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WINTER 1997

Measuring fiber orientation anisotropy in paper using infrared ellipsometry

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PROBING PAPER SURFACES

Practically speaking, paper is a two-dimensional network of cellulose fibers. The orientation distribution of these fibers controls the mechanical properties of a sheet. Both the extent of fiber orientation anisotropy and the preferential fiber direction angle are important parameters. Numerous techniques have been proposed and used to determine these parameters. Some of the more common are: Image analysis, ultrasound propagation, zero span tensile strength ratio, X-ray diffraction, light diffraction and light diffusion.

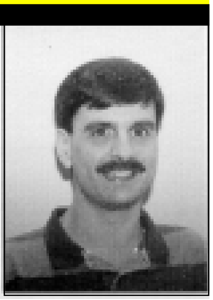
Only the ultrasound propagation and light diffusion techniques have led to practical commercial laboratory devices. However, these techniques show poor spatial resolution and only provide information on the average fiber orientation averaged through the sheet. Most papers show considerable differences from top side to bottom side and this two-sidedness is the source of many dimensional stability problems.

The photoelastic modulator (PEM) is the basis of a new technique which addresses this problem. The instrument quickly measures the principal direction and strength of the infrared birefringence, properties closely correlated with the elastic stiffness of the sheet, which is in turn controlled by the fiber orientation distribution. These readings are basically equivalent to ultrasonic-type measurements with the added advantages of non-contact, two-sidedness, high speed, and high spatial resolution. Surface roughness, which is often a problem in ellipsometric work, is used to advantage: The surface of the sample need not be precisely aligned since the light diffusion cone is fairly broad.

MEASURING THE BIREFRINGENCE OF PAPER

Paper is strongly absorbing in the infrared spectral region with an absorption band centered on 10 μm, a region easily accessible with the CO_2 laser. This absorption is caused by helical cellulosic fibrils that are wound along the fiber axis. As a result, non-uniform fiber orientation and strain distributions will cause a large birefringence: the intensity of reflected light changes as the polarization direction of the incident light beam rotates. Because the penetration depth is small, only a layer approximately 20 μm thick is probed and the two sides of a sample can be measured independently. However, because the surface of paper is very rough, the birefringence cannot be obtained from simple reflectivity measurements of the type used in conventional ellipsometry (e.g. rotating polarizer): Changes in intensity and polarization of the reflected light are dominated by surface scattering and, consequently, the underlying birefringence is not easily observed.

