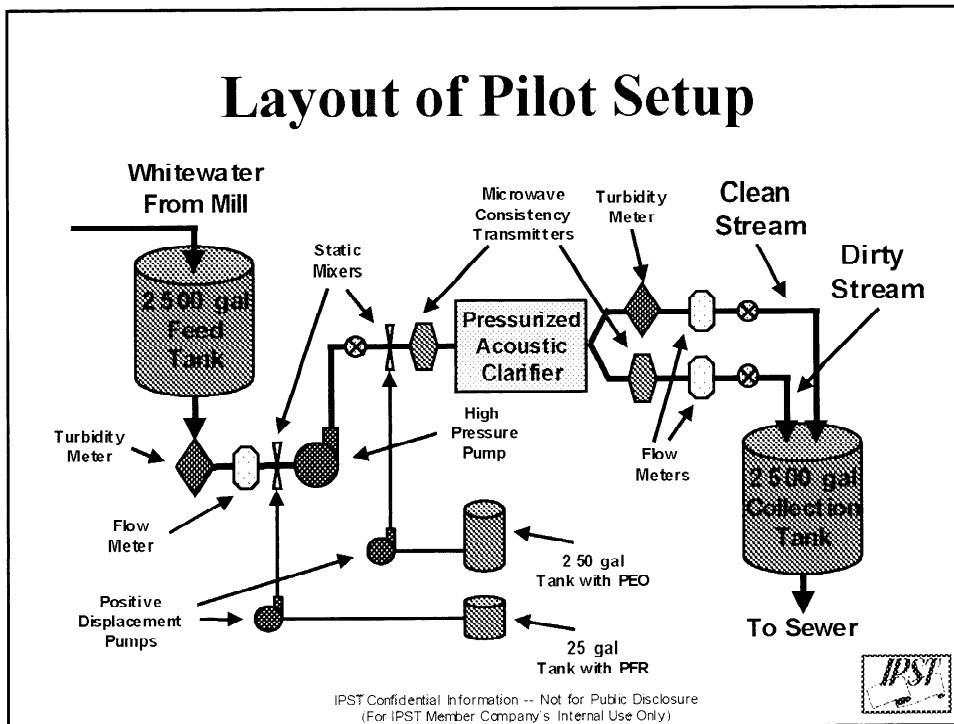


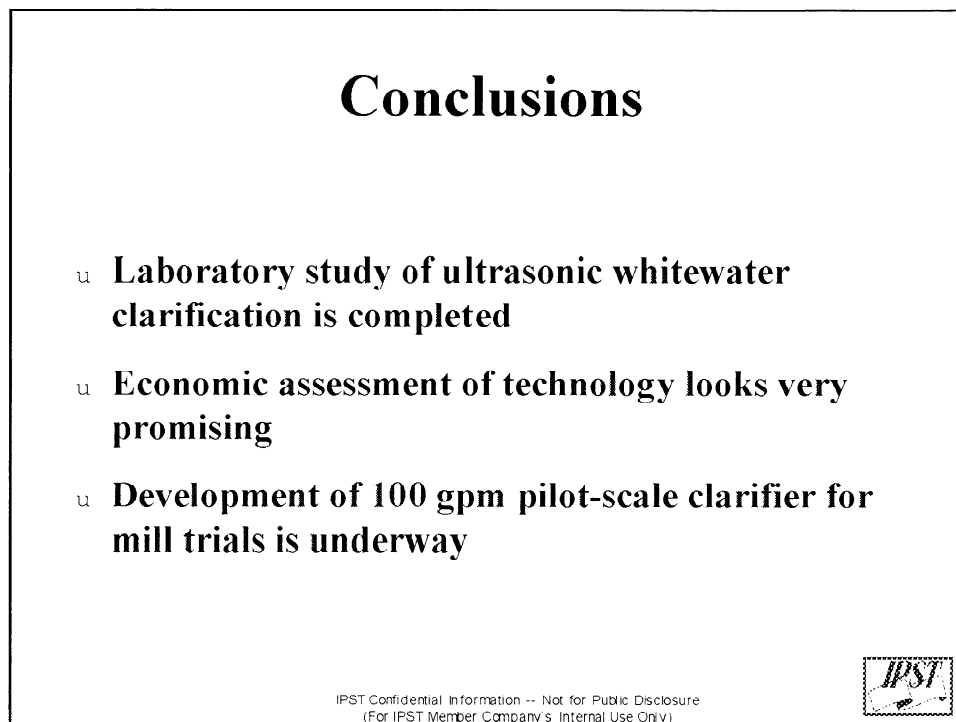
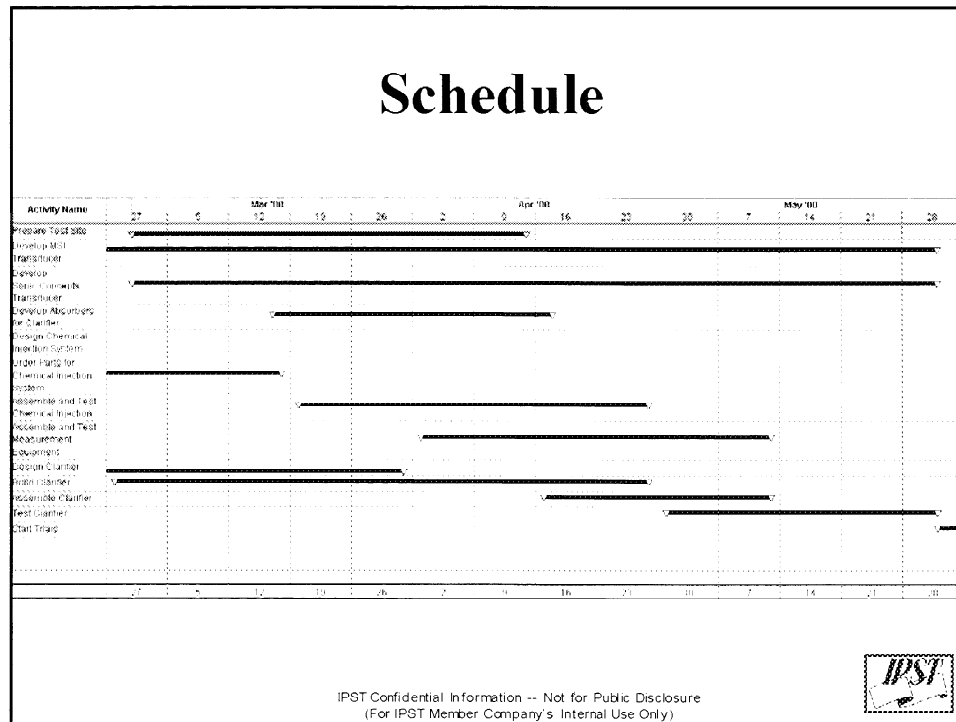
## Mill Demonstration

- Development of 100 gpm pilot clarifier
- Real-time observations using different whitewater streams
- Economic Analysis

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## Acknowledgments

- u **IPST**: Joe Gerhardstein, Jimmy Jong, Feler Bose, Dolon Silimon, Yulin Deng, Zegui Yan
- u **Beloit Pulping**: Jack Milliken, David Grimes
- u **SP Newsprint**: Jim Ramp
- u **Funding**: IPST, US Department of Energy-Office of Industrial Technologies, and State of Georgia

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ON-LINE MEASUREMENT OF PAPER PROPERTIES

STATUS REPORT

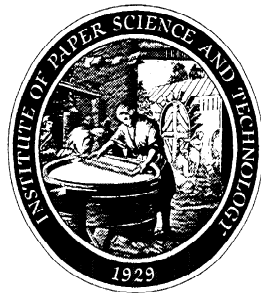
FOR

PROJECT F007

Mac Hall  
Ted Jackson  
Andy Brown

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# On-line Measurement Of Paper Properties



Project F007

Mac Hall  
Ted Jackson  
Andy Brown

On-line Measurement Of Paper Properties

## ***Relevant Research Lines***

Line 10 - Reduce energy / reducing reprocessing

Line 12 - Develop on-line sensors to relate  
manufacturing variables to process  
and product

On-line Measurement Of Paper Properties  
***FY 99-00 Funding***

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DFRC F007 - \$56,000

DOE-OIT - \$98,800

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Project Participants with DOE-OIT  
Cooperative Agreement  
DE-FC02-95CE41156

- Institute of Paper Science & Technology
  - Atlanta, Georgia
- ABB Industrial Systems Inc
  - Columbus, Ohio
- Georgia-Pacific Corporation
  - Host Mill, Cedar Springs, Georgia
- Herty Foundation
  - Savannah, Georgia



## On-line Measurement Of Paper Properties

### ***Project Personnel***

#### **ABB Personnel:**

**Bradley Pankonin**

#### **G-P Cedar Springs Mill Personnel:**

**Doug Jimmerson**

#### **PAC Subcommittee:**

**Thomas Rodencal, Georgia-Pacific**

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## On-line Measurement Of Paper Properties

### ***Goals & Schedule***

	Jan-Mar 99	Apr-Jun 99	Jul-Sep 99	Oct-Dec 99
1. Integrate ZD sensor with ABB data process system	-----	-----	-----	
2. Verify on-machine sensors (1) In-Plane (2) ZD	-----1	-----2		
3. Determine correlation of ultrasonic measurements with mill grade specifications	---	-----	-----	-----
4. On-machine measurement/ process relationships		-----	-----	-----

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## On-line Measurement Of Paper Properties

### Status of Goals For FY99-00

1. This goal was cancelled when DOE/Agenda 2020 reduced previously approved project funding.
2. Demonstrated successful sensor operation for extended runs scanning linerboard at speeds up to 1800 ft/min.
3. Completed bump-test runs involving 25 successive reels of 69# and of 55# linerboard. Compared measurements of end-of-reel strips with on-machine data.
4. Compared on-machine data with process

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## On-line Measurement Of Paper Properties

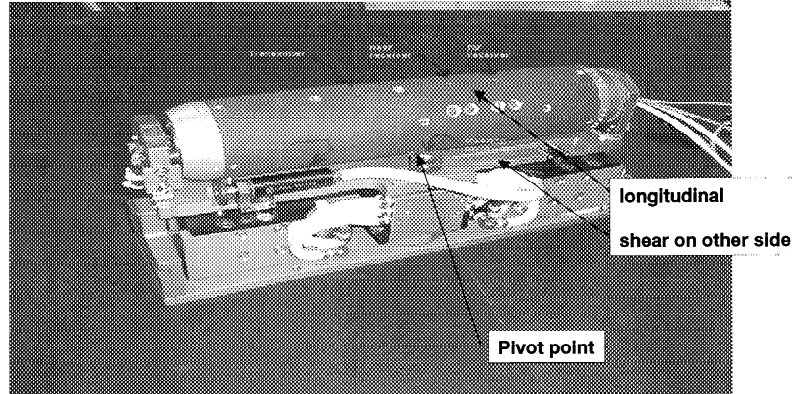
### *Discussion*

- 
- Review of on-machine sensor system
  - 69# & 55# Trials
  - Cross Direction In-plane Stiffness, Ring Crush, & STFI Profiles
  - ZD measurements relation to refining, calendering, & plybond

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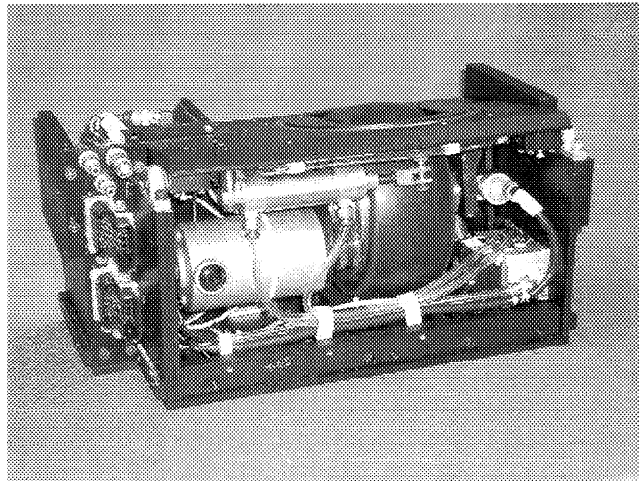
## *Sensor Overview* - Lower Sensor Module Rotates in Contact With Sheet

$$V = \Delta d / \Delta t$$



- Three transducer ultrasonic transit time (velocity) measurement
- Air motor assist reduces drag on the process
- CD Shear and CD Longitudinal transducer sets 180° apart with one measurement each every 5° of process

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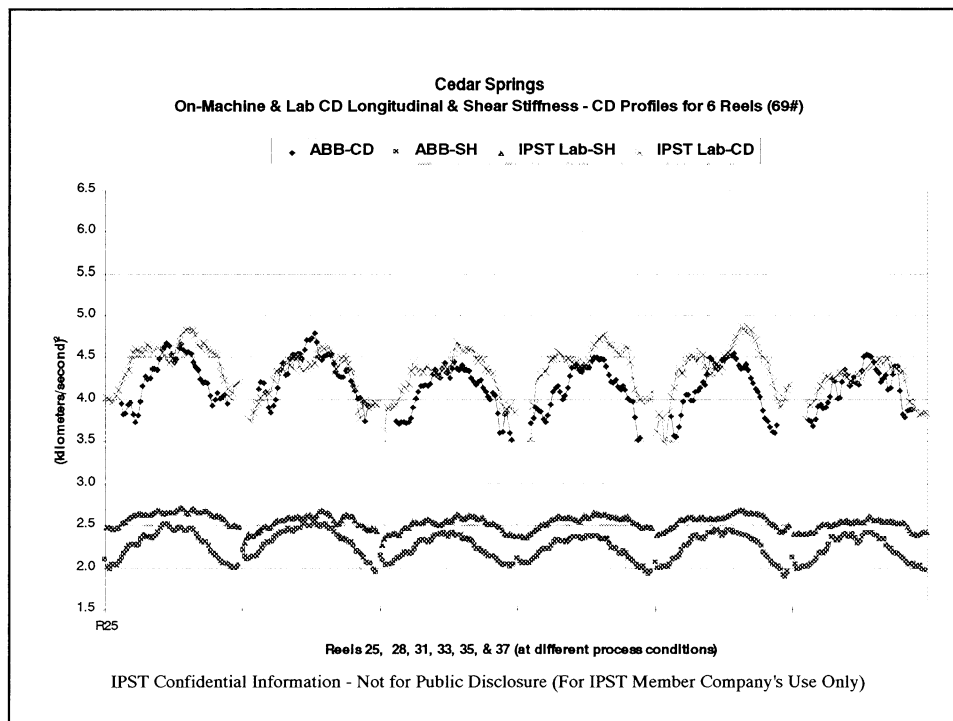


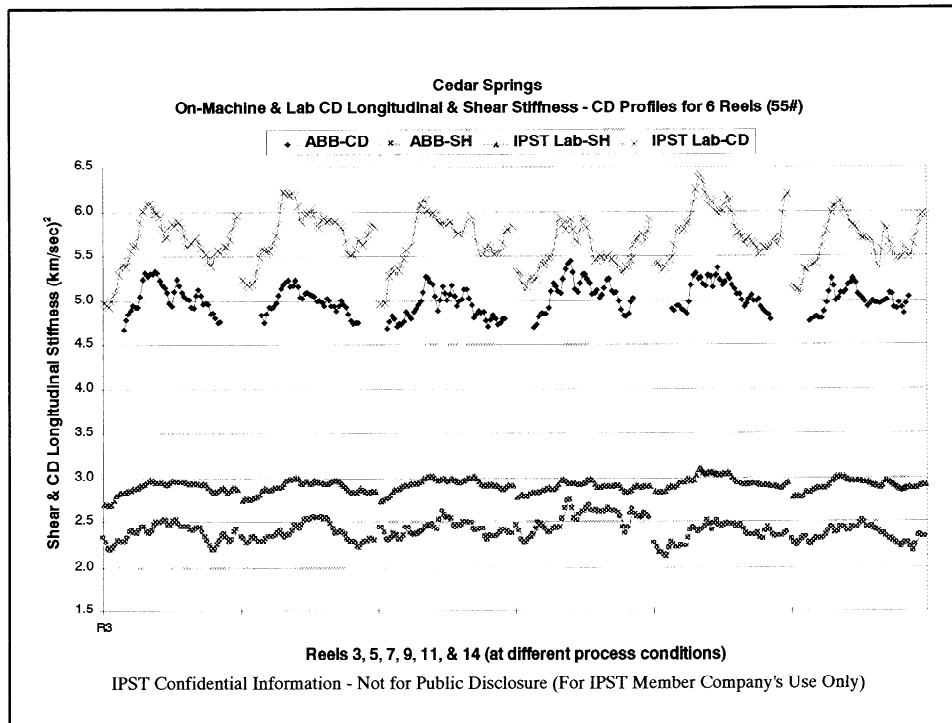
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## Process Variables Manipulated

1. Machine Speed
2. Rush/Drag
3. Number of Calender Stack Nips
4. % Top Stock Coverage
5. Basis Weight
6. % Moisture
7. Base Refiner Load
8. Broke Flow
9. DLK Flow

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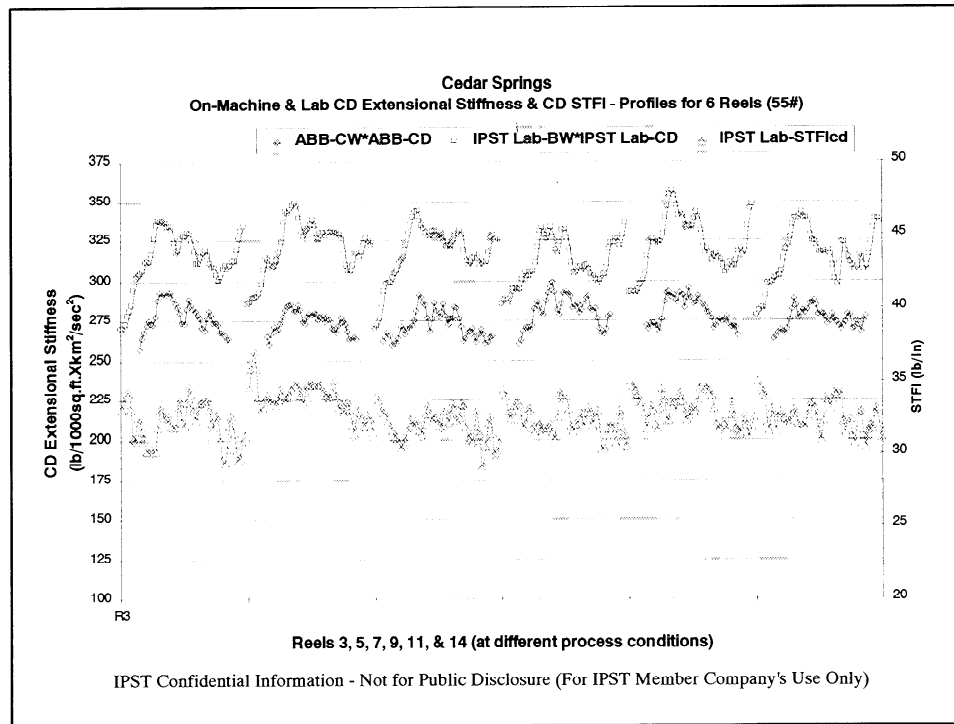
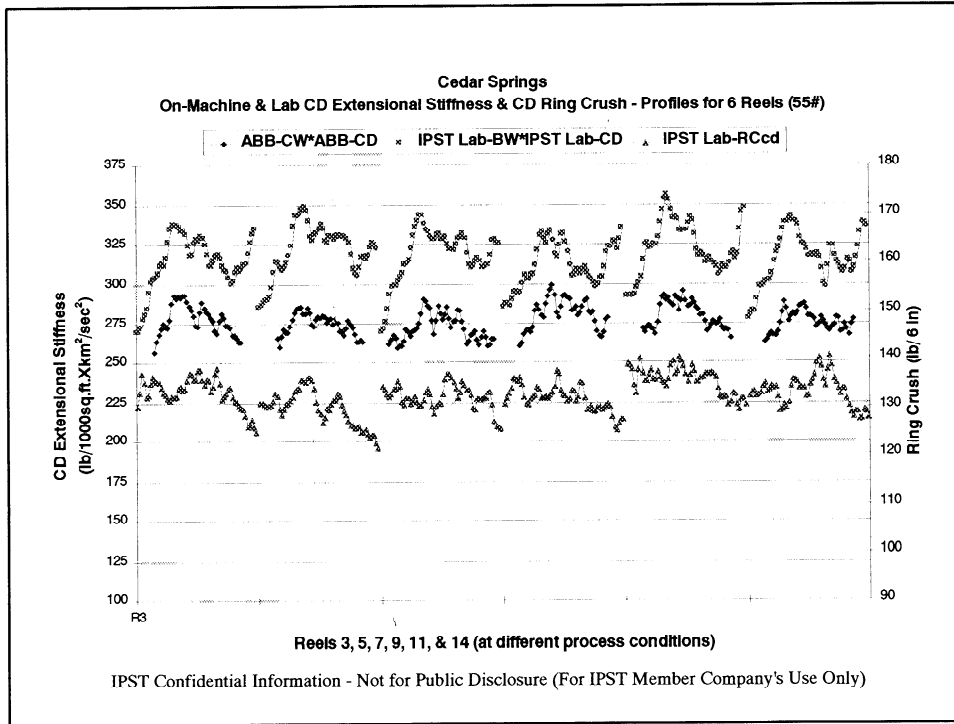


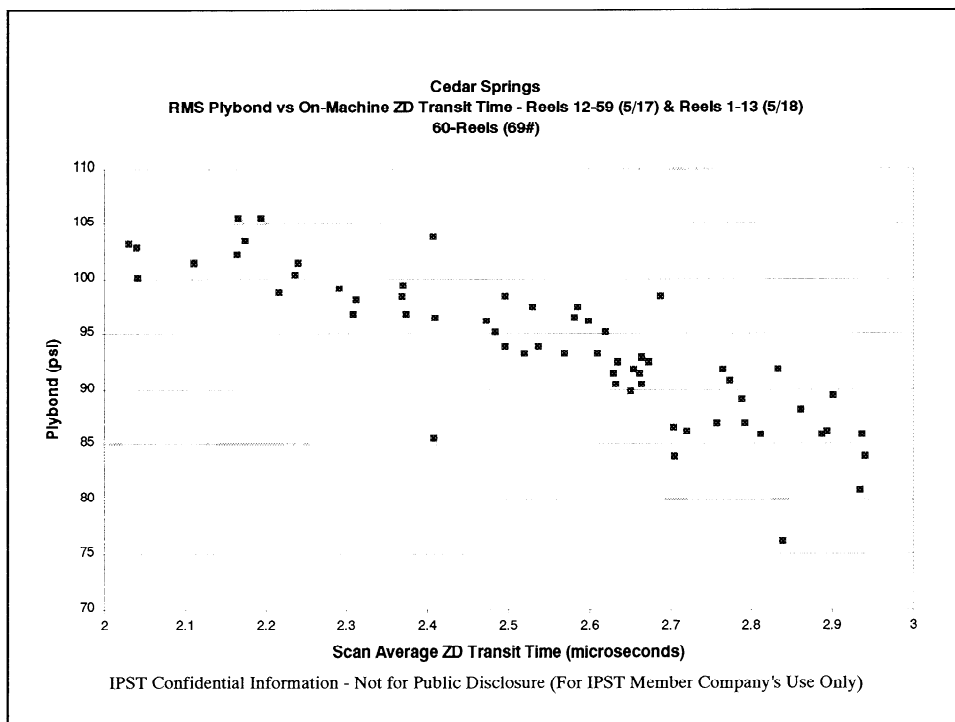
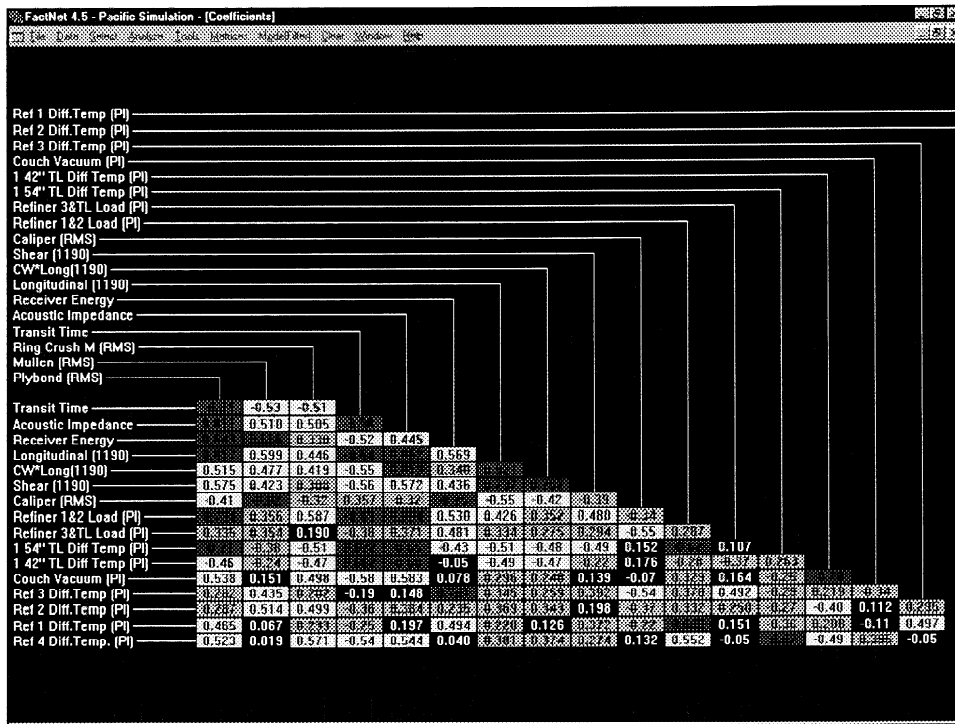


