AN ABSTRACT OF THE THESIS OF

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James B. Wilson

Dielectric quantification of material properties is a technology well established in many industries. The application of this concept to the forest products industry to measure adhesive cure, however, has been belated in part due to a lack of proven technology directed at industrial processes and products. It is of great interest to manufacturers to minimize production costs and maximize output. This means being able to identify the minimum time required to cure composite panel products during hot pressing.

In the hot-pressing process, material is currently pressed based on a conservative schedule that is actually longer than necessary. The schedule provides what temperatures and pressures are to be used throughout the press cycle to ensure resin cure (cross-linking). In the case of urea-formaldehyde, the resin is subject to strength loss if heat remains applied too long. The objective of the schedule is to cure the resin to an acceptable level

and remove the product before degradation can occur. It is difficult to exactly predict the optimum point given all the variables to consider such as panel thickness; moisture and time; press pressure and temperature; and particle geometry, etc. This is where dielectric monitoring can help.

Since the critical variable is degree of resin cure, it is logical to design a monitoring system that measures and utilizes it in a feedback control system. As the resin is curing, the molecules and ions become interlaced in a lattice structure during polymerization, reducing rotational and migrational mobility. Rotational and migrational mobility can be quantified dielectrically by applying an oscillating electric field to the material. Included in any monitored dielectric quantity is the effect due to moisture and wood. Studies were conducted to determine their contribution to the readings.

Particleboard panels were manufactured in a laboratory environment and monitored with a dielectric system developed by the author. Three different adhesives were used - urea formaldehyde (UF), phenol formaldehyde (PF), and polymeric diphenolmethane diisocyanate (isocyanate, MDI). Dielectric response curves were obtained for each of the resins, and internal bond strength (IB) measurements were taken throughout the curing process of the boards. IB data were charted with dielectric data to show characteristics in the dielectric response curves that could be used to indicate cure status.

The dielectric response curves show very encouraging peaks, valleys, and inflection points that seem to correspond to respective cure data. These

characteristics could be incorporated into a full-scale system and used in an industrial setting to control and optimize press operations.

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Dielectric Cure Monitoring of Composite Panels During Hot Pressing: A Fundamental Understanding

by

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TABLE OF CONTENTS

Page

INTRODUCTION 1

OBJECTIVES 4

BACKGROUND/LITERATURE REVIEW 5

Brief History of Dielectrics 5

Principles of Dielectrics 19

PROBLEM STATEMENT 29

Current Methods to Manufacture Particleboard 29

Potential Benefit of Dielectrics 30

EXPERIMENTAL DESIGN 32

Preparation Considerations 32

Materials 40

Methods 41

RESULTS 56

Dielectric Response of Materials Involved 56

Internal Bond Strength Values 62

Dielectric Response Curves of the Three Adhesives 66

TABLE OF CONTENTS (Continued)

Page

DISCUSSION 69

Compilation of Dielectric and IB Data 69

Physical Chemistry in Dielectric Response Curves 70

CONCLUSIONS 78

Experiment Summary 78

Recommendations for Future Research 79

BIBLIOGRAPHY 80

APPENDICES 85

LIST OF FIGURES

Figure		Page
1.	Dielectric data recorded for MDI (Wolcott, 1995a)	9
2.	Dielectric data recorded for MDI (Wolcott, 1995b)	10
3.	Dielectric data recorded for UF (Wolcott, 1995b)	10
4.	Dielectric data recorded for UF (Magill, 2000)	12
5.	Configuration diagrams for sensors 1 and 2	13
6.	Diagram of interdigitated sensor operation theory	14
7.	Chemical diagram for UF (Skeist, 1990)	15
8.	Chemical diagram for PF (Skeist, 1990)	16
9.	Chemical diagram of MDI (Skeist, 1990)	17
10.	Standard representation of IB data (Humphrey, 1999)	18
11.	Cartesian representation of capacitive and resistive current components	22
12.	Ionic and molecular orientation in response to alternating electromagnetic field (plates show relative charges)	26
13.	IB cure rate data plotted against cure temperature for UF (derived from data reported in Humphrey, 1999)	35
14.	In–press monitoring setup	43
15.	Inherent system noise of an oven dried panel in the hot press (UF-MDI circuit setup)	44

LIST OF FIGURES (continued)

Figure **Page**

16. Resistive and capacitive component waveforms 46

17. Electrical monitoring circuit for UF boards 47

18. Electrical monitoring circuit for MDI boards 49

19. Electrical monitoring circuit for PF boards 51

20. Sample removal pattern from each board produced 55

21. Dielectric response curve of dry board – no moisture or adhesive, UF-MDI setup 57

22. Dielectric response curve of dry board – no moisture or adhesive, PF setup 57

23. Dielectric response curve of 10% MC board without adhesive, UF-MDI circuit setup 59

24. Dielectric response curve of 10% MC board without adhesive, PF circuit setup 59

25. Top, center and bottom temperature data with RTD 60

26. IB cure regions defined 63

27. Actual and ideal IB data for UF boards 64

28. Actual and ideal IB data for PF boards 65

29. Actual and ideal IB data for MDI boards 65

30. Dielectric response curve for UF boards 67

31. Dielectric response curve for PF boards 67

LIST OF FIGURES (continued)

Figure		Page
32.	Dielectric response curve for MDI boards	68
33.	Dielectric and IB data for UF	70
34.	Dielectric data recorded by Wolcott and Rials for UF (Wolcott, 1995a)	72
35.	Dielectric and IB data for MDI	74
36.	Dielectric and IB data for PF	76
37.	Dielectric and IB data for PF, magnified scale	77

LIST OF TABLES

Table		Page
1.	Summary of methods to remove heat from panels	36
2.	Statistical results for paired two sample t-test of samples from two panels to determine significant difference	37
3.	Summary of panel and adhesive properties	41
4.	Summary of process parameters and respective units	54
5.	Adhesive cure time data	66

LIST OF APPENDIX FIGURES

Figure		Page
38.	Hierarchy diagram of LabVIEW VIs working together to collect and process data	87
39.	Panel view of collect and send software VI	88
40.	Diagram view of collect and send software VI	89
41.	Panel view of signal process software VI	90
42.	Diagram view of process software VI page 1	91
43.	Diagram view of process software VI page 2	92
44.	Panel view of display and record software VI	93
45.	Diagram view of display and record software VI	94
46.	Individual UF board dielectric response curves	96
47.	Individual PF board dielectric response curves	96
48.	Individual MDI board dielectric response curves	97

LIST OF SYMBOLS USED

Symbol	**Definition**
EM	Electromagnetic
IB	Internal Bond Strength
LVL	Laminated Veneer Lumber
MC	Moisture Content % (Oven-dry basis)
MDF	Medium Density Fiberboard
MDI	Isocyanate
OSB	Oriented Strand Board
PC	Personal Computer (IBM compatible)
PF	Phenol-Formaldehyde
RTD	Relative Tan Delta
UF	Urea-Formaldehyde
VI	Virtual Instrument (a LabVIEW term for software program)

Dielectric Cure Monitoring of Composite Panels During Hot Pressing: A Fundamental Understanding

INTRODUCTION

As early as the 1950's, the field of dielectrics was explored as a measurement and control technique in the forest products industry (Miller and Cole, 1957). Dielectric monitoring involves the electrical measurement of the behavior of molecules and ions in a substance. Changing properties in the measured material correspond to changes in the dielectric signal measured. Until recently few research papers were written on the topic as it applies to forest products.

In 1975, William James of the Forest Products Laboratory in Madison, Wisconsin published a comprehensive paper on the dielectric properties of wood (James, 1975). He looked at variables such as temperature, frequency, moisture content, and grain variation. This became the basis for much of the subsequent research that followed in this field.

Research on monitoring of pure adhesive cure is fairly comprehensive as it applies to the forest products industry. Commercial systems are available and fairly common in use. Miller and Cole used dielectrics to monitor adhesives in 1957. In 1997, Kranbuehl published a report on monitoring epoxy/graphite systems using a Hewlett-Packard device.

Even more research exists in using dielectrics for moisture content determination. Nanassy (1972) looked at the "dielectric measurement of moist wood in a sealed system." James published again in 1983 concerning "dielectric properties of lumber loads in a dry kiln," followed by a publication in 1986 exploring "the interaction of electrode design and moisture gradients in dielectric measurements on wood." Quarles and Milota (1991) reported on the "influence of kiln temperature and density on the performance of in-line moisture meters," examining how dielectric moisture meters were affected by these factors. There are many papers published on this topic, and it is a technology widely implemented in the industry today.

In the field of *in-situ* monitoring of adhesive cure, however, there is remarkably little published data available. Robert Rubitschun (1981) conducted a study using an Audrey II dielectric spectrometer made by Tetrahedron Associates in an effort to monitor cure in plywood glue lines. While ultimately unsuccessful, it may have been simply an equipment issue that prevented any feasible readings. He had difficulties with the phenol formaldehyde (PF) adhesive in that the instrument readings began varying uncontrollably when it was introduced. Rials had similar problems with PF resin using a Eumetrics System III Micro-Dielectric Analyzer (Rials, 2001). The instrument was unable to produce dielectric readings, as they were out of range. The Eumetrics system did not have an adjustment to compensate for the wide range of resistance values between the adhesives studied.

Wolcott and Rials published two papers in 1995, one concerning in-situ monitoring of isocyanate (MDI) adhesives, and the other including both urea formaldehyde (UF) and MDI. In both studies an Eumetrics dielectric analyzer was used to obtain data. The results obtained in those studies are covered in a later section.

OBJECTIVES

The general objective of this research was to develop a dielectric system for monitoring adhesive cure during hot pressing and explore its application to particleboard formed with three different adhesives - urea formaldehyde, phenol formaldehyde, and isocyanate.

The specific objectives were:

1. Develop a dielectric system for in-press monitoring of adhesive cure, consisting of an electrical circuit, data acquisition hardware, and custom software program.
2. Develop a dielectric monitoring system for which all three adhesives stay within range for the duration of pressing cycle.
3. Further the knowledge available academically in the field of dielectric cure monitoring of forest products.
4. Match internal bond (IB) strength data and cure progression dielectric data for a panel during hot pressing.

BACKGROUND/LITERATURE REVIEW

Brief History of Dielectrics

It is important to differentiate between the concepts of dielectric drying/curing and dielectric monitoring. Dielectric drying of wood and adhesive curing rely on the molecular rotation experienced in an alternating electric field and the resulting friction to generate the heat necessary to dry wood or cure adhesive in the product (Resnik, 1997; Wilson, 1987). Dielectric drying is different than dielectric monitoring in that its purpose is to impart large quantities of energy to the material. In dielectric drying the objective is to optimize the frequency and voltage and maximize the energy converted into heat utilizing the resulting movement of molecules and ions (Brown, 1947). Dielectric monitoring seeks to maximize the electrical signals monitored, and to find the combination of frequency and electric properties that best represent molecular and ionic movement.

Dielectrics of wood

William James published in 1975 what is probably the most comprehensive look at the dielectric properties of wood given various conditions. His research covered Douglas-fir, oak and hardboard. At a frequency of 100 Hz, a temperature of 90°C and a moisture content (MC,

oven dry basis) of 12%, James reports a value of 66 for the tan delta (a dielectric property defined later) of hardboard. Holding the other variables constant and reducing the moisture content to zero, yielded a tan delta value of 0.11.

James (1985) also investigated mechanical stress and its effect on the dielectric properties of wood. At and above 12% MC parallel to the grain, mechanical stress affected dielectric readings. Below 12% MC, however, the reading did not react to mechanical stress, precluding its use as a drying stress monitor.

Dielectrics of Moisture

The field of moisture content determination by dielectric means is well developed, having been in use for commercial products for years. There is much information available on the use of handheld devices, stationary monitors, full-scale dryers and more. As the approximate dielectric constant (another material property defined later) of oven dry wood is 2, and the dielectric constant of water is 80.4, it becomes evident that water will dominate the reading of a wood-water system. Devices that measure moisture content generally use frequencies between 3 and 30 MHz, but can be in the GHz range (King and Basuel, 1993; Resnik et.al., 1997).

In 1993, King and Basuel published a paper on using microwaves to measure moisture content of composite panels. They developed a device that was designed to be used in-line to determine the total basis weight of reconstituted composite panels.

Adhesive Cure Monitoring

Another field of importance to composite panel manufacture is adhesive cure. There are many papers in a multitude of journals on dielectric monitoring of adhesives, films, polymers, etc. (Kranbuehl, 1997; Ungarish et.al., 1991; Fritzen et.al., 1977; Yalof, 1975). However, very little research has been applied to the adhesives used in forest products manufacture. Some literature on the exploration of this issue can be found in Miller and Cole (1957) and Geimer (1996).

Miller and Cole (1957) looked at catalyzed and uncatalyzed urea, resorcinol, phenol-resorcinol and melamine type commercial glues. They used a 1 MHz excitation frequency to obtain the data, compared to 100 Hz in this study, and as a result, the numbers were not directly comparable.

Dielectric Monitoring of Wood/Adhesive Systems

There is little published data at the time of this report on cure monitoring of composite panel systems using dielectrics. A few research

papers exist, and industry has expended some time and energy on the issue, but their results are not generally available in the public domain.

Plywood adhesive cure monitoring was examined by Rubitschun (1981). Rubitschun used an Audrey II dielectrometer to monitor a PF adhesive under laboratory conditions, but the results of that study were ultimately inconclusive.

