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ABSTRACT

The Lawrence Berkeley National Laboratory (LBNL) has been developing sensors for the pulp and paper industry that uses a magnetic field. The applications for magnetic sensors that have studied include 1) sensors for the measurement of the water and ice content of wood chips entering the pulping mill, 2) sensors for measuring the water content and other constituents of the black liquor leaving the paper digester, and 3) sensors for measuring paper thickness and water content as the paper is being processed. These tasks can be done using nuclear magnetic resonance (NMR). The magnetic field used for doing the NMR can come from either permanent magnets or superconducting magnets. The choice of the magnet is dependent on a number of factors, which include the size of the sample and field strength needed to do the sensing task at hand. This paper describes some superconducting magnet options that can be used in the pulp and paper industry.

INTRODUCTION

The Lawrence Berkeley National Laboratory has been involved with developing sensors for the pulp and paper industry since 1997. The paper industry uses energy and water in large quantities. The industry has been involved in the process of modernizing its facilities to improve efficiency and minimize the effect the plants have on the environment. Modern paper plants recycle water, pulp, and the chemicals used to pulp the wood entering the plant and provide the color and finish needed in modern papers. A modern paper plant can be a net energy producer. The organic material that is separated from the cellulose (lignin and other volatile chemicals) during the pulping process is used to generate heat for producing steam and electricity for use within the plant. Energy that may be left over can be sold as steam heat or as electricity to the power grid. The amount of net energy produced by the plant depends on the plant efficiency and the amount of feedstock wood that enters the pulp mill.

Modern paper plants can produce over 1500 metric tons of paper per day on a single production line. These plants use continuous pulping instead of batch pulping. This means that wood in the form of wood chips, water, and pulping chemicals enter the digester on a continuous basis. A slurry of cellulose and water (called brownstock) leaves the pulp mill to go to a bleaching plant or directly to a papermaking machine. The black liquor left over from the pulping process is separated into recycled pulping chemicals and organic fuel used to generate heat and electricity for the mill. A paper machine that generates up to 1500 metric tons of paper a day will do so in a sheet that is up to 12 meters wide. This sheet travels at process velocities up to 20 meters per second, depending on the paper basis weight (mass per unit area). A simplified paper plant schematic diagram is shown in Figure 1.