ABOUT THE SCIENTISTS

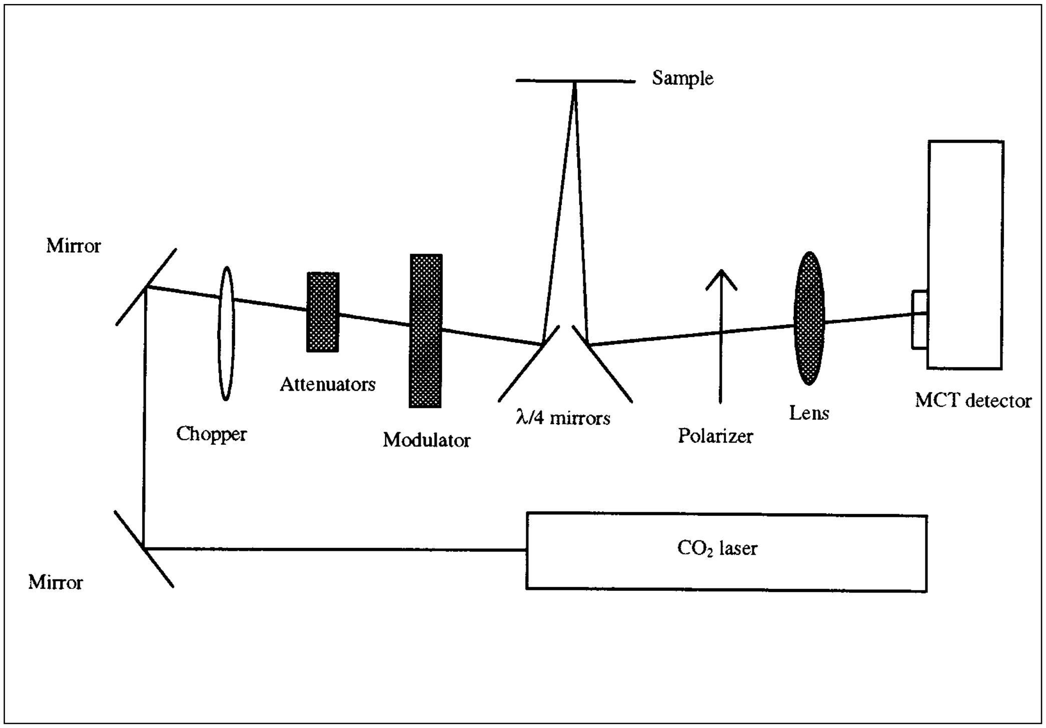


Originally from the Ottowa area, **Pierre Bernard** (left) chose Laval University in Quebec for its strong program in lasers and photonics, as well as its proximity to nearby ski centers. He received his Ph.D. in laser spectroscopy from Laval's Physics Department in 1982. He worked on the Tokomak fusion reactor project at Hydro-Quebec's research institute in Varennes, and then joined the University of Ottawa's Physics Department where he worked in semiconductor laser spectroscopy and the growth and characterization of ternary crystals.

Dr. Bernard began working at the National Optics Institute (NOI) in 1988. NOI is a nonprofit private corporation set up by the provincial and Canadian federal governments to promote the use of optics and lasers in industry. "We're only partially funded by the government," Bernard explains. "Year after year a larger portion of our operating budget must come from external revenue like sales of components or equipment or R&D contracts."

(Continued on page 6)

Figure 1. Optical configuration of the surface anisotropy measuring instrument.



These surface roughness effects also plague phase-modulated ellipsometry. But, when the PEM is employed, a simple calibration procedure can be used which allows us to work with highly diffusing surfaces such as paper and even roughly sawed wood. Figure 1 shows the basic elements of the instrument.

The technique is similar to the approach used by Shirley Johnson [1] to measure simultaneously the birefringence magnitude and direction of sheared colloidal suspensions. An important difference is the use of the near-normal reflected light instead of the transmitted light. Other differences include the use of 45-degree quarter-wave mirrors and special mounts that allow computer-controlled precise adjustment of the first quarter-wave mirror, the polarizer, and focusing lens.

The setup is described as follows. The 3 Watt linearly polarized laser beam goes through a variable attenuator to reduce the power incident on the sample to an acceptable level. The amplitude of the beam is modulated at low frequency (400 Hz) with a mechanical chopper, while its state of polarization is modulated at 50 kHz (ω) with the PEM which has its axis at 45 degrees with respect to the incident beam polarization. The beam is then incident on a quarter wave mirror and directed to the sample. The energy reflected by the sample is redirected by a second quarter-wave mirror, goes through an analyzer oriented at 45 degrees and is focused onto a HgCdTe photodetector. The configuration is such that the angles of incidence on