Wolcott and Rials had two publications in 1995, both of which focused on dielectric monitoring of composite panels, one in the Forest Products Journal (FPJ), and the other in the Proceedings of the Washington State University International Particleboard/Composite Materials Symposium (WSU). The article in the FPJ reported on particleboard made with MDI and monitored with a Eumetrics System III Micro-Dielectric Analyzer. An interdigitated sensor was embedded in the particleboard furnish prior to pressing and excited at 100 Hz to measure dielectric loss or ϵ'', defined as " . . . a measure of the energy expended to align dipoles and transport ions" (Wolcott and Rials, 1995a). Panel readings obtained were similar to small controlled samples. The MDI component of the dielectric signal seemed to dominate, showing promise for the system (Figure 1). The sampling rate was not very fast, however, and the authors suggested increasing it in future research to better detect the early part of the cure dielectric response curves.

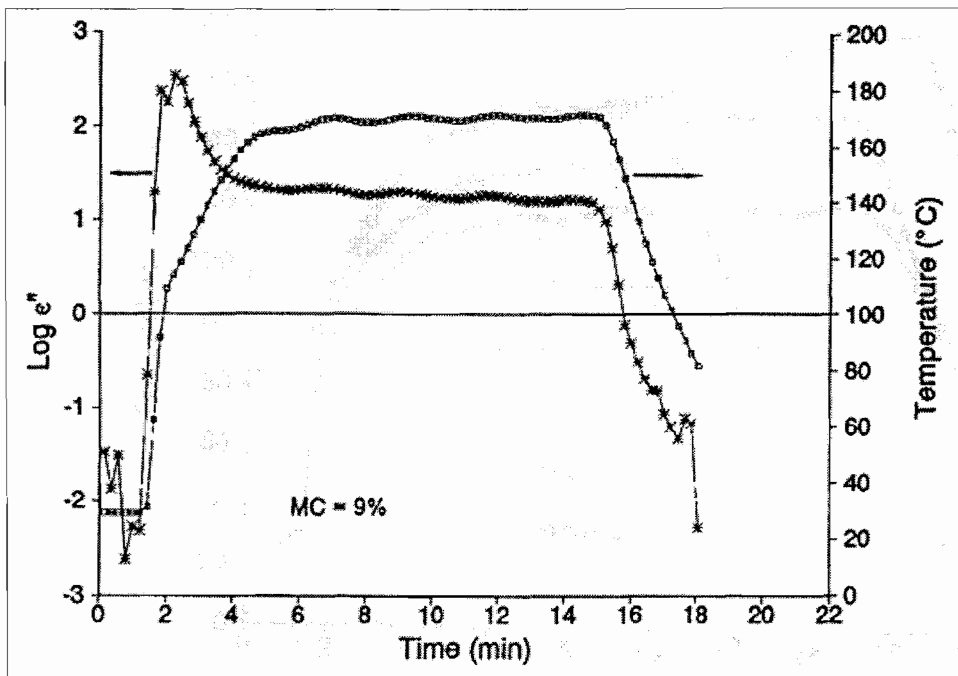

Figure 1 - Dielectric data recorded for MDI (Wolcott and Rials, 1995a)

In the Washington Symposium Proceedings, boards made with MDI and UF were manufactured and monitored. Five excitation frequencies between 10^{-1} and 10^{3} Hz were used with the same equipment and sensor as before. This time, however, electrical conductivity was measured and plotted on a log scale. Three sensors were used for each board, one in the core (50% mark), one halfway between the surface and the core (25% mark), and one at the face (10% mark). The sampling rate was the same as the previous study. Results were obtained for both adhesives, and the graphs are displayed in Figures 2 and 3. The MDI was concluded to show that water

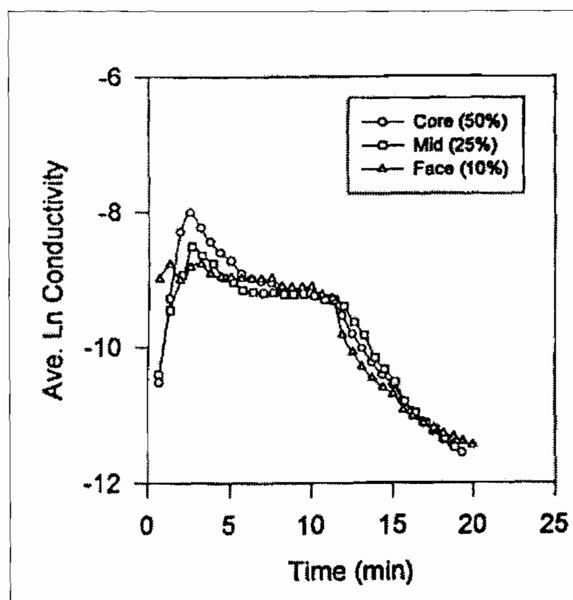

Figure 2 - Dielectric data recorded for MDI (Wolcott and Rials, 1995b)

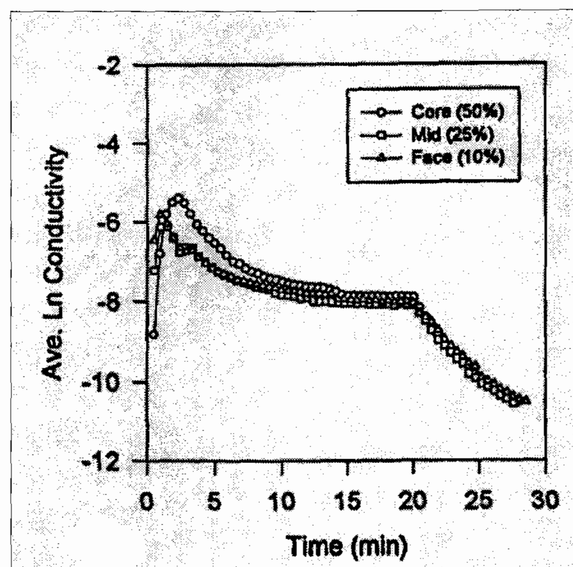

Figure 3 - Dielectric data recorded for UF (Wolcott and Rials, 1995b)

consumption by the adhesive during cure dominated the signal, and showed good results. As the UF adhesive had a greater amount of water present, it was less certain whether the conductivity reading measured adhesive or water. Ultimately, the adhesives studied had different response curve characteristics.

In 1999, Richard Magill and Steve Sauter published an abstract on the results of a dielectric monitoring system (Magill and Sauter, 1999). UF bonded particleboard was manufactured in a laboratory press. No graphs were presented, but it is claimed that both conductance and capacitance measurements – dielectric values – were recorded for adhesive cure. Features in the curves were said to correlate to the adhesive system.

A follow-up study was published by Richard Magill in which the dielectric monitoring system developed was operated on a production hot press (Magill, 2000). Particleboard was manufactured with UF adhesive, and impedance (a ratio of conductance to capacitance of system) data recorded. Figure 4 shows an example of the data collected by the system used. The individual features of the dielectric response curve were described and defined as a means of controlling the pressing operation. As a result of this system, Signature Control Systems (manufacturer and vendor of the equipment) expects a more uniform product with fewer blows and delaminations. They expect a reduced press cycle time which will translate into money saved for the manufacturer.

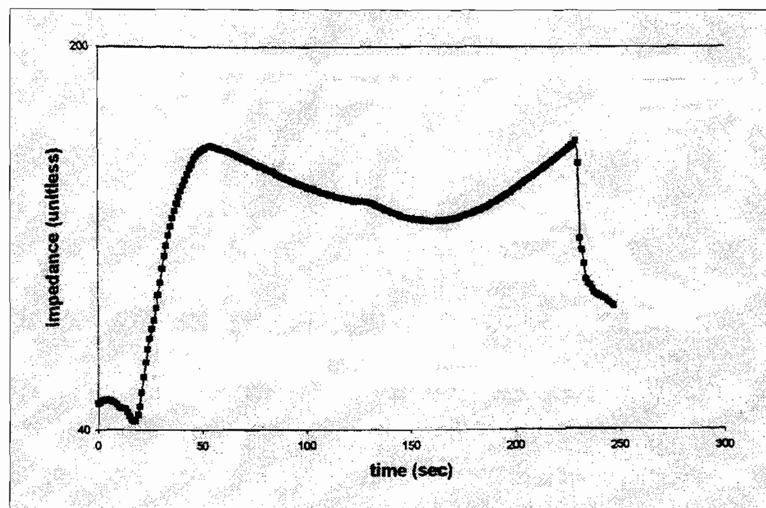

Figure 4 - Dielectric data recorded for UF (Magill, 2000)

Sensor Configuration

There are two fundamental types of sensor configurations to choose from when monitoring materials via dielectrics. The first is the classical two-plate configuration, where you have two flat plates with the test material between them. One plate is applied with an alternating voltage, and the other is grounded (as in Figure 5, Sensor 1).

The other sensor design is a recent development. It utilizes a printed circuit board with conductors interleaved like fingers, but not touching (see Figure 5, Sensor 2) (Wolcott and Rials, 1995a).

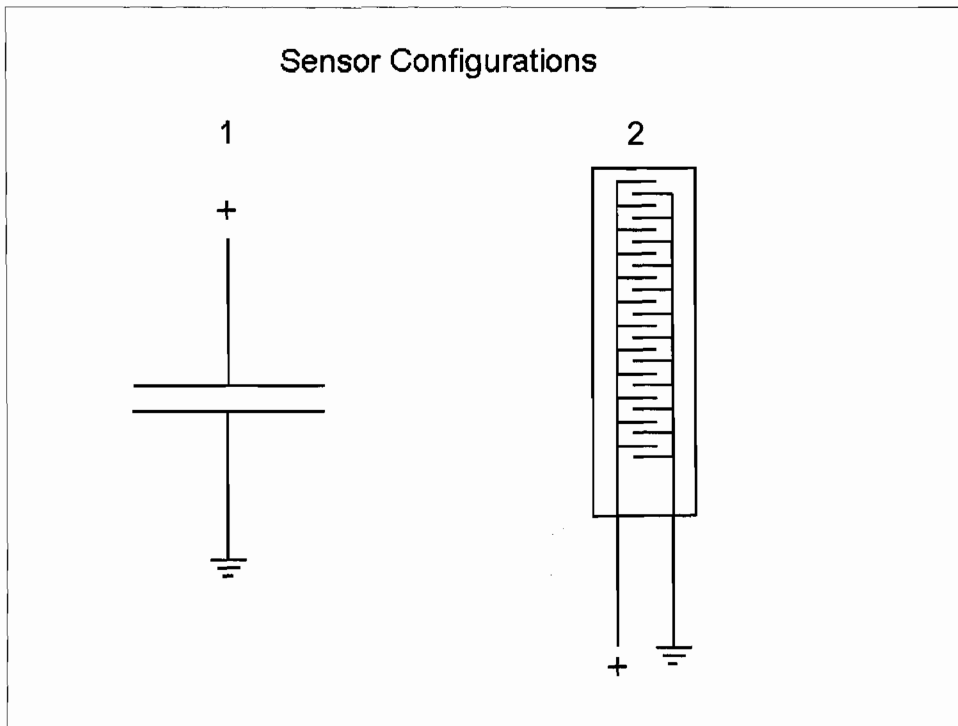

Figure 5 - Configuration diagrams for sensors 1 and 2

Sensor 2 is also known as an interdigitated electrode. The idea behind it is to submerge or sandwich it in the material to be tested and a "fringe effect" penetrates and measures the dielectric properties. Essentially, the field lines outside the capacitor are what is making the reading (Figure 6).

For this experiment, the sensor requirements were to be non-intrusive and non-marking to the product. As the interdigitated electrode sensor by design has a sensitivity depth of a few millimeters to either surface, it was decided to implement the first sensor design. A sensor the exact dimension of the panels was designed and fabricated. It left no surface markings, and was

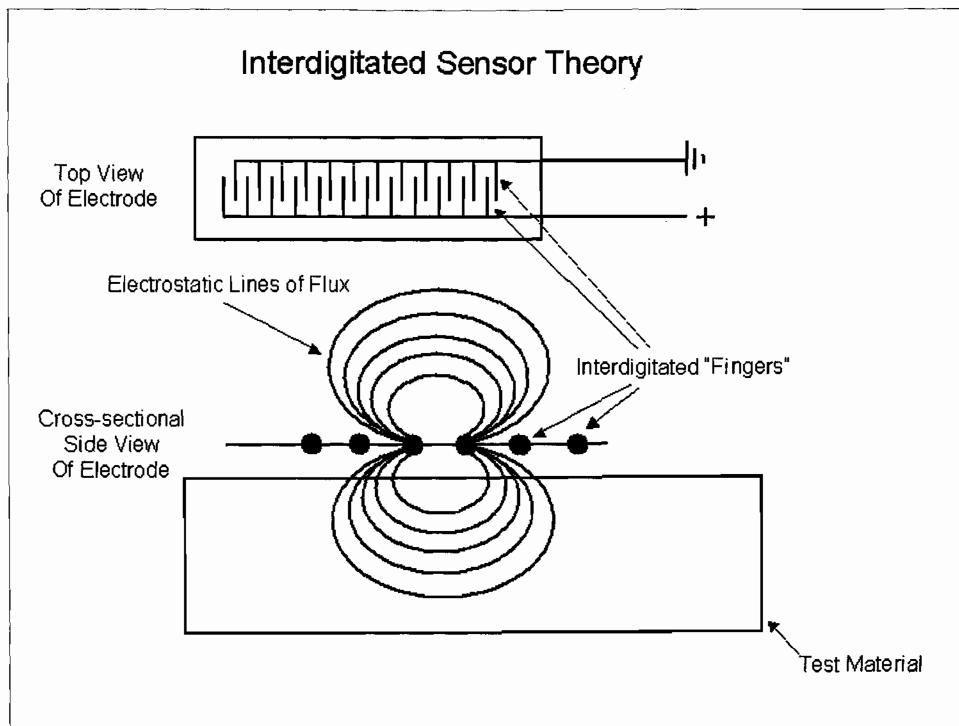

Figure 6 - Diagram of interdigitated sensor operation theory

indefinitely reusable. The disadvantage to the large sensor setup was the requirement of electrical isolation, which meant adding layers of Teflon sheeting and an extra set of aluminum caul plates. This material impeded heat transfer into the panels.