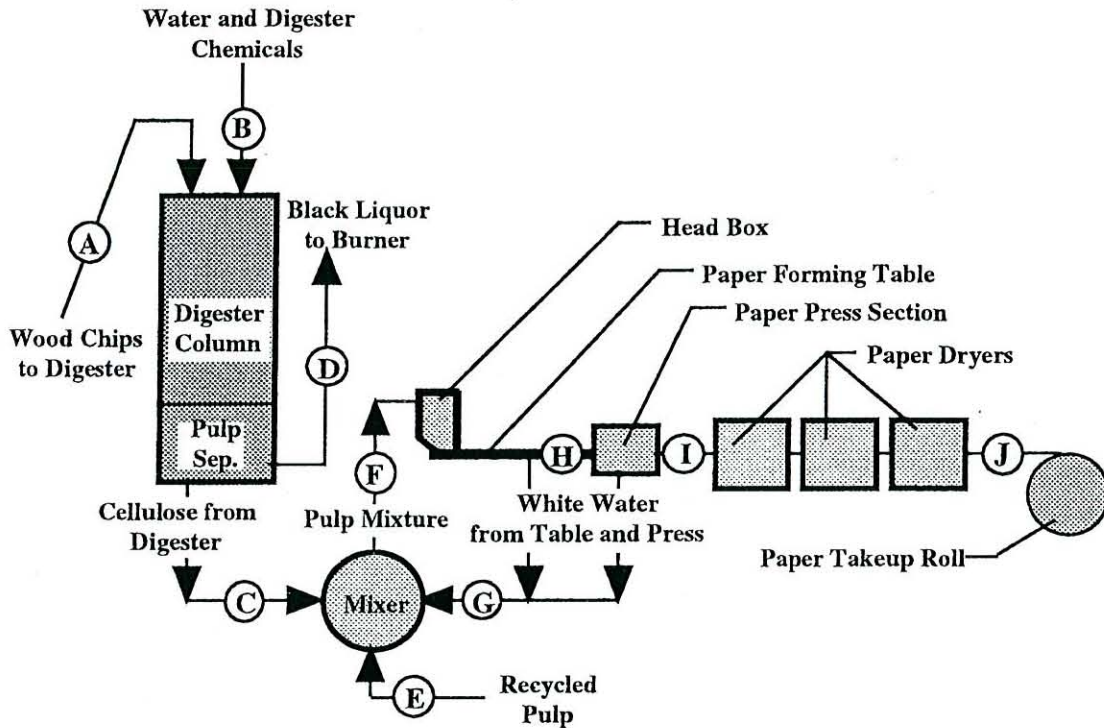


FIGURE 1. A Schematic Representation of a Single Large Paper Plant for Producing Liner Board (an unbleached paper used for cardboard). The circled letters in the figure correspond to the state points shown in Table 1 below. Stream B consists of a mixture of pulping chemicals (most sodium hydroxide and sodium sulfate) and water. Stream C is predominately water and unbleached cellulose. Stream D consists of lignin, lignocellulose, liginosulfonate, pulping chemicals and water. Streams at points F, H, I, and J are mostly cellulose and water. Other chemicals may be added at point I to increase the durability of the finished paper.

Table 1 below shows the mass flow major constituents of the flow streams at the state points shown in Figure 1 above. The final column shows the velocity of the stream at the state point shown in Figure 1 above. Pulping occurs in the digester. The pulp, which primarily cellulose and water enters the mixer where recycled pulp and water are added. The pulp stream leaves mixer and goes into the head box (at point F) as 99.5-percent water and 0.5-percent cellulose. Paper leaves the forming table (at point H) as a sheet that is 40 percent water. The pressing and drying steps result in a finished paper that is 6 to 8-percent water.

TABLE 1. Constituent Mass Flow and Product Velocity in Various Places in the Mill Shown in Figure 1 (The schematic plant shown in Figure 1 produces 1500 metric tons of unbleached liner-board per day. The mass flows shown in Table 1 represent that level of paper production.

Point	Cellulose Flow (kg s ⁻¹)	Lignin Flow (kg s ⁻¹)	Water Flow (kg s ⁻¹)	Total Flow (kg s ⁻¹)	Product V (m s ⁻¹)
A	8.0 to 16.0	2.0 to 10.7	1.1 to 8.9	11.5 to 35.6	~0.3
B	----	----	10.9 to 67.7	10.9 to 67.7	~2.0
C	8.0 to 16.0	small	8.0 to 16.0	16.0 to 32.0	~1.1
D	small	2.0 to 10.7	3.0 to 60.6	5.0 to 71.3	~2.5
E	0.0 to 8.0	small	~4.0	4.0 to 12.0	~1.0
F	16.0	small	~3178	~3194	~2.5
G	small	----	~3174	~3174	~2.5
H	16.0	small	~11	~27	8 to 20
I	16.0	small	~4	~20	8 to 20
J	16.0	small	~1.2	~17.2	8 to 20

SENSING TASKS THAT CAN INVOLVE THE USE OF MAGNETS

The LBNL team identified several sensing tasks that could benefit from using a magnetic field and nuclear magnetic resonance (NMR)[1]. These tasks included: 1) water content measurements on wood chips entering the plant, 2) identification of various wood species in the incoming chip stream, 3) a measurement of the moisture content of the pulp (brownstock) leaving the digester, 4) a measurement of the lignin and moisture content of the black liquor leaving the digester, and 5) measurements of moisture, caliper (paper thickness) and basis weight of paper passing from the press through the dryer.

This led to a series of experiments in the UC Berkeley College of Chemistry NMR test facility. These measurement were done in a 300 MHz NMR magnet that generates 7.05 T. The magnet has a 0.1 ppm (0.1 parts per million) good field region in spherical region with a diameter in the range from 5 to 8 mm. The LBNL team measured the moisture content of wood, commercial pulp and paper over a range of moisture contents. Good agreement with moisture content measurements made by oven drying were achieved.