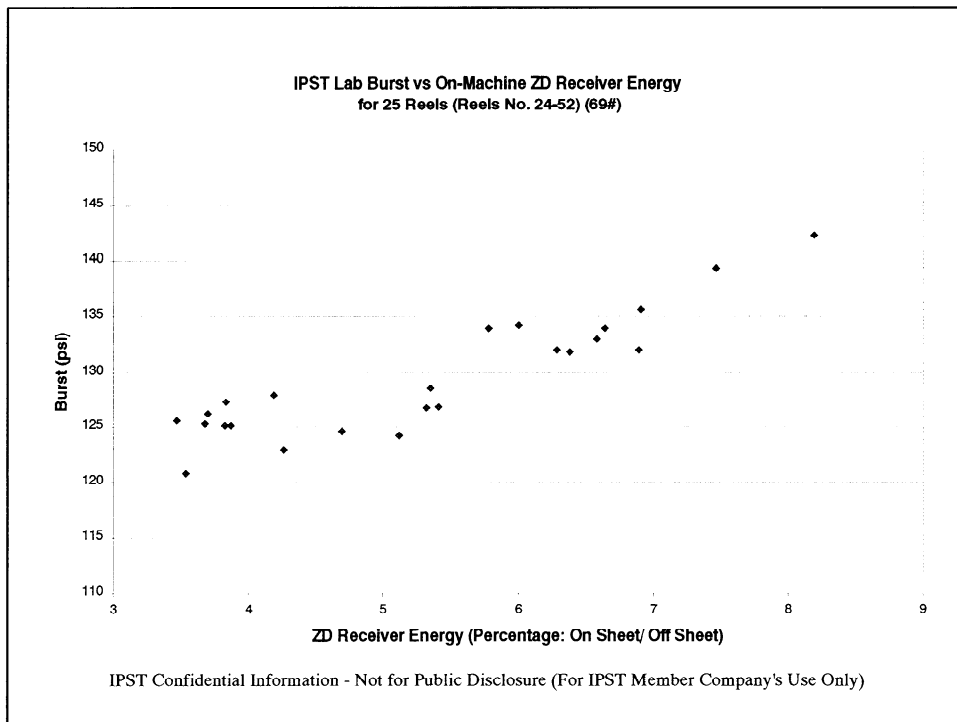
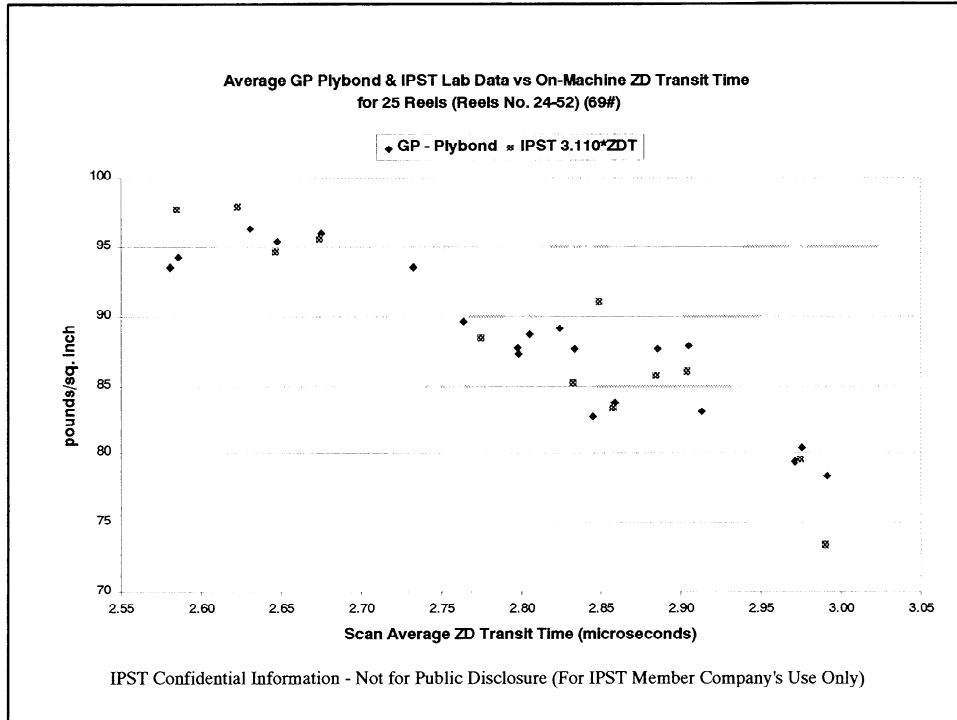
## On-line Measurement Of Paper Properties **Observation for the Paper Physicist:**

**In-Plane Cross Direction Stiffness  
Profiles measured on-machine show  
greater “degree of frown” than Lab  
measurements on end-of-reel strips**

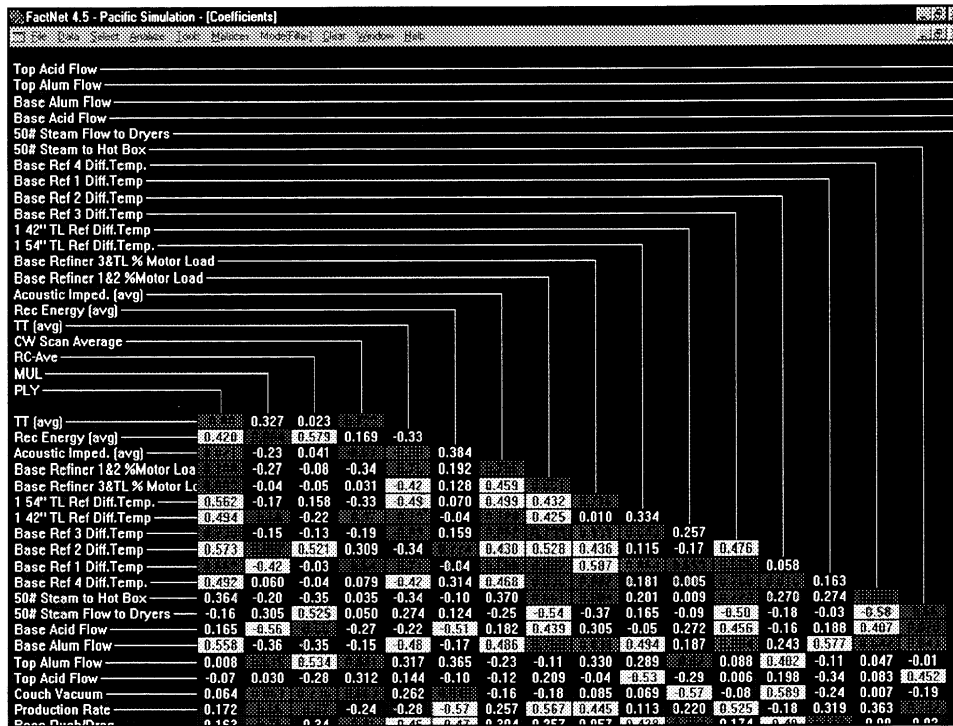
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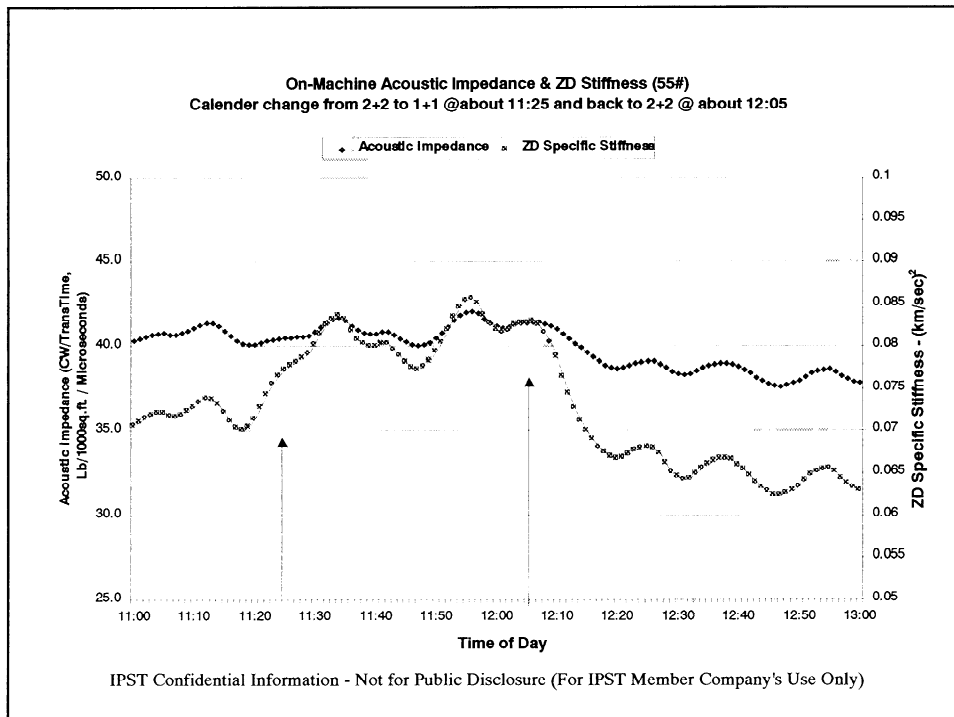
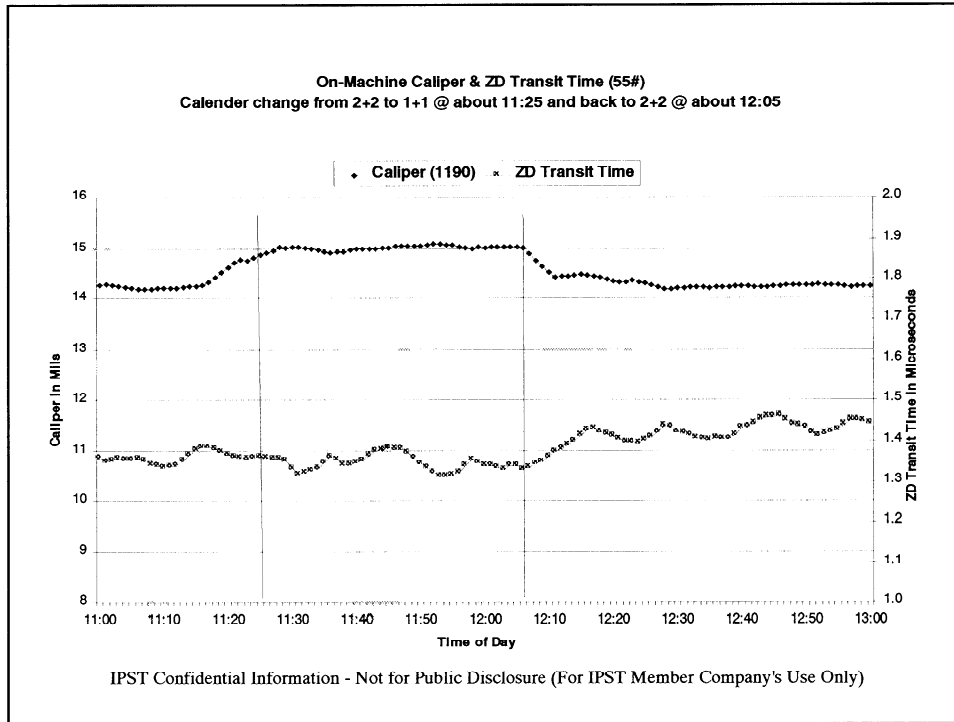


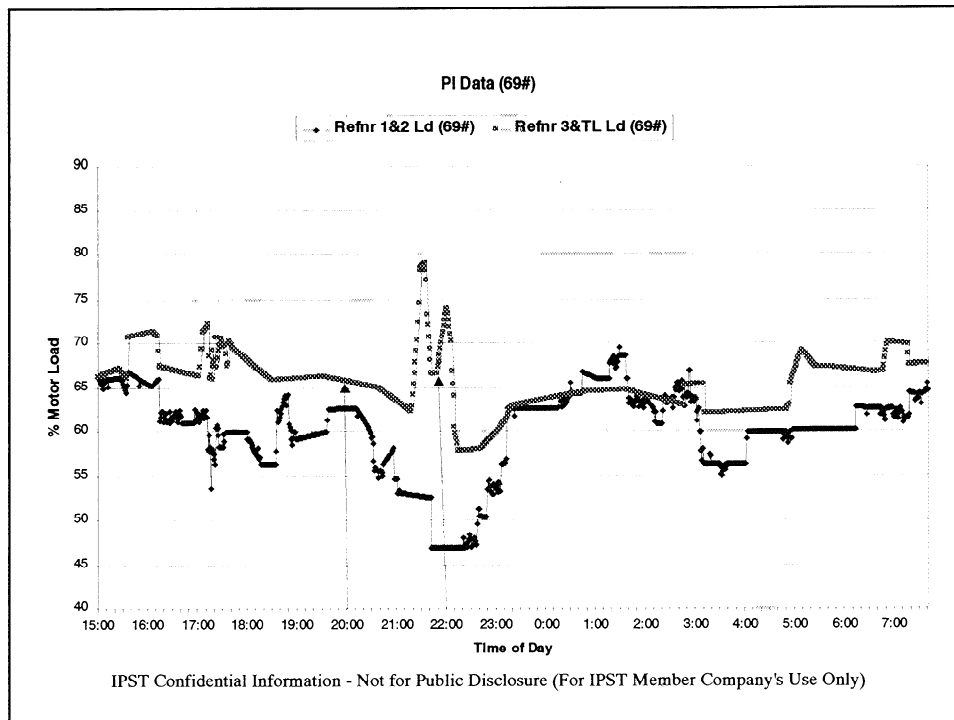
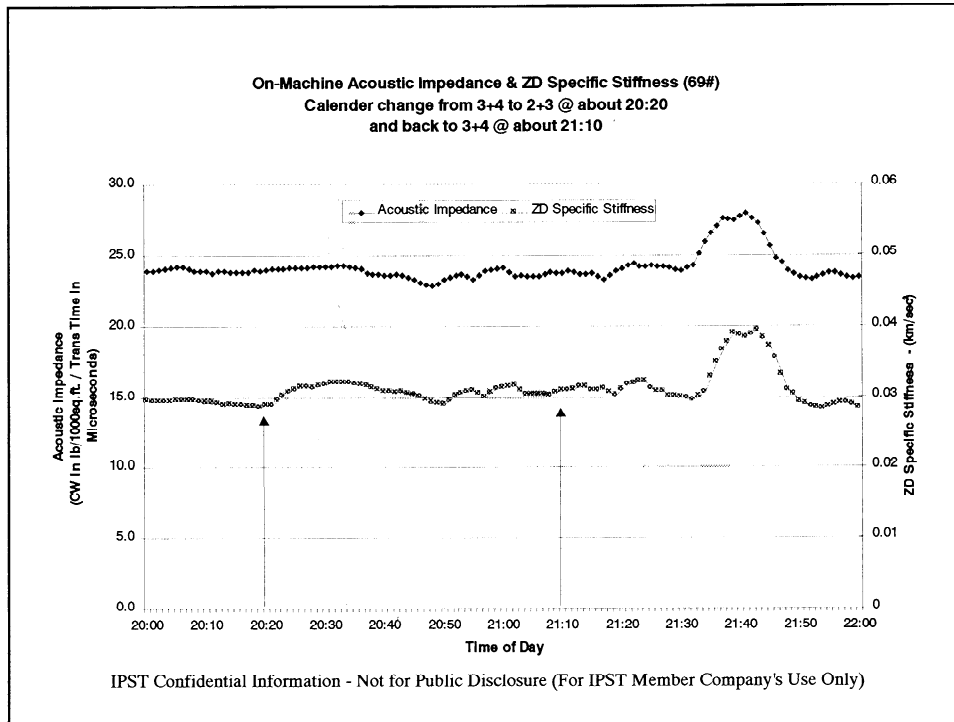


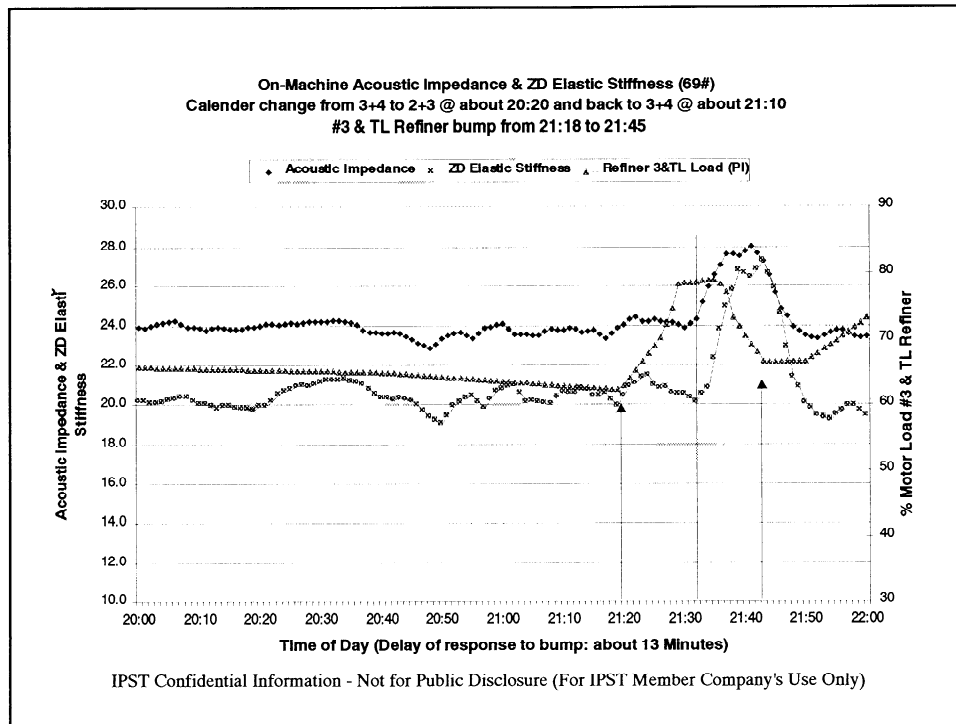
## Process Variables Which Affect ZD Web Properties

- FURNISH
- » REFINING
- WET PRESSING
- WET STRAINING
- » CALENDERING

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## On-line Measurement Of Paper Properties Summary of Results

### The On-Machine ZD Sensor System works

**ZD Measurement are sensitive to:**

**Refining  
Calendering**

**ZD Measurement correlate with:**

**ZDT (plybond)  
Burst (Mullen)**

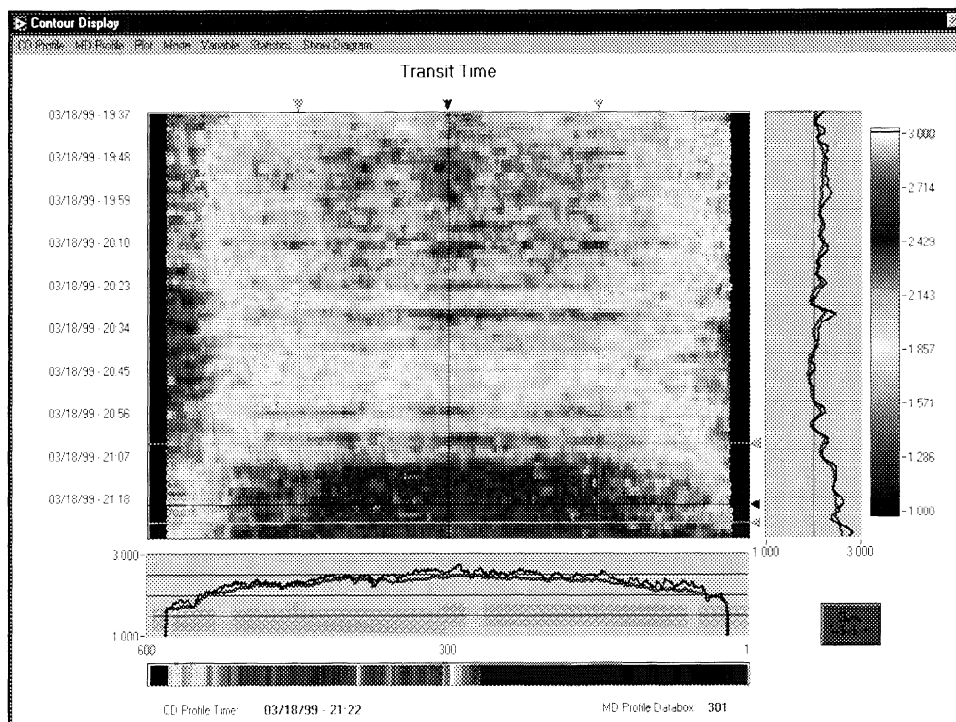
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## On-line Measurement Of Paper Properties

### Conclusions

- **Successfully Demonstrated the Out-of-Plane Ultrasonic Technology on a Commercial Paper Machine.**
- **Sensitivity of ZD Measurements to ZD Tensile and Plybond show potential value to the mill because rolls that do not meet the Plybond specification have to be reprocessed. Significant saving may be obtained by acceptable first quality.**

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## On-line Measurement Of Paper Properties Conclusion/Recommendation

- **Display of ZD data to operator both as time trend of scan average and contour map of scan-by-scan cross-web profiles could provide early warning of process upsets and constant visibility of product status, helping the operator maintain stable and efficient process operation.**



NON-CONTACT LASER ULTRASONIC MEASUREMENTS

STATUS REPORT

FOR

PROJECT F031

John Waterhouse  
Chuck Habeger  
Emmanuel Lafond  
Jimmy Jong  
Ted Jackson  
Joe Gerhardstein

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## NON-CONTACT LASER ULTRASONIC STIFFNESS MEASUREMENTS Project F031

Charles Habeger, Emmanuel Lafond, Jimmy  
Jong, Joe Gerhardstein, Ted Jackson,  
John F. Waterhouse

March 7, 2000

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## PROJECT OBJECTIVE

Design and construct an automated  
laboratory laser-ultrasonic stiffness instrument

### Relevant Research Lines:

#### **11. Convertability and End Use Performance.**

Improve the ratio of product performance to cost for pulp and paper products 25% by developing: models, algorithms, and functional samples of fibrous structures and coatings which describe and demonstrate improved convertability and end-use performance; break-through papermaking and coating processes which can produce the innovative webs with greater uniformity than that achieved by current processes.

#### **12. Sensors and Process Control.**

Reduce pulp and paper product costs by 25% through increased productivity and improved pulp, paper, and product uniformity achieved with developments in sensors and process controls.

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## **PROJECT DELIVERABLES**

1. Techniques for laser ultrasonic signal detection and analysis on paper.
2. An automated apparatus to do routine laboratory testing.

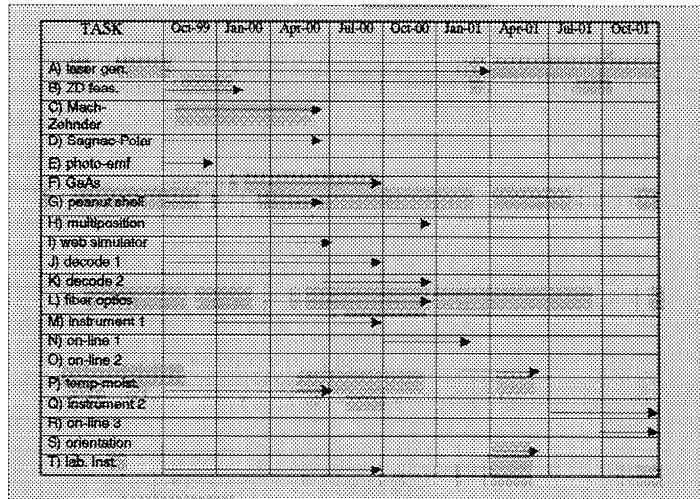
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## **RELATED WORK**

- DOE DE-FC07-971D13578 (IPST Project No. 4184).
- Budget FY 00-00 Dues: \$81,849  
DOE/OIT: \$327,397

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## PROJECT SCHEDULE 4184



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## PROJECT SCHEDULE F031

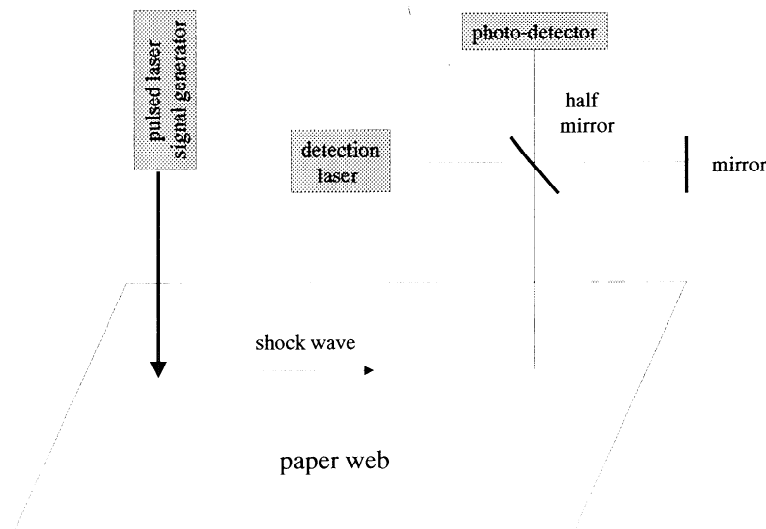
Task Descriptions	1999 Apr - June	1999 July - Sept	1999 Oct - Dec	2000 Jan - Mar	2000 Apr - June
1. Signal analysis methods					
2. Instrument Design					

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## 4184 Online Laser Ultrasonics Update and F031-4184 Signal Analysis Work

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### Laser Ultrasonics Schematic



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## Online Laser Ultrasonics Status

### Excitation Sources

LBNL is experimenting with CO<sub>2</sub> laser excitation.

They are trying different frequencies and pulse width in an effort to get more energy into sheet with less marking

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## Online Laser Ultrasonics Status

### Detection Interferometry

- A) Lason photo-emf system  
evaluated at IPST (deemed usable with minor paper marking, but not optimal)
- B) Mach-Zehnder scanning mirror system  
under development at LBNL
- C) GaAs TWM interferometer with scanning mirror  
under development at IPST

In about 1 month, Emmanuel Lafond will finish GaAs assessment, travel to LBNL, work on Mach Zehnder.

Then, we will decide as to best interferometer for first trials.