Adhesive Chemistry

As each adhesive cures, they experience distinctly different chemical reactions (Skeist, 1990). UF experiences a condensation reaction, releasing

water as it polymerizes into chains. The polymer is bonded to wood by hydrogen bonding, no covalent bonds are formed with the adhesive (Figure 7).

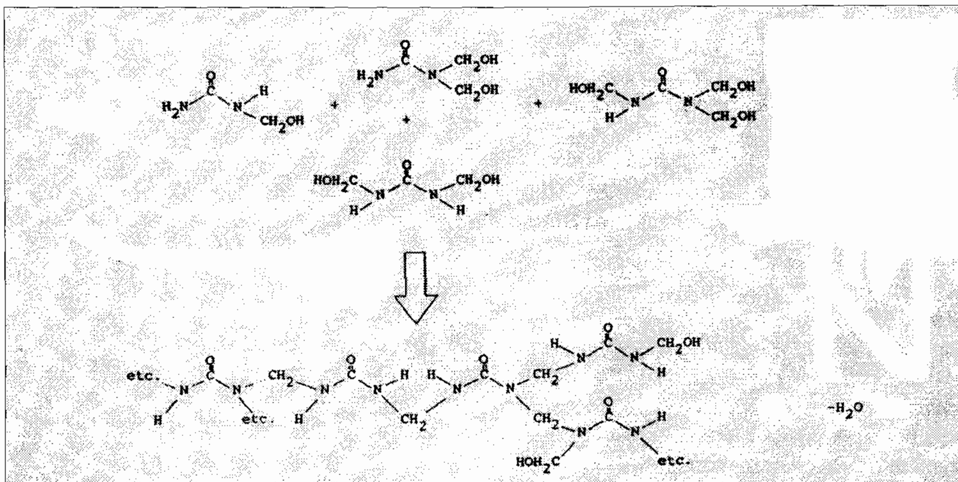

Figure 7 - Chemical diagram for UF (Skeist, 1990)

PF polymerizes in much the same way as UF. For each methyl hydroxy group attached to the benzene ring that reacts with the ortho or para hydrogen on another ring, water is released and the chain lengthens (Skeist, 1990). Again, there is no chemical bonding to the wood, just weaker hydrogen bonding (Figure 8).

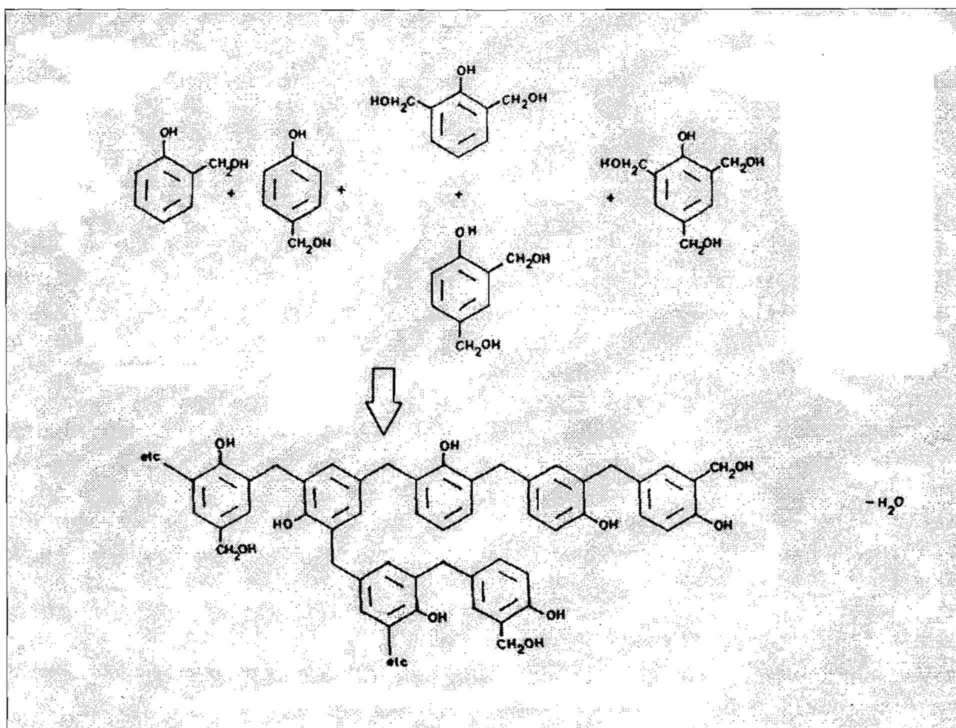

Figure 8 - Chemical diagram for PF (Skeist, 1990)

MDI is very reactive, combining with hydroxyl groups and water. Given the chance, it will also react with some metals. It actually consumes water during its reaction phase and in doing so releases CO_2 (Figure 9). The –N=C=O functional group reacts with hydroxyl groups on water and wood, forming strong chemical bonds (Skeist, 1990).

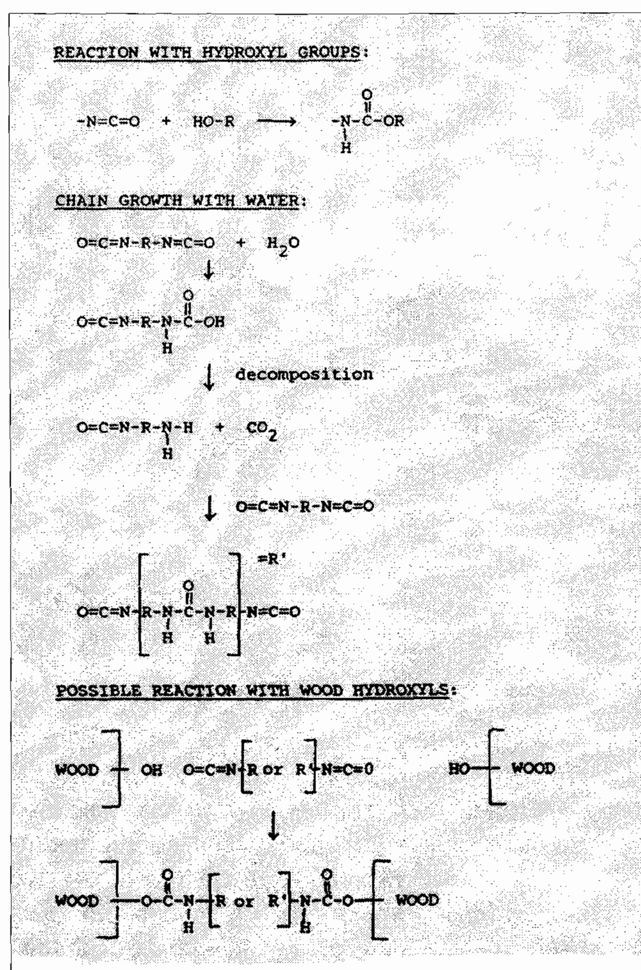

Figure 9 - Chemical diagram for MDI
(Skeist, 1990)

IB Value Representation

Looking to other authors, (Humphrey, 1999) IB data seems to follow a very consistent pattern of linear regions (Figure 10). (The bond strength data in that figure is not the same as IB strength, but the displaying principles are the same.)

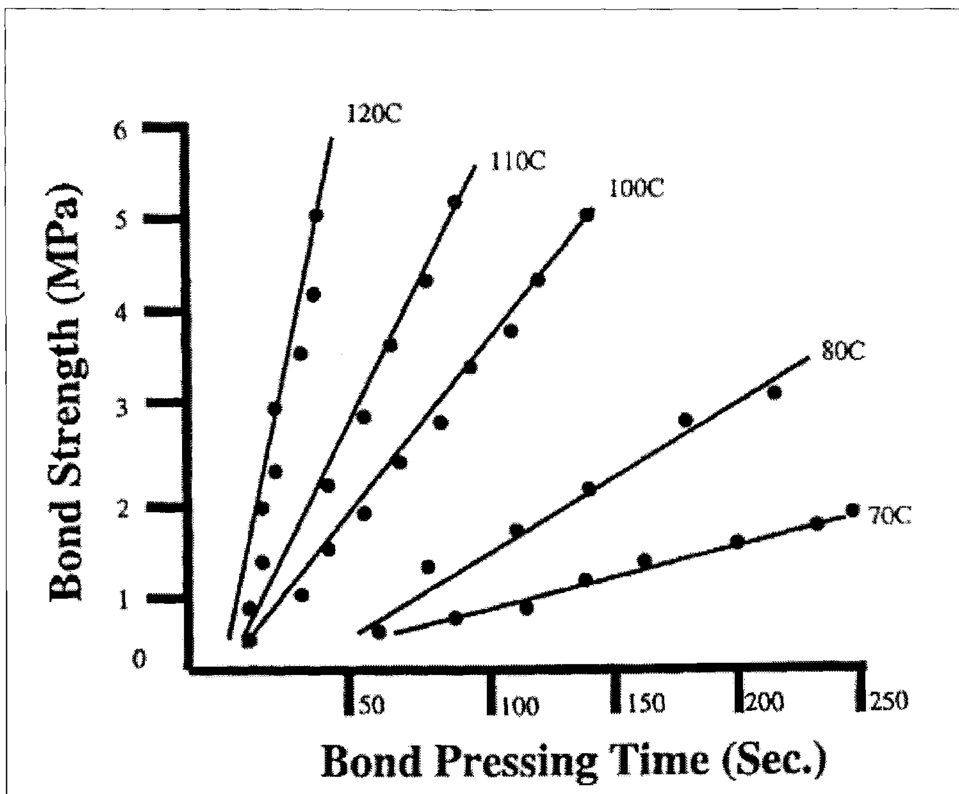

Figure 10 - Standard representation of IB data (Humphrey, 1999)

Patents Pertaining to Dielectric Curing and Cure Monitoring

Many patents exist based on the technology of dielectric monitoring. In general they pertain to cure monitoring of polymeric materials. In 1988, David Day and Marvin Bromberg received a patent on a monitoring apparatus for polymeric materials. A non-contacting capacitance probe for dielectric cure monitoring was patented by Paul Gammell in 1995, again not specifically for any one type of polymeric material. Steven Liang and Joseph Urquhart-Foster

(1996) patented a device that senses dielectric properties in a thermoplastic winding process.

Dielectric patents abound not only in monitoring, but curing as well. For example, George Harris et.al. (1999) patented a means of curing adhesives in engineered wood products using microwaves. Andrew Zsolnay et. al. was granted a patent in 1983 for a system that cures polymeric materials.

These patents all operate on the principle of dielectrics, and are for monitoring and/or curing polymers. Not one patent surfaced in the search for a dielectric cure monitor as it specifically applied to in-situ manufacture of wood composite panels.

Principles of Dielectrics

Definitions

A dielectric is simply defined as a poor conductor of electricity but an efficient supporter of an electrostatic field. While the poor conductivity aspect of a dielectric keeps electric current to a minimum, the material itself polarizes in an electric field, maintaining the electrostatic lines of flux. Thus energy can be stored in a dielectric material, making capacitors possible (Cutnell and Johnson, 1998).

Generally, conductors have a range of resistance between 10^{-8} and 10^{-5} $\Omega \cdot m$, semiconductors between 10^{-6} and 10^{+9} $\Omega \cdot m$, and dielectrics between 10^{+7} and 10^{+17} $\Omega \cdot m$. (Torgovnikov, 1993) Dielectric measurements are made by applying an alternating voltage to conductive plates on either side of the material under test. This in effect creates a capacitor, with the material as the dielectric.