A crude measure of the NMR structure of black liquor was made. Peaks for various chemicals were seen. Figure 2 below shows measurements made on the black liquor from the digester of a paper mill. The measurements show that one can differentiate between various components in the liquid part of the black liquor. In order to make the measurements shown in Figure 2, the uniformity of the magnetic field had to be of the order of 0.1 ppm. When the paper industry was presented with the results, shown in Figure 2, it was not clear that this sort of data could be used to control paper plant processing.

Moisture content and caliper was measured for the sheets of filter paper in a stack, using NMR imaging techniques. These measurements were done for very small samples with a range of masses between 3 and 30 mg. Individual sheets in the stack could be seen. The sheet thickness could be determined to a few microns. Experiments to differentiate between species of wood were tried, without success. The results of the UC Berkeley tests were encouraging because they showed that sensors using NMR could be made to work on the feed stock and product of a paper plant.

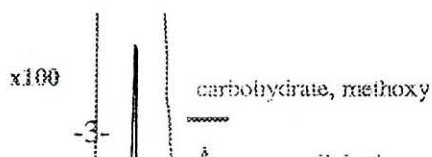


FIGURE 2. A measurement of the constituents in Black liquor from a Pulpig Digester. The sharp peak in the center is the water NMR peak. The magnified peak is above the unmagnified peak that shows almost no structure. The other peaks can be seen on either side of the water peak in the magnified signature are from fragments of lignin and cellulose that are dissolved in the black liquor.

If one is going to use NMR sensors in a paper mill, the mass of the sample must be increased five to six orders of magnitude over the lab sample masses. The good field region must be large enough to allow the sample to be measured. For chips going into a digester, the minimum sample size approaches 100 mm. For black liquor and the cellulose stock leaving the digester, the same sort of sample size is needed. To measure basis weight caliper and moisture content of already formed paper one has to be able to scan a sheet of paper moving 20 meters per second over a width up to 12 m (about 40 feet). After much discussion with the industry, it became clear that their priorities were moisture content measurements of chips and the black liquor stream. Also very important is the measurement of moisture content and caliper simultaneously with a measurement of the paper basis weight.

When the idea of using superconducting magnets to generate the magnetic fields was presented, to the paper industry, but it was vetoed. Cryostats with liquid helium are incompatible with a paper plant. This paper will make a case for reconsidering that decision.

WATER CONTENT MEASUREMENTS AT LBNL

In order to meet the stated requirements of the paper industry, LBNL decided to do its measurements at low fields using a permanent magnet. The first such magnet used was a samarium cobalt magnet that generated an induction of 0.47 T in a direction that was perpendicular to the direction of flow for wood chips or black liquor. The chips or black liquor was carried in a TFE pipe, which was 19 mm in diameter. The 20 MHz RF coils were wound around TFE pipe, which contributes no NMR signal of its own. The magnet good field region was about 18 mm in diameter by 25 mm long. Good field for this magnet is defined as about plus minus 10 ppm.

The samarium-cobalt permanent magnet provided a reasonably uniform. However, the field from the samarium-cobalt magnet did change with temperature about -300 ppm per

degree C of temperature increase. As permanent magnet materials go, samarium-cobalt has a rather low temperature coefficient. To do the first experiments, the magnet and the sample were put into a temperature-controlled box. Since the magnet mass was of the order of 85 kg, the temperature control time constant was very long. A simple control system could keep the RF frequency from drifting during the NMR measurements.