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In six months we expect to

- 1) Develop fiber optic distribution methods
- 2) Combine the best excitation with the best detection options
- 3) Integrate with ABB mounting and flutter control system
- 4) Experiment on a moving, open web

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### F031 Laboratory Instrument

#### Tasks

- 1) Develop a method of determining paper elastic parameters from laser-ultrasonic signals (overlaps with on-line efforts)
- 2) Configure lasers and optics (probably TWM GaAs system) for a laboratory unit
- 3) Build fiber optics delivery systems
- 4) Provide means for manipulation and application of sample to the laser-ultrasonic detector
- 5) Implement computer control manipulation and signal analysis

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### Ao Signal Analysis

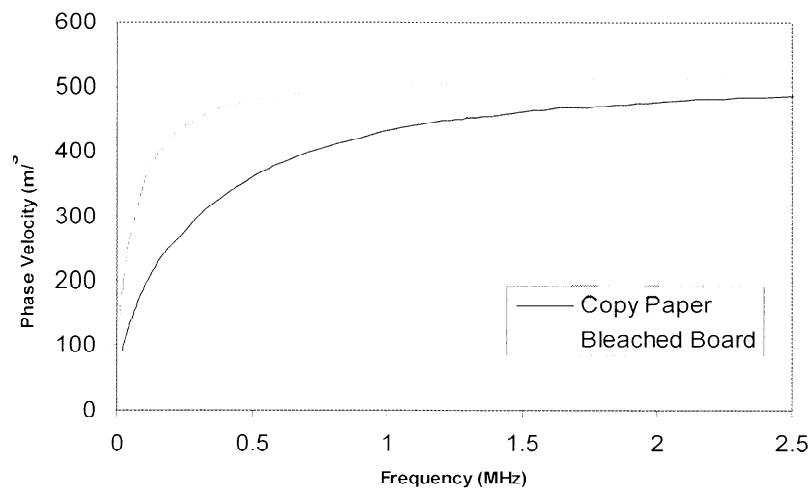
Standard In-Plane Contact transducer methods  
best detect symmetric ultrasonic plate waves.

- 1) motion is mainly in-plane
- 2) propagation is nondispersive
- 3) time-of-flight velocity measurements possible

Laser-ultrasonic transducer methods  
best detect antisymmetric Ao plate waves.

- 1) motion are mainly out-of-plane
- 2) propagation is dispersive
- 3) time-of-flight velocity measurements improbable

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Typical Ao Dispersion Curves for a Lightweight Paper and a Paperboard

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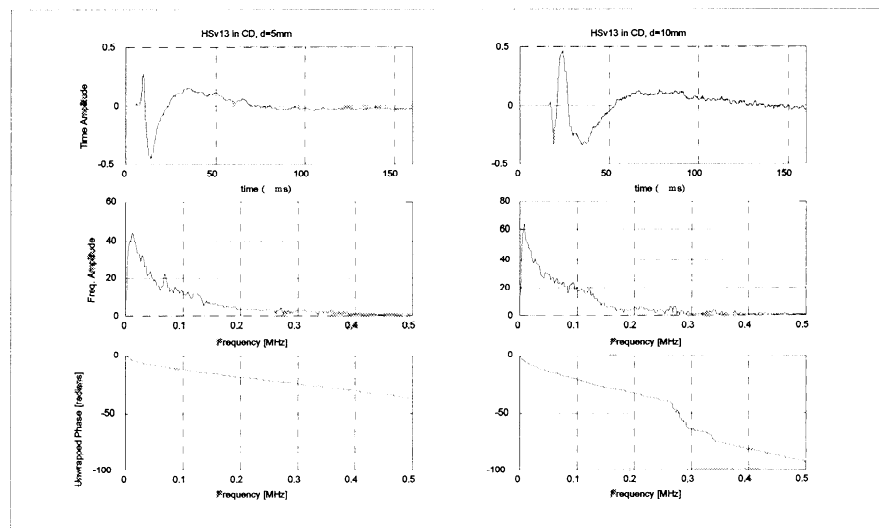
The Ao dispersion curve is a complicated function of the elastic parameters in the plane of propagation

At low frequency it depends mostly on bending stiffness of the plate

At intermediate frequency it depends mostly on bending stiffness and out-of-plane shear rigidity

At high frequency it becomes a surface wave.

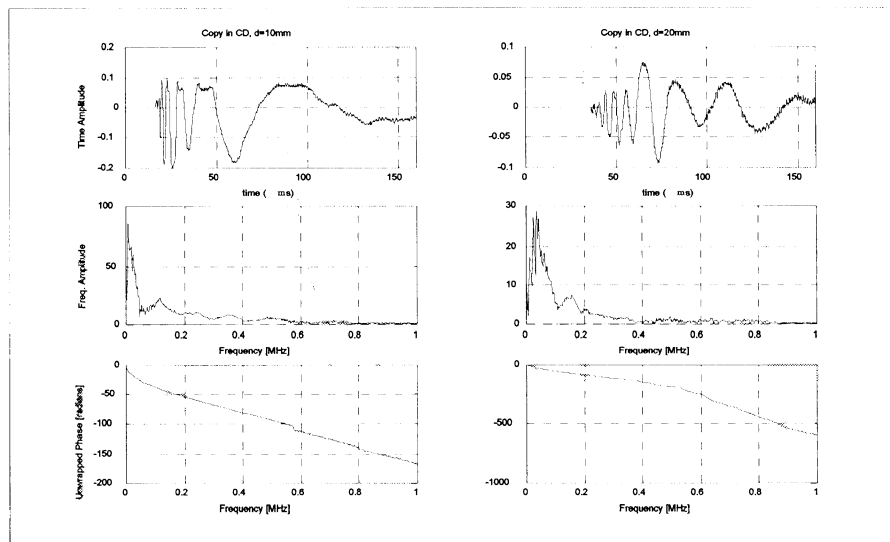
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Time and Frequency Domain Laser Ultrasonic Signals  
Propagating in the Cross direction on a Paperboard Handsheet

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Time and Frequency Domain Laser Ultrasonic Signals  
Propagating in the Cross direction on a Lightweight Paper

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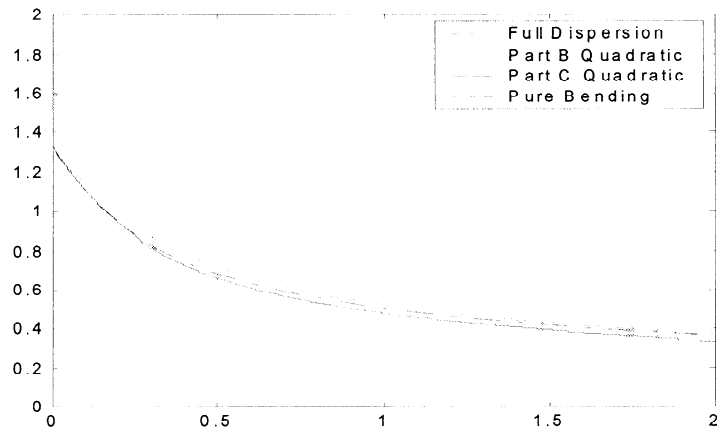
We have developed a simple quadratic equation that gives the low frequency Ao dispersion curve in terms of

bending stiffness / basis weight

and

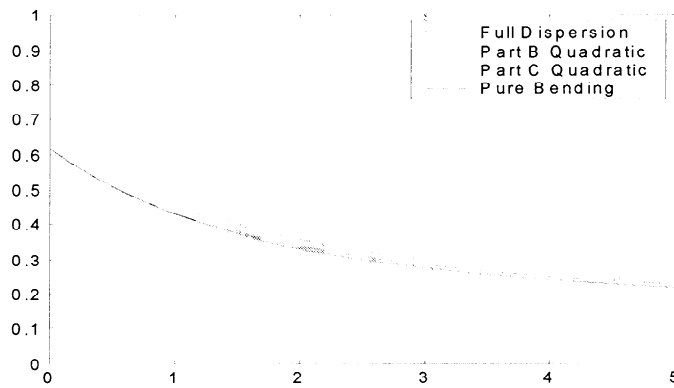
out-of-plane shear rigidity / basis weight.

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A  $c/f^{1/2}$  Dispersion Curve for a Paper Board  
Compared with Bending and Bending Plus Shear Approximations

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A  $c/f^{1/2}$  Dispersion Curve for a Lightweight Paper  
Compared with Bending and Bending Plus Shear Approximations

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### Experimental Determination of an Ao Dispersion Curves

- 1) capture signals at two separations
- 2) do Fourier analysis
- 3) look at phase as a function of frequency
- 4) “unwrap”
- 5) calculate phase velocity from phase at two separations

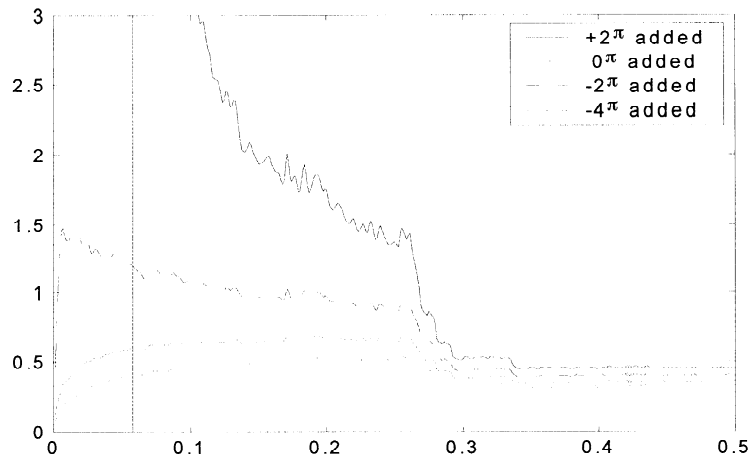
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Fourier analysis allows phase differences to be determined within integer multiples of  $2\pi$ .

“Unwrapping” means determining the correct  $2\pi$  multiple

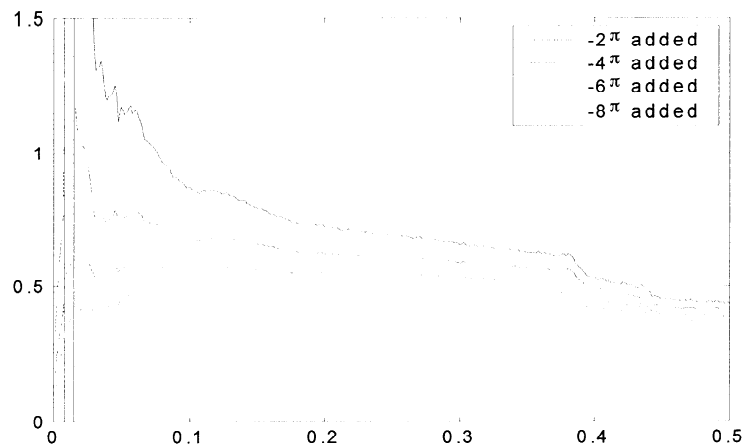
We do this from the shape of the possible  $c/f^{1/2}$  dispersion curves

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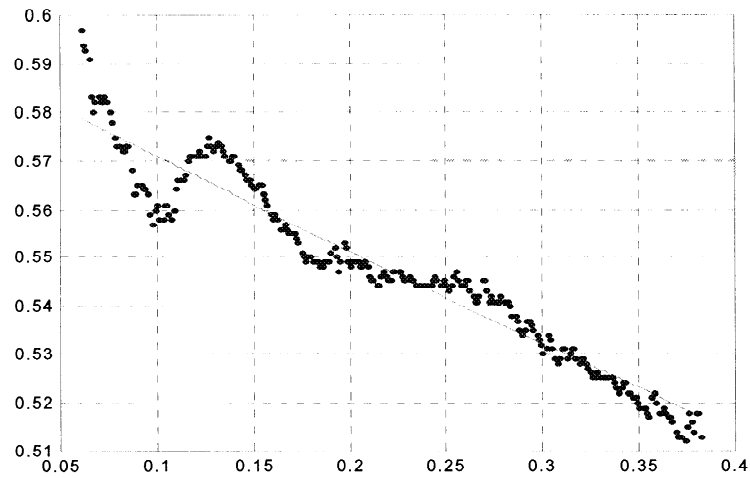
Experimental Bleached Paperboard  $c/f^{1/2}$  Dispersion Curves  
Assuming Different Unwrappings

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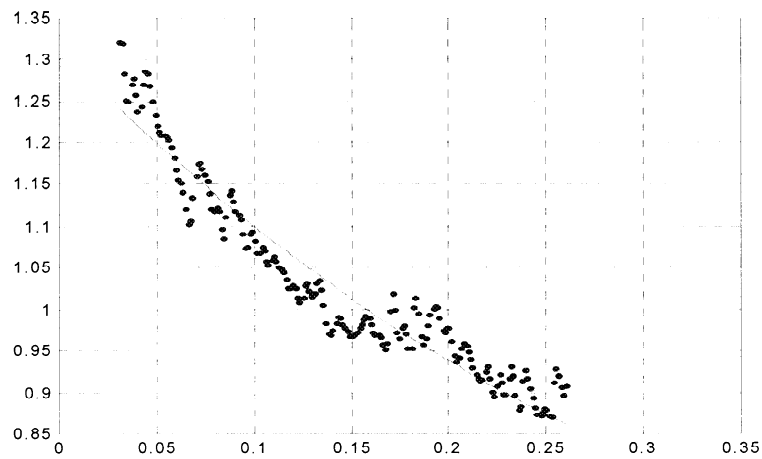
Experimental Lightweight Paper  $c/f^{1/2}$  Dispersion Curves  
Assuming Different Unwrappings

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A Least-Squared Fit of an Eqn. (1) Dispersion Curve  
to Experimental Lightweight Paper Phase Velocities

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A Least-Squared Fit of an Eqn. (1) Dispersion Curve  
to Experimental Bleached Paperboard Phase Velocities

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### Comparison of contact and laser ultrasonic parameters

#### Laser-Ultrasonic Measurements

	Bending stiffness	Shear rigidity
copy paper	$2.5 \times 10^{-4} \text{ Nm}$	$2.0 \times 10^4 \text{ N/m}$
board	$1.6 \times 10^{-2} \text{ Nm}$	$5.0 \times 10^4 \text{ N/m}$

#### Guesses from Contact Ultrasonics and caliper

	Bending stiffness	Shear rigidity
copy paper	$2.9 \times 10^{-4} \text{ Nm}$	$1.9 \times 10^4 \text{ N/m}$
board	$1.6 \times 10^{-2} \text{ Nm}$	$5.1 \times 10^4 \text{ N/m}$

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FUNDAMENTALS OF DIMENSIONAL STABILITY

STATUS REPORT

FOR

PROJECT F020

Douglas Coffin  
Barry Hojjatie  
Kennisha Collins

Institute of Paper Science and Technology  
500 10<sup>th</sup> Street, N. W.  
Atlanta, Georgia 30318



# **Dimensional Stability: Fundamentals of Cockle**

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***Spring PAC 2000***

**Douglas W. Coffin**



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## **Dimensional Stability *Project Personnel***

---

**Doug Coffin**

**Barry Hojjatie**

**Kennisha Collins**

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**Dimensional Stability**  
***Relevant Research Line***

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**11 Convertibility and End-Use Performance**

Improve the ratio of product performance to cost for pulp and paper products 25% by developing: models, algorithms and functional samples of fibrous structures and coatings, which describe and demonstrate improved convertibility and end-use performance.

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**Dimensional Stability**  
***Research Objectives***

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Reduce the amount of paper rejected because of cockle by identifying the causes of cockle and determining corrective measures to prevent cockle.

To develop a science-based understanding of the dimensional stability of paper and paperboard.

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## Dimensional Stability

### ***Project Deliverables***

---

- Literature review of cockle: F020 Report 2, 1996
- Fundamental mechanism of cockle and review of buckling: F020 Report 1, 2, 3
- Analysis of hygro buckling: (*Int. J. Nonlinear Mechanics* 40(6) 1999.)
- Method to quantify cockle: (Shadow Moiré equipment, IPST)
- Report of Handsheet Studies
- Cockle Technical Guide

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## Dimensional Stability

### ***Spring 2000 Status***

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- Handsheet studies (In progress).
- Joint study with project F02102 (In progress).
- Summarize results and prepare final project report. (Not started)

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## Dimensional Stability

### *Milestones for 1999*

---

- Improved Shadow Moiré system
- Controlled method of altering formation
- Improved drying methods with F02102 simulator
- Developed method to evaluate copier cockle

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## Overall Project Name

### *Schedule*

---

Task Descriptions (example)	1999 Apr- Jun	1999 July- Sept	1999 Oct- Dec	2000 Jan- Mar	2000 Apr- Jun	2000 Jul- Sept	2000 Oct- Dec	2000 Apr- Mar
1. cockle during drying	-----	-----	-----	-----	-----	-----X		
2. cockle in converting	-----	-----			-----	-----	-----X	
4. part work with F02102			-----	-----X		-----	-----	-----X
5. Analysis of results							-----	-----
6. Final report								-----

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**Overall Project Name**  
***Future Deliverables***

---

- Report of handsheet studies
- Cockle Technical Guide
  - Identify different types of cockle
  - identify mechanisms
  - identify likely causes and remedies

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**Review of Progress for  
March 1999 - March 2000**

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## Outline

- Handsheet Studies
  - Formation (chemical and mechanical modification)
- Effect of Drying Strategy
  - Joint work with F02102
- Effect of Restraint
  - Stress analysis of a “baggy edge”

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## Handsheet Studies

- Objective: Investigate the effect of process and sheet variables on degree of cockle
  - Cockle developed during drying
  - Cockle developed after drying

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## Handsheets: Previous Findings

- Restraint during drying is a significant factor, increase restraint decrease cockle
- Cockle increased as formation worsened
- cockle improved with lower drying temperature, increased grammage

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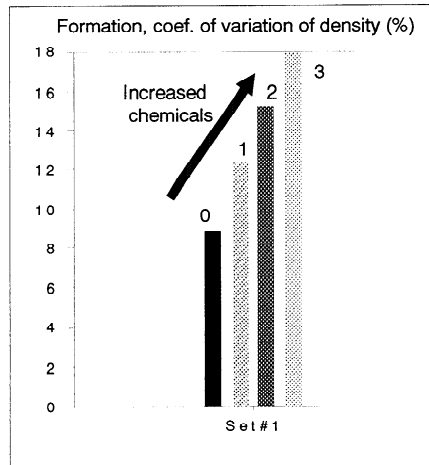
## Focused Study on Formation

- Effect of formation on severity of cockle
  - Formation controlled by chemicals
  - Formation controlled by mechanical means
- Cockle
  - During drying
  - After re-exposure to high RH

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## Chemical Modification of Formation

Equal mixture of  
Polyethylene Oxide  
(PEO) Phenolic  
Formaldehyde  
Resin (PFR)



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## Mechanical Modification of Formation

Increased forming consistency, delayed drainage

	<u>Formation</u>	<u>S.D.</u>
Standard Forming:	14.7	1.39
Modified forming:	5.6	0.15

Formation: Standard Deviation of grammage within sheet  
S. D.: standard deviation of formation for several sheets

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## Making Initially Flat Sheets

Degree of Cockle (Standard Deviation of height, mils)

	<u>Full Restraint</u>	<u>Partial Restraint</u>
Standard Formation	7	12
Modified formation,	7, 3	21

Sheets with poor formation can be made flat  
Problem: getting consistently dried flat sheets

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## Drying Study

- Joint work with F02102
- Sheets dried partly on drying simulator
  - uniform heat transfer
  - nonuniform heat transfer
- cockle severity versus exiting moisture

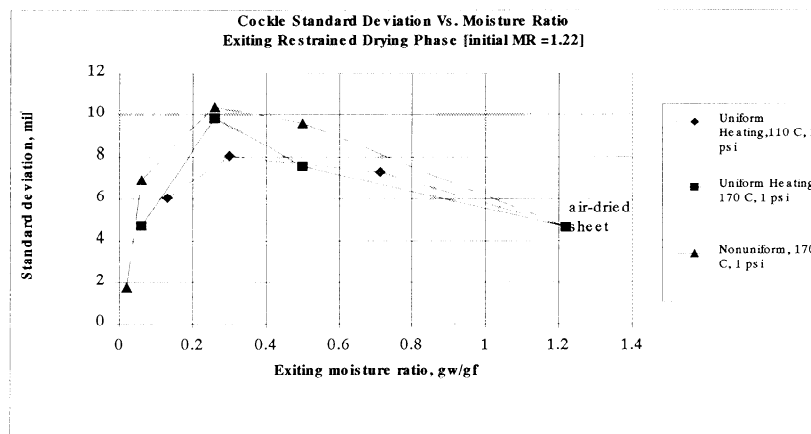
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## “Copy Paper” Furnish

- 70 gsm sheets
- 75% BHWK/25% BSWK; PCC(15%)  
Optical Brightener (2 lbs./ton), AKD (3 lbs./ton) Starch(12 lbs./ton), Retention Aid (2 lbs./ton)
- 450 CSF
- Tensile Ratio: about 2.0
- Initial Solids Level: 45% (via pressing)

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## Cockle versus Exiting Moisture Content



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## Summary of Findings

- Sheets restrained until dried had least cockle
- Sheets dried with nonuniform heat transfer had slightly more cockle
- Sheets partially dried on simulator had worst cockle

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## Possible Explanations

- Sheets that are partially dried on simulator have worst cockle because
  - Larger nonuniformities caused by heat
  - More rapid drying leads to larger stresses

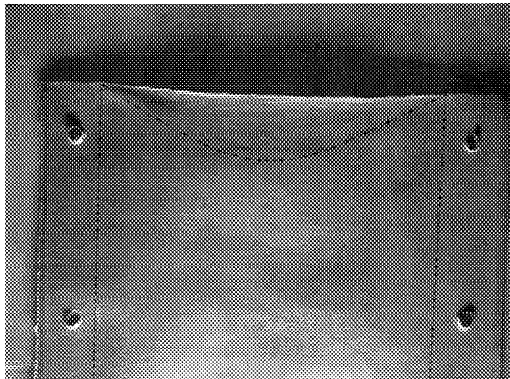
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## A Look at *Baggy Edges*

- Occurrence of cockle greater at CD edges of web
- CD Edges sometimes appear baggy, loose, grainy
- Related to nonuniform shrinkage

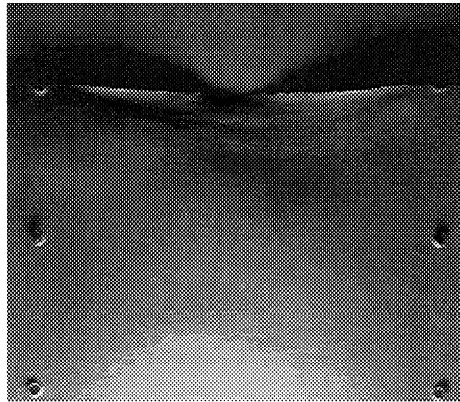
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## Another *Baggy Edge*



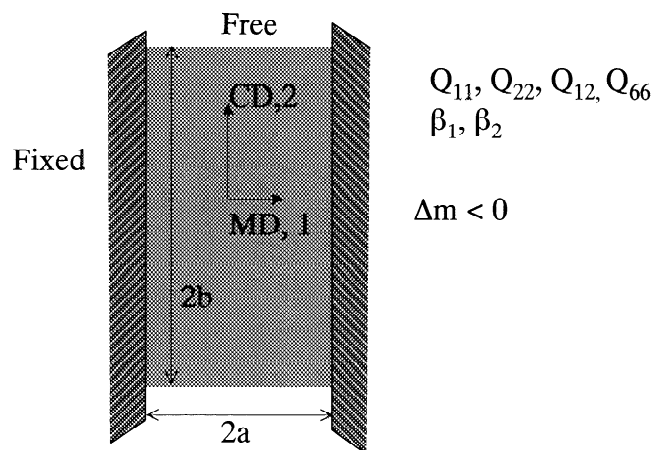
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## Example with Nonflat Edge



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## Analysis of Problem



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## Approximate Stress Distribution

$$\sigma_{MD} = -\Delta m [Q_{11}\beta_1 + Q_{12}\beta_2 + (Q_{12}\beta_1 + Q_{22}\beta_2)\Psi_1(x, y, Q_{ij}, a, b)]$$

$$\sigma_{CD} = -\Delta m [Q_{12}\beta_1 + Q_{22}\beta_2]\Psi_2(x, y, Q_{ij}, a, b)$$

$$\sigma_{CD} = -\Delta m [Q_{12}\beta_1 + Q_{22}\beta_2]\Psi_3(x, y, Q_{ij}, a, b)$$

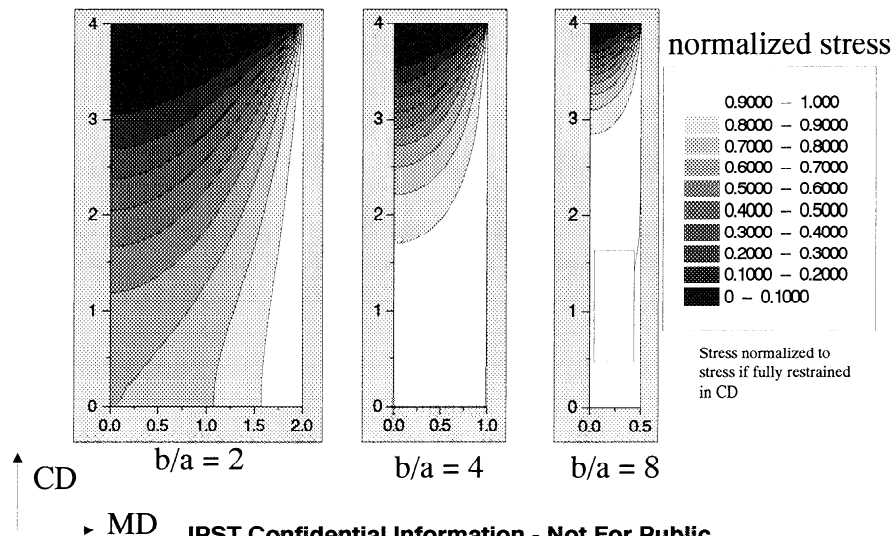
Stresses proportional to change in moisture.