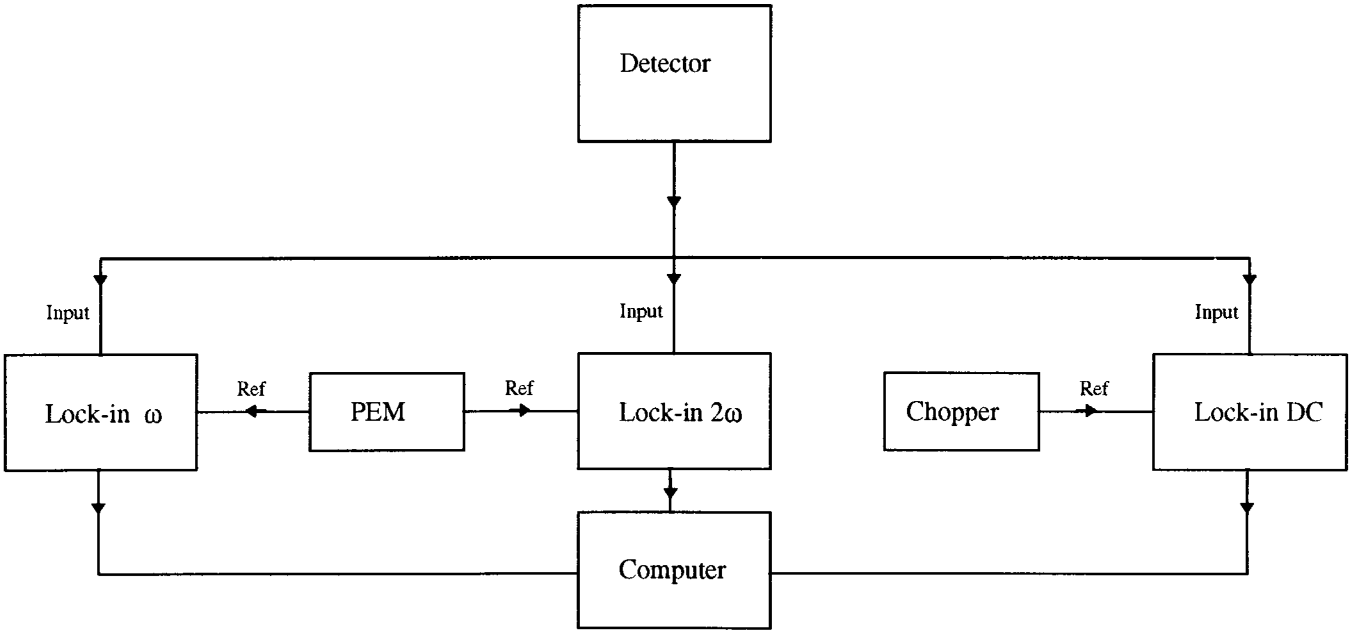


Figure 2. Electronic block diagram for signal detection.

the quarter wave mirrors are kept close to 45 degrees, while the angle of incidence on the sample is about 4 degrees from normal. The signal from the detector is Fourier analyzed to give the birefringence magnitude and the orientation angle. The Fourier analysis is performed with lock-in amplifiers at ω and 2ω. Another lock-in operating at 400 Hz, called the "DC" lock-in, is used to measure the total intensity reflected by the sample.

Figure 2 shows the electronic block diagram. The signal measured can be divided into three parts I_{DC}, I_{ω} and $I_{2\omega}$. These terms are related to the birefringence magnitude A and orientation angle Θ by the following equations:

$$I_{DC} = r(1+ A^2 - A\cos(\Theta)J_0(\phi)),$$
$$I_{\omega} = 2Ar\sin(\Theta)\, J_1(\phi) + s_{\omega},$$
$$I_{2\omega} = -2Ar\cos(\Theta)\, J_2(\phi)+ s_{2\omega},$$

where ϕ = peak retardation amplitude of the PEM; J_0, J_1 and J_2 are the Bessel functions; A = the magnitude of the birefringence; Θ = the orientation angle of the birefringence; and, s_{ω} and $s_{2\omega}$ are unwanted signal contributions due to surface scattering effects. The retardation amplitude produced by the modulator is set at 2.405 radians, where $J_0 = 0$.

Paper is a relatively rough surface at the operating wavelength and, as mentioned above, this causes a problem for conventional ellipsometry. On the other hand, with the above setup, surface scattering causes additive signals at ω and 2ω that can be compensated with small angular adjustments of the first quarter-wave mirror and polarizer. The result is a relatively simple calibration procedure that is repeated only when the roughness of the surface being studied is changed. A small calibration sample is placed on a high-speed rotating platform. This simulates an isotropic sample and the quarter wave mirror and polarizer are adjusted to bring the ω and 2ω signals to zero. This straightforward calibration procedure is very effective even for rough wood surfaces. Once the instrument is calibrated, the three signals (I_{DC}, I_{ω}, $I_{2\omega}$) are used to calculate the birefringence magnitude and orientation. The birefringence magnitude is a direct measure of the fiber orientation anisotropy, while the orientation gives the preferential fiber direction.