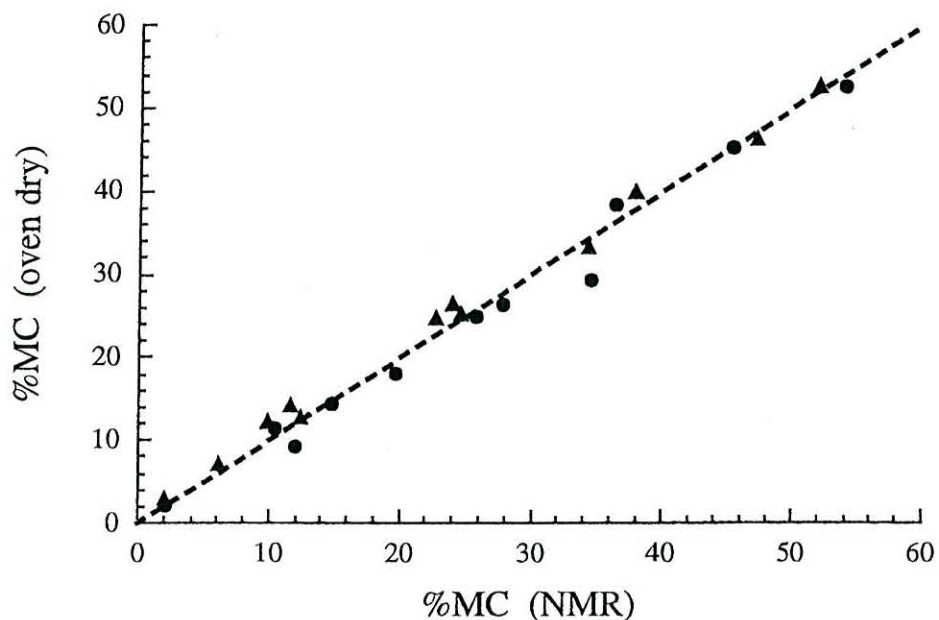


FIGURE 3. Moisture Content Measured using NMR compared to the Moisture Content of the Same Sample Measured by Drying the Same Sample in an Oven under Reduced Pressure.

Figure 3 shows the water content of wood chips measured by NMR as a function of the water content measured by drying. There is very good agreement of the NMR measurement with the drying measurements up to very high moisture contents. A number of experiments have been done on wood chips that contain water and a mixture of water and ice. Doing these experiments using 20 MHz and 300 MHz NMR suggests that a change in the RF frequency will permit one to do more than just measure liquid water content. Preliminary measurements suggest that water the form of ice can be differentiated from water and cellulose.

The percent of lignin and other constituents was measured black liquor by measuring the fraction of water and subtracting it from one. It has been demonstrated that one can measure the energy content of black liquor using NMR. The LBNL experimental work shows that NMR can be used for sensors in a paper plant. In order for the sensors to work well, the mass of the samples must be increased another two to three orders of magnitude. If anything, the field quality should be an order of magnitude better than LBNL could get from its permanent magnet. Since the sample size must grow a factor of six to eight, the mass of a permanent magnet used to measure that sample will grow more than two orders of magnitude. The paper industry should clearly consider the use of superconducting magnets as part of NMR sensors.

SUPERCONDUCTING MAGNETS FOR MEASURING MOISTURE CONTENT OF WOOD CHIPS AND BLACK LIQUOR CONSTITUENTS

There is a practical size limit associated with permanent magnets with a large volume of good field. If the magnet used for the LBNL experiment were scaled to produce a good field diameter of 120 mm and a good field length of 160 mm (a good field to ± 10 ppm), its mass would be over 20 metric tons. The cost of the magnet scales to first order as the magnet mass. As a result, the cost of the permanent magnet would likely exceed 1.5 M\$. A superconducting magnet that can operate at field levels up to 2T with field uniformity within a 120-mm diameter sphere of better than 10 ppm can be built for a fraction of the cost of a large permanent magnet.

Besides cost, a superconducting magnet offers other benefits over a large permanent magnet. These include: 1) The field stability is better than 1 part in 10^8 over a period hour when the magnet is persistent. 2) The magnetic field does not vary with ambient temperature. 3) Depending on how the magnet is shimmed, the field uniformity within the good field volume can be as good as 0.1 ppm. 4) The magnetic induction within the good field region can vary from 0.1 T (4.2 MHz for hydrogen) to 2.0 T (85 MHz for hydrogen). 5) NMR can be done using other atoms, such as sodium 23. Some interesting work could be done with large samples of the liquor in the digester using sodium 23 NMR at 2 T, where the resonant frequency is 22.52 MHz. The sensitivity and signal to noise ratio for sodium 23 at 2 T is similar to hydrogen at 0.53 T. So one can expect to get good sodium results if the field is 2T.