Function of MD, CD shrinkages and stiffnesses

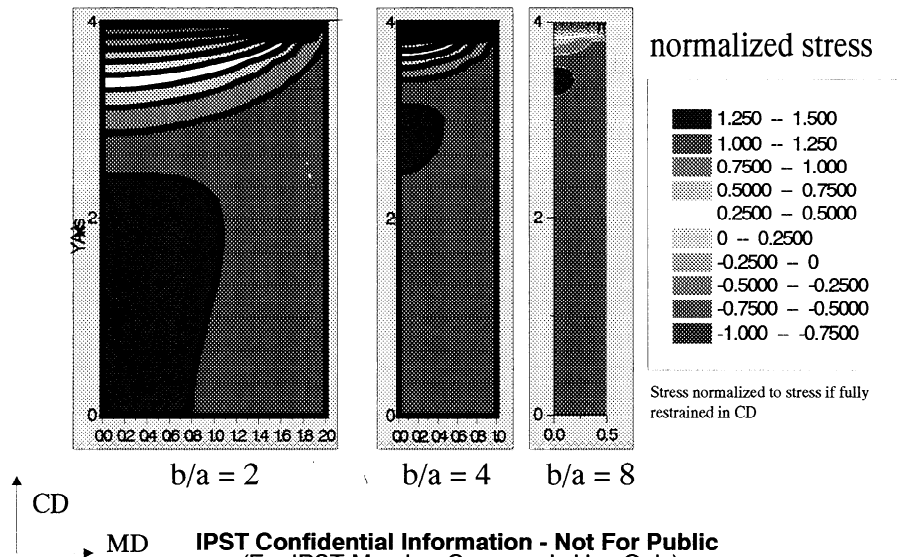
Note, relaxed condition of zero MD displacement along MD edge, prescribed zero strain instead.

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## CD Stresses in One Quadrant



## MD Stresses in one Quadrant



## *Baggy Edge Findings*

- CD stresses are much reduced along MD edges
- MD stress reduced along CD edges
- Edge dries under much less restraint
- As you reduce the open draw, reduce CD width of baggy edge

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## Consequence of Baggy Edge

- Because CD edges are under reduced MD and CD restraint
  - increased occurrence of cockle
  - excessive shrinkage causes rough surface
  - edges will have loose or baggy appearance

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## Summary

- Handsheets
  - All in place, except improve control of drying.
  - Joint work with F02102 going well, provides better control of drying.
  - Cockle Guide. Identifying cockles, wrinkles and the like. Developing simple analyses, looking for solutions.

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FUNDAMENTALS OF ACCELERATED CREEP

STATUS REPORT

FOR

PROJECT F026

Charles Habeger  
Douglas Coffin  
Barry Hojjatie  
Kennisha Collins  
Miranda Bliss

Institute of Paper Science and Technology  
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Atlanta, Georgia 30318

**F026 "Fundamentals of Accelerated Creep"****FY 99-00 \$108,000****Research Lines: 11****DOE/OIT - none****Staff:** Chuck Habeger, Doug Coffin, Barry Hojjatie, and Kennisha Collins**Objective:**

- Establish that sorption induced stress gradients and intensification of creep at high load are the root cause of accelerated creep.
- Determine the influence of mechanical conditioning on the behavior of paper under sustained load.

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**PROJECT DELIVERABLES:**

1. Published papers which convincingly argue that our mechanism is the universal explanation for accelerated creep.
2. Strategies for forming creep resistant papers.

**PROJECT SCHEDULE:**

TASK		00 1 <sup>st</sup> quarter	00 2 <sup>nd</sup> quarter	00 3 <sup>rd</sup> quarter	00 4 <sup>th</sup> quarter
Compression	Work hardening		-----		
Work Hardening	Compression test		-----		
Testing	Compression work hard			-----	-----
Residual stress	Identify dopants	-----	-----		
X ray analysis	Do 1 <sup>st</sup> ORNL tests		-----	-----	
	Start residual stress analysis			-----	-----

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## **MAJOR ACCOMPLISHMENTS SINCE LAST REPORT**

Three papers (the fundamentals of accelerated creep, the effects of recycling on accelerated creep, and the influence of pulp blends and multi-ply sheet structure on accelerated creep) submitted for publication.

Developed a formal explanation for the transient loss tangent phenomenon using the same sorption-induced stress concentration approach. (publication in preparation).

Our accelerated creep work showed that residual stress play a major, unappreciated role in paper creep; therefore, we initiated a joint project with Oak Ridge National Laboratory to use X ray analysis to determine residual stresses in paper.

Demonstrated that work hardening by accelerated creep reduces accelerated creep and is resistant to mechanical relaxation.

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### **Tensile Accelerated Creep Work Hardening**

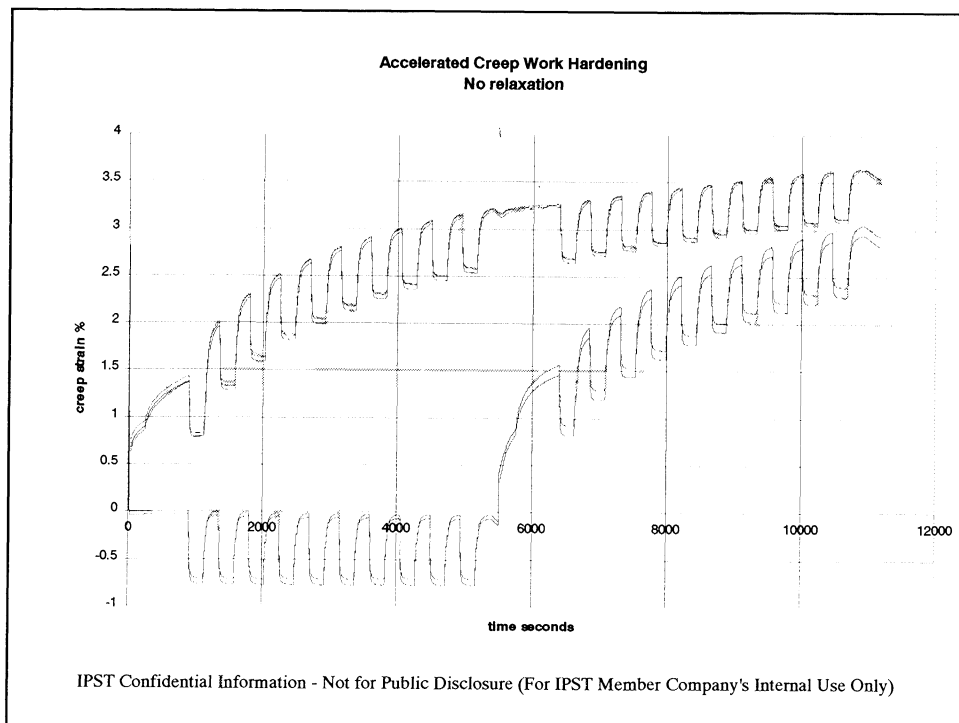
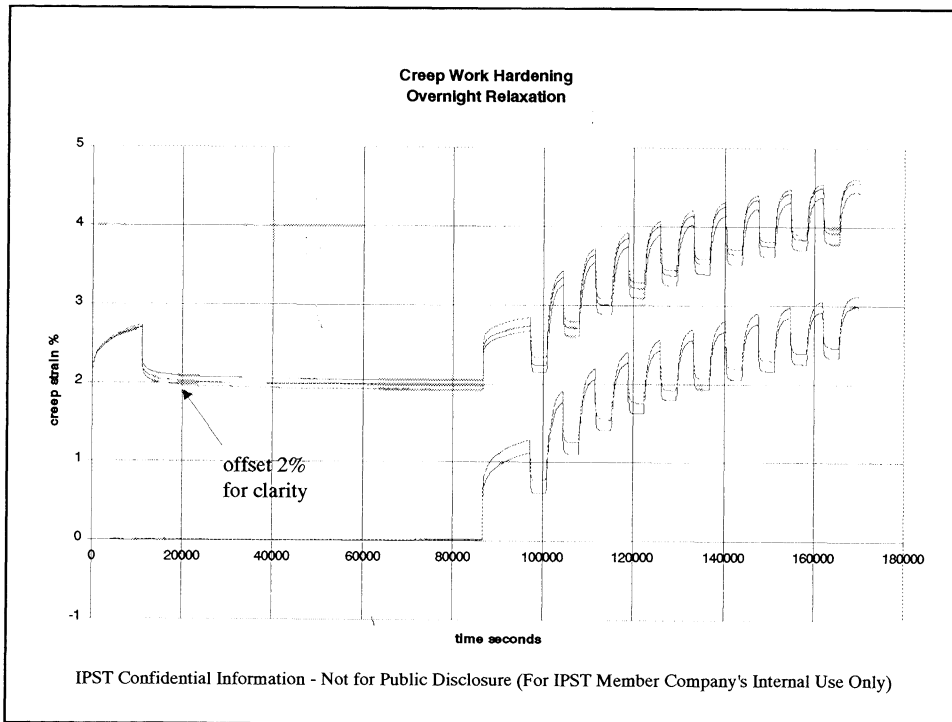
#### **Results**

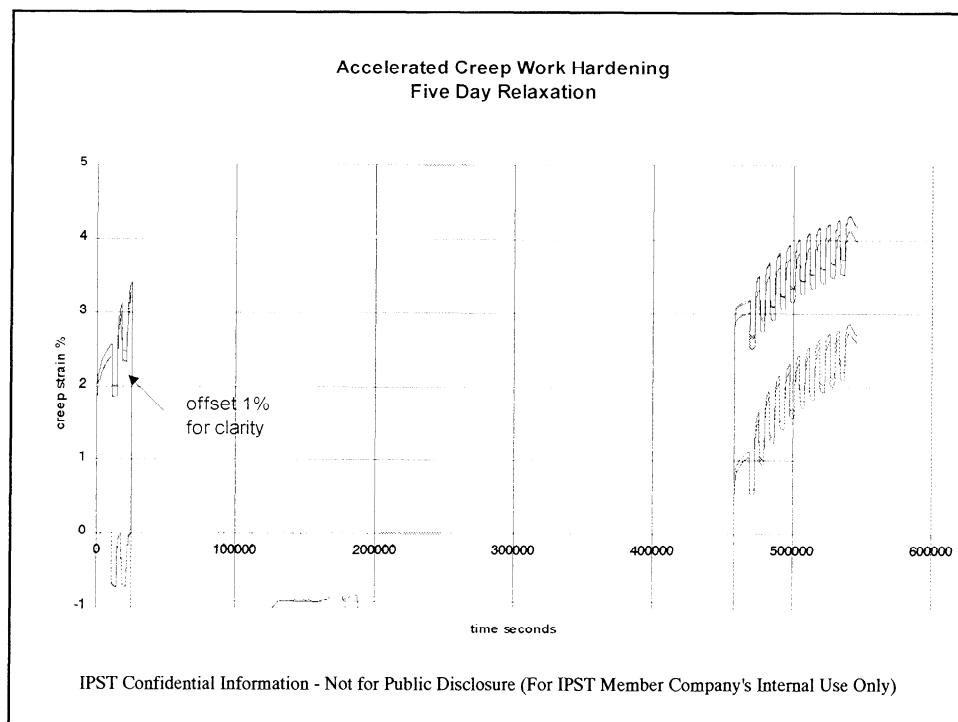
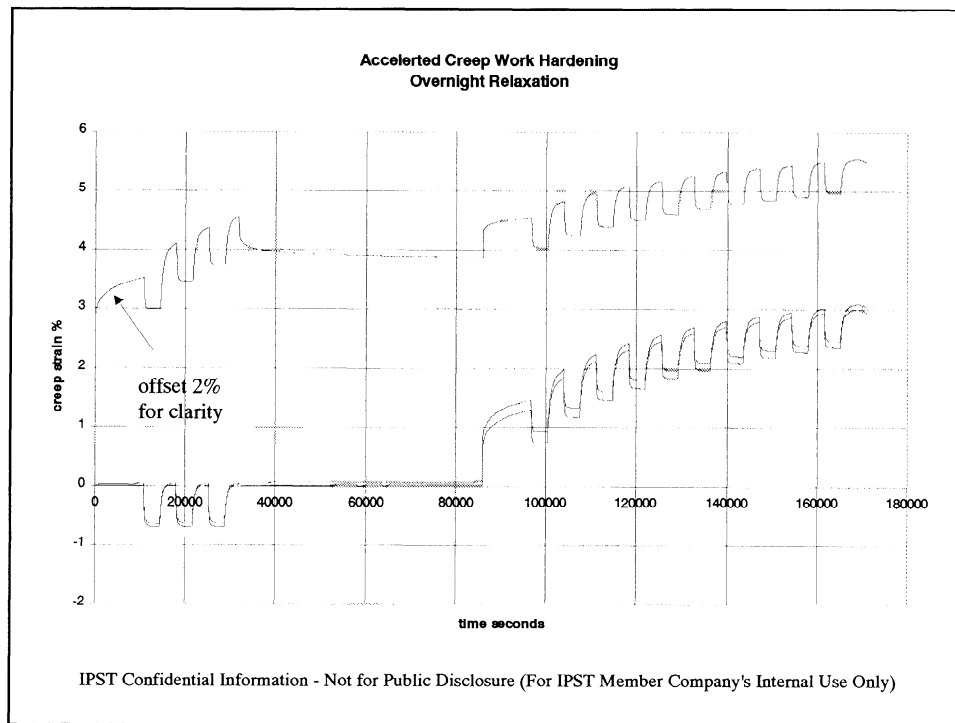
- (1) Creep work hardening produces a creep resistant paper, but does not reduce accelerated creep.
- (2) Accelerated creep work hardening produces an accelerated creep resistant paper

#### **Conclusion**

It is possible to make papers that are stable in variable humidities without treatment to reduce moisture uptake

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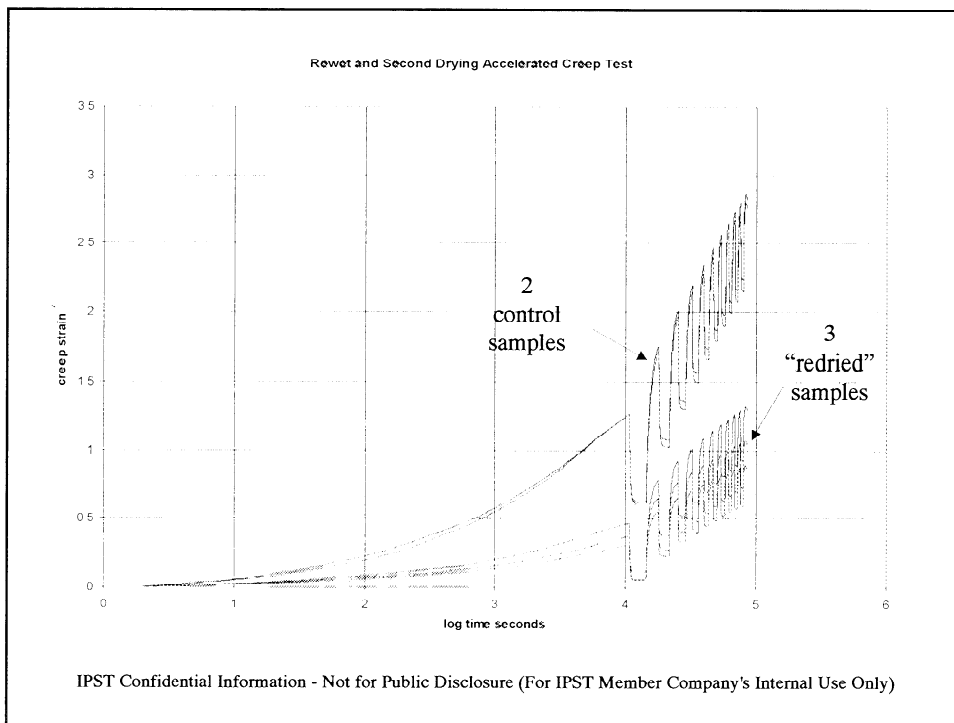


### Tensile accelerated creep work hardening

- 1) does not change tensile strength
- 2) increases tensile stiffness
- 3) reduces tensile stretch and TEA
- 4) may slightly reduce compression strength

Accelerated creep work hardened papers will not be stronger,  
but could be more creep rigid,  
and less prone to cyclic humidity box-lean failure.

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### Remaining Questions

- 1) How does tensile a.c. work hardening affect compression accelerated creep?
- 2) How does compression a.c. work hardening affect compression accelerated creep?
- 3) Can these potential benefits be realized from high-speed drying and /or wetting under load?

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### X-ray Paper Residual Stress Determinations Joint Project with Oak Ridge National Laboratory

From our fundamental analysis of accelerated creep, we believe that residual stresses play an hitherto unappreciated role in the mechanical response of paper.

Especially true for nonlinear, long-time processes, residual stresses can greatly increase paper compliance

Example: paper in compression in middle and tension near surfaces



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X-ray technique have been successfully used  
to determine residual stresses in metals

Polymers don't have the necessary  
sharp, properly-placed x-ray peaks

However, residual stresses have been measured in  
polymers with metallic crystal particle dopants

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Using x-ray equipment, expertise, and computational facilities  
at Oak Ridge National Laboratory,  
we will attempt to measure residual stresses  
in doped paper sheets.

This should be sensitive to through-sheet level residual stresses,  
but not intra-fiber or local between-fiber  
residual stress distributions.

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### **Dopant Requirements**

- 1) random orientation of crystal lattices
- 2) 1 to 10 micron crystallite size
- 3) load sharing with fibers
- 4) about 10-15% by weight concentration in a 1 mm thick sheet

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### **Candidate Doping Techniques**

- 1) lumen loading with metal particles
- 2) lumen loading of  $\text{TiO}_2$  or  $\text{CaCO}_3$  particles
- 3) cell wave precipitation of  $\text{CaCO}_3$
- 4) Kymene induced attachment of metal particles to fiber surface

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### **Research Plan**

- 1) Produce doped sheets at IPST
- 2) Evaluate for x-ray peak quality and load sharing potential at ORNL
- 3) Choose the best doping technique, and calibrate peak location to fiber strain
- 4) Investigate the influence of sheet manufacture, moisture conditioning, and load conditioning on the residual stress distribution in paper

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### **Loss Tangent Sorption Transients**

We have argued vociferously that accelerated creep is a result of sorption induced stress concentrations.

When small amplitude sinusoidal loads are used to measured complex moduli during and after sorption, loss tangent are found to be unusually large.

This phenomenon is commonly associated with accelerated creep.

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We believe that these loss tangent transients are also a result of sorption-induced stress concentrations.

We, Doug in particular, are developing a mathematical argument to demonstrate that part of the reported loss tangent transient phenomenon is an artifact of the experiment and that part is a real phenomenon coming from sorption-induced stress concentrations.

An intermediate iteration of this argument is included in the PAC report.

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### **Accelerated Creep in Fibers**

With some exceptions, fibers don't do accelerated creep, whereas structures made of fibers do.

We argue that this is because fibers are not susceptible to heterogeneity-driven-accelerated creep and they sorb too fast to experience moisture-gradient driven accelerated creep.

If sorption time and moisture cycle time are of the same order, fibers should do accelerated creep

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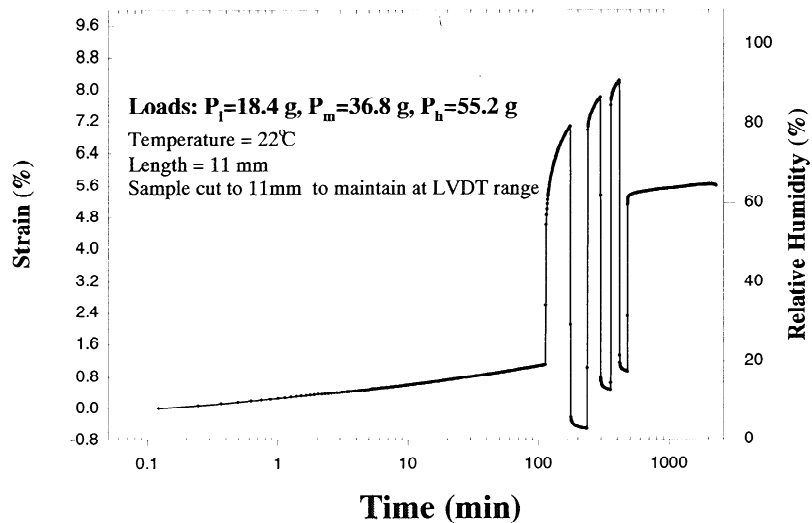
We are doing experiments on Nylon 6,6 fibers of different denier and at different humidity cycle times to demonstrate that accelerated creep will appear in fibers as fibers become larger and cycle times faster

Preliminary results are encouraging.

But, we have had experimental difficulties with creep repeatability which we have solved.

However, difficulties with humidity control have not been resolved.

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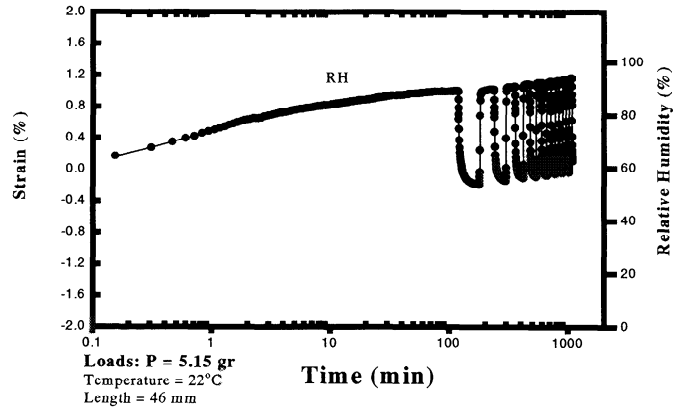


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### Creep of Nylon Fiber Denier 6, Cyclic RH

File: Nyld6f3

Note: Nylon Fiber, Denier 6 subjected to  $P=7.7$  gr at 90% for 2 hrs then conditioned ( $P=0$  gr,  $RH=90\%$ ) for about 2 hrs before this run.

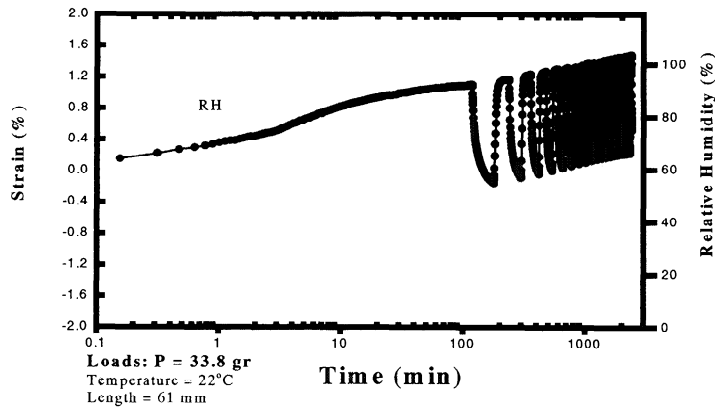


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### Creep Nylon Fiber Denier 40, Cyclic RH

File: Nyld40f4

Note: Nylon Fiber, Denier 40 subjected to  $P=50.7$  gr at 90% for 2 hrs then conditioned ( $P=0$  gr,  $RH=90\%$ ) for about 10 hrs before this run.



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For this work,  
we are abandoning the fiber creep tester

We are modifying the paper creep tester  
(which has superior humidity control)  
to accommodate fiber testing

**MICROMECHANICS OF FIBER NETWORKS**

**STATUS REPORT**

**FOR**

**PROJECT F023**

**Martin Ostoja-Starzewski  
Andrew N. Woods**

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Atlanta, Georgia 30318**



**FUNDAMENTALS OF  
MICROMECHANICS OF FIBER  
NETWORKS**

*Spring PAC 2000*

---

**Martin Ostoja**



**FUNDAMENTALS OF MICROMECHANICS  
OF FIBER NETWORKS**

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**IPST Personnel**

**PI: Martin Ostoja,**

**Staff: A. Woods, T. Jackson**

**PAC Subcommittee**

**A. Colasurdo, W. Hamad, E. Stewart**

**FUNDAMENTALS OF  
MICROMECHANICS OF FIBER NETWORKS**  
*Research Line #11*

---

Improve the ratio of product performance to cost for pulp and paper products 25% by developing: models, algorithms, and functional samples of fibrous structures and coatings which describe and demonstrate improved convertibility and end-use performance; breakthrough papermaking and coating processes which can produce innovative webs with greater uniformity than that achieved by current processes.

**FUNDAMENTALS OF  
MICROMECHANICS OF FIBER NETWORKS**  
*Project Deliverables*

---

- ❁ Computer models and algorithms for:
  - #1: 2-D and 3-D fiber network
  - ❁ #2: Mechanics of a single fiber as an elastic, multi-layer composite
  - ❁ #3: Statistical description of formation
  - ❁ #4: Effects of formation on stress wave propagation in paper

**FUNDAMENTALS OF  
MICROMECHANICS OF FIBER  
NETWORKS**

*Spring PAC 2000*

---

**Martin Ostoja**



**FUNDAMENTALS OF MICROMECHANICS  
OF FIBER NETWORKS**

---

**IPST Personnel**

**PI: Martin Ostoja,**

**Staff: A. Woods, T. Jackson**

**PAC Subcommittee**

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**FUNDAMENTALS OF  
MICROMECHANICS OF FIBER NETWORKS**  
*Research Line #11*

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Improve the ratio of product performance to cost for pulp and paper products 25% by developing: models, algorithms, and functional samples of fibrous structures and coatings which describe and demonstrate improved convertibility and end-use performance; breakthrough papermaking and coating processes which can produce innovative webs with greater uniformity than that achieved by current processes.

**FUNDAMENTALS OF  
MICROMECHANICS OF FIBER NETWORKS**  
*Project Deliverables*

---

- ❁ Computer models and algorithms for:
  - #1: 2-D and 3-D fiber network
  - ❁ #2: Mechanics of a single fiber as an elastic, multi-layer composite
  - ❁ #3: Statistical description of formation
  - ❁ #4: Effects of formation on stress wave propagation in paper

## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

### *Fall ' 99 Status of Goals*

---

- ✿ Goal #1: Extension, optimization, and acceleration of the computer models: **completed**
- ✿ Goal #2: Further verification of the fiber network model using laboratory experiments: **partially completed**
- ✿ Goal #3: Investigation of optimal formation patterns for best mechanical properties of paper: **partially completed**
- ✿ Goal #4 Investigation of the effect of strength additives on the overall strength of paper: **completed**

## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

### *Fall ' 99 Status of Goals*

---

- ✿ Goal #5: Inclusion of single fiber mechanics in the network model: **completed**
- ✿ Goal #6: Correlation of mass distribution with crack damage patterns: **investigated by the student**  
**changed to:** statistical description of formation
- ✿ Goal #7: Biaxial tests and biaxial failure envelopes:  
**changed to:** wave propagation studies

## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

### *Related Work*

---

- ✿ Government funded projects that are linked to this project with fiscal year funding level: none
- ✿ Other DFRC projects that are linked to this project with fiscal year funding level: none
- ✿ Student projects that are linked to this project: Jaime Castro's Ph.D. thesis

## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

### *Related Work*

---

- ✿ Government funded projects that are not linked to this project:
  1. "Multiscale Mechanics of Paper," \$180,000, National Science Foundation, 1997-2000.
  2. "Towards Optimal Performance of Cellulose Fiber Networks," \$100,000, U.S. Department of Agriculture, 1999-2001.

## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

### *Deliverables Now Available*

---

❖ **All computer programs, and algorithms for fiber network mechanics**

- ❖ a member company can use computer programs and algorithms after a demonstration session
- ❖ the benefit: rapid prediction of various material-structure properties

## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

### *Capabilities of the Fiber Network Model*

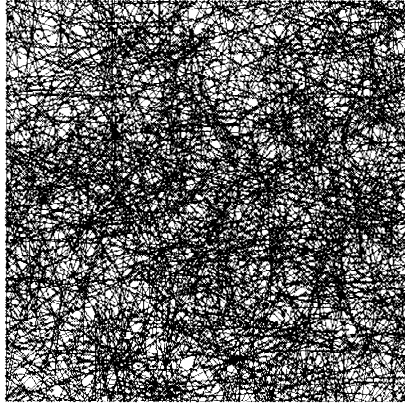
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- |  |              |
|--|--------------|
| ❖ MD-CD stiffness and strength/toughness:            | yes          |
| ❖ Z-direction stiffness and strength:                | in fall 2000 |
| ❖ Crack propagation in the network:                  | yes          |
| ❖ Zero-span tensile:                                 | yes          |
| ❖ STFI (unless coupled with a finite element model): | no           |
| ❖ Delamination:                                      | in 2001      |
| ❖ Viscoplasticity of network (creep)                 | in 2001/02   |
| ❖ Effects of formation:                              | yes          |
| ❖ Effects of mixing several fiber types:             | yes          |
| ❖ Effects of variability in fiber properties:        | yes          |
| ❖ Effects of additives:                              | yes          |

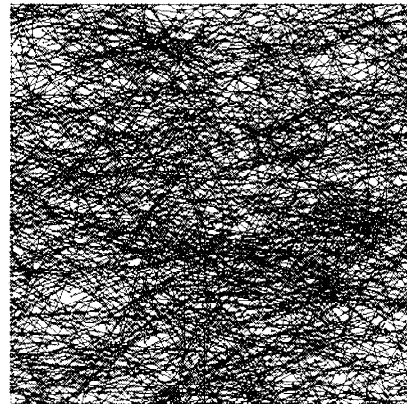
## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

### *Capabilities of the Fiber Network Model*

---



2,500 fibers:    handsheet

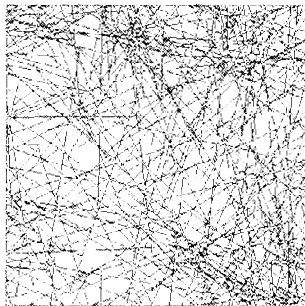


machine made

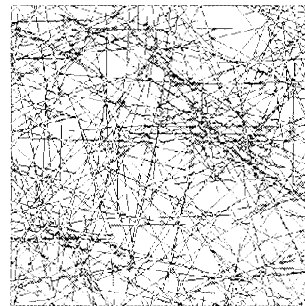
## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

***Discussion:*** Experimental validation of  
network models

---



long fibers, bad formation



short fibers, good formation

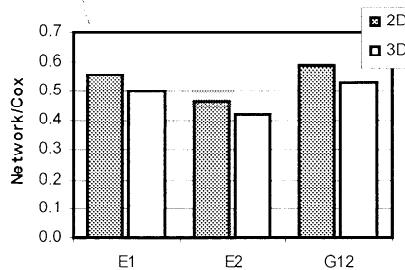
**A decrease in stiffness and its scatter**



## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

***Discussion:*** Effect of Z-displacements  
i.e., treat paper as a 2-D or a 3-D body?

---



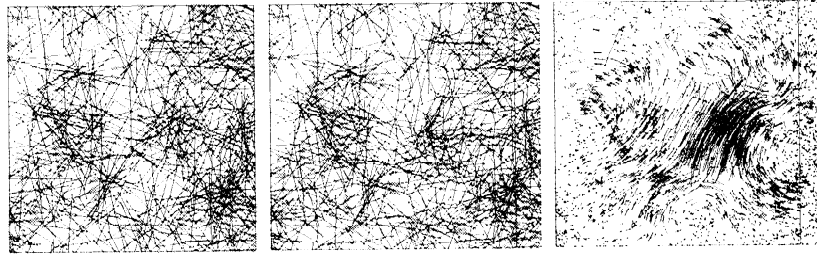
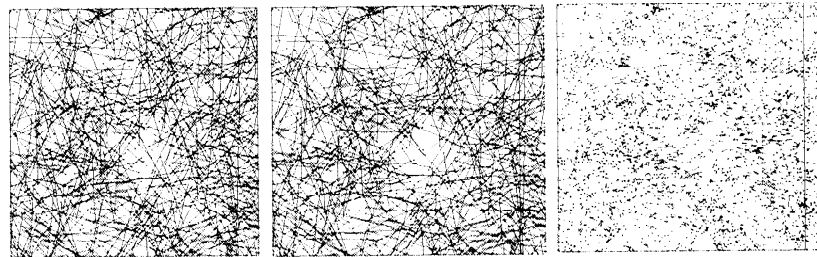
In-plane effective elastic properties for 3-D and 2-D analyses, normalized by the formulas of Cox (1952); averages for ten specimens.

## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

***Discussion:*** Effect of formation on paper mechanics

---

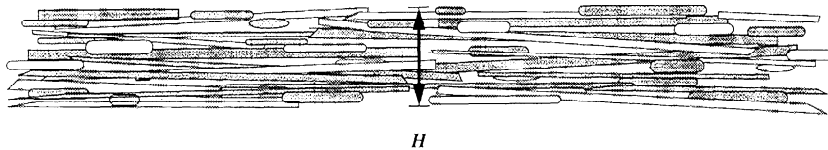
The difference between the deformation field from the computational mechanics approach and the affine (linear) displacement field is dependent on formation. The uniform strain assumption should not be expected to produce good paper strength predictions when network geometry is inhomogeneous, as it is with any but the mildest degree of flocculation.

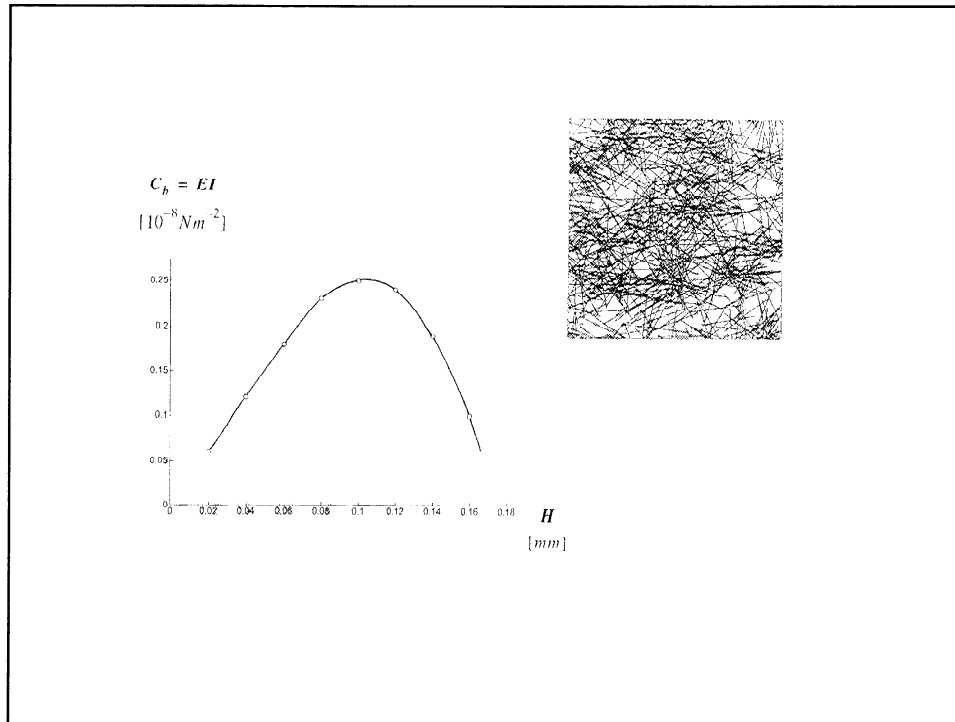
a) floc parameter  $b = 2.0$ b) floc parameter  $b = 0.4$ 

## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

***Discussion:*** Optimal thickness of paper for  
highest bending stiffness

---





## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

***Discussion:*** Effect of strength additives  
i.e., include or neglect fiber-fiber bond flexibility?

---

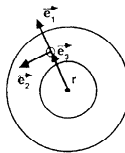
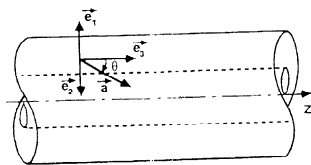
**Depending on the end-properties desired, it may be desirable to add flexibility to the fiber-fiber bonds. Specific effects on elasticity and strength may be predicted with the computer model:  
toughness increase, softening, ...**

## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

***Discussion:*** Mechanics of a single fiber as an  
elastic, multi-layer composite

---

**Determination of chiral effect in a fiber due to helically  
wound fibrils**



$$\begin{bmatrix} F \\ M \end{bmatrix} = \begin{bmatrix} C_{11} & C_{12} \\ C_{21} & C_{22} \end{bmatrix} \begin{bmatrix} \epsilon \\ \beta \end{bmatrix}$$

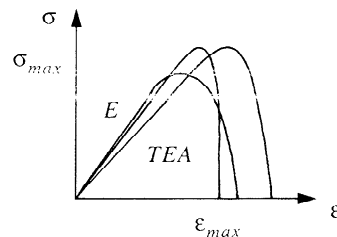
## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

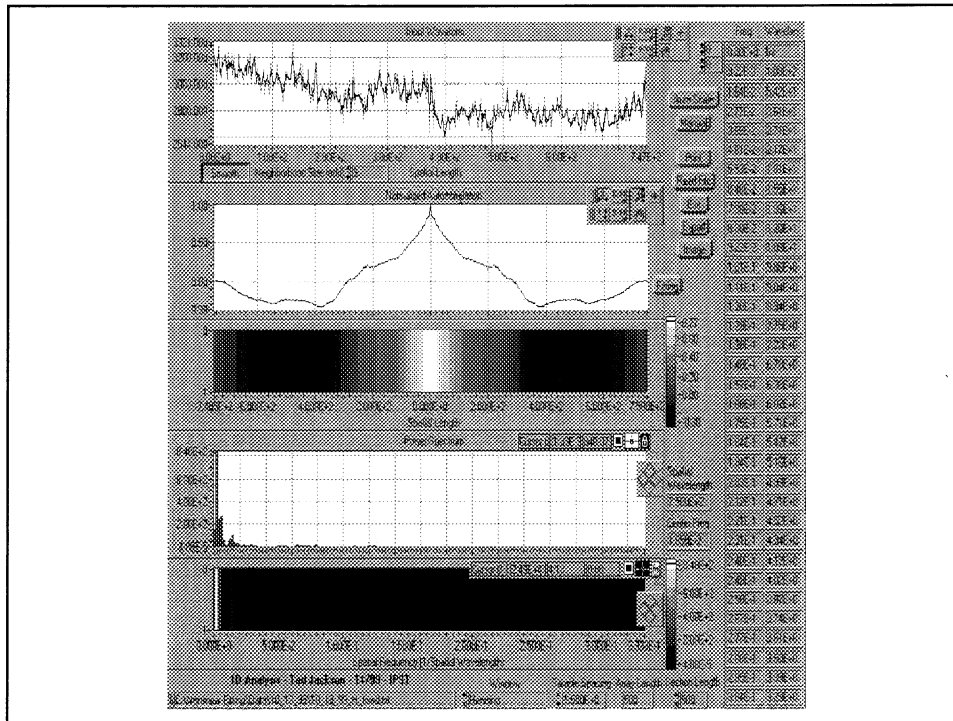
***Discussion:*** Statistical description of formation

---

**Determination of statistical fluctuations of stiffness  
and strength of paper on scales of millimeters  
through tens of meters.**

*Cause:*  
*turbulence on the wire , ...*





## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

***Discussion:*** Sensitivity of wave propagation to  
paper formation

---

**A multitude of scales:**

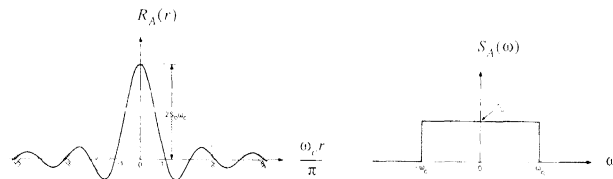
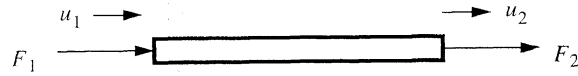
- fibril-bundle structure randomness of single fiber's properties,
- fiber microstructure disorder of flocs' geometry,
- streaks imperfection on scales of meters in MD and CD,
- imperfection on scales of meters in MD and CD.

**A need to understand these effects in:**

- dynamic response of paper webs (flutter, runnability, ...),
- ultrasonic measurements.

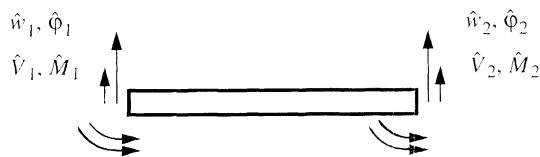
$$\frac{\partial^2 u}{\partial x^2} - \frac{1}{c^2} \frac{\partial^2 u}{\partial t^2} = 0$$

$$\frac{\partial}{\partial x} [A(x)E(x) \frac{\partial u}{\partial x}] - \rho(x)A(x) \frac{\partial^2 u}{\partial t^2} = 0$$



$$KG(x)A(x) \frac{\partial}{\partial x} \left[ \frac{\partial}{\partial x} w - \varphi \right] - \rho(x)A(x) \frac{\partial^2 w}{\partial t^2} = 0$$

$$E(x)I(x) \frac{\partial^2 \varphi}{\partial x^2} + KG(x)A(x) \left[ \frac{\partial}{\partial x} w - \varphi \right] - \rho(x)I(x) \frac{\partial^2 \varphi}{\partial t^2} = 0$$



**Significance:**

- (i) Strong sensitivity of elastodynamic response to formation imperfections.
- (ii) Different effects of imperfection in mass density than imperfection in stiffnesses or cross-sectional area. Depending on the wavelength, there is a tendency to diffuse the resonance frequency around that of the reference (idealized), homogeneous material.

**FUNDAMENTALS OF  
MICROMECHANICS OF FIBER NETWORKS**  
*Goals For Fall 2000*

---

- ✿ #1: Inclusion of inelastic effects (shrinkage, drying, ...) in the single fiber model
  - analysis,
  - parametric studies via the fiber network program
- ✿ #2: Dynamics of fiber and floc deposition on the wire as a basis of formation of paper
- ✿ #3: Fine-tuning of correlation functions of statistical non-uniformity of paper webs by further experiments

## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

### *Schedule*

GOAL	APR	MAY	JUN	JUL	AUG	SEPT
Goal #1: Inclusion of inelastic effects (shrinkage, drying, ...) in the single fiber model	-----	-----	-----	-----	-----	-----
Goal #2: Dynamics of fiber and floc deposition on the wire as a basis of formation of paper		-----	-----	-----	-----	-----
Goal #3: Fine-tuning of correlation functions of statistical non-uniformity of paper webs via further experiments		-----	-----	-----	-----	-----Y

## FUNDAMENTALS OF MICROMECHANICS OF FIBER NETWORKS

### *Patent And Publications*

✿ Patents: none

✿ Planned publications:

on all the subjects discussed

a conference paper on waves/formation  
submitted to the 2000 Paper Physics  
Conference



IMPROVING THE REFINING OF CHEMICAL PULPS

STATUS REPORT

FOR

PROJECT F024

John Waterhouse  
Hiroki Nanko

Institute of Paper Science and Technology  
500 10<sup>th</sup> Street, N. W.  
Atlanta, Georgia 30318

## **SLIDE SUMMARY AND NOTES**

### **Slide 1**

#### **IMPROVING THE REFINING OF CHEMICAL PULPS**

##### **Project F024**

John F. Waterhouse

Derek Page

Hiroki Nanko

Miranda Bliss

March 7, 2000

### **Slide 2**

#### **PROJECT OBJECTIVE**

Determine how changes in fiber structure and the means to produce them, are related to improved paper machine productivity, paper quality, and reduced energy consumption.

##### Relevant Research Lines:

11. Convertability and End Use Performance.

[Note this also impacts research line 10 Reduced net energy consumption]

### **Slide 3**

#### **PROJECT DELIVERABLES**

1. Methodology for determining a pulp's response to refining.
2. Tools for improved pulp characterization.
3. Strategies for reducing energy consumption and improving paper-machine productivity.

### **Slide 4**

#### **SUMMARY OF ACTIVITY**

(October 1999 - March 2000)

- Measurement of a fibers propensity to "cutting"
- Used 12" Sprout-Waldron disk refiner to investigate fiber "cutting"
- Established a model for improving the (disk) refining process using the Invention Machine software

### **Slide 5**

#### **INTER-UNIT COOPERATION**

### **Slide 6**

#### **F013 AND F024 PROJECT OBJECTIVES**

### **Slide 7**

#### **COOPERATIVE PROJECT BETWEEN PULPING & BLEACHING AND PAPER PHYSICS UNITS**

[Wet, Dry, & Re-wet Zero-span measurements comparing Kraft & Kraft-Oxygen delig. pulps]

**Slide 8**

**VARIATION OF WET/DRY ZERO SPAN RATIO WITH KAPPA No. [ CED]**

**Slide 9**

**VARIATION OF WET/DRY ZERO SPAN RATIO WITH KAPPA No. [ DED]**

**Slide 10**

**CURL VARIATION WITH MIXER TYPE [Temperature 90 & 110°C]**

**Slide 11**

**CHANGE IN TENSILE INDEX WITH SEVERITY OF REFINING [after Kerekes]**

**Slide 12**

**CHANGE IN TENSILE INDEX WITH SEVERITY OF REFINING [after Croney ]**

**Slide 13**

**FIBER LENGTH VARIATION WITH SPECIFIC EDGE LOAD**

[Based on data of Kibblewhite, R.P. Fundamental Research Symposium 93]

**Slide 14**

**SIMULATING PULPING & BLEACHING DEGRADATION PROCESSES**

[BULK ACID HYDROLYSIS - 2.6 M HCl @ 2.5% T= 45°C]

**Slide 15**

**FIBER STRENGTH DEPENDENCE ON CELL. CONTENT & DEGRADATION**

[includes results for both vapor phase and bulk HCl treatment]

**Slide 16**

**Variation of Wet/Dry Zero Span Ratio  
with Bulk HCl Treatment**

**Slide 17**

**NEVER DRIED ZERO SPAN STRENGTH**

[Note it is the fiber breaking load that is important in relationship to fiber "cutting"]

**Slide 18**

**SPROUT-WALDRON 12" DISK REFINER - [Freeness Variation]**

**Slide 19**

**SPROUT-WALDRON 12" DISK REFINER - [FQA Fiber Length Variation]**

**Slide 20**

**SPROUT-WALDRON 12" DISK REFINER - [FQA Curl Variation]**

**Slide 21**

**SPROUT-WALDRON 12" DISK REFINER - [FAQ Fines Variation]**

**Slide 22**

**UNREFINED PULP FINES - Control 3.08 % Bulk HCl Treated 6.01%  
[photomicrographs]**

**Slide 23**

**REFINED PULP FINES** - Control 10.3% Bulk HCl Treated 28.1%  
[Photomicrographs]

**Slide 24**

**PULP AND FINES PROPERTIES** - [Pulp & fines pH & fines Zeta Potential]

**Slide 25**

**FIBER "CUTTING" MECHANISMS** - [Iribane, J. & Schroeder, L.]

**Slide 26**

**PROPERTIES OF SELECTED MILL PULPS**

**Slide 27**

**ZERO-SPAN PERFORMANCE OF A MILL PULP**

[Wet & Dry Zero-span strengths from different pulp mill locations]

**Slide 28**

**ZERO-SPAN PERFORMANCE OF A MILL PULP**

[Wet/Dry Zero-span strengths from different pulp mill locations]

**Slide 29**

**ZERO-SPAN PERFORMANCE OF A MILL PULP**

[Zero-span strengths of ECF and TCF pulps & fiber characteristics]

**Slide 30****SUMMARY OF FINDINGS**

- Curl induced during pulping in a peg mixer occurs with kraft-oxygen delignified pulps.
- A significant reduction in never-dried zero-span strength is found with bulk HCl treatment.
- Significant "cutting" was found in the 12" S.W. Disk Refiner for a pulp subjected to 30 minutes HCl treatment.
- Significant reductions in never-dried zero-span strength were found when compared with dry zero-span strength for a number of mill pulps.

**Slide 31****SUMMARY OF FINDINGS**

- A measure of the loss in zero-span strength due to chemical and mechanical factors is the ratio of never-dried to dried zero-span strength.
- Never-dried fiber strength is proposed as being directly related to a fiber's propensity to "cutting" during refining.

**Slide 32****FUTURE WORK**

- Determine the impact of a pulp's propensity to "cutting" on water removal and paper property development for selected mill pulps.
- Evaluate the potential of using low consistency turbulent flow for producing desirable changes in fiber structure.

**IMPROVING THE REFINING OF CHEMICAL PULPS**  
**Project F024**

John F. Waterhouse

Derek Page

Hiroki Nanko

Miranda Bliss

March 7, 2000

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## **PROJECT OBJECTIVE**

Determine how changes in fiber structure and the means to produce them, are related to improved paper machine productivity, paper quality, and reduced energy consumption.

Relevant Research Lines:

11. Convertability and End Use Performance.

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## **PROJECT DELIVERABLES**

1. Methodology for determining a pulp's response to refining.
2. Tools for improved pulp characterization.
3. Strategies for reducing energy consumption and improving paper-machine productivity.

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## **SUMMARY OF ACTIVITY**

(October 1999 - March 2000)

- Measurement of a fibers propensity to “cutting”
- Used 12” Sprout-Waldron disk refiner to investigate fiber “cutting”
- Established a model for improving the (disk) refining process using the Invention Machine software

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## INTER-UNIT CO-OPERATION

- **Pulping and Bleaching (F013)**

Tom McDonough

Chuck Courchene

Craig Jackson

- **Paper Physics (F024)**

John Waterhouse

Derek Page

Hiroki Nanko

Miranda Bliss

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## F013 and F024 Project Objectives

- **(F013)** - To evaluate the effects of delignification processes and ECF bleaching on fiber properties, paper properties , and refining behavior.
- **(F024)** - Determine how changes in fiber structure and the means to produce them, are related to improved paper machine productivity, paper quality, and reduced energy consumption.

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## COOPERATIVE PROJECT BETWEEN PULPING & BLEACHING AND PAPER PHYSICS UNITS

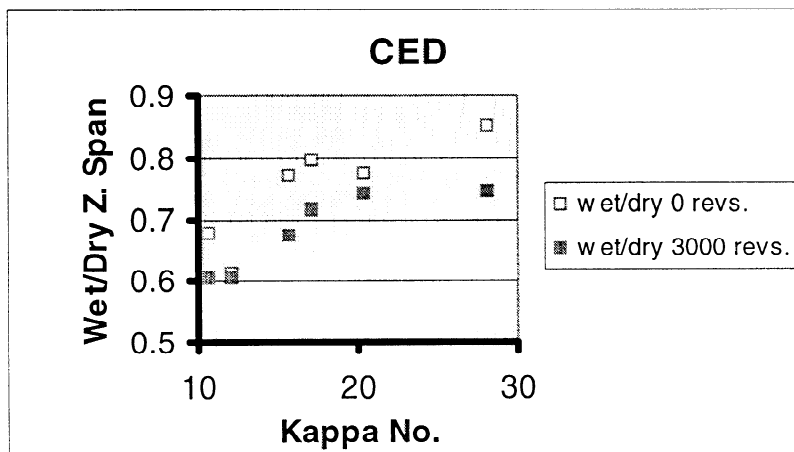
### Kraft versus Kraft-Oxygen Delignification

Unbleached Zero Span Strength Nm/g

Material & Cook	Unbl. Kappa no	Wet		Dry		Rewet	
		0 revs	3000 revs	0 revs	3000 revs	0 revs	3000 revs
S.P. chips $\Rightarrow$ conventional kraft	110.1	104.7	122.5	112.4	132.5	113.5	131.2
S.P. chips $\Rightarrow$ conventional kraft	28.1	120.6	129.8	140.6	151.1	129.3	136.2
S.P. chips $\Rightarrow$ conventional kraft	17.1	114.7	114.5	140.0	150.0	128.3	126.9
kappa 30 $\Rightarrow$ O oxygen	20.4	82.7	107.4	98.3	138.6	89.4	127.0
kappa 30 $\Rightarrow$ O oxygen	15.7	89.4	104.1	107.9	144.5	90	120.1
kappa 30 $\Rightarrow$ O oxygen	12.1	73.4	99.4	97.4	136.0	75.3	107.1
kappa 30 $\Rightarrow$ O oxygen	10.7	69.2	87.3	95.7	144.5	71.6	103.7

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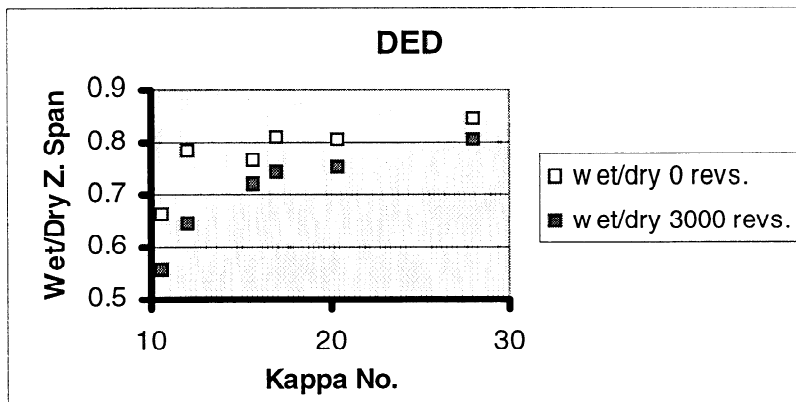
### VARIATION OF WET/DRY ZERO SPAN RATIO WITH KAPPA No.