SELECTED EXAMPLES

Figure 3 shows results for a simple but convincing example. The measured infrared angle and birefringence are plotted versus the orientation of a paper sample placed on a rotating platform. The correlation is excellent and the measurement process is very rapid (3 Hz).

Figure 4 illustrates the results of a more extended set of measurements. Several samples previously tested with the ultrasonic velocity technique were measured over a large area on both sides. In order to compare results from the two techniques, a mean value for both sides of each sample must be calculated. This is because the infrared ellipsometry technique gives a surface measurement with a spatial resolution of about 5 mm, while the ultrasonic velocity gives a bulk measurement with a spatial resolution of about 75 mm. The correlation is excellent, showing that the infrared ellipsometry technique measures a parameter closely related to the strain distribution.

The advantage of the infrared ellipsometry technique over the other approaches is its capability to perform surface measurements with spatial resolutions unachievable with other techniques. That advantage is put to good use in the next example.

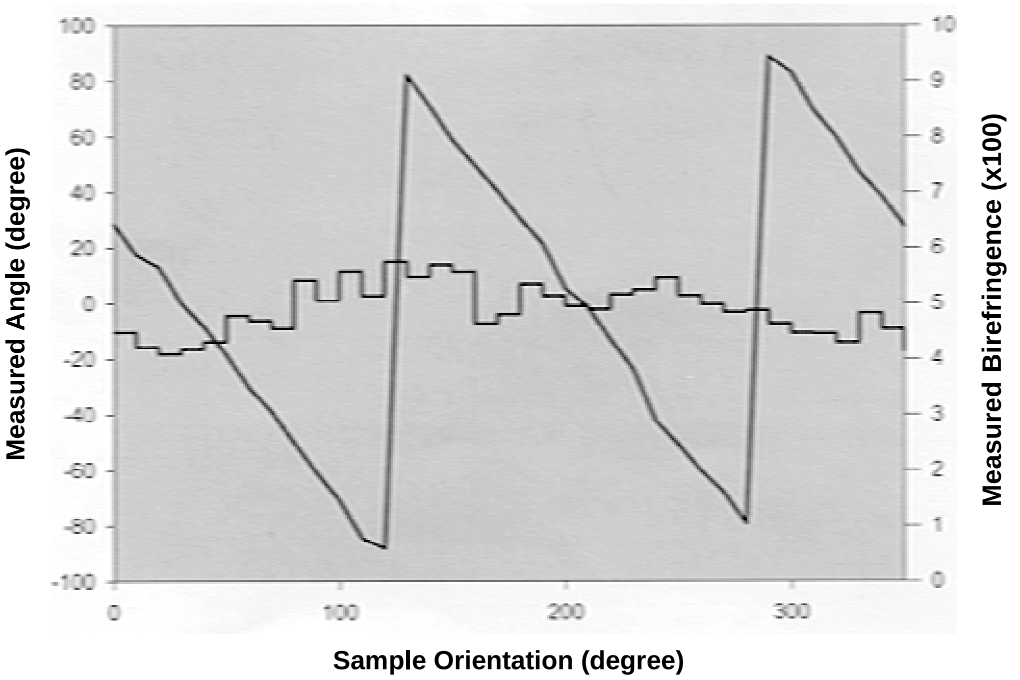


Figure 3. A simple example illustrating measurement of both birefringence magnitude and orientation axis for a rotating paper sample.