The major change that has occurred with superconducting magnet technology that may make it acceptable to the pulp and paper industry is the elimination of liquid cryogenes within the magnet. A 2-T magnet system can be cooled using a 4 K cryo-cooler. A pulse tube refrigerator may be an attractive machine for cooling a NMR magnet system[2]. Like the Gifford McMahon coolers, a pulse tube cooler will also provide cooling at 40 to 50 K for cooling shields and the top of high temperature superconductor (HTS) leads. The combination of cryo-coolers that operate at 4 K and HTS leads allows magnets to operate reliably in a paper mill environment.

Magnets for sensors can operate in persistent mode. Once the magnet has been charged to the desired current, the leads can be disconnected. The part of the lead sticking

out of the cryostat (with its protection resistor) can be covered to protect the electrical connection from the moisture that is associated with the paper making process.

Figure 4 shows a cross section of a spherical superconducting solenoid that is capable of delivering a field quality of better than 1 ppm in a region that is 100 mm in diameter[3,4]. Stray field is an issue, so the solenoid shown in Figure 4 is actively shielded[5]. The stray field 1 meter from the magnet center is less than 0.0003 T. At the outside boundary of the cryostat the stray field is less than 0.03 T. Table 1 presents a number of the parameters for the solenoid shown in Figure 4.

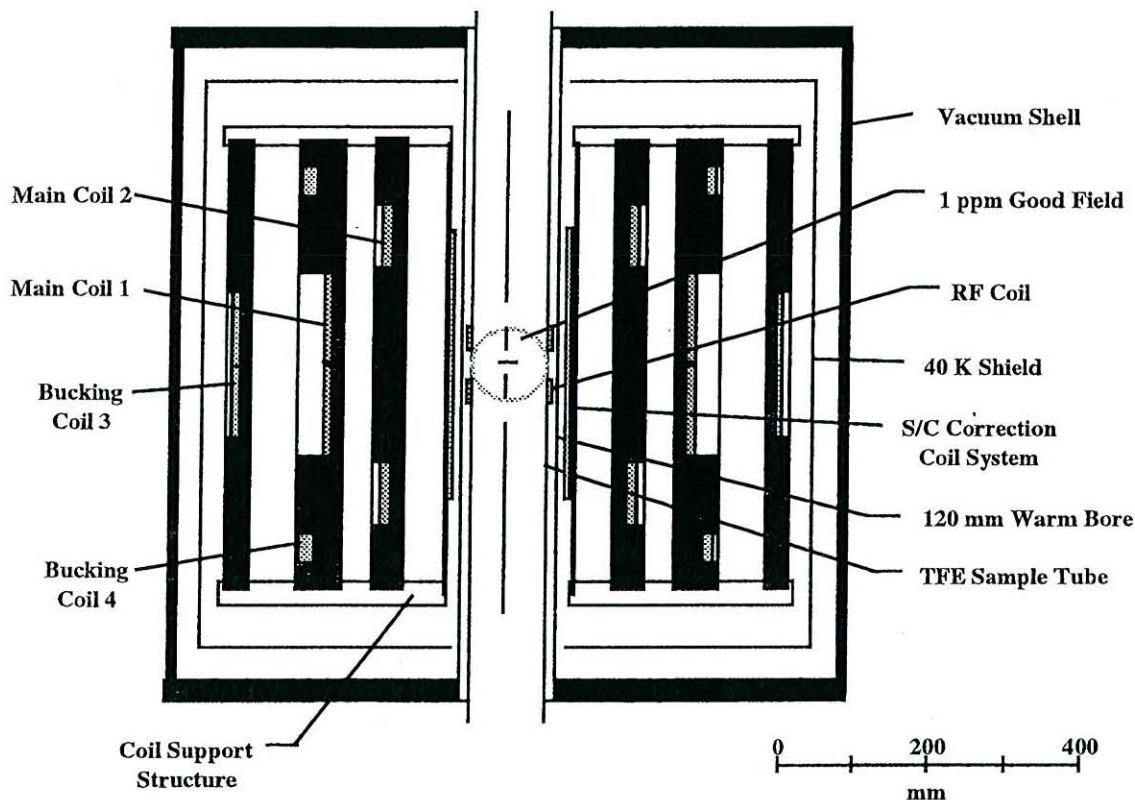


FIGURE 4. A Cross Section of a Nearly Spherical Actively Shielded 2 T Superconducting Solenoid with a 1 Part per Million Good Field Region that is 100 mm in Diameter. This magnet is suitable for measuring the moisture content of wood chips, pulp or black liquor in a pipe that is 120 mm in diameter. The magnet can be used to separate various species in black liquor in a region that is 80 to 90 mm in diameter. All of the coils would operate in persistent mode including the field correction coils. The cryostat support system is not shown. The magnet bore is shown as vertical, but it could be horizontal. The 4 K cryo-cooler is not shown.