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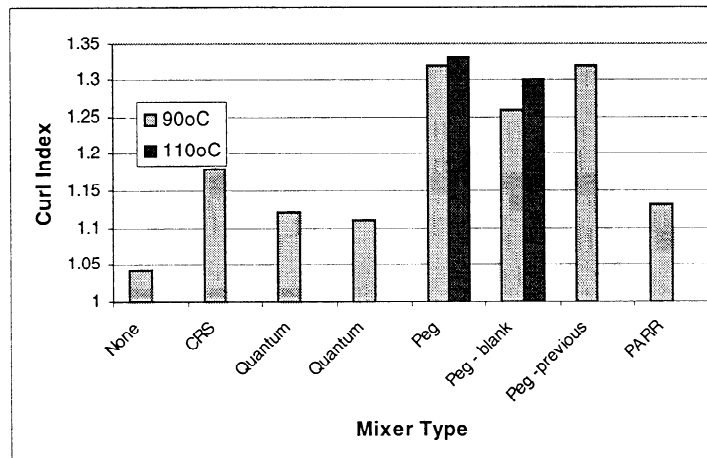


## VARIATION OF WET/DRY ZERO SPAN RATIO WITH KAPPA No.



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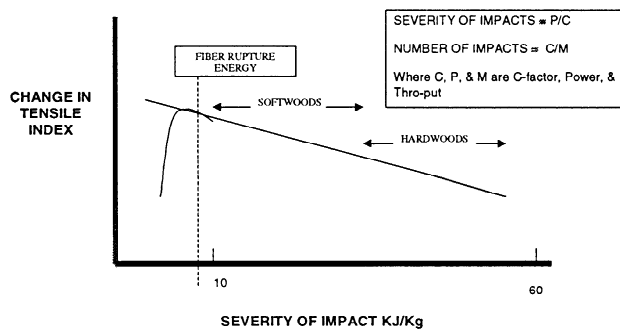
## CURL VARIATION WITH MIXER TYPE



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# SEVERITY OF REFINING

CHANGE IN TENSILE INDEX WITH SEVERITY OF REFINING AT CONSTANT SPECIFIC ENERGY  
(AFTER KEREKES, R.)



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Taken from Croney, C., Ouellet, D., & Kerekes, R.J.

5th International Paper & Board Industry Conference Scientific & Technical Advances in Refining, Vienna, Austria April, 1999

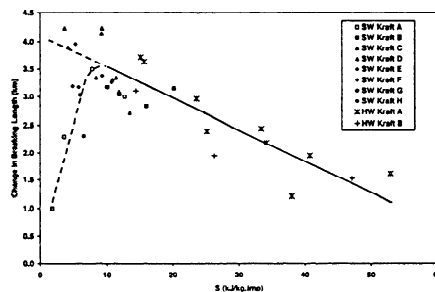
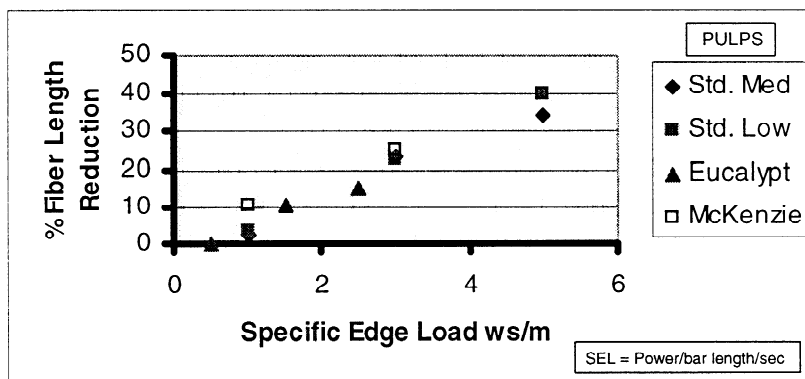


Figure 12: Change in breaking length as a function of Specific Energy per Impact (S) for hardwood and softwood pulps at  $E=120$  kWh/t.

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## VARATION OF FIBER LENGTH REDUCTION WITH SPECIFIC EDGE LOAD

Based on data of Kibblewhite, R.P. Fundamental Research Symposium 93



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## SIMULATING PULPING & BLEACHING DEGRADATION PROCESSES

### BULK ACID HYDROLYSIS

2.6 M HCl @ 2.5% T= 45°C

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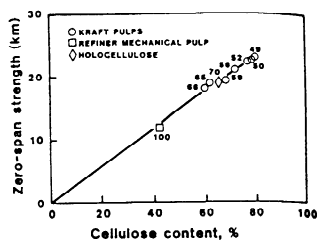


Fig. 1. Zero-span strength of well-bonded sheets of black spruce pulps plotted against cellulose content. The figures indicate % yield (Page et al. 1983).

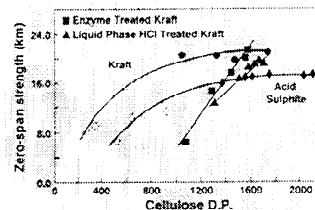
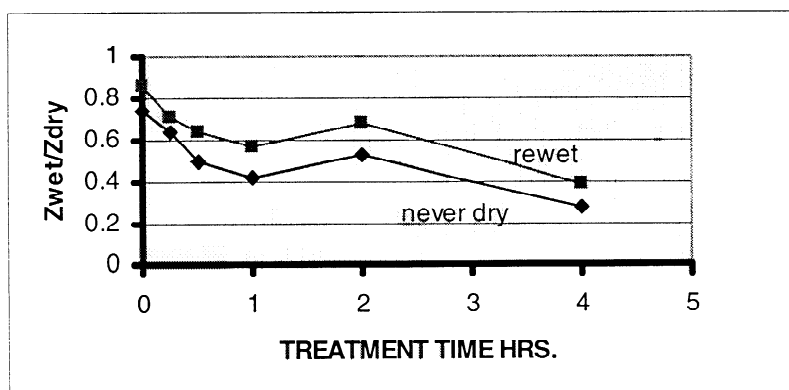


Fig. 4. Zero-span strength of hand sheets of black spruce pulps as a function of D.P. of cellulose. The dotted symbols denote samples treated in the vapour-phase with hydrochloric acid.

Data of Gurnagul, N., Page, D.H., & Paice, M.G. in Nordic Pulp & Paper Research J. No. 3:152-154 (1992)

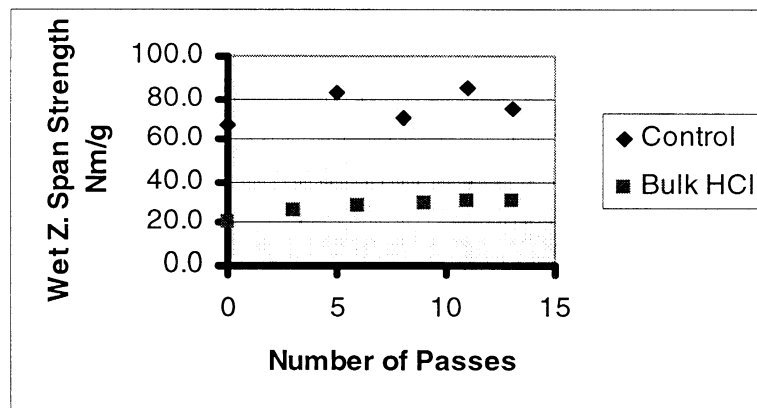
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### Variation of Wet/Dry Zero Span Ratio with Bulk HCl Treatment



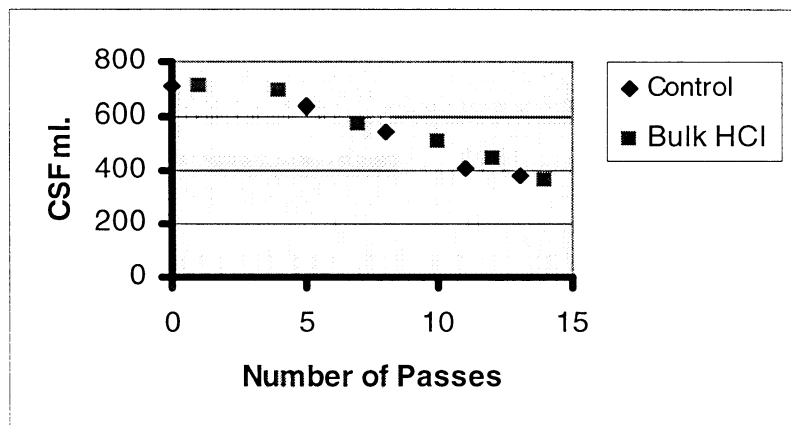
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## NEVER DRIED ZERO SPAN STRENGTH



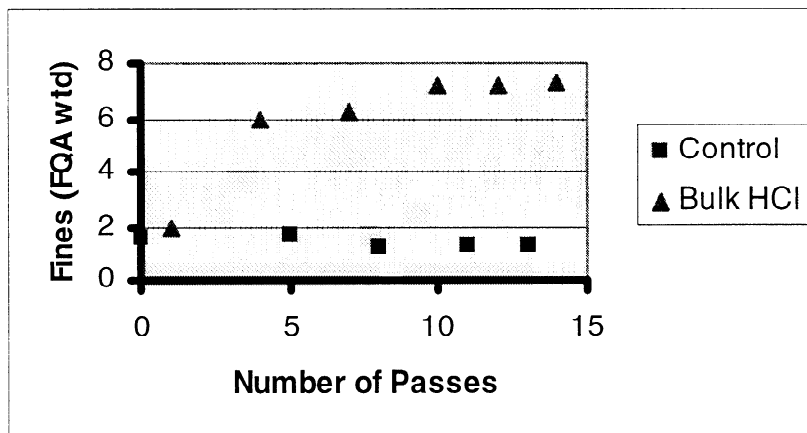
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## SPROUT-WALDRON 12" DISK REFINER FREENESS VARIATION



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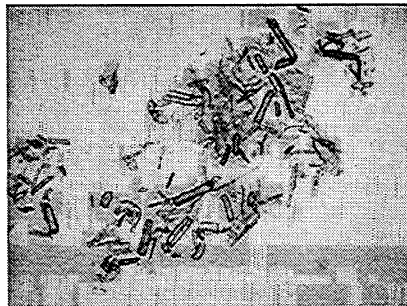
## SPROUT-WALDRON 12" DISK REFINER FQA FINES VARIATION



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## UNREFINED PULP FINES

Control 3.08 %    Bulk HCl Treated 6.01%

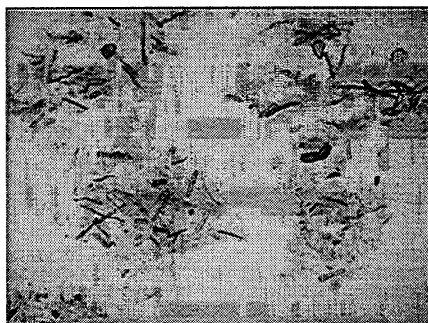


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## REFINED PULP FINES

Control 10.3%

Bulk HCl Treated 28.1%



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## PULP AND FINES PROPERTIES

PASSES/ SAMPLE	% FINES	Pulp pH	Fines pH	Zeta Potential
0/ Untreated	3.08	5.46	5.72	-
13/Untreated	10.3	5.23	5.69	-20.9
0/Bulk HCl	6.01	3.34	4.20	-
13/Bulk HCl	28.1	3.32	4.24	-8.2

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Taken from Iribane, J. and Schroeder, L.R.

Proceeding 1999 TAPPI International Paper Physics Conference , San Diego, CA September 1999.

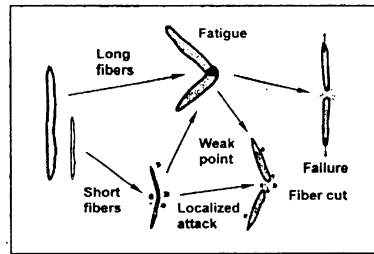


Figure 11. Proposed paths of weak-point formation and resulting effects.

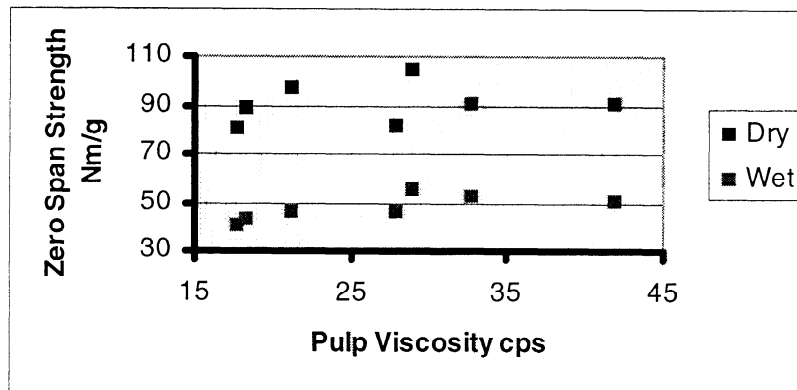
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## PROPERTIES OF SELECTED MILL PULPS

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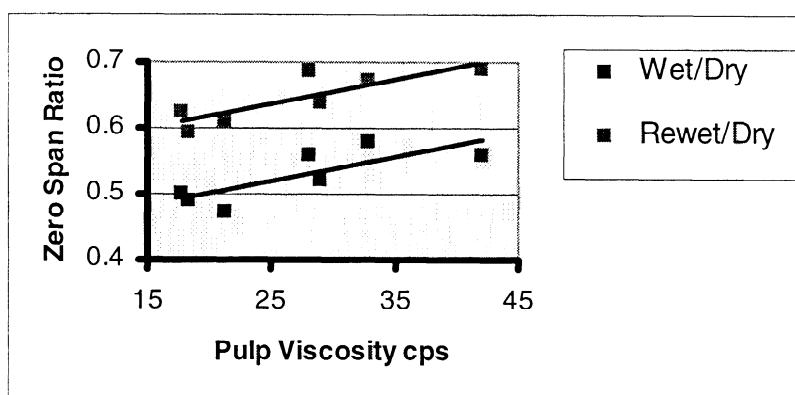


## ZERO-SPAN PERFORMANCE OF A MILL PULP



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## ZERO-SPAN PERFORMANCE OF A MILL PULP



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## ZERO-SPAN PERFORMANCE OF A MILL PULP

PROPERTY	PULP ECF		PULP TCF	
	0 revs.	3000 revs.	0 revs.	3000 revs.
<b>CSF ml</b>	672	404	654	354
<b>ZS wet Nm/g</b>	104.7	129.6	93.4	101.3
<b>ZS dry Nm/g</b>	111.6	168.5	117.4	144.4
<b>ZSwet/ZSdry</b>	0.938	0.770	0.795	0.702
<b>Length wtd.</b>	2.26	2.38	2.27	2.32
<b>Curl arith.</b>	0.107	0.057	0.118	0.057
<b>% fines wtd.</b>	1.10	0.805	1.01	0.63

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## SUMMARY OF FINDINGS

- Curl induced during pulping in a peg mixer occurs with kraft-oxygen delignified pulps.
- A significant reduction in never-dried zero-span strength is found with bulk HCl treatment.
- Significant “cutting” was found in the 12” S.W. Disk Refiner for a pulp subjected to 30 minutes HCl treatment.
- Significant reductions in never-dried zero-span strength were found when compared with dry zero-span strength for a number of mill pulps.

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## **SUMMARY OF FINDINGS**

- A measure of the loss in zero-span strength due to chemical and mechanical factors is the ratio of never-dried to dried zero-span strength.
- Never-dried fiber strength is proposed as being directly related to a fiber's propensity to "cutting" during refining.

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## **FUTURE WORK**

- Determine the impact of a pulp's propensity to "cutting" on water removal and paper property development for selected mill pulps.
- Evaluate the potential of using low consistency turbulent flow for producing desirable changes in fiber structure.

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**FUNDAMENTALS OF INTERFIBER BONDING**

**STATUS REPORT**

**FOR**

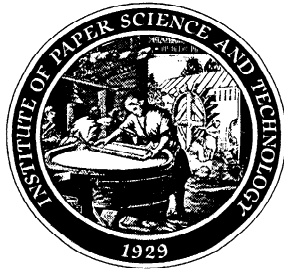
**PROJECT F025**

**Hiroki Nanko  
Shaobo Pan**

**Institute of Paper Science and Technology  
500 10<sup>th</sup> Street, N. W.  
Atlanta, Georgia 30318**

# **Fundamentals of Interfiber Bonding (F-025)**

***Spring PAC 2000***



**Hiroki Nanko**

**Shaobo Pan**

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## **Fundamentals of Interfiber Bonding**

***Project Personnel***

### **•IPST Personnel**

**Hiroki Nanko, PI**

**Shaobo Pan, Staff**

### **•PAC Subcommittee Personnel**

**Anthony Colasurdo, Chair**

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## **Fundamentals of Interfiber Bonding**

### ***Relevant Research Line***

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**Line 11 – Improve the ratio of product performance to cost for pulp and paper products by 25%**

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## **Fundamentals of Interfiber Bonding**

### ***Project Deliverables***

---

- Microscopy techniques for visualizing papermaking polymers
- Strategy for improving the effectiveness of bonding agents
- Insight for designing new bonding agents

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## **Fundamentals of Interfiber Bonding**

### ***Related Work***

---

- Student Project:  
By Michelyn McNeal

“Visualization of Guar Gum Adsorption to Pulp”

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## **Fundamentals of Interfiber Bonding**

### ***Fall ' 99 Status***

---

- C-PAM visualization was completed
- Guar gum visualization was started
- C-starch visualization was started
- Examination of bond failure was planned

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## **Fundamentals of Interfiber Bonding**

### ***Tasks/Status for Spring 2000***

---

- Cationic guar gum visualization is near completion
- Cationic starch visualization is near completion
- Examination of bond failure will begin in summer

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## **Fundamentals of Interfiber Bonding**

### ***Discussion***

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- Visualization of cationic starch adsorption
- Visualization of cationic guar gum adsorption

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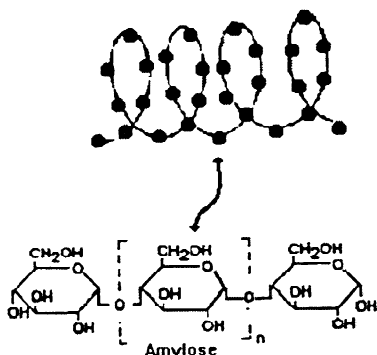




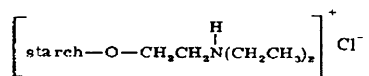
## Cationic Starch

### [Starch Structure]

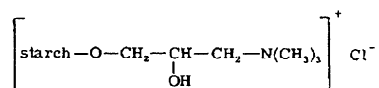
#### Amylose



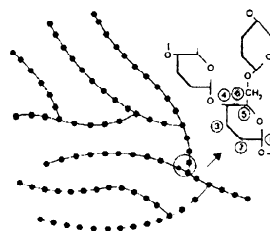
#### Tertiary Aminoalkyl Ethers of Starch



#### Quaternary Ammonium Starch Ethers

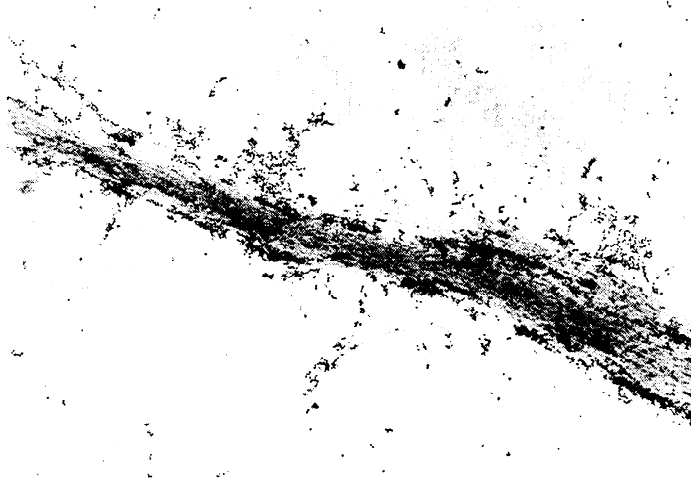


#### Amylopectin



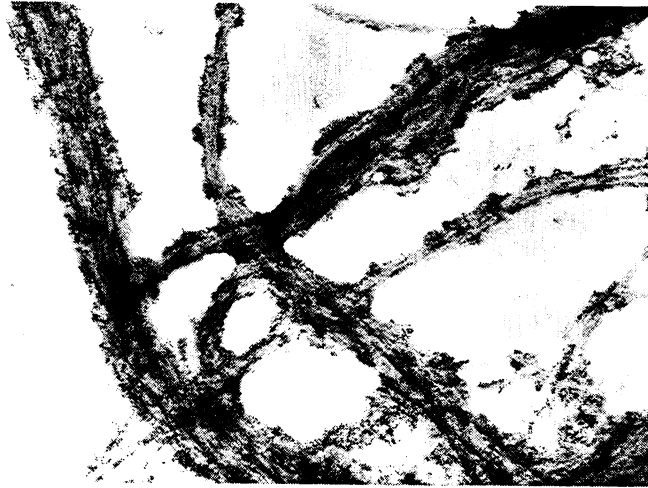
## Previous Results

### Cationic Corn Starch (0.5%)



## ***Previous Results***

***Cationic Corn Starch (0.5%)***

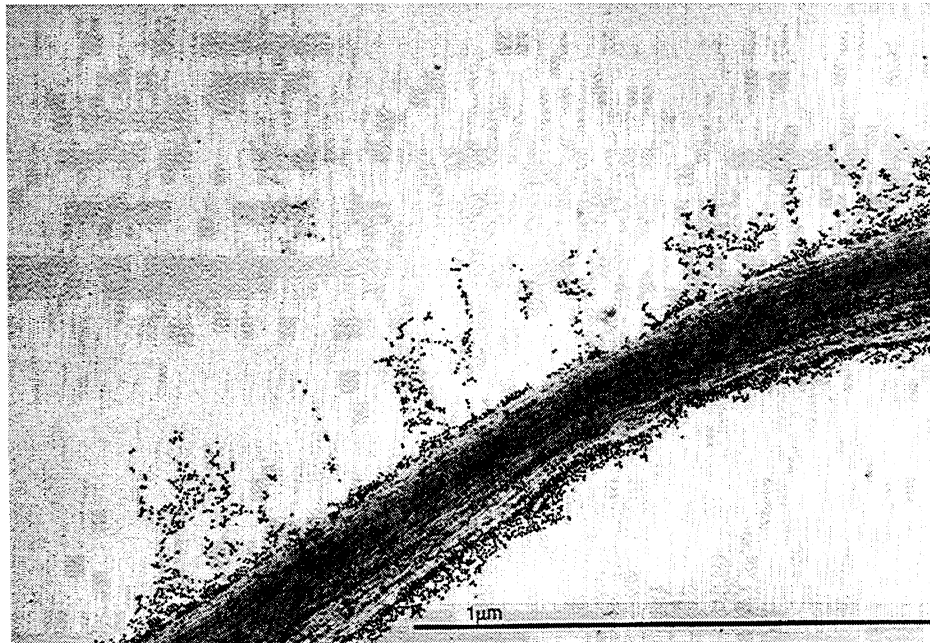


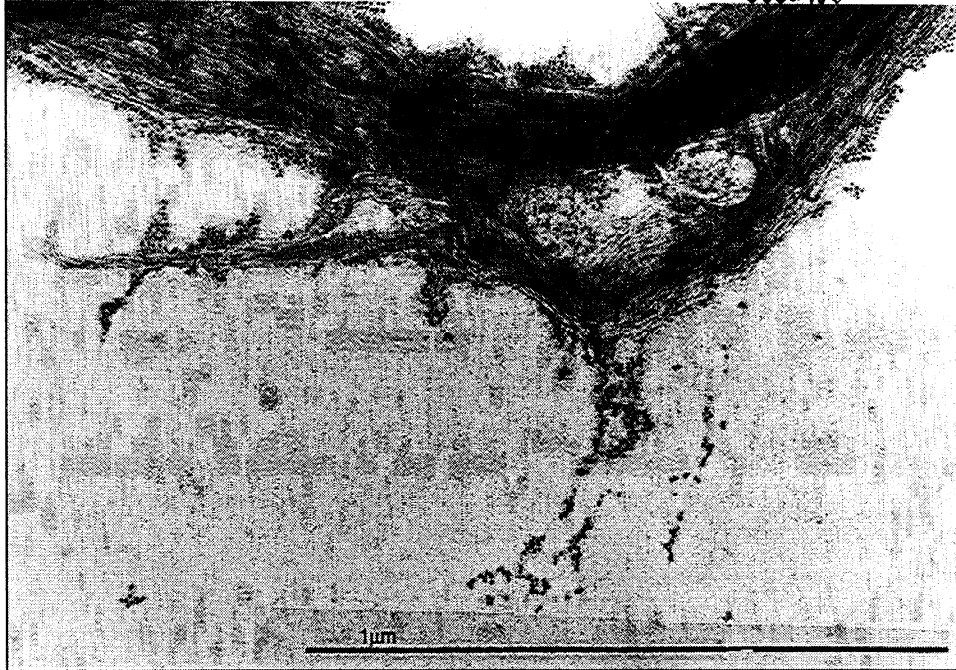
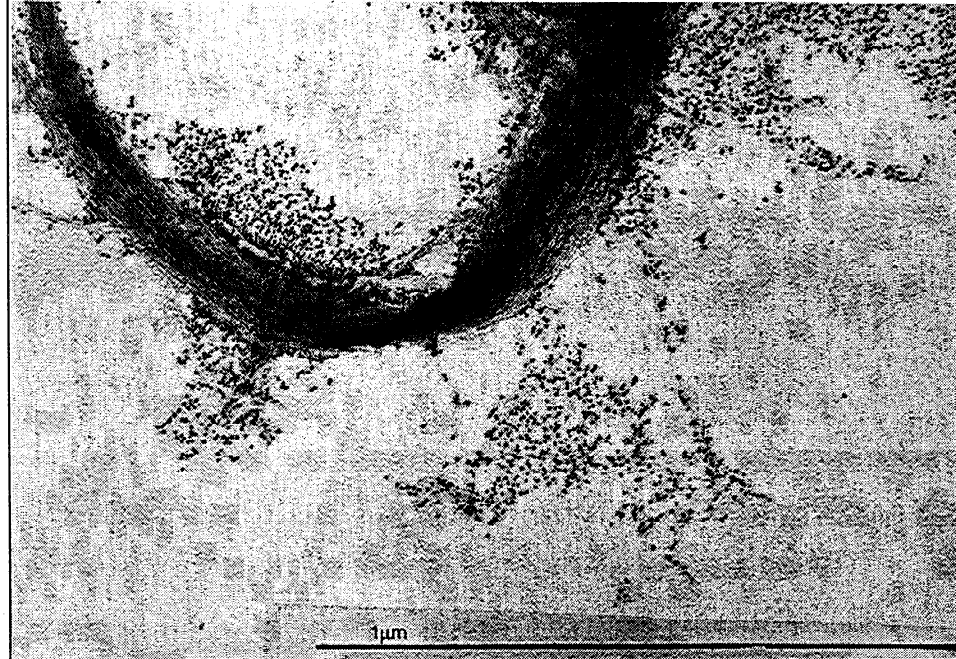
### ***Characteristics of Cationic Starch***