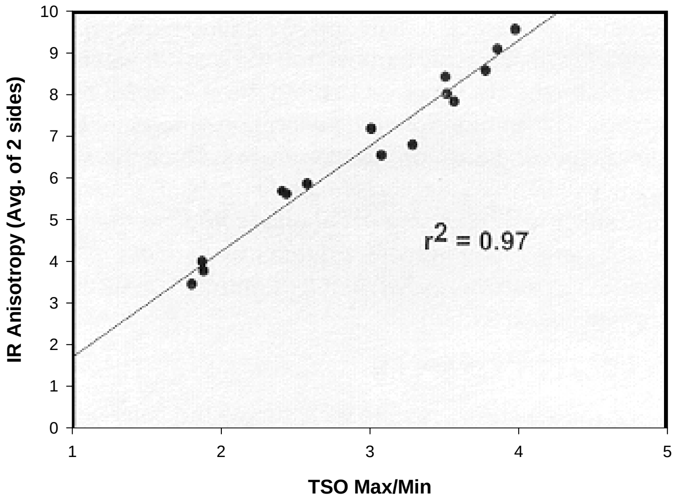


Figure 4. Infrared birefringence of paper surfaces correlates very well with ultrasonic velocity anisotropy ratio.

Figure 5 illustrates a two-dimensional mapping of a paper sample known to have small scale dimensional stability problems. A surface of 25 cm (cross direction) by 15 cm (machine direction) was measured with a spatial resolution of 5 mm. The three images on the left are for side one of the sample, while the ones on the right are for side two. The images at the top show two-dimensional mapping plots of the fiber orientation anisotropy, the center images show two-dimensional Fourier transforms and the bottom images show the power spectral densities after averaging the data along the paper machine direction. The machine direction is usually the preferential fiber direction but variations in properties are more likely to occur in the cross direction. The Fourier analysis shows periodic variations of the measured properties along the cross direction, variations associated with problems on the paper machine. It is also important to notice that these "streaks" occur mainly on the bottom side of the paper, while the top side is fairly uniform. This type of problem had been predicted but never observed [2] for lack of a suitable instrument.

Finally, Figure 6 illustrates that this technique can also be used with wood samples. It shows measurements of the infrared preferential angle and birefringence around and within a knot defect. Information on grain angles is used in modeling the impact of defects on the lumber mechanical properties. It was recently suggested that this instrument could also be used to detect the presence of compression wood and this is presently being investigated.

REFERENCES

[1] Johnson, S. J., "Simultaneous Dichroism and Birefringence Measurement of Dilute Colloidal Suspensions in Transient Shear Flow", Engineer Degree Thesis, Chemical Engineering Department, Stanford University, September 1984.

[2] Aidun, C. K., "A Fundamental Opportunity to Improve Paper Forming", TAPPI Journal, vol. 79, p.55 1996.

Figure 5. Two-dimensional mapping of both sides of a paper sample showing streaks in the birefringence for the wire side.