TABLE 2. Basic Parameters for a 2 T Solenoid with a 100 mm Diameter Good Field Region (See Figure 4)

Magnet Parameter	
Cryostat Warm Bore (mm)	120
Cryostat Outside Diameter (mm)	900
Cryostat Length (mm)	900
Design Central Induction B_0 (T)	2.0
Length 1 ppm Good Field Region (mm)	100
Diameter 1 ppm Good Field Region (mm)	100
Superconducting Shim Set Inside Diameter (mm)	140
Smallest Main Magnet Coil Inside Diameter (mm)	316

Largest Bucking Coil Outside Diameter (mm)	725
Total Number of Coils	8
Number of Turns in the Magnet	5720
Magnet Design Current at B_0 (A)	405.04
Magnet Stored Energy at B_0 (kJ)	235
Magnet System Self Inductance (H)	2.86
S/C plus Matrix Current Density at B_0 ($A\ mm^{-2}$)	396
E J Quench Protection Criteria ($A^2\ m^{-4}\ J$)	3.69×10^{22}
Solenoid Quench Protection Method	Quench Back
Distance from Center to the 0.0003 T Line (mm)	920

The RF coil is saddle shaped and produces a dipole field that is perpendicular to the flux lines in the good field region of the solenoid. Correct placement of the RF coil can improve its efficiency, so that a maximum signal can be obtained from the sample, which will have a mass from 0.4 to 0.7 kg.

The unresolved issue is the effect of NMR activation time T_1 during moisture content measurements. Pure water can have an activation time as long as 10 s. Water in contact with wood chips has a much shorter activation time. If the water is outside of the chip, the activation time can be as long as 800 ms. Water that is inside the chip has an activation time of the order of 120 to 150 ms. The activation time for pure Cellulose is less than 60 ms. Activation time is an issue if accurate measurements of moisture content are to be made in the sample. This suggests that the sample either has to travel slowly (less than $0.2\ m\ s^{-1}$) through the magnet, or the material has to be placed into the magnet and measured while not moving. When one takes only a sample of the material, there is always a question of whether that sample is a valid sample of what is in the flow stream.

The magnet shown in Figure 4 and Table 2 will have a total mass of around 350-kg. Since a superconducting solenoid has a much lower mass than a large permanent magnet, the solenoid appears to be a cost effective alternative.

SUPERCONDUCTING MAGNETS FOR MEASURING PAPER THICKNESS

The measurement of paper thickness requires a magnetic field gradient. The magnet and the RF coil that can only be on one side of the moving paper sheet. The paper should not touch the magnet or the sensing head. The magnet and head should scan across the sheet of paper (up to 12-meters in width) as the paper goes through at speeds up to $20\ m\ s^{-1}$. A four-cell gradient magnet is shown in Figure 5. The coil that creates the RF pulse and picks up the signal from the moisture in the paper is located in the gradient field, between the second and third cells. The magnet shown in Figure 5 is about a meter long (in the direction perpendicular to the page). The sensing RF coil shown in Figure 5 is about 100 mm long. The paper should see between 600 and 700 mm of activation field (at about 2 T) before the paper enters the region that sees the RF pulse. Separate RF coils may be used to send the RF pulse and receive the RF echo back from the moisture in the paper.