Calculation of the Relative Dielectric Constant

The configuration of the capacitor has significance to any readings obtained. Capacitance readings are dependent on the area of the plates, the distance between them (assuming it is constant simplifies the model), the frequency at which the measurement is taken, and the RMS voltage applied to the cathode. The relationship between these variables is as follows: (Cowan, 1968; Cutnell and Johnson, 1998)

$$C = \frac{Ic}{2 \bullet \pi \bullet F \bullet V}$$

Where

C = Capacitance of system (F)

I_C = Capacitive current (A)

F = Frequency (Hz)

V = RMS Voltage (V)

A number of properties are measured by this configuration: loss tangent (tan delta), relative permittivity, and relative dielectric constant. The two fundamental response measurements are resistive current and capacitive current. These currents are represented in the Cartesian System by a complex number, with resistive current (I_R) in the real domain, and the capacitive current (I_C) in the imaginary domain (capacitance is negative and assigned to quadrant IV by convention). The angle between them is the phase angle, delta (or δ, see Figure 11). In a capacitor filled with a lossy (high degree of resistive current) dielectric, a portion of energy is converted into heat and dissipated. This portion of energy lost is the loss tangent and is a ratio defined as: (Torgovnikov, 1993)

$$\tan\delta = \frac{I_C}{I_R}$$

Where

I_R = Resistive current

I_C = Capacitive current

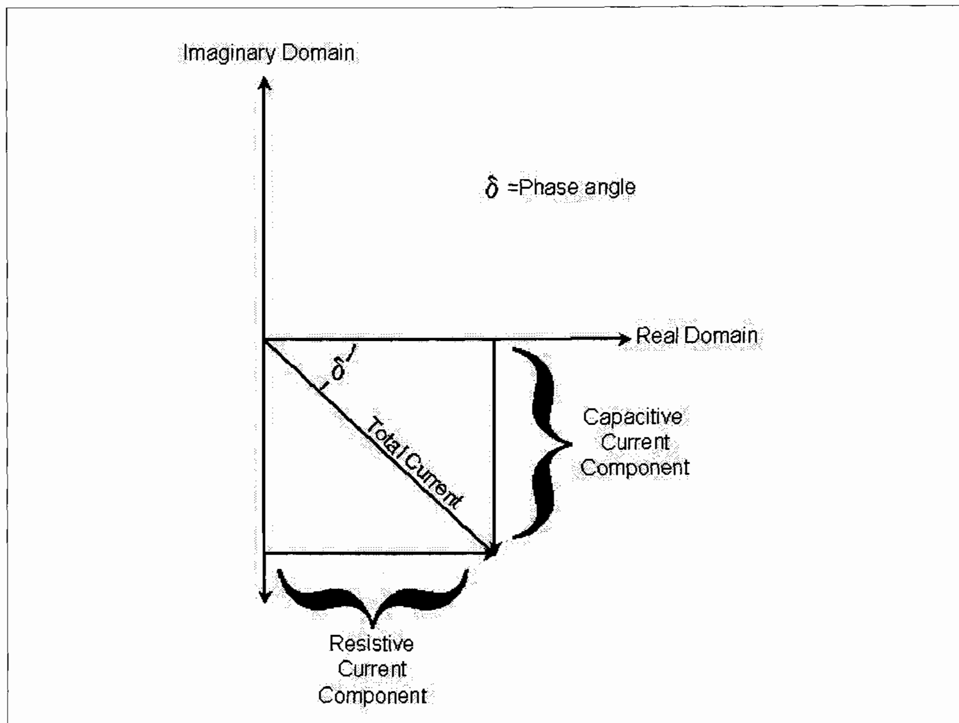

Figure 11 - Cartesian representation of capacitive and resistive current components

The relative permittivity of a dielectric is a ratio of the capacitor system with the dielectric between the plates to the same capacitor filled with a vacuum.

$$\varepsilon' = \frac{C}{C_0}$$

Where

ϵ' = permittivity (unitless)

C = Capacitance of system (F)

C_0 = Capacitance of vacuum (F)

While electrical engineers call ϵ' relative permittivity, in the field of physics it is synonymous with and will be referred to in this paper as the relative dielectric constant (Daniel, 1967). In order to calculate ϵ', C_0 must be known. This can be calculated by:

$$C_0 = E_0 \bullet \frac{A}{d}$$

Where

$E_0 = 8.85*10^{-12}$ (F/m)

A = Area of a capacitor plate (m^2)

d = distance between plates (m)

Thus by knowing the area of the capacitor plates, the distance between them, the angle between the capacitive current phase and the resistive current phase, and the voltage, one can calculate the relative dielectric constant of the material in question.

$$K_e = \frac{\dfrac{I \bullet \cos(\pi/2 - \theta)}{2 \bullet \pi \bullet F \bullet V}}{E_0 \bullet \dfrac{A}{D}}$$

Where

I = Total current

θ = Phase angle

F = Frequency (Hz)

V = Voltage across capacitor

E_0 = physical constant

A = Capacitor plate area (m^2)

D = Distance between plates (m)

K_e = Dielectric constant

For this study the dielectric reading obtained by the system was tan delta, obtained by taking the tangent of the measured phase angle (phase difference between capacitive and resistive current waveforms) in radians.

With any capacitor, there are two methods of applying voltage: static and alternating. With static voltage, a constant charge is applied to the cathode of the capacitor, and it charges up. Once the source is removed, the capacitor discharges at a rate depending on the material between the plates. A lossy material will allow a faster discharge.

The other application method involves a voltage that oscillates between equal positive and negative charges. The frequency of the alternating voltage used can have a cycle time that is slower than or faster than the time required to charge the capacitor to a predetermined percent. For example, if it takes a capacitor 1×10^{-3} seconds to reach 95% of full charge with a static charge, but you apply an alternating voltage at 2 KHz, the capacitor can only reach about 50% of full charge before the voltage reverses and it discharges. Increasing the applied voltage frequency reduces the percent the capacitor charges, and reducing the frequency increases it.

Molecular Action in Changing Electric Fields

Dielectric monitoring involves quantifying the phase lag between the capacitive and resistive currents. Following is a brief description that will assist in understanding what happens at the molecular level during hot pressing of composite panels.

Wood is a polymer matrix comprised of a multitude of compounds. Water is bound up in the matrix (cell walls), the amount dependent on environmental conditions. Present in wood substance are four primary types of molecules: cellulose, hemicellulose, lignin and water. When an electric field is applied to this matrix, the dipolar molecules have a tendency to align

themselves along the electrostatic lines of flux in accordance with their polar charges, as described in Figure 12 (Yalof and Brisbin, 1973).

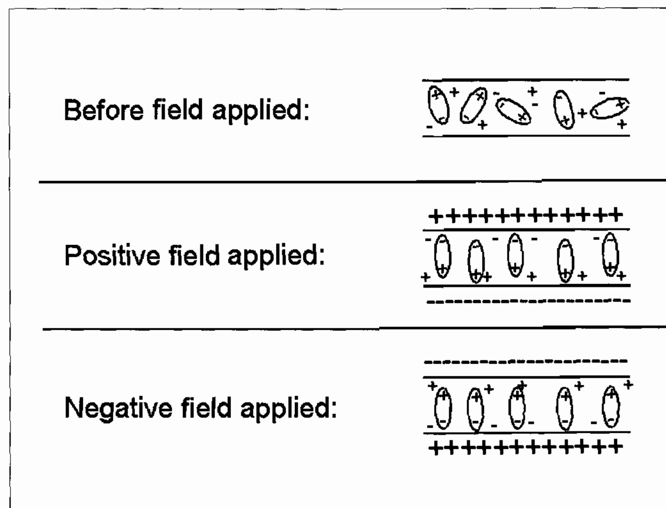

Figure 12 - Ionic and molecular orientation in response to alternating electro-magnetic field (plates show relative charges)

The time required for a molecule to reorient (reaction time) is finite and specific to each type of molecule. Different molecules have different relaxation times, and in a heterogenous material such as wood, this affects the overall readings.

In addition to simple molecules in the polymer matrix are ions. Present in differing quantities from one adhesive to another, ions are charged particles that migrate toward the plate of opposite charge subject to the alternating field

(Figure 12). The mobility of these ions is dependent on the structure of the matrix, the type of molecules present and the strength of the electrostatic field (to name a few factors.) It is also governed by the frequency at which the field is alternating, as the ions travel a finite amount of distance over a finite amount of time.

As molecular relaxation time and ion mobility is time-dependent, the frequency at which the field is oscillating will have a direct impact on the readings obtained. For example, if the field is reversing polarity 10 times per second (Hz) the molecules have sufficient time to reorient and the ions will progress a greater distance. If the field polarity is changing at 100 KHz, the molecules have insufficient time to completely align themselves before the polarity changes again, and the ions will progress a proportionately shorter distance before changing direction. Thus two electrostatic fields of identical strength but differing frequencies will yield phase lags that are not identical. For purposes that will be addressed in another section, this experiment will restrict itself to a single test frequency.

Parameters Affecting the Dielectric Constant

As the relative dielectric constant is a combination of all materials in the capacitor, it stands to reason that every type of molecule and the proportions thereof between the plates contribute to the reading. Being a composite material, particleboard furnish contains wood, adhesive, and moisture. The

dielectric constants of each of these vary. Dry wood ranges from 2.0-3.0 (Tsoumis, 1991), adhesive from 3.5-6.9, and the dielectric constant of water is 80.4 (Cutnell and Johnson, 1998).

PROBLEM STATEMENT

Current Methods to Manufacture Particleboard

Composite panels have been manufactured for decades from the first plywood plants to the invention of particleboard and medium density fiberboard (MDF) and more recently, oriented strand board (OSB). In manufacturing each product, adhesives are necessary to bind the wood fibers together and maintain physical integrity through conditions specified by their respective grading agencies. Phenol-formaldehyde, urea-formaldehyde, and isocyanate are the primary adhesives used to do this.

Each of these adhesives is thermosetting (requiring heat to cross-link), and requires different conditions to cure to optimum strength. These conditions are correct temperature, pressure, pressing time, amount of adhesive, and amount of water present in the furnish. If these are fulfilled, the adhesive can maintain panel integrity according to the requirements of the respective grading agencies. If the panel is subjected to excess heat or time, the manufacturer loses productivity. With insufficient heat or time, the adhesive does not fully cure, resulting in a substandard product.

In order to guarantee sufficient adhesive cure, it is standard procedure to follow schedules that are conservative, overestimating the length of time to cure. This results in inevitable extra costs and lost productivity. Press

schedules are developed recursively, with estimated parameters and an included margin of safety. Panels are produced and tested at the point of cure when the press was opened. Adjustments are made such as length of press time, and the process begins again until the schedule fits the factors suitably. The resulting schedule is valid (except for the lost time, labor and material used in development) until a variable in the process changes. If material density changes, platen temperatures drift, moisture content of furnish changes, etc.; the schedule is no longer optimized for the new conditions and an entirely new schedule must be developed. This translates into time lost developing the new schedule and substandard products manufactured under sub-optimal conditions. It also means greater variation in material properties between panels.

Potential Benefit of Dielectrics

Despite this shortcoming, there is little application of real-time monitoring in industry. Possible explanations of this are that new technology has been either too expensive or simply under explored for practical application. Ideally, a process such as press operation would not be governed by schedules that attempt to guess cure times but would rather monitor actual cure progression, using feedback control set to predetermined limits.

The technology to do this has existed for decades. Dielectric cure

monitoring has been successfully applied in a number of industries including plastics and adhesives for bond monitored products (Day and Bromberg, 1988; Kranbuehl, 1997). As it measures direct material properties, it could theoretically be used to monitor the degree of adhesive cure for any wood-based panel product currently in production. Thus, a computer or operator would know the moment a panel has reached sufficient internal bond strength due to adhesive cure, and end the cycle at that point. This would not only guarantee optimized bond strength development, but would also save time and improve efficiency for the process in general. Both advantages would mean cost savings for the manufacturer.

EXPERIMENTAL DESIGN

Preparation Considerations

This study is focused on the manufacture of particleboard (as opposed to plywood, medium density fiberboard (MDF), or other types of composite wood panels) because it is relatively easy to simulate industrial conditions for manufacturing particleboard. MDF raw material is more difficult to produce in the laboratory (density variations in the panel are more extreme and a tumbling drum is ineffective for spraying moisture and adhesive), as is oriented strand board (OSB). OSB is a difficult material to use to manufacture small panels with any degree of consistency.

The experimental design of this project relied on defining many variables, deciding which to hold static, and which to vary. Those that were to be varied required a degree of experimentation, to determine their variability. The primary objective of the experimental design was to eliminate as many sources of variability as possible, while yielding data that would be valuable in reaching valid conclusions.

As the technology from this project is intended to be potentially applicable to industrial processes, it was important to maintain parameters similar to real-life pressing operations. Ideally, a full-size hot press would have been used, and a completely non-intrusive sensor designed, such that

the process of manufacturing particleboard could be duplicated exactly. The experiments were performed in a laboratory, and a 91 cm x 91 cm computer controlled hot press was used. The electrical sensor setup used was in addition to the standard caul plates (aluminum sheets used to maintain mat integrity until press closes), which impeded heat transfer into the panels. The delayed heat transfer effect of the sensors was the same for every board manufactured, thus their contribution was ignored, and the overall readings understood to be a relative value. This allowed for the potential of improved sensor design in the future, expecting to see similar (but not duplicate) results as those of this project.

This project was designed as a general overview of the potential of the system to the composite panel industry, and analyzed the three adhesives used in manufacturing particleboard - PF, UF, and MDI. Particleboard was selected as the primary focus due to its homogeneity, and its relative ease of manufacture. Every panel was designed to have a density of 720 kg/m^3 (45 lb/ft^3) and a thickness of 12.7 mm (.5 in). The platen temperatures during pressing for the three adhesives were 160°C for UF and MDI, and 205°C for PF.

Heat and Moisture Contribution to Dielectric Reading

A preliminary study was conducted to determine the effect that heat and moisture content had on the dielectric reading. Particleboard furnish was oven dried and formed into mats with thermocouples in the center. They were then pressed at either 160°C or 205°C for about 800 seconds and monitored for RTD and temperature. Next, loose furnish was hydrated to the same moisture content as the boards with adhesive would be (10% MC), with a thermocouple in the centerline and pressed. The resulting RTD was again monitored and recorded, yielding temperature and RTD versus time curves.

A Process to Determine IB Strength

All IB strength measurements were assigned time values based on the amount of time spent curing. Timing began at the point the press closed, providing an absolute reference.