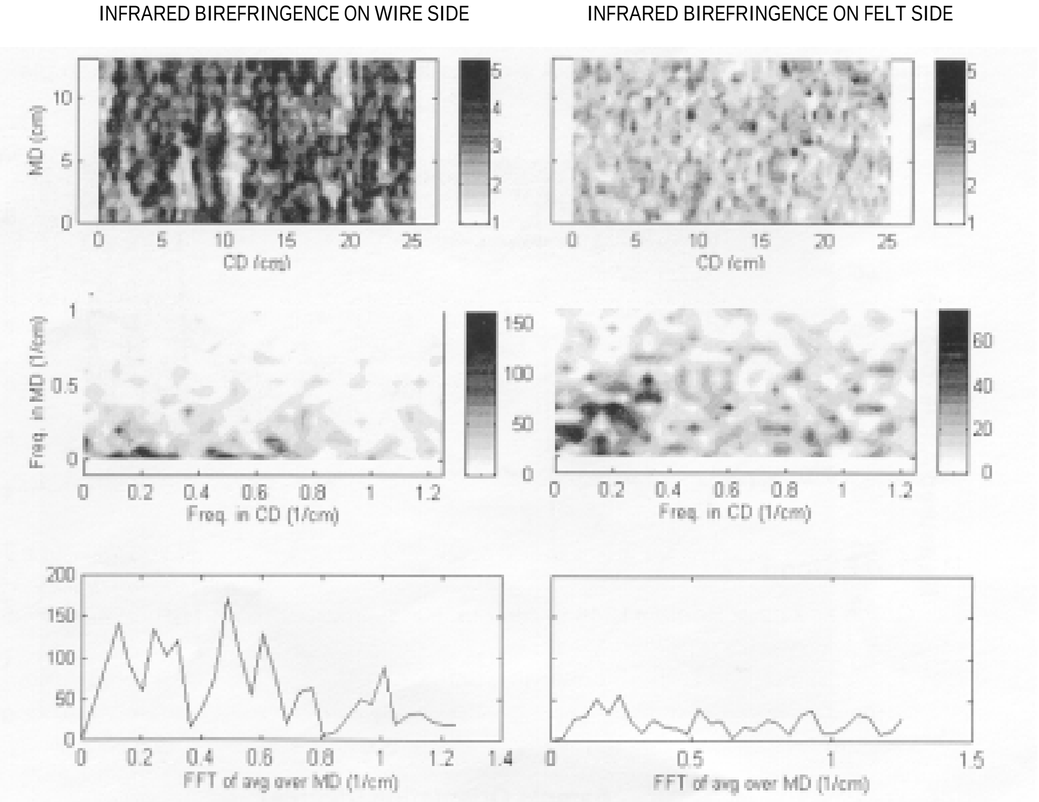
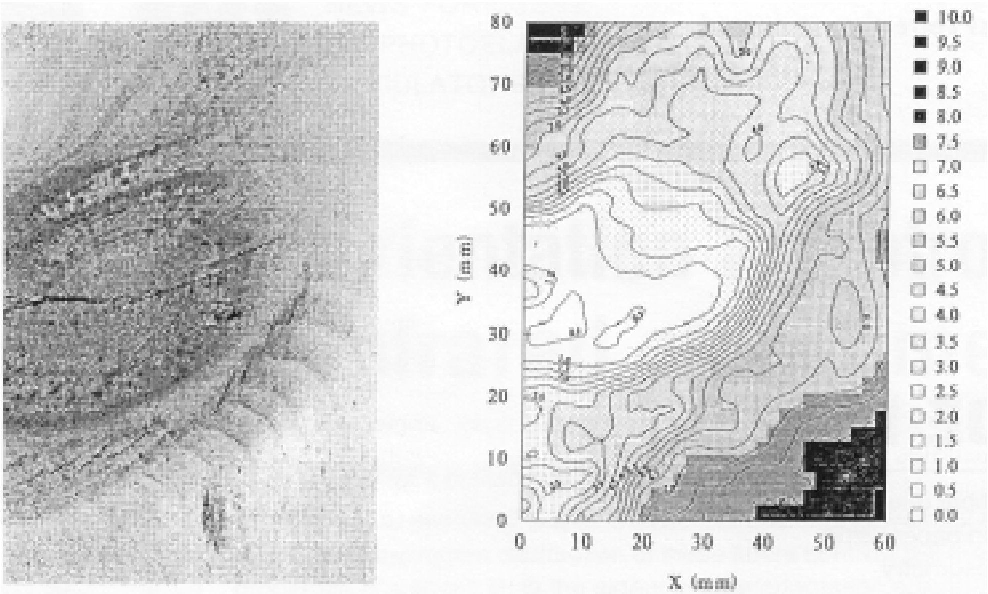
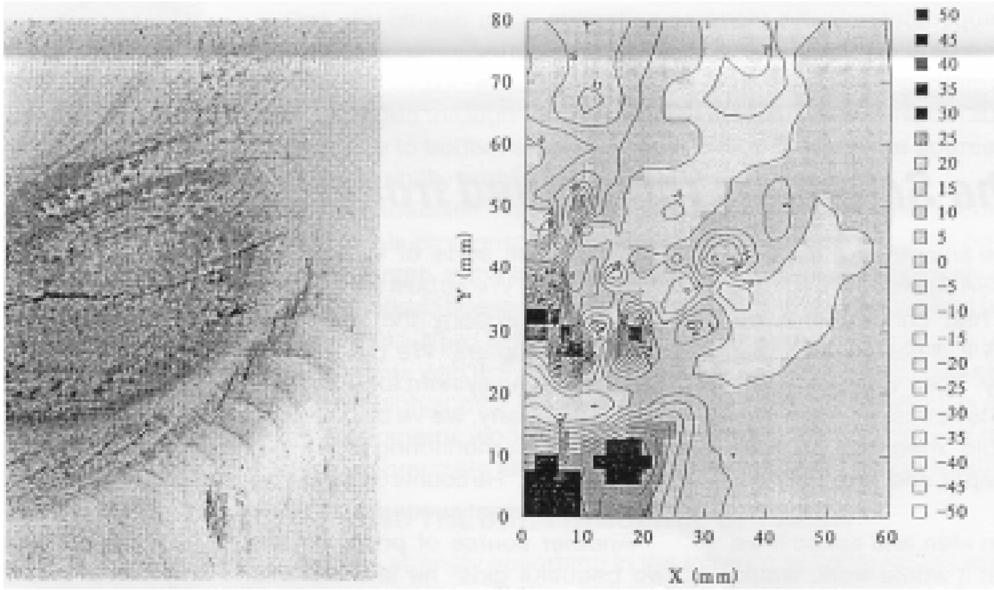