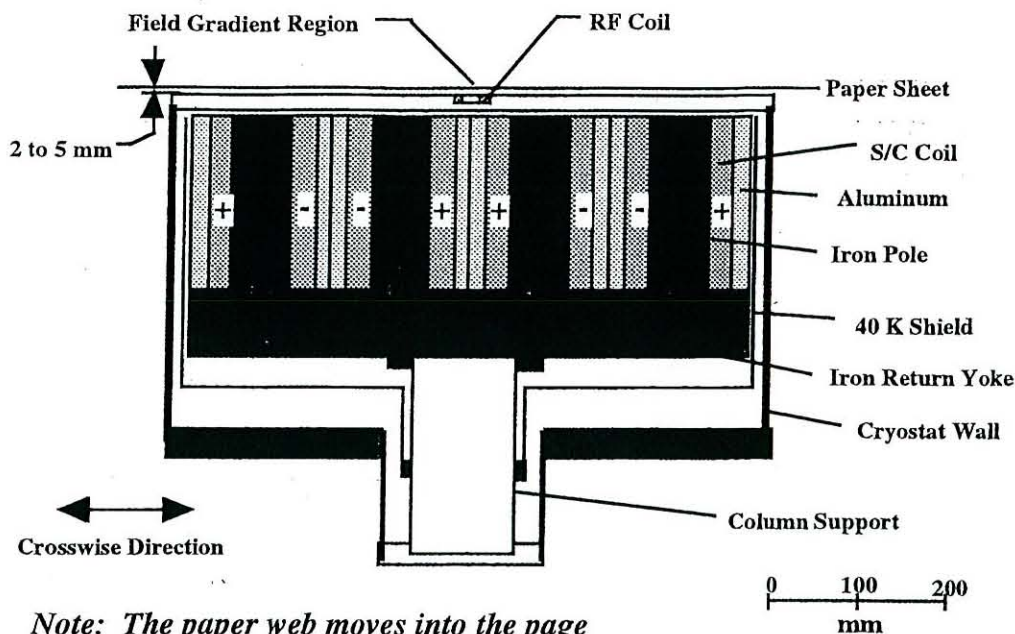


FIGURE 5. A Cross Section of a Superconducting Gradient Magnet for Measuring Paper Thickness is shown in Figure 5. This magnet and NMR system can be combined with a basis weight measurement device to scan across the web to measure paper moisture content and thickness. The magnet and RF coils are on one side of the paper.

In the region of the RF coil, there is a field gradient that is perpendicular to the paper. If this gradient is 50 T per meter, the variation of the RF frequency from a 50-micron thick sheet of paper will be about 100 kHz. One should be able determine changes in the RF frequency to about 2 kHz. As a result, one should be able to measure the paper thickness to 1-micron.

One of the biggest problems is the NMR activation time for the water in the paper. At a paper velocity of 20-m s^{-1} , the paper will be in the magnetic field only about 35 ms before the RF pulse flips the hydrogen atoms. This means that the moisture in the paper is only about a quarter activated. Thus the magnitude of the NMR signal from the water in the paper is reduced by a factor of four. The 120 to 150 ms activation time combined with a small sample (about 90 mg for a 10 cm^2 sample of 20 weight paper) means that the NMR signal will be small. Using a superconducting magnet to increase the magnetic field in the paper will increase the strength of the NMR signal while reducing noise.

CONCLUDING COMMENTS

Experiments at LBNL have demonstrated that good-quality measurements of the moisture content in wood chips and black liquor can be made using low field NMR. The permanent magnets used for the LBNL measurements can not easily be scaled to a size that is attractive for direct use in a paper mill. The use of superconducting magnets becomes cost effective for sample sizes above 50 mm. A superconducting magnet will produce a more uniform magnetic field over a larger volume than will a permanent magnet. Larger magnetic fields would be attractive for doing sodium 23 NMR as well as hydrogen NMR. The field uniformity available in a superconducting magnet will allow the constituents of black liquor to be examined during the digestion process.

One of the immediate uses of NMR could be to provide an accurate measurement of the moisture content of purchased pulp and chips. In a large paper mill (1500 metric tons of paper per day of production), a one percent error in the measurement of the moisture

content of wood or pulp can result in about 0.5 million dollars per year extra being paid for pulp or chips. A reliable way of measuring moisture content in purchased pulp should pay for itself in less than one year.

Superconducting gradient magnet may provide a way of measuring paper thickness at the same time its basis weight is being measured using beta ray attenuation. NMR might also be used to measure the moisture content of the paper along with its caliper.

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