In order to obtain IB strength values during adhesive cure, boards had to be made in specific time increments. That is, if a board had to be cured for 130 seconds (from the point of press closure), the time the adhesive spent at an adequate temperature had to reflect that as accurately as possible. To obtain such results, the timer would begin at the time zero reference, the board remained in the press until 130 seconds had passed, and then the press promptly opened. As each of the three adhesives used was

thermosetting, they would continue to cure even when removed from the press. Dependent on the heat transfer properties of the wood, it could take ten minutes or more for the center of the board to drop below the necessary temperature (80°C was chosen to be the point that adhesive cure slowed sufficiently - see Figure 13). A technique needed to be developed in order to quickly remove heat from each board as they came out of the press without damaging effects to panel properties.

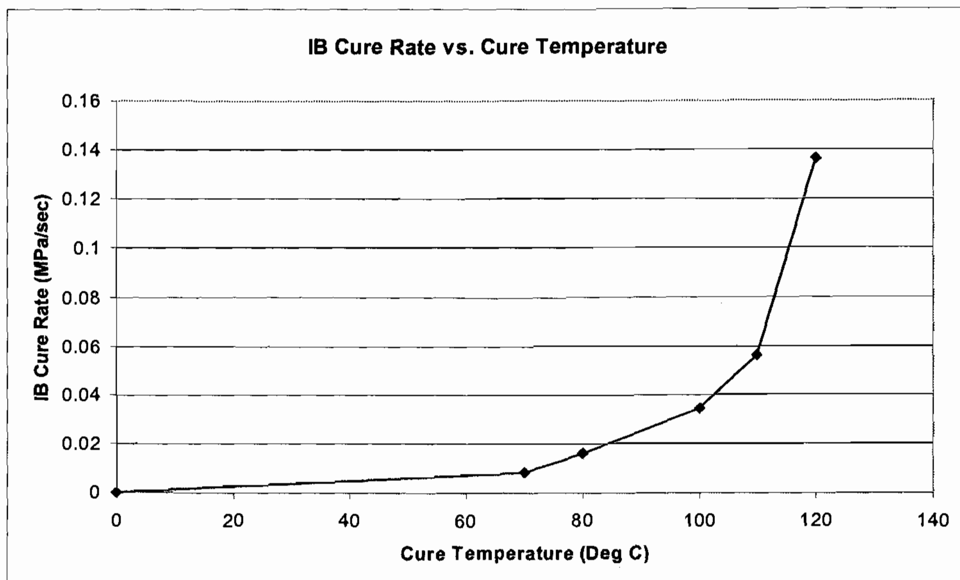

Figure 13 - IB cure rate data plotted against cure temperature for UF (derived from data reported in Humphrey, 1999)

Heat Removal Challenge

This was a formidable problem, requiring experimentation to determine how best to accomplish this task. Explored were ideas such as directing

high-pressure air over the top and bottom of the panel, placing the panel in a freezer, applying frozen aluminum plates to both sides of the panel, and different applications of liquid nitrogen (N_2). The construction of a polystyrene box sealed with silicone adhesive and filled with N_2 proved to be the fastest method of removing heat without damaging the board (Table 1).

Means of heat removal from Centerline	**Centerline Rate of heat removal (°C/sec)**
Polystyrene box containing N_2	-1.1506
Liquid N_2 poured on board	-1.113
Liquid N_2 heat exchanger 100 psi	-0.7091
Pressurized air in wood box	-0.4761
Frozen Al plates pressed to sides of panel	-0.4103
Freezer, with moving air	-0.3062
With pressurized air	-0.2837
Liquid N_2 with heat exchanger 35 psi	-0.2505
Liquid N_2 air cool	-0.1722
Freezer, no moving air	-0.1349
Air cooled	-0.0603

Table 1 - Summary of methods to remove heat from panels

After finding the fastest method of removing heat, it remained to be determined whether the application of N_2 directly to the panel affected

strength characteristics. Two boards were manufactured–remaining in the press for 800 seconds to ensure full cure, one allowed to cool in a freezer and the other submerged in N_2 in the polystyrene box. The boards were cut into 16 samples each and IB tests (according to ASTM D1037-99) were performed on all 32 samples. The results of that test are in Table 2. The P-value was .0166, and as .05 or less is accepted to indicate significant difference, some

Paired two sample t-test for means		
Internal Bond Strength	Freezer-Cooled Board	N_2 Cooled Board
Mean (MPa)	1.0049	0.8971
Variance	0.0161	0.0211
Observations	16	16
Hypothesized Mean Difference	0	
df	15	
P-Value	.0001	

Table 2 - Statistical results for paired two sample t-test of samples from two panels to determine significant difference

strength loss (about 11%) occurred from this technique. As adhesive levels were such that ultimate cure strength for each fully cured panel was far higher than required, it was determined that this method was the best. Rate of heat removal seemed more important than obtaining even higher strengths from

the panels. Every board tested for strength in this experiment was cooled by the method described above, and it was acknowledged that each one could have a slight strength loss as a result. That was not important, as the idea of this project was to determine whether bond strength formation could be monitored at all. The underlying principle is the same, and relative strength values were far more important than absolute numbers.

IB Data Presentation

Solving the heat removal issue was vital to this experiment. While the fastest method feasible was developed and utilized, there remained a period of time between the point of panel removal from the press, and when the core of the board had cooled to sufficiently slow the adhesive curing. In short, the panels did not stop curing immediately upon removal from the press, but rather cured for a period of time until the heat in the core was lower. The N_2 process served to minimize this lag time, but could not eliminate it. Therefore, every IB sample measured had 77 (UF and MDI) or 95 (PF) seconds added to the time it spent in the press, depending on the adhesive used. This adjusted for the time the adhesive actually spent curing with sufficient heat in the panel.

Reviewing the available literature, this has not been applied before, so in theory it should prove a more accurate means of presenting data.

Board and Sample Number Determination

Next, the number of panels for each type of adhesive had to be defined. Based on past experience, the window of likely cure (time period that IB strength increases from 0 to maximum) was estimated to be 240 seconds in length.

As little data was found regarding cure times for adhesives at different temperatures, an estimate was made as to when the cure began. This was expected to occur at or after 120 seconds following press closure. Twenty UF and MDI boards, and 28 PF boards were made in increasing 5 second increments after that 120 second point for each adhesive. Two boards of each adhesive were cured for 800 seconds and tested for IB strength. A total of 74 boards were tested for IB strength for the entire experiment.

Sixteen test specimens per panel were taken from the center of each panel to determine average IB. To determine the sample size per board, statistical help was enlisted. Rogelio Decasa and Vicente Monleon, both graduate students in the statistics department, acted as consultants on this project. Based on a preliminary study in which all 16 samples were broken from two boards, a 95% confidence interval was calculated depending on the number of samples tested. If all 16 samples of a panel were tested and the mean value calculated, a 95% confidence interval of +/-0.02275 MPa was the result. If four were tested, a 95% confidence interval of +/-0.03999 MPA resulted. As the larger confidence interval represented about 9% of the

industrial strength requirements as opposed to 5% for the smaller confidence interval, the larger confidence interval was accepted and four samples per panel were measured for IB strength.

A random number generator was used to select the order in which each panel was pressed within adhesive types. This was done to eliminate any built-in error due to sequential manufacture.

Materials

The material used in this study was core furnish from the Duraflake Division of Willamette Industries, Inc., and consisted of an unknown mix of western softwood species. It was obtained directly from the dryer at about 8% MC and was subsequently dried to ovendry in a laboratory oven.

For this project the reasoning behind oven drying the furnish lies in the fact that three different adhesives were studied. Each adhesive applied contributed differing amounts of moisture to the material, and it was important that the total moisture content of the formed mats entering the press were all at 10% MC. It was important to keep the total MC of the mats the same as moisture contributes such a large amount to the dielectric reading. Moisture was not a variable in this study. Thus the furnish was dried and rehydrated to a moisture content particular to the type of board being manufactured.

The panels and adhesives had the following properties (Table 3):

	Property	UF	PF	MDI
Panel Properties	Board Dimensions	30 x 30 x 1.27 cm (12 x 12 x ½ in)	30 x 30 x 1.27 cm (12 x 12 x ½ in)	30 x 30 x 1.27 cm (12 x 12 x ½ in)
	Target Board Density	720 kg/m^3 (45 lb/ft^3)	720 kg/m^3 (45 lb/ft^3)	720 kg/m^3 (45 lb/ft^3)
	Wood Furnish	Core, Duraflake	Core, Duraflake	Core, Duraflake
	Stratification	None	None	None
	MC % of Mat into Press	10	10	10
Adhesive Properties	Press Temperature to Cure	160°C	205°C	160°C
	Adhesive solids % based on Oven Dry Wood Weight	8	8	5
	MC % of Mat Prior to Adhesive	6.2	4.4	10
	Adhesive % Solids	65.5	57.1	100
	Adhesive Manufacturer	Neste	Georgia-Pacific	Huntsman

Table 3 - Summary of panel and adhesive properties

Methods

Adhesive/Moisture Application

A rotary drum style tumbler was used for uniform application of moisture and adhesive. To apply the liquids, a Binks model 21 air spray gun

was used at 0.24 MPa (35 psi) and an application rate of 80 g/min. Care was taken to administer the same amount of liquid from board to board, as variation in those amounts would potentially cause large fluctuations in the dielectric signal. Deionized water was found to result in a more consistent signal for the same amount of water in a particular panel and was used exclusively.

Press layup

Prior to pressing, 30 x 30 cm (12 x 12 in) mats were formed via a homogenous layup box. All boards were formed to a target density of 720 kg/m^3 (45 lbs/ft^3) and pressed to a thickness of 12.7 mm (½ in). The layup configuration of the caul plates, insulating layers and capacitor plates is shown in Figure 14.

The press used was computer controlled by displacement, programmed to close to the same panel thickness at the same rate for every board. This ensured a consistent density profile from one board to the next. The top and bottom caul plates were 4.0 mm thick aluminum. Between the caul and capacitor plates was a sheet of .80 mm Teflon© (Polytetrafluorethylene, PTFE), serving as an electrically insulating layer. This was necessary as the top and bottom platens shared a common electrical ground. The capacitor plates were the same material as the caul plates, cut to 30.5 cm by 30.5 cm.

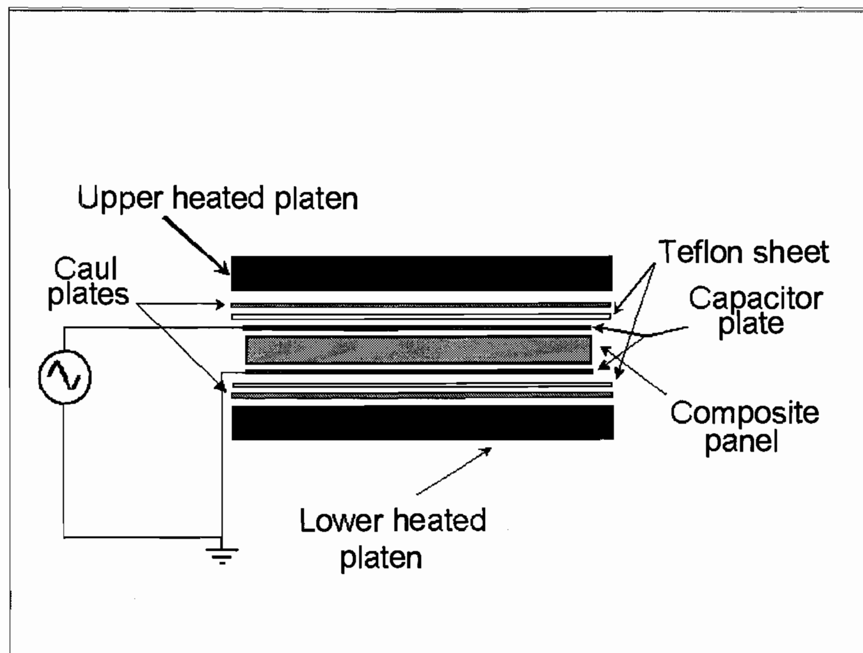

Figure 14 - In–press monitoring setup

Description of Monitoring System

In a perfect world, the system developed for monitoring dielectric properties would quantify the phase angle between the alternating voltages, calculate the tan delta, and display that value for control purposes. It would connect to the two leads of the in-press capacitor, and determine the actual tan delta value of the material between the plates. As no such system was known to exist at the start of this project, it remained to construct one to the specifications required by the materials to be tested.