Figure 6. High resolution scan around a knot in a wood sample.

INFRARED ANISOTROPY



INFRARED ANGLE



EVENTS AND INFORMATION

PEM at Pittcon

Come see us at Booth #1846 in Atlanta. Hinds Instruments will have a booth with PEM-90 product information and customer support at the Pittsburgh Conference, March 17-20. Ted Oakberg, Bob Wang, and Peggy Smith will be on hand to answer your questions and listen to your ideas. There will also be a demonstration of the PEM-90 modulator and controller.

If you will not be attending Pittcon, but want to visit with us, we will also be at CLEO (Baltimore, May 20-22) and SPIE (San Diego, July 27-August 1).

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About the Scientists (continued from page 1)

The surface anisotropy measurement system (SAM) was just such a venture. "We had contact with researchers from a Swedish pulp and paper company. They were telling us that they would love this type of instrument that could measure surface properties of paper and fiber orientation."

"We had an idea and asked if we could show that it would work, would they buy the equipment. That's how it all started. They said 'OK' and we showed them it was possible and they bought the instrument. We retain all the rights on the technology," he adds.

As a senior researcher in the Laser Systems Technology Group at NOI, Dr. Bernard has been involved in numerous projects ranging from laser vision through turbid media to distributed fiber optics sensors and applied spectroscopy for environmental monitoring.

"We do all sorts of things," Dr. Bernard says. "We've built several systems for a company that produces fiber-optic couplers. We did a laser-based polishing system for them. For another company, we've built several instruments for monitoring for an aluminum factory." He counts SAM among his most important successes.

Another source of pride are the "two beautiful girls" he and his wife have adopted from China. "We just came back from getting our second daughter a few months ago," he says. Big sister is three and a half.

Alain Charlebois (photo on right, p. 1) earned his M.Sc. degree at the University of Ottawa in Solid State Physics and has been working at NOI since 1989. "I wanted to be an engineer," he began, "but I guess I ended up a physicist – not too far off. I was very interested in physics in high school and we had good science teachers."

A researcher in the laser applications group at NOI, Charlebois has been involved in the field of submillimeter lasers, developing lasers at very long wavelengths to measure fiber orientation, but with a different technique than SAM. Although, he says, "My main project has been the surface anisotropy measurement system."

"This instrument (SAM) is becoming more and more interesting to people," Charlebois states. "More and more people think that the problems of stability in paper are related either to the differences between the two sides or very quick variations of the property as a function of position. You need to measure at the surface to detect these problems and with a good spatial resolution," he adds.

Besides lasers and SAM, Charlebois enjoys jogging and ice hockey.