#### ***Adsorption*** (common features to corn and potato starches)

- Non-uniform adsorption to the fibrils
- Adsorption forming stretched strands
- Adsorption forming either loose or dense agglomerations

#### **Short Strands with Dense Agglomerations (Corn)**



**Dense Agglomerations and Strands (Potato)****Loose agglomerations (corn)**

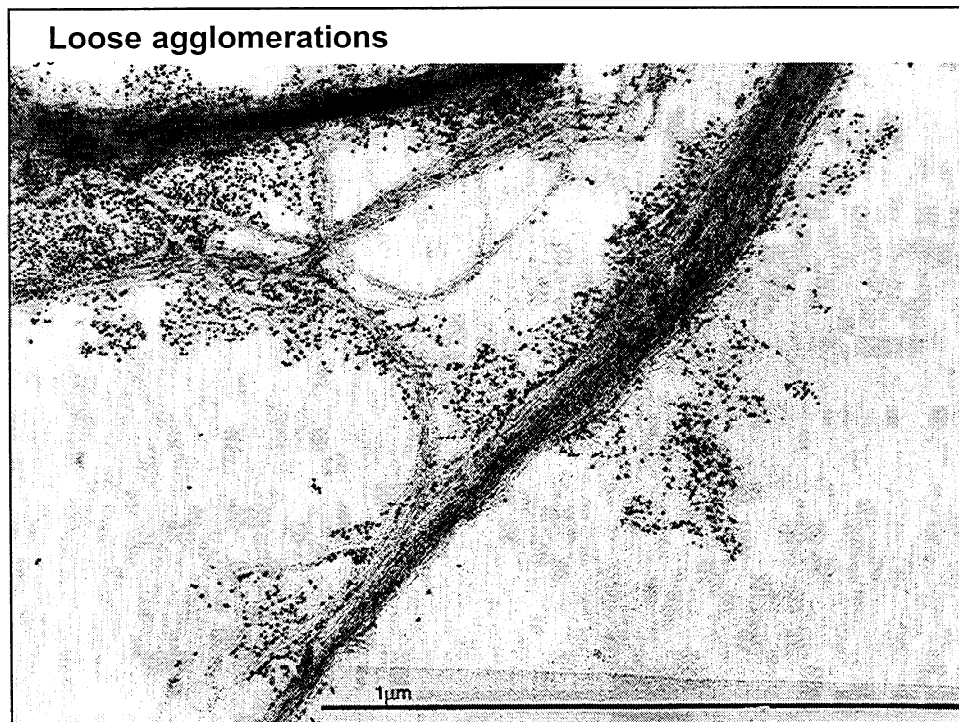
### ***Characteristics of Cationic Corn Starch Adsorption***

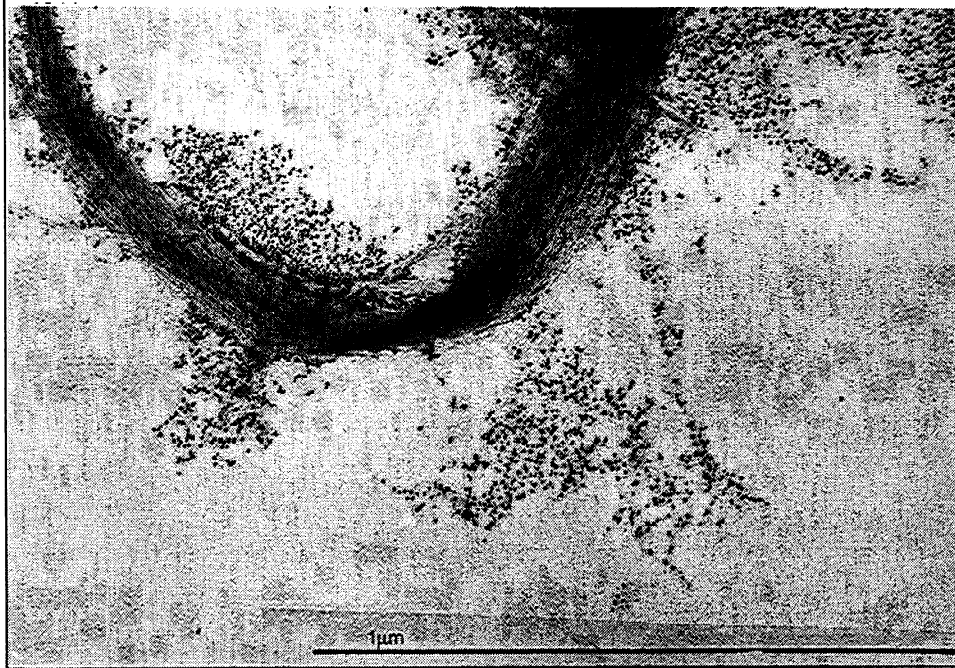
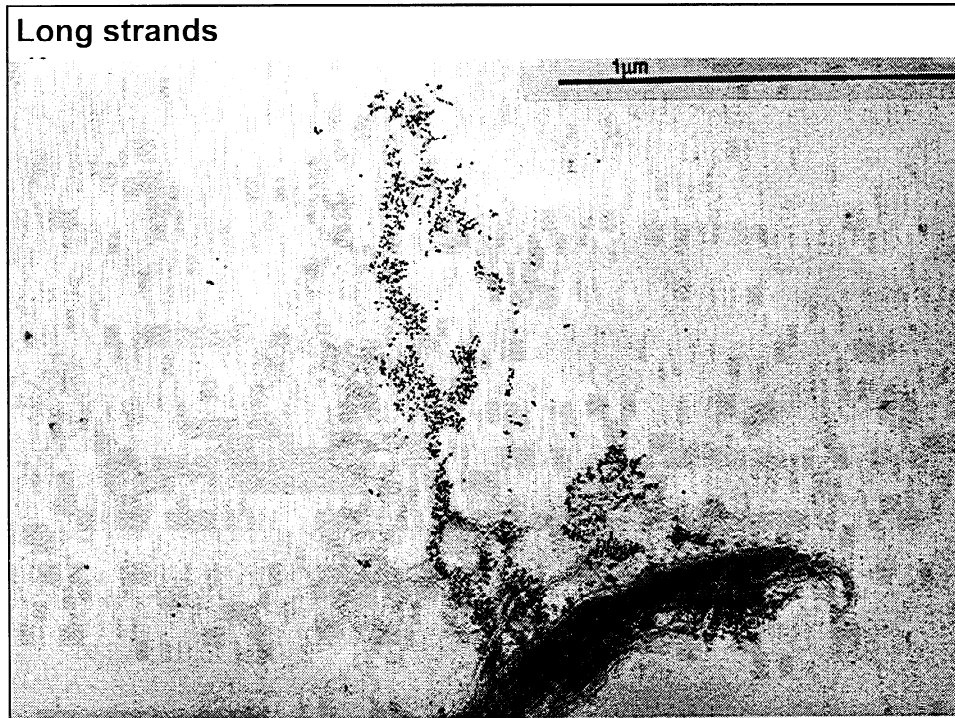
*Dosage: 1.0%*

*Charge density: 0.35% N*

*Mixing: level 3 for 1 minute*

- **Starch is adsorbed in a loosely agglomerated form**
- **Formation of short strands**



**Loose agglomerations and short strands****Long strands**

### ***Characteristics of Cationic Potato Starch Adsorption***

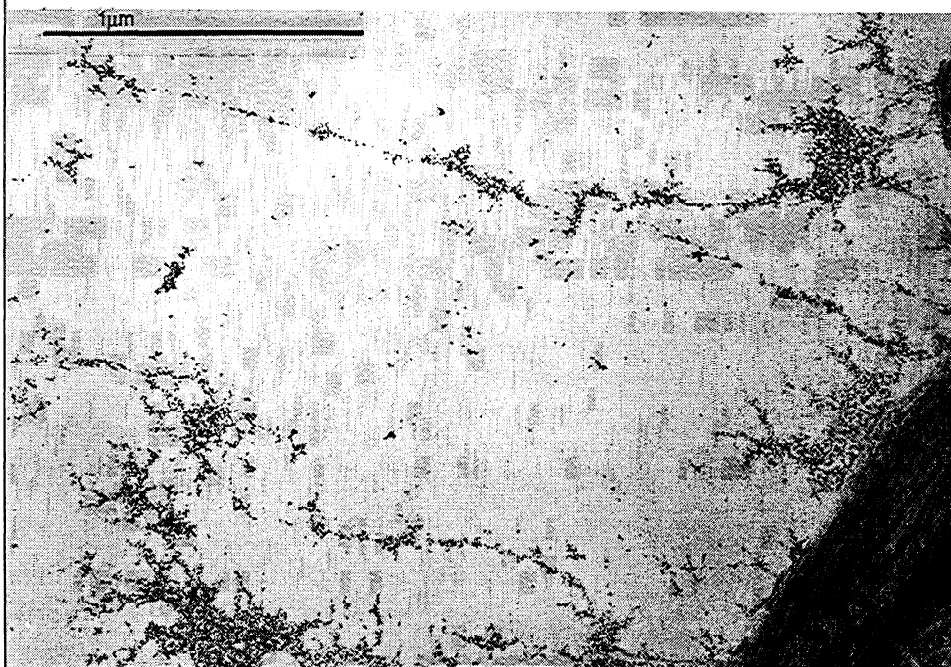
*Dosage: 1.0%*

*Charge density: 0.35% N*

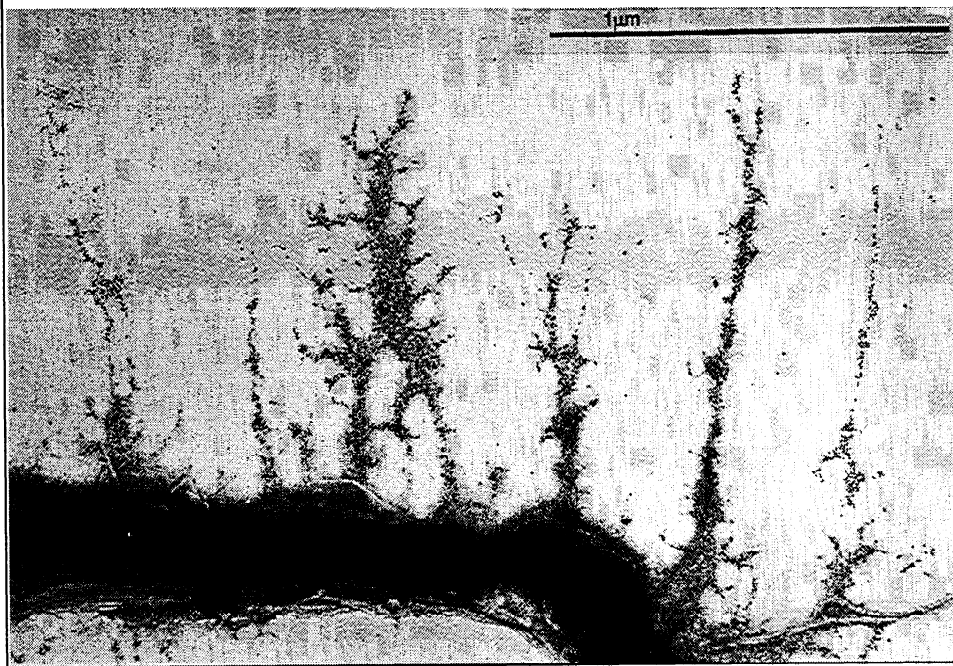
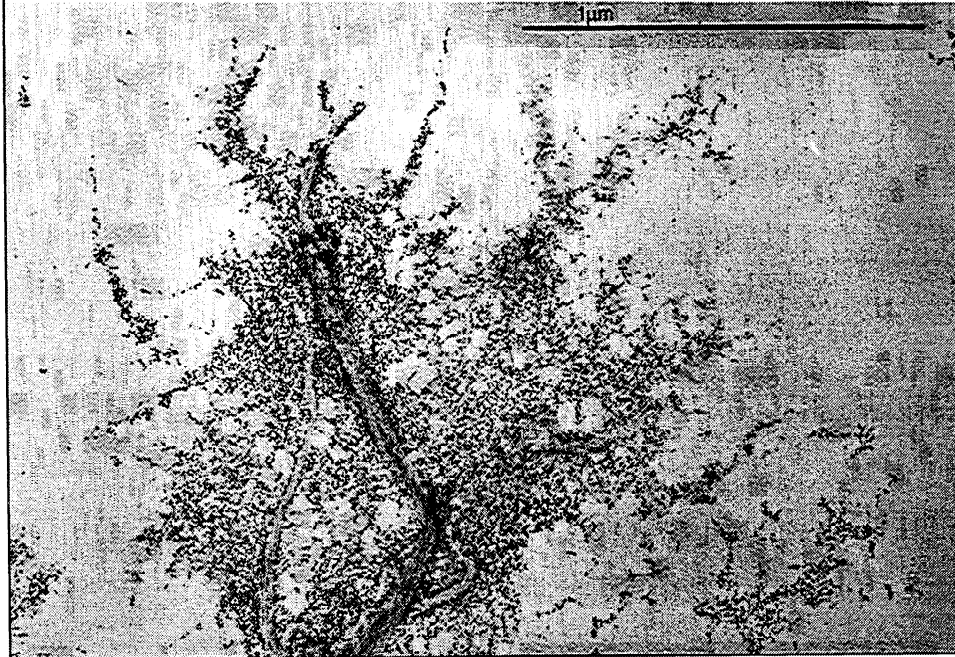
*Mixing: level 3 for 1 minute*

- **Frequent formation of long strands (relative to corn starch).**
- **Adsorption of larger agglomerations than with cationic corn starch.**

**Long strands**



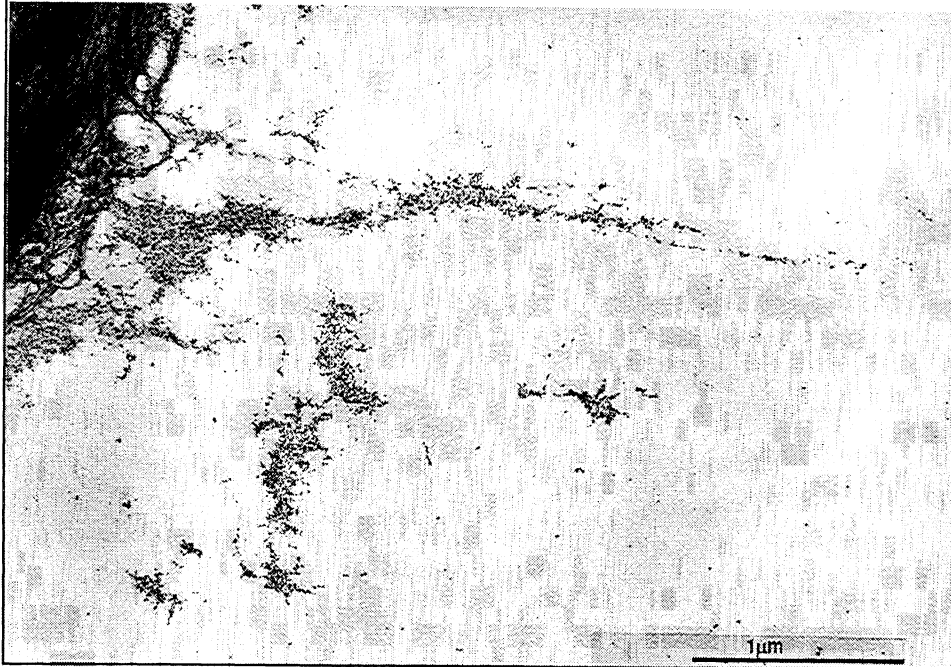


**Strands****Large agglomeration**

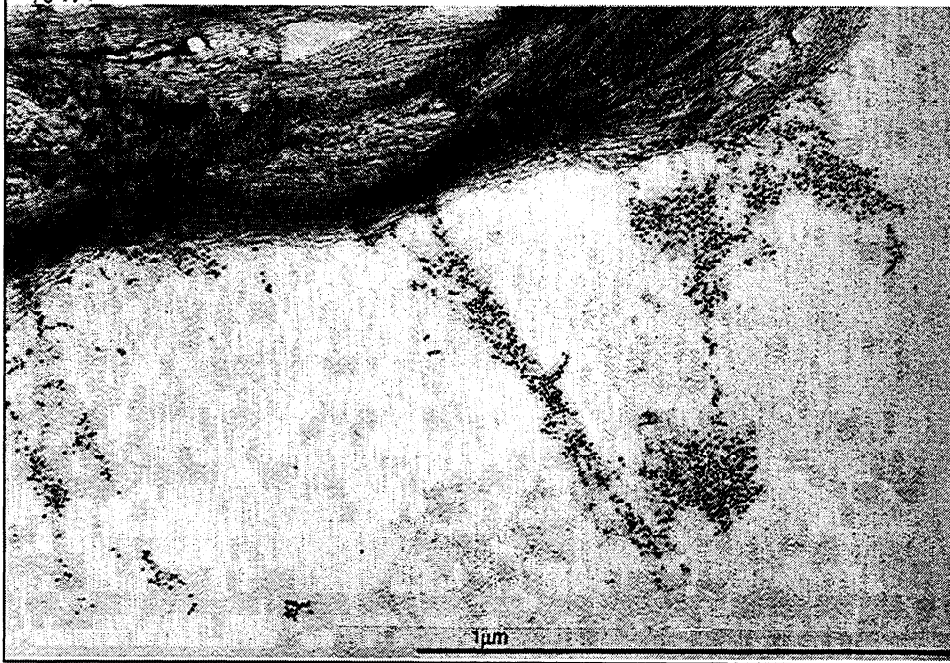
### ***Effect of Mixing Conditions (Potato)***

- **Low shear / short time:**  
long strands  
loose agglomerations
- **Low shear / long time:**  
shorter, fewer strands  
loose agglomerations
- **High shear / long time:**  
shorter, fewer strands  
dense agglomerations

**Mixing: scale 3 for 1 min**



**Mixing: scale 3 for 20 min**



**Mixing: scale 4 for 20 min**



## ***Effect of Dosage (Potato)***

*Dosage: 0.5, 1.0, 1.5 %*

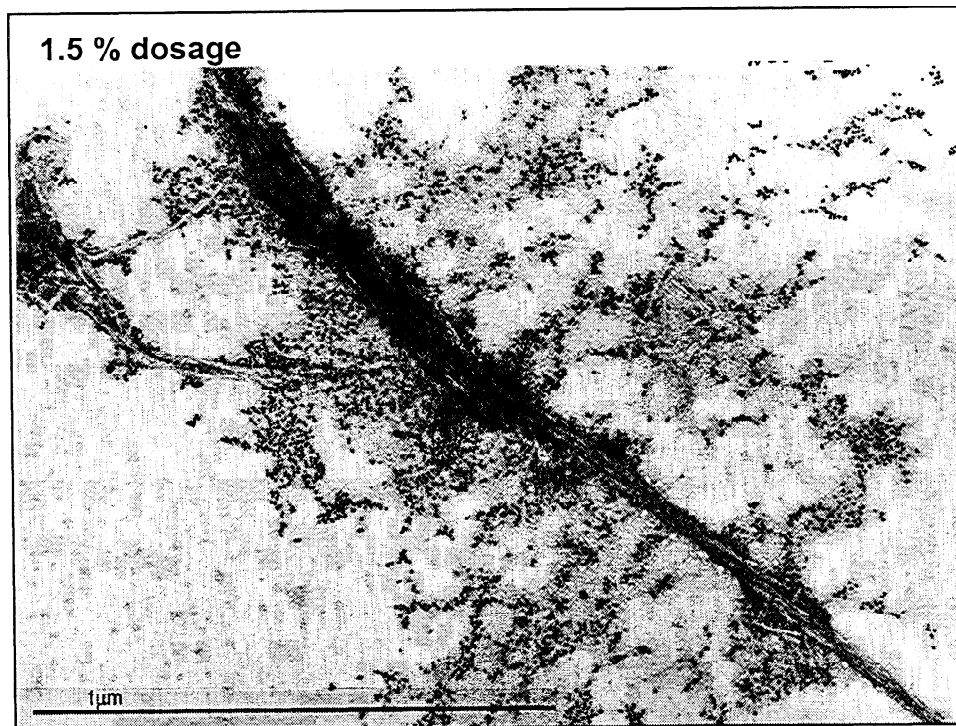
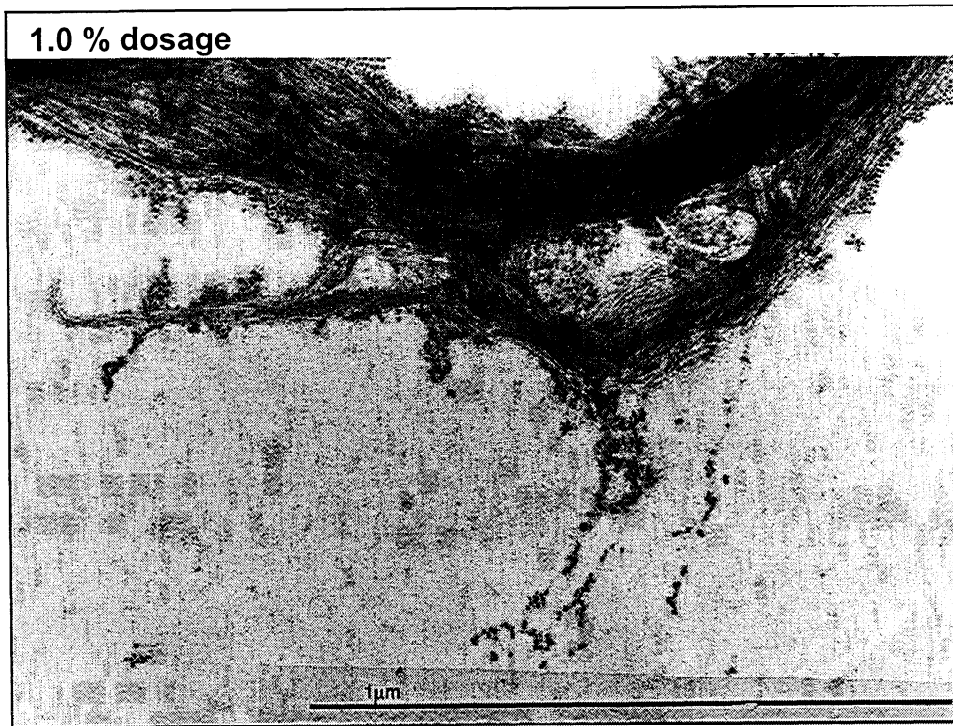
*Charge density: 0.35%N*

*Mixing: level 4 for 20 min*

- **0.5% Dosage:**
  - Short strand formation
  - Small agglomerations on the fibrils
- **1.0% Dosage:**
  - Long strand formation
  - Higher coverage by small starch agglomerations
- **1.5% Dosage:**
  - Increased frequency of long strand formation
  - Higher coverage by large agglomerations

**0.5 % dosage**





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### ***Effect of Charge Density (Potato)***