In reality, constructing a system such as this requires the use of cables, imperfect resistors and capacitors that have inherent error, and environments that have substantial electromagnetic (EM) noise and require filters (see Figure 15). The use of shielded cabling served to reduce the induced signals from the environment, but added capacitance as they are simply two conductors separated by a dielectric material. Proper grounding techniques

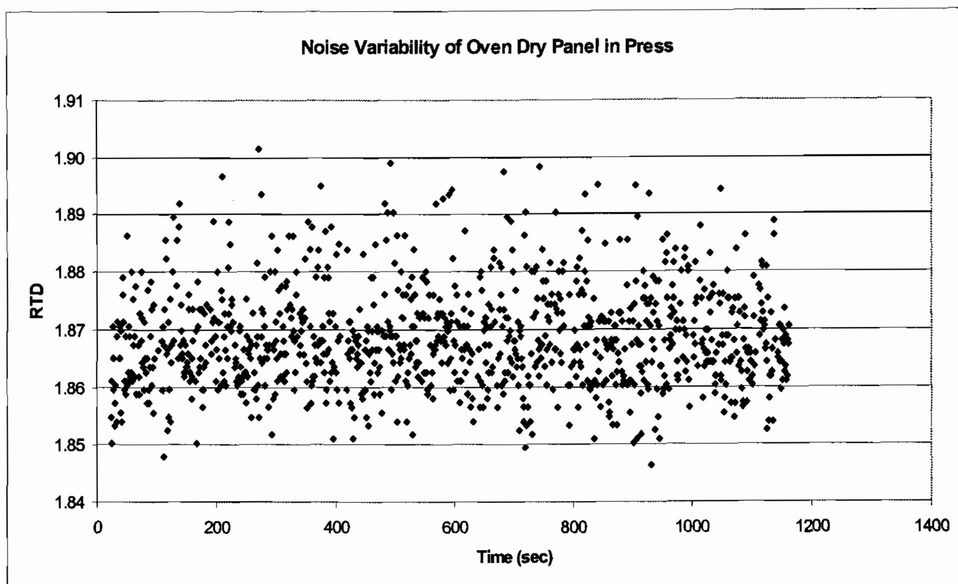

Figure 15 - Inherent system noise of an oven dried panel in the hot press (UF-MDI circuit setup)

were followed with the cable shielding. All of these factors (cabling, data acquisition card, EM noise) added unknown amounts of resistance and capacitance, which offset all readings made by the system. This rendered the phase angle reading into a relative value, useful only for direct comparison to

other values made by this system. All values reported in this project are in units of relative tan delta (RTD). It is still possible, however, to look for general trends in the data and isolate different characteristics that indicate reaction to chemical events. In that respect, it is worth comparing to results obtained by other authors.

Electrical Monitoring Circuits

The electrical circuits used to make the dielectric measurements were custom designed for this project. Each adhesive had its own particular electric characteristics and required different circuit designs in order to stay in range of the system. Following is a description of the circuit requirements for all three adhesives.

Common to all adhesives was the necessity to build a voltage dividing circuit. The purpose of the voltage divider was to provide monitoring capability for both the capacitive and resistive components. The software required both waveforms to be similar in amplitude to successfully regress the sampled data and find the difference between them. A voltage drop was necessary across both the capacitor setup, and a resistor in series with the setup, in order to obtain the necessary out-of-phase waveforms as shown in Figure 16. The resistive component (waveform 1, Figure 16) was sampled at 50KHz across nodes 1 and 2 of Figures 17, 18 and 19, while the capacitive component

(voltage waveform 2, Figure 16) was sampled at 50KHz across nodes 2 and 3 of Figures 17, 18 and 19.

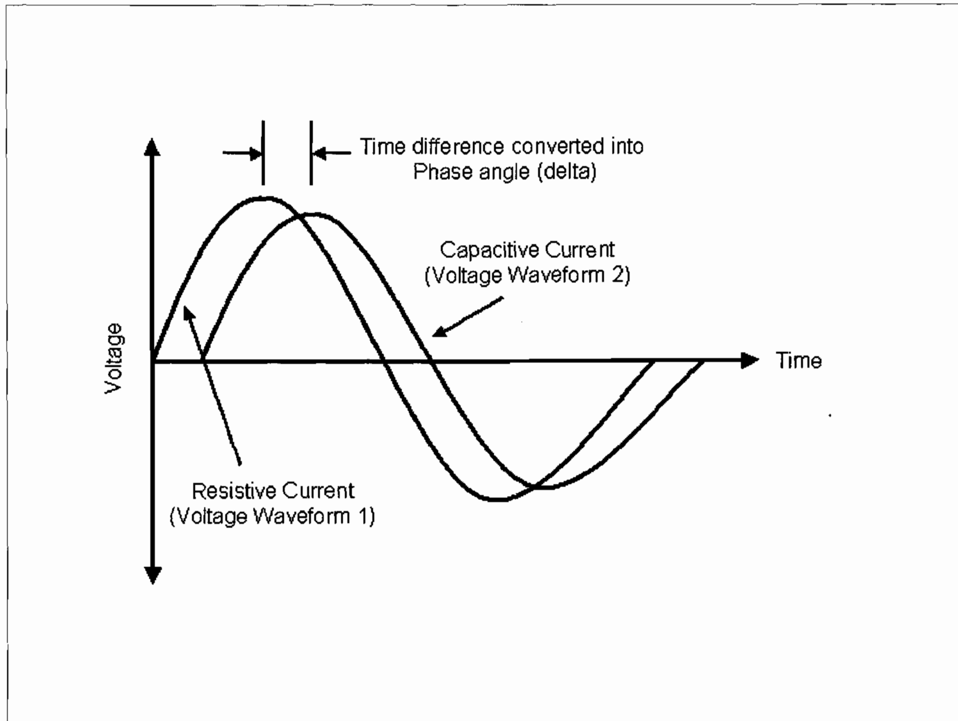

Figure 16 - Resistive and capacitive component waveforms

The software measured the time difference (Δt) between the two waveforms, and using their frequency (100 Hz) calculated the phase angle.

$$\delta = \delta t \text{ sec} \bullet 360 \frac{\text{deg}}{\text{cycle}} \bullet 100 \frac{\text{cycle}}{\text{sec}}$$

Where:

δ = phase angle

δt = time difference between waveforms (sec)

UF Monitoring Circuit

The UF adhesive required the simplest circuit design. With the basic voltage divider circuit, the capacitance and resistance of the panels produced remained within the range of the system (Figure 17). A viable signal was

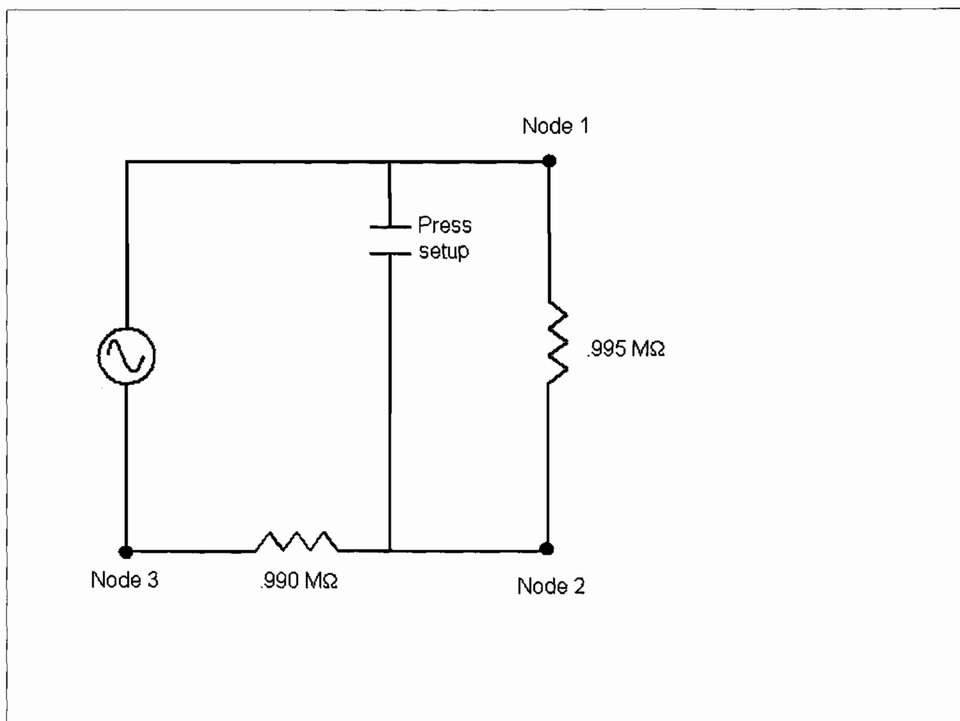

Figure 17 - Electrical monitoring circuit for UF boards

measured from the time the press closed to the time it was opened (the press cycle).

MDI Monitoring Circuit

As the first boards were made, it became evident that the signals were out of range of the monitoring system. The nature of the MDI caused the panels to have different capacitance values. This should have simply offset the RTD by a constant, but this was not the case. A high frequency signal appeared in the 100 Hz cycles, preventing the software from interpreting them into RTD values. The addition of a 1600 pF capacitor between the charged plate and ground successfully filtered out that noise and allowed the software to process the waveforms again (Figure 18). By deduction, the boards made with UF adhesive had enough inherent capacitance that an external filter was not necessary.

At this point it was a concern that the addition of a capacitor for noise filtering would alter the value of the RTD. Further research revealed that adding a capacitor that bridged the entire voltage divider would not alter the capacitance at any point within it. To test this hypothesis, panels at 10% MC were made and monitored under both circumstances, with and without the additional capacitor. With the moisturized panels, adding the capacitor actually reintroduced the high frequency noise to the waveforms. Therefore, no data could be recorded on how the extra capacitor altered the signal. The

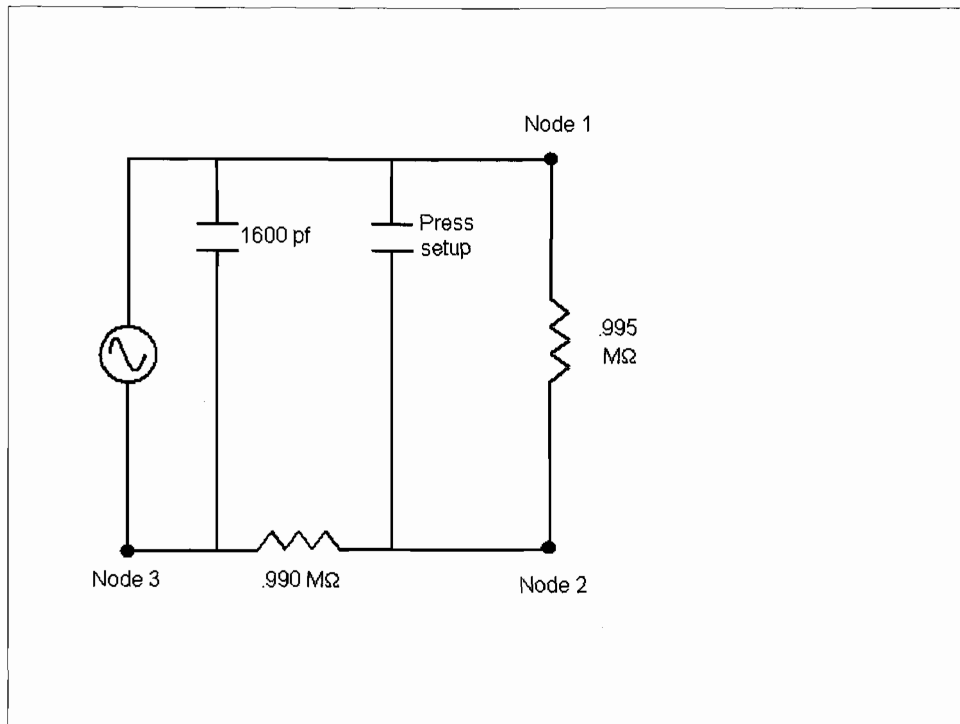

Figure 18 - Electrical monitoring circuit for MDI boards

only comparison that can be made is that the UF and MDI boards approached the same RTD value of about 2.16 at the end of the pressing cycle. This evidence supports the idea that there was no offset between the readings made with the MDI and UF circuits. Circuit theory predicts that adding a capacitor in this function would have no effect on the divider circuit components.

PF Monitoring Circuit

Next, panels were manufactured with PF adhesive and monitored with the existing dielectric circuits. The amplitudes of the two waveforms were so disparate that the software could not regress them and solve for the zero crossings (the point at which waveform amplitudes are momentarily zero), determine the time difference between them and thus calculate RTD. The resistance of PF boards had dropped drastically from those made with the other adhesives. The circuit needed alteration to bring the waveforms back into a range that the software could interpret.

As the exact amount of resistance needed was not known, a potentiometer with a range of 0-5 MΩ was inserted in parallel with the press capacitor to raise the resistance again (Figure 19). After some experimentation it was determined that a value of 3.30 MΩ allowed the software to process both waveforms successfully for the duration of the pressing cycle.

Unlike the capacitor previously inserted for the MDI panels, the potentiometer did change the constants of the system. It had the effect of increasing the resistive current leg of the phase angle/RTD value (see Figure 12). This rendered the numbers recorded for PF boards incomparable to those recorded for MDI and UF boards. Based on that knowledge, the circuits used to monitor the UF and MDI panels will be referred to as the "UF-MDI setup" (as there is no offset between them), and the circuit used to monitor

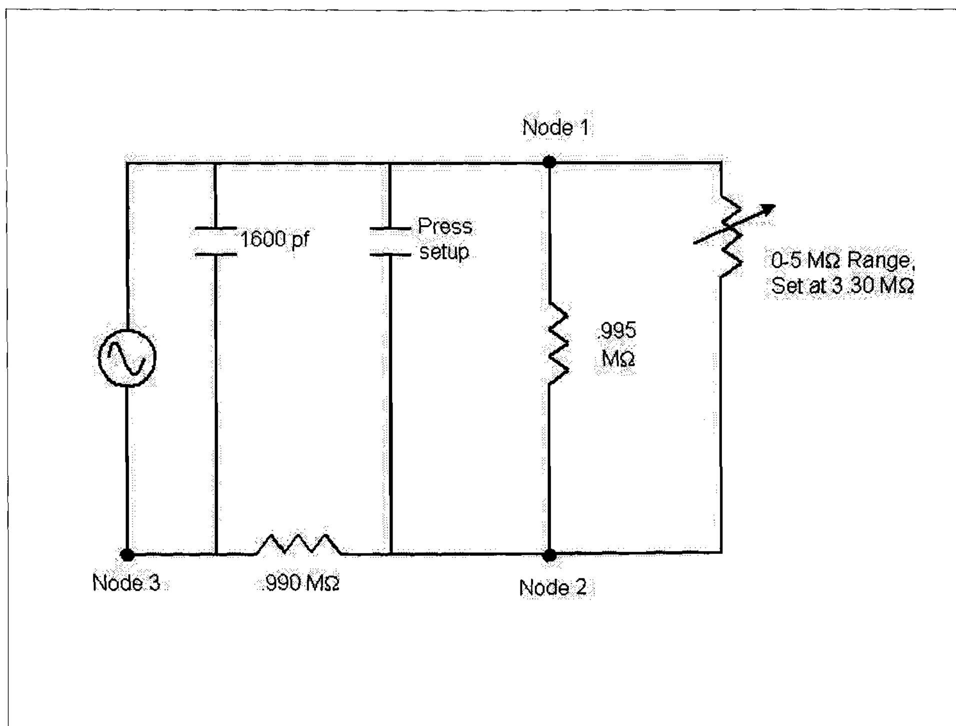

Figure 19 - Electrical monitoring circuit for PF boards

the PF panels will be referred to as the "PF setup." In this way, the offset due to the potentiometer is recognized.

Signal Frequency

The excitation frequency of the system had to be determined before the hardware was obtained and the analyzing/recording software written. Conceivably, a frequency sweep would have been performed, potentially yielding a wealth of data about the spectroscopy of the material as a dielectric. However, to make this project achievable, a single frequency was selected for

sampling. As a general rule, frequencies of 10KHz and lower tend to emphasize the effect of ion mobilization, and frequencies of 10KHz and higher tend to emphasize the rotation of dipoles (Torgovnikov, 1993). Due to the range of tan delta values obtained by James in 1975 by capacitance bridge, the 100 Hz frequency seemed most suited to the values detectable by the proposed hardware and software. A tan delta value of .11 (ovendry hardboard at 100 Hz) was at the lower threshold of the range predicted by a sensitivity study performed prior to the project. Based on that study, 100 Hz was the frequency used in the capacitor to make dielectric readings.

Data Acquisition Hardware/Software

The PC data acquisition hardware used was a National Instruments PCI 7030-6030E. It was a 16 bit card with 16 analog and eight digital channels, with up to 100 kS/s sampling rate, connected to an RT (real-time) board. The RT board contained its own 133 MHz processor and 8 Mb of DRAM. Once the software program was downloaded to the card and initiated, it functioned independently of the operating system. This prevented interference from the Windows environment and would continue to operate as long as power was supplied to the card. The independence of the data acquisition from the PC's operating system was important due to the sampling frequency (the card operated at 50 KHz/channel) and the precision required to obtain meaningful results.

LabVIEW 5.1's graphical user interface language "G" was used to write and run the software on the computer. In essence it sampled the dual waveforms and determined the phase angle between them, saving that data to a file while displaying it in graphical form on the monitor for real time feedback. That data was later converted to RTD in spreadsheet form.

The computer that controlled the hot press also recorded data related to press parameters during the pressing of each board. It recorded such information as top and bottom platen temperatures, press opening position and pressure applied. That data was merged with the dielectric data into one file per board.

Each board was also measured for temperature in the centerline. Thermocouple wire (type T, 36 gauge, Teflon coated) was used to measure that data. The thermocouple was connected to a Campbell 21x data logger which relayed the data to a separate PC. That PC saved the data to a file which was then brought over and merged with the other two data sets for that board. This gave a complete picture of all variables in the process relative to each other.

The variables monitored and recorded for each board are listed below in Table 4:

Process Parameter	Unit
Dielectric value	RTD
Centerline Temperature	°C
Platen Temperatures (Upper and Lower, not reported)	°C
Press Force (not reported)	MN
Press Opening Dimension (not reported)	mm

Table 4 - Summary of process parameters and respective units

IB Strength Setup

An Instron universal testing machine was used to obtain IB strength numbers, along with a 454 kg load cell and Campbell 21x data acquisition system. ASTM D1037-99 was the governing standard for this procedure. Sample blocks were cut to 50.8 mm by 50.8 mm and affixed by hot-melt adhesive to aluminum grip connectors. The blocks were pulled apart at a rate of .48 mm/min. As mentioned earlier, the IB data was shown in simplified graphs of three linear sections (Figure 10).

The sample blocks for IB testing were cut from the panels made in the dielectric setup. The panels were 30.5 cm by 30.5 cm. To ensure quality samples, an area 20.3 cm by 20.3 cm in the center of each board was

designated for use. That area was further sectioned into 16 blocks, 5.1 cm by 5.1 cm each. Figure 20 shows the layout described. The blocks were labeled by board number and assigned a column letter and row number so that the samples selected and tested could be reinserted into a spreadsheet matrix corresponding to their original location in the board.

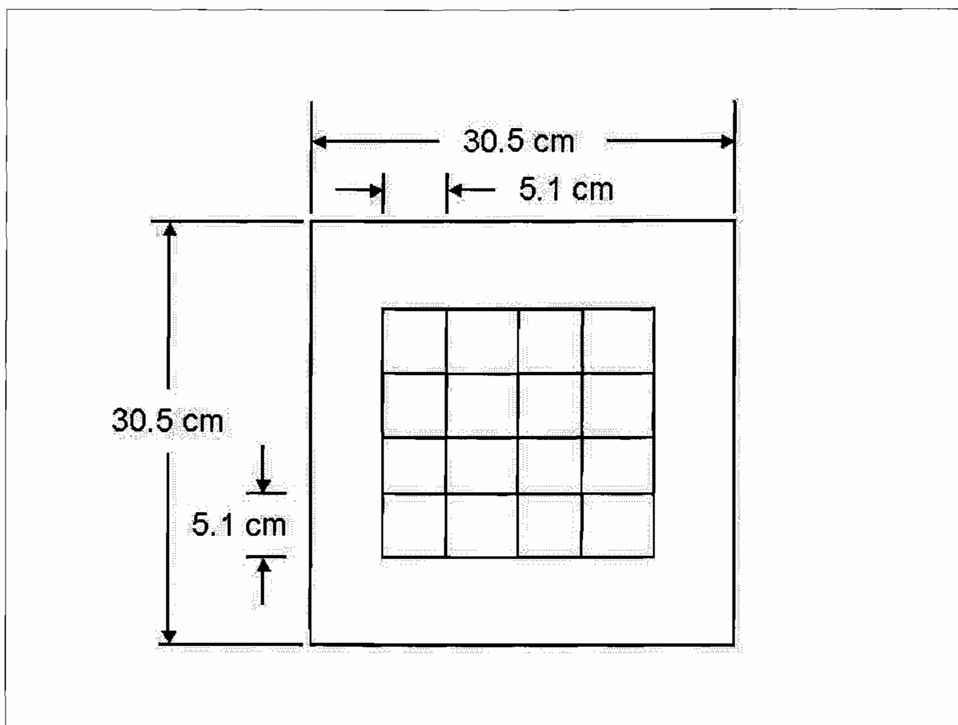

Figure 20 - Sample removal pattern from each board produced

RESULTS

Dielectric Response of Materials Involved

Dry Furnish

Measuring the dielectric properties of the entire panel between the capacitor plates is a composite reading of all material–wood, water and adhesive. Every molecule and ion present is a contributing factor to the RTD reading obtained. The application of heat changes the dielectric properties of wood. Moisture has an enormous influence on the RTD value. The adhesive itself interacts with the wood and water and has its own RTD value at any one point in time. Ideally the response curve monitored would be the adhesive itself without the effects of wood and moisture, but this was not possible. Instead, the dielectric response curves for all three adhesives used must be analyzed to reveal the information desired to determine the state of cure. The effects of the temperature/wood interaction on RTD for the UF-MDI and PF setup circuits with no adhesive are displayed in Figures 21 and 22 respectively. Noting the difference between them serves to support the theory that adding the potentiometer to the PF circuit setup changed the constants of the system.

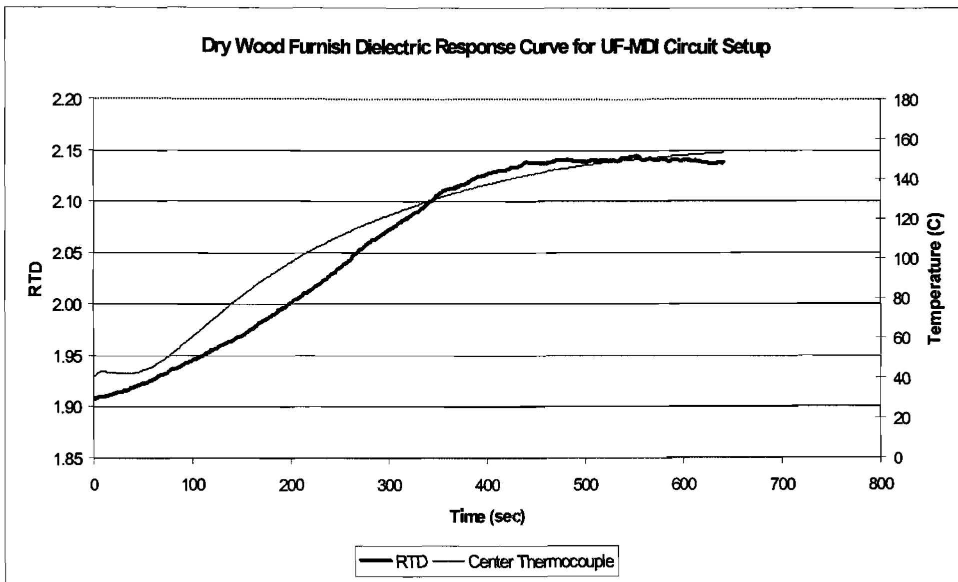

Figure 21 - Dielectric response curve of dry board–no moisture or adhesive, UF-MDI circuit setup

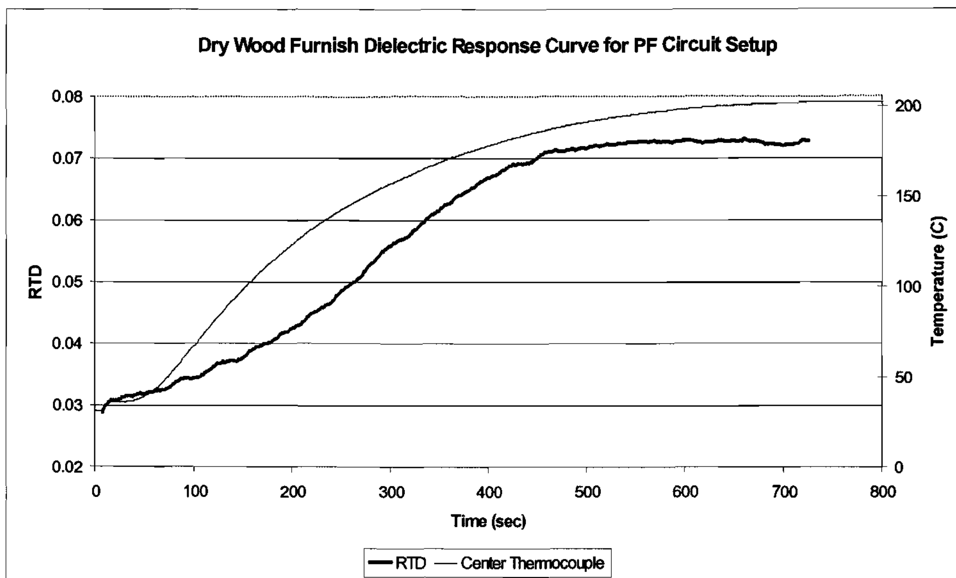

Figure 22 - Dielectric response curve of dry board–no moisture or adhesive, PF circuit setup

Furnish at 10% MC

The dielectric response curves are very similar in shape. This is important in that it shows the two setups are similar. Next the contribution of the moisture in each board is examined. Each type of board with adhesive was engineered to have a total MC of 10%. The amount of water in the emulsion was calculated for UF and PF. MDI was 100% solids, and therefore had no moisture in it. Whatever remained to bring the mixture up to 10% MC was added to the furnish in the tumbler prior to the addition of adhesive. To determine the dielectric response curve of 10% MC, that amount of moisture was added to dry furnish and formed into particleboard without adhesive. It was done for both electrical setups. Figures 23 and 24 show the results of that study.

These figures show a general similarity, both following a pattern of peaking at about 117 seconds. The logical assumption is that the sudden reversal of the curves is due to water flashing into vapor at that point. It would be expected that the point would correspond directly to 100°C on the temperature curve, but instead it matches with about 80°C. This temperature curve, however, is not an accurate representation of the entire panel.

Due to the heat transfer properties of wood furnish, the surfaces heat up much faster than the core of the panel. Eliminated early on in the process

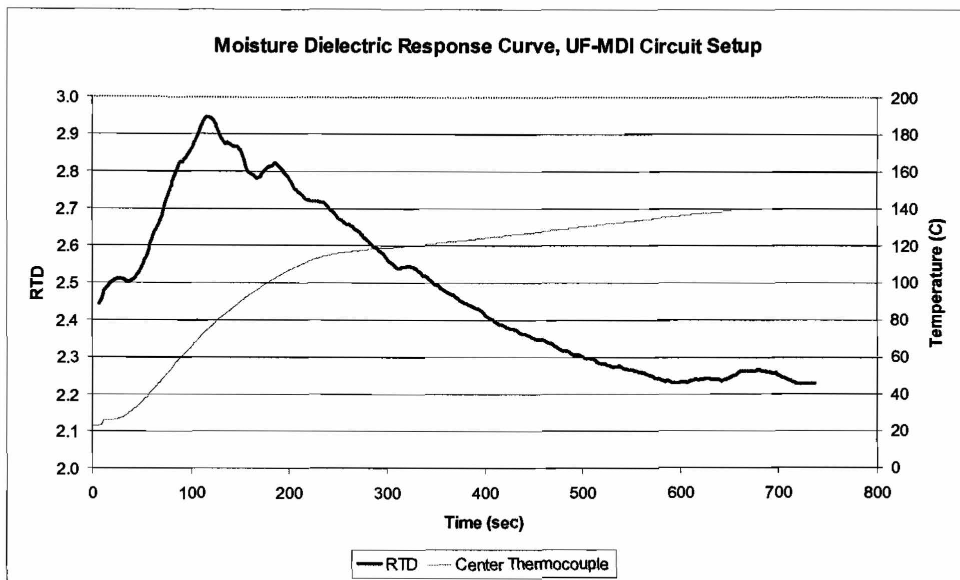

Figure 23 - Dielectric response curve of 10% MC board without adhesive, UF-MDI circuit setup

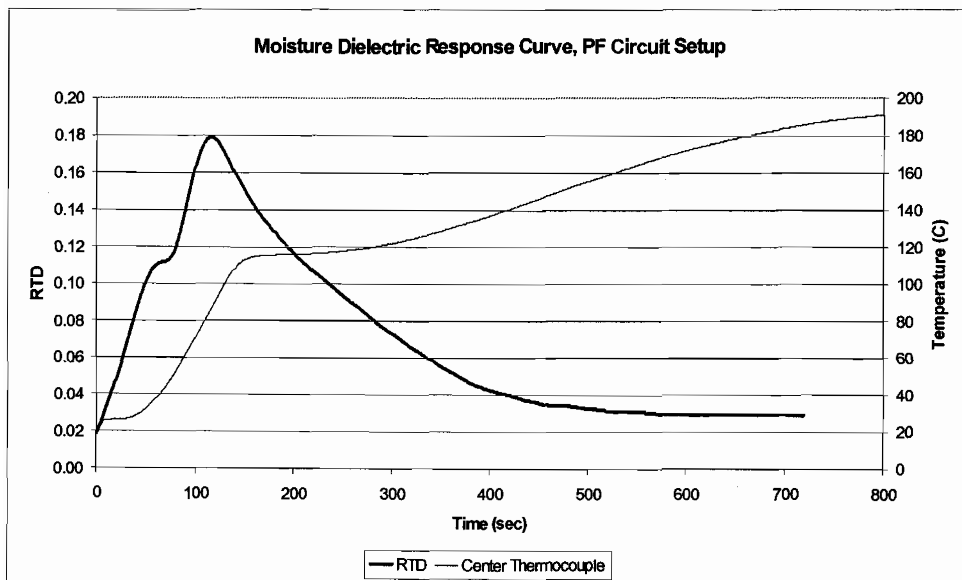

Figure 24 - Dielectric response curve of 10% MC board without adhesive, PF circuit setup

of taking data, were surface thermocouples that gave a broader picture of the heat conditions. It was suspected they interfered with the electric signals, so they weren't used. There is some early data on what to expect from board to board for heat gradient properties, and that is shown in Figure 25. The dielectric response curve spike occurs at 80°C at the centerline, but the panel surfaces are at 130°C (due to 8 mm of aluminum caul plates and .84 mm of Teflon sheeting inhibiting heat transfer from the 160°C platens to the panel surface). Water flashes into steam beginning at the surface, progresses inward, and when the ratio of vapor to liquid water reaches a critical point (it happens to be at the 80°C centerline point for this thickness of board, species, density, etc.) the contribution of the vapor to the dielectric response curve

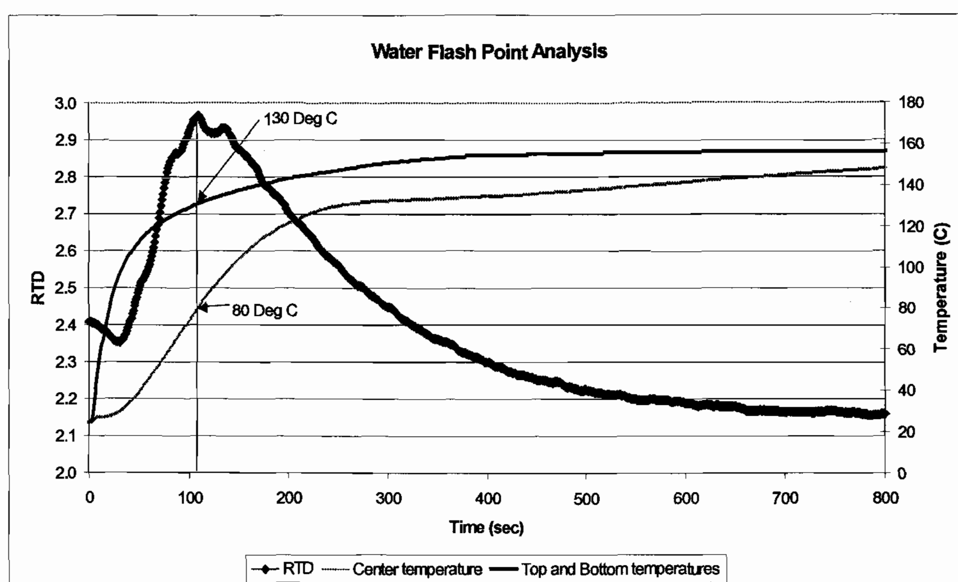

Figure 25 - Top, center and bottom temperature data with RTD

dominates the liquid water component. At that point it reverses direction and begins falling.

The rate at which the RTD of PF setup-monitored boards falls (-.000433 RTD/sec at 250 seconds) is different from the rate at which the RTD of UF-MDI setup-monitored boards falls (-.002063 RTD/sec at 250 seconds). This is likely due to differences in target temperatures and to a lesser degree, offsets in the system. The PF setup moisture dielectric response curve also has a characteristic inflection point at about 71 seconds and 41°C at the centerline. A similar inflection point occurs in the UF-MDI setup at 33 seconds and 30°C. The differing temperatures at which the inflection points happen are in part due to the platen temperatures. One possible explanation for the different temperatures at which they occurred is the two adhesives had glass transition points at those temperatures. For the PF boards, the platens were at 205°C during pressing. For the UF and MDI boards, they were at 160°C. The UF-MDI circuit setup curve approaches an RTD value of about 2.16, and the PF circuit setup curve approaches an RTD value of about 0.03, both asymptotically.

As PF required a higher cure temperature than UF and MDI, the respective temperatures were used for the moisture readings in Figures 24 and 25. As the water in the panel begins to heat up, it starts to flash into steam at the boiling point. As pressure builds in the panel, the boiling point rises to a point higher than 100°C. Once the boiling point has been reached,

much of the incoming heat energy is used by the heat of evaporation, resulting in a leveling out effect in the temperature curve. After most of the water has been converted to steam and is in the process of leaving the board, the temperature resumes rising at an increasing rate. In the panels pressed at 205°C, the boiling point is reached sooner than those pressed at 160°C (about 120 seconds).

At first glance, one might expect the RTD to shift to the left with the temperature curve, for the PF system. In reality, it stayed about the same as the UF-MDI system. This is likely due to the fact that the water in the surfaces of the panel reaches the boiling point almost instantly in both setups. The heat then progresses inward at a rate dependent on the cure temperature and transfer properties of the wood and remaining moisture.

Internal Bond Strength Values

In order to check that dielectric response curves monitored for the three adhesives corresponded to their cure (the crosslinking process); boards were cured to specific time increments and the process described earlier was used to cool them quickly. After being cut up and selecting four random IB samples from each panel, the samples were broken on a universal testing machine. Referring back to Figure 10, it makes sense to simplify and display the results in three linear regions (pre-cure, duration cure and post-cure) instead of one

continuous "S" shape (see Figure 26). That representation was beneficial for comparing the IB values obtained in this project to the dielectric data, so it was adopted as seen in the results section. It is important to note that the samples were conditioned to about 8% moisture content at ambient conditions prior to testing. All IB strength plots are on the same scale for ease of comparison, and each data point represents four IB samples measured per panel. The begin-cure points were measured with a high amount of confidence. The end-cure points, however, were found with only one or two panels per adhesive, and are not very robust. These figures are simplified for

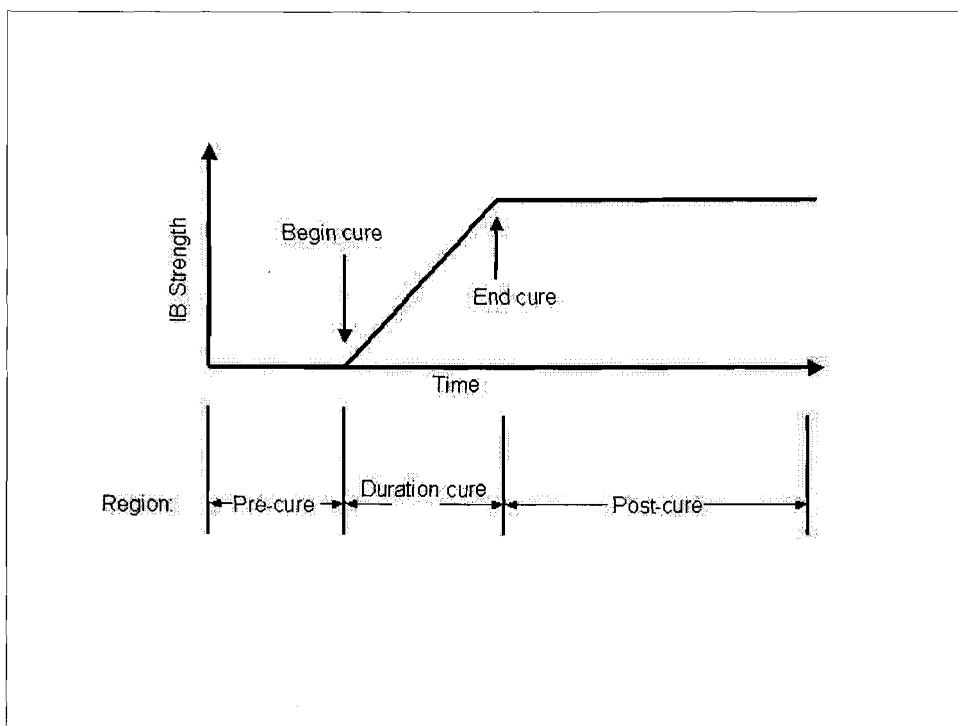

Figure 26 - IB cure regions defined

displaying purposes. The results of those tests are outlined in the following Figures 27, 28 and 29.

As the industrial standard for particleboard is 0.448 MPa (65 psi - ASTM D1037-99), all boards met and exceeded that value handily when fully cured. In fact, the MDI boards were seven times stronger than required. The three adhesives exhibited differing characteristics from each other in that they achieved differing maximum bond strengths, and begin-cure and end-cure times. The begin-cure point is defined as the time when the IB strength starts to increase. The end-cure point is when the IB strength levels out (see Figure 20). Duration cure time is the difference between the two - the amount of time it takes the adhesive to go from zero to complete cure (Table 5).

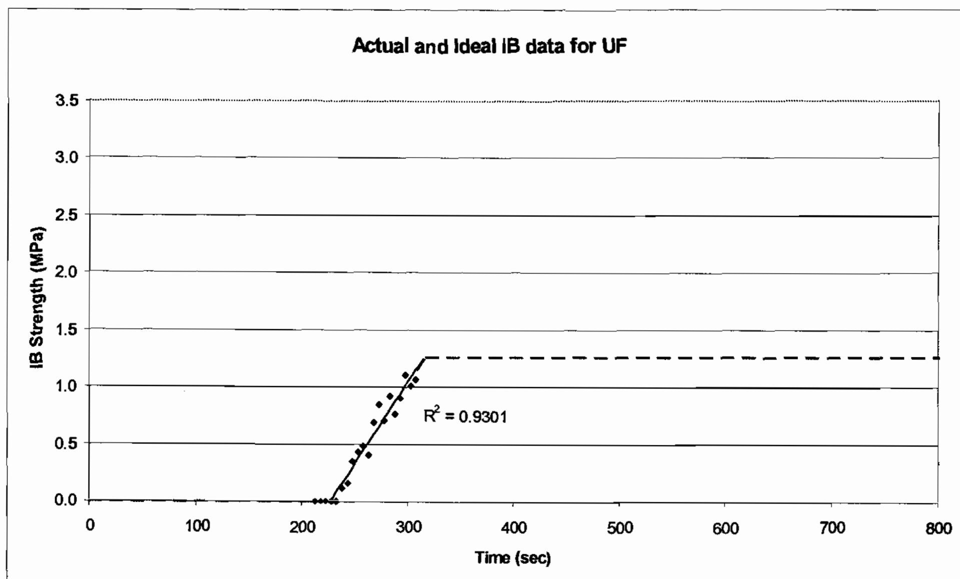

Figure 27 - Actual and ideal IB data for UF boards