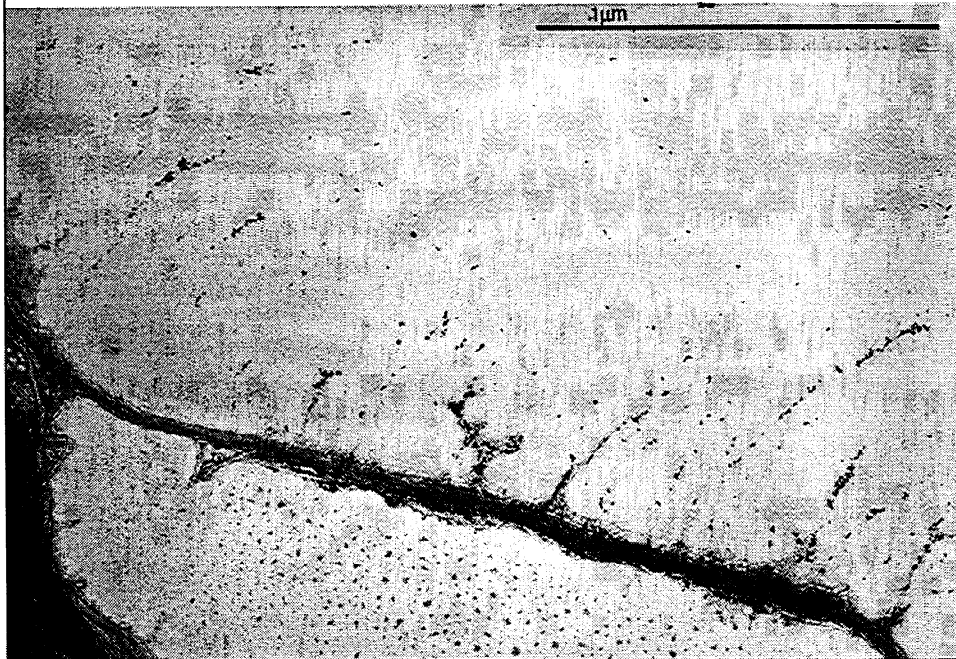
Dosage: 1.0 %

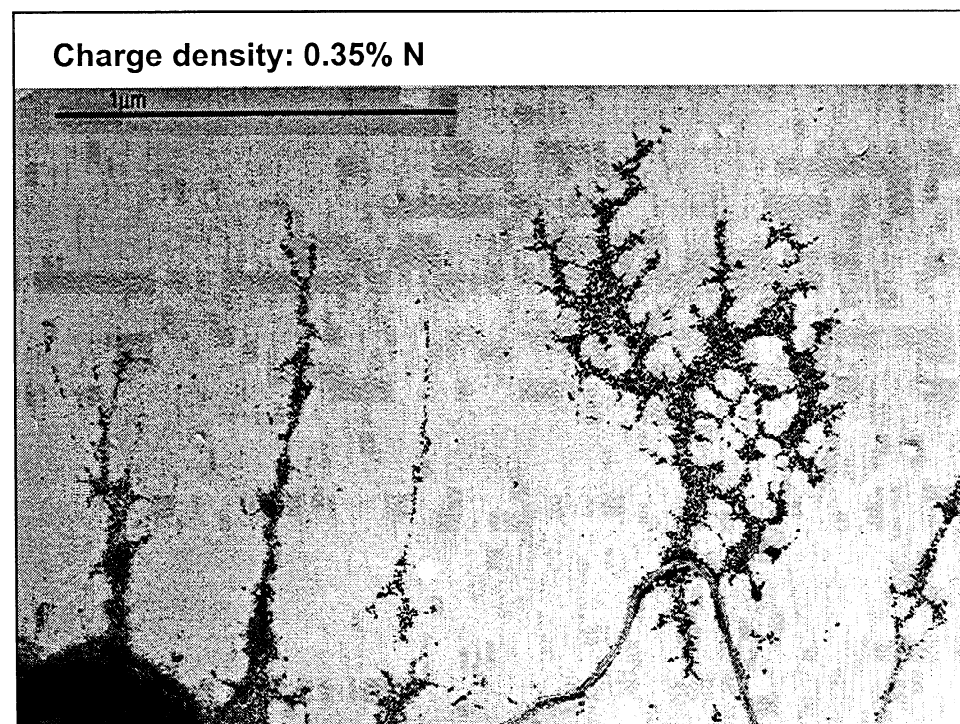
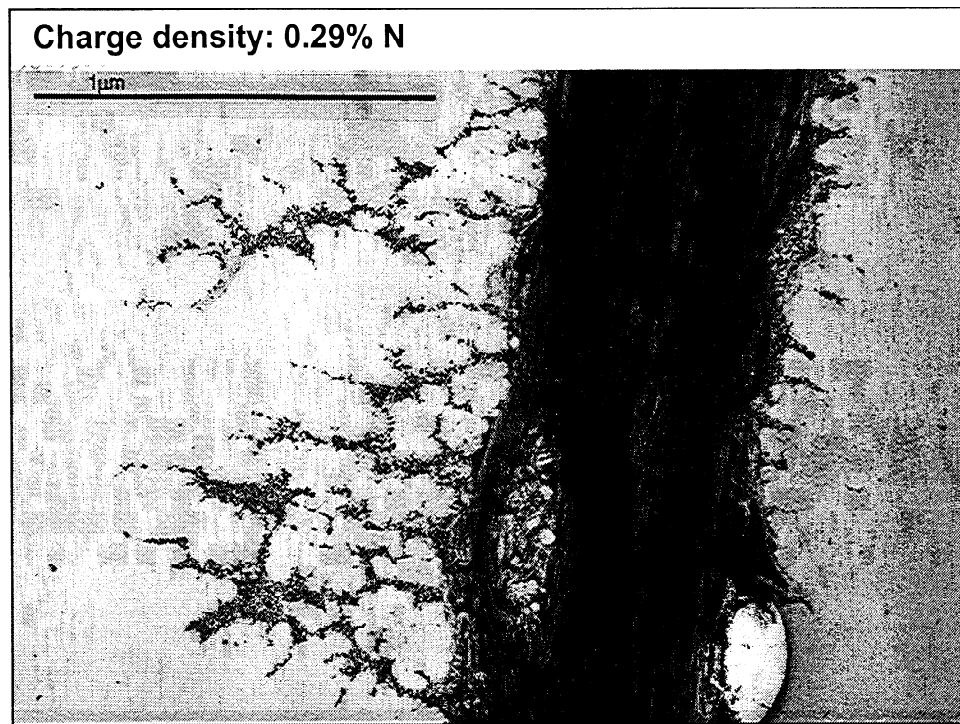
Charge density: 0.15, 0.29, 0.35%N

Mixing: level 4 for 20 min

- Strands become more agglomerated with increasing charge density
- Greater starch adsorption occurs at higher charge density

Charge density: 0.15% N





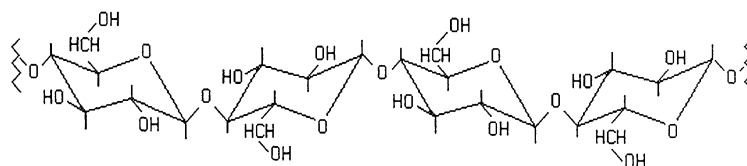
## ***Conclusions***

- **Cationic starch is adsorbed in an agglomerated form.**
- **Agglomerated strands are non-linear and non-uniform.**
- **Increasing dosage increases the size of agglomeration.**
- **Increasing shear densifies the starch agglomerations.**
- **Potato starch is more readily adsorbed, forms longer strands and larger agglomerations than corn starch.**

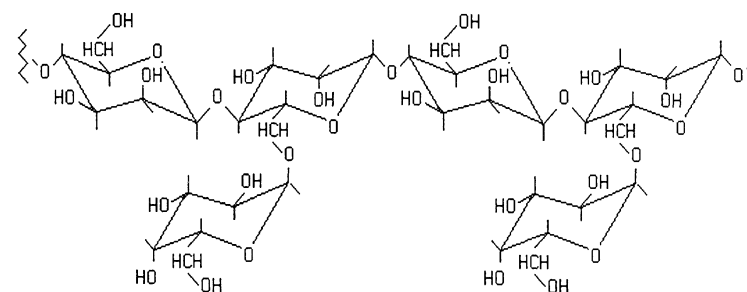


### ***Guar Gum***

CELLULOSE



GUAR



### ***Cationic Guar Gum Adsorption Behavior***

- **Mixing time**
- **Increasing shear**
- **Increasing dosage**

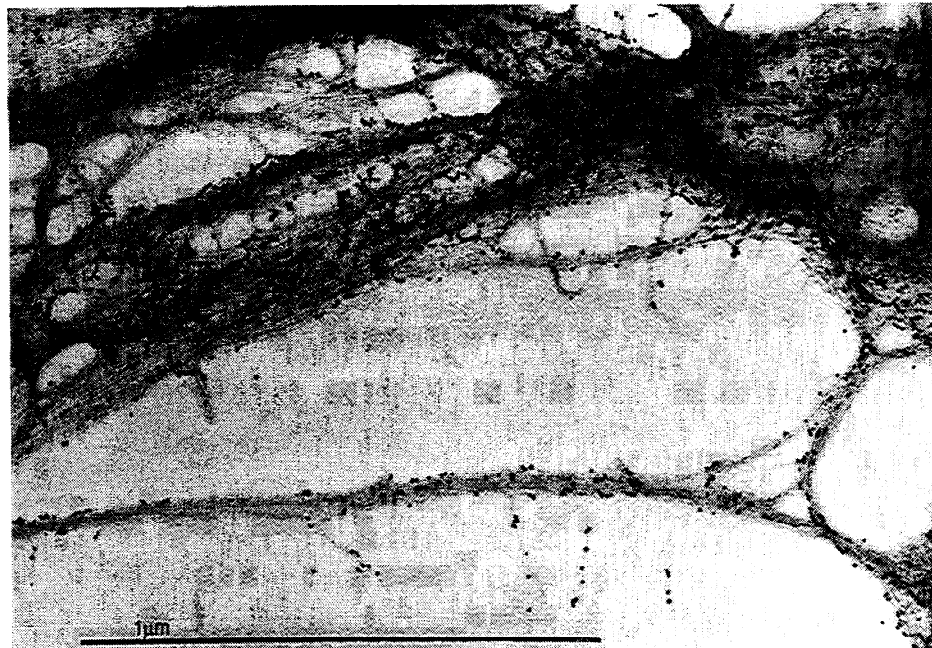
### ***Effect of Mixing Time***

***Dosage: 0.15% (3 #/T)***

***Propeller stirrer: 500 rpm***

- **0.5 minute**
- **1 minute**
- **2 minutes**
  
- **Surface adsorption of guar increases with mixing time**
- **Strands become longer and thicker**

**0.5 Minute**



**1 Minute****2 Minutes**

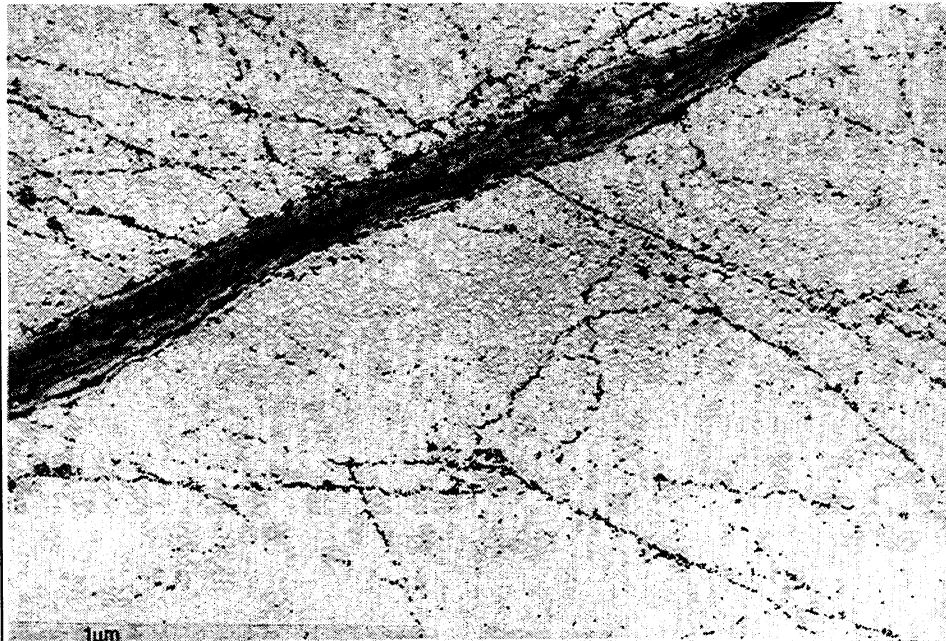
### ***Effect of Increasing Shear***

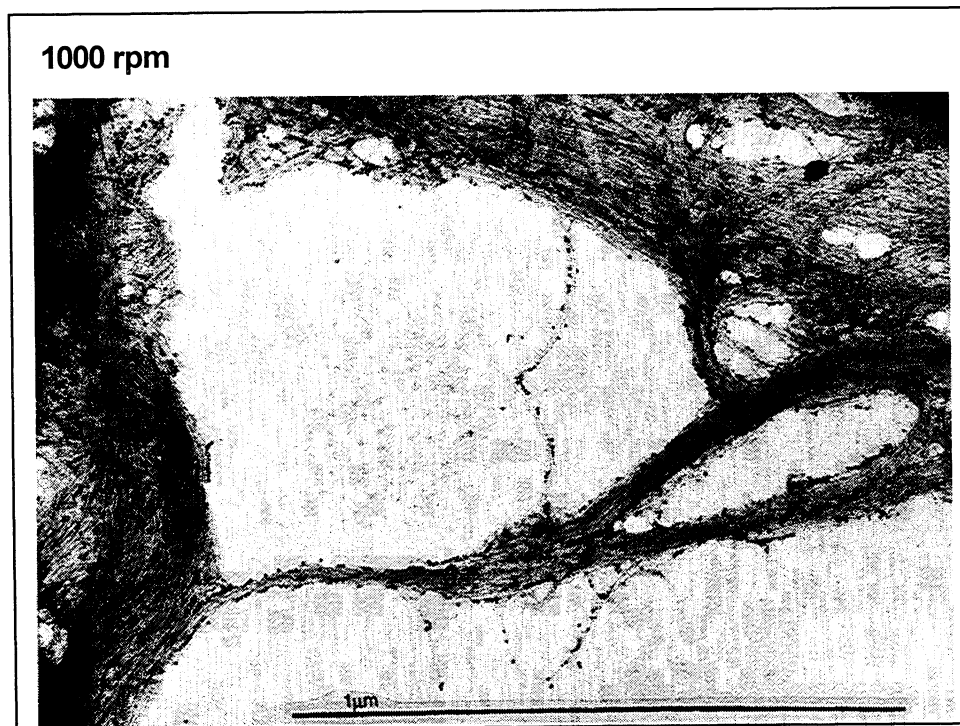
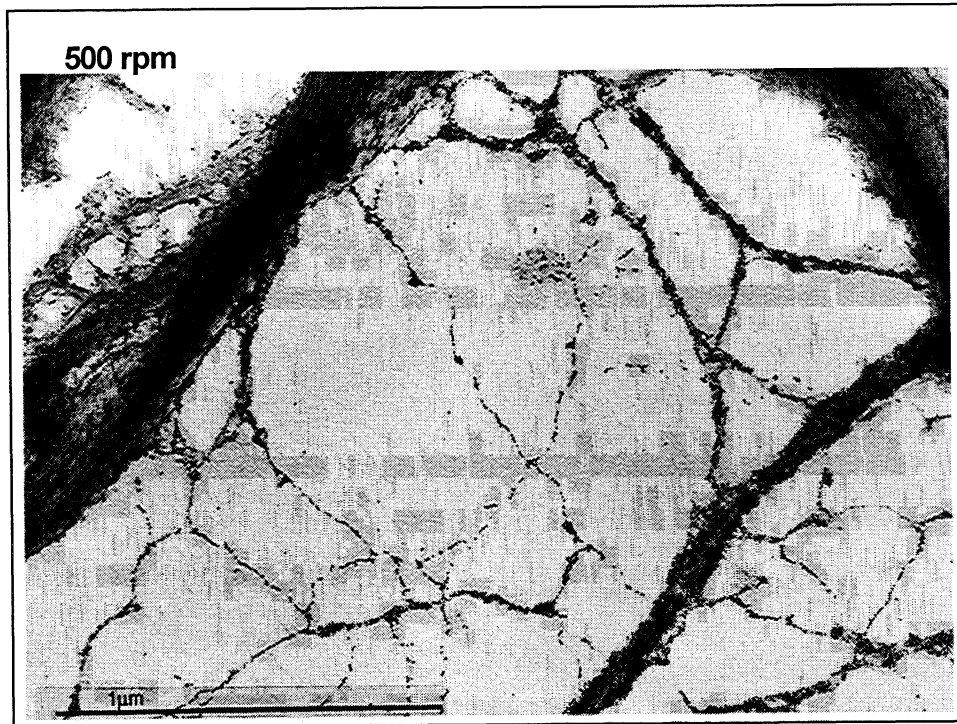
***Dosage: 0.15% (3 #/T)***

***Mixing Time: 2 minutes***

- 200 rpm
- 500 rpm
- 1000 rpm
- From 200 to 500 rpm, guar adsorption increases and strands become thicker and less linear.
- Guar strands are rarely found after exposure to extreme shear

**200 rpm**





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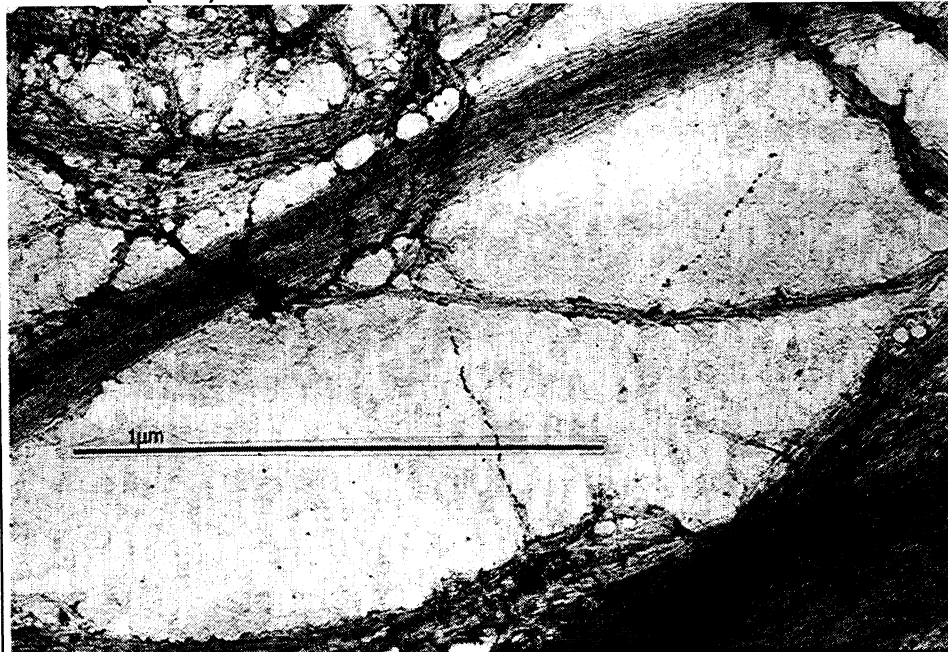
### ***Effect of Increasing Dosage***

***Propeller Speed: 500 rpm***

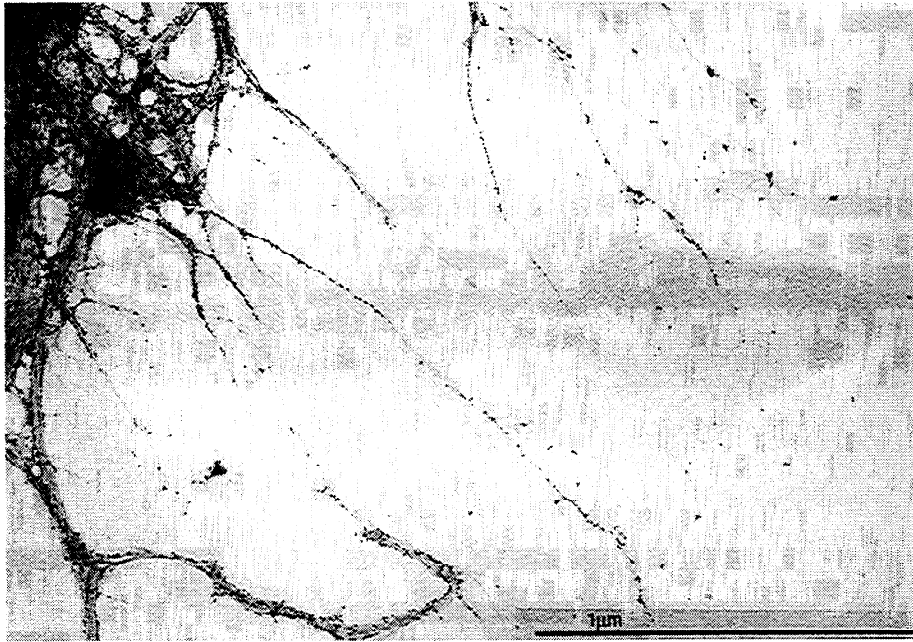
***Mixing Time: 1 minute***

- 0.05 % (1 #/T)      ▪ 0.25 % (5 #/T)
- 0.15 % (3 #/T)      ▪ 0.50 % (10 #/T)
  
- Guar adsorption increases with the dosage
- Strands become both longer and thicker
- Agglomerated guar becomes more prevalent
- At high doses, guar strands tend to form a network

**0.05 % (1 #/T)**



**0.15 % (3 #T)**

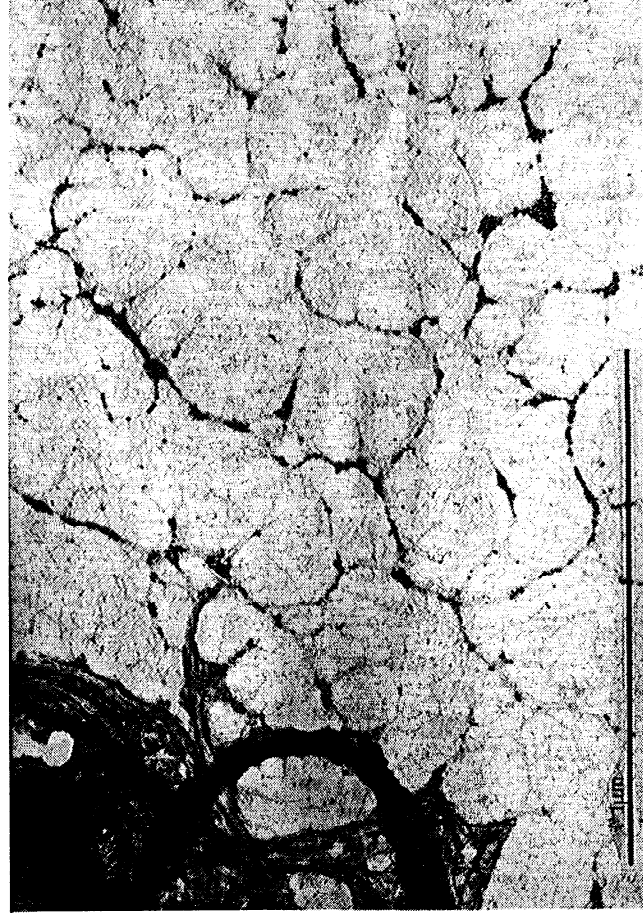




0.5 % (10 #Π)



0.5 % (10 #Π)





## ***Conclusions***

- **Cationic guar gum forms thin, linear, extended strands at low dosage.**
- **Strands thicken as the dosage increases.**
- **The strands form networks at high concentrations.**

## ***Ongoing Study***

- **Thick Stock Guar Addition Followed by Dilution**
  - **Mixing Time**
  - **Dosage**
- **Papermaking and Strength Testing**
- **Other Guar Derivatives**

## Fundamentals of Interfiber Bonding

### *General Conclusions*

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- The staining method developed for visualizing cationic polyacrylamide works well for cationic starches and guar.
- The influence of several conditions on the adsorption behavior of these bonding agents can be analyzed using this method.

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## Overall Project Name

### *Schedule*

---

Goals	Apr	May	Jun	Jul	Aug	Sep
Visualize Starch	----	-----	---x			
Visualize Gums	----x					
Bond Failure				-----	-----	-----

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LIQUID SUBSTRATE INTERACTIONS

STATUS REPORT

FOR

PROJECT F044

Wayne Robbins  
Tabitha Horton

Institute of Paper Science and Technology  
500 10<sup>th</sup> Street, N. W.  
Atlanta, Georgia 30318

## Liquid/Substrate Interactions Spring PAC 2000

Principle Investigator: Wayne Robbins

Staff: Tabitha Horton

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## Relevant Research Line

**Improve the ratio of product performance to cost for pulp and paper products 25% by developing:**

- + models, algorithms, and functional samples of fibrous structures and coatings which describe and demonstrate improved convertibility and end-use performance**
- + break-through papermaking and coating processes which can produce the innovative webs with greater uniformity than that achieved by current processes.**

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## Relevant Research Line, cont.

Develop and implement relationships between materials & manufacturing variables AND paper structure, properties, & uniformity

Develop and implement relationships between paper structure, properties, & uniformity AND end use performance and convertibility

Improved papermaking processes

Improved converting processes

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## Deliverables

Model(s) describing how substrate properties affect interactions of liquid. (June 2001)

Including: inks (initial emphasis), size press liquids, coatings, among others (later).

Measurement techniques, direct and traceable, that can be used over more than one grade range. (June 2000)

Liquid application techniques that simulate full scale processes. (June 2000)

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## Additional Support

There is presently no support beyond the PAC funding for this project.

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## Background

The project was staffed and work was initiated by 30 September 1999.

### **GOALS FOR FY 99-00:**

- 1) Complete a baseline literature review
- 2) Complete selection of substrate characterization methods.

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**FY 99-00 GOALS, cont.**

- 3) Complete selection of liquid application techniques.
- 4) Evaluate the JMP Statistical Discovery program as a tool to evaluate major effects.

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## Progress Against Goals

**Goal # 1:** Complete baseline literature survey.

**Status:** Complete

**Significant Findings:**

- 1) No shortage of relevant theory!
- 2) Application of “theory to practice” is limited by one variable experiments or poor multivariable experimental design.

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## Progress Against Goals, cont.

**Goal # 2:** Complete selection of substrate characterization methods.

**Status:** In Progress

**Significant Findings:**

- 1) Both profilometric and interferometric methods of roughness determination are traceable, but the profilometric method is more representative of the bulk properties of the sheet

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## Significant Findings, cont.

- 2) The profilometric method appears sensitive over several grade ranges.
- 3) Surface roughness and contact angle, water appear confounded and sensitive to moisture content over a range of grades.

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## Work In Progress

- 1) Design and conduct an experiment to understand contact angle and roughness independently.
- 2) Establish the relationship between Apparent Density pore size distribution measurements.
- 3) Establish traceability for contact angle, water and single fiber contact angle measurements.

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## Progress Against Goals, cont.

**Goal # 3:** Complete selection of liquid application techniques.

**Status:** In Progress

**Significant Findings:** None to date.

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## Work In Progress

A screening experiment was performed where substrate properties and liquid viscosity were varied.

Vanadium salted liquids were applied using gravure and Bristow Wheel liquid application techniques.

Analyses are in progress to establish the depth of penetration, and ultimately the substrate properties dominating liquid penetration.

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## Progress Against Goals, cont.

**Goal # 4:** Evaluate the JMP Statistical Discovery program as a tool to identify major effects.

**Status:** Complete

**Significant Findings:**

1) The program was used successfully to evaluate a data set involving the printability of linerboard, and resulted in identification of major and minor effects.

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## FY 00-01 Goals

Perform designed experiments using the independent variables:

1) Substrate

pore structure  
roughness  
surface energy

2) Liquid

viscosity  
surface tension  
volume

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3) Liquid Application  
pressure

In an effort to achieve early deliverables, substrate type, liquid volume, viscosity and pressure applications will be limited to uncoated paper and board printing applications.

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## Issues

- 1) Collection of roll quantity substrates that represent a range of grades that are printed using flexographic, offset and gravure methods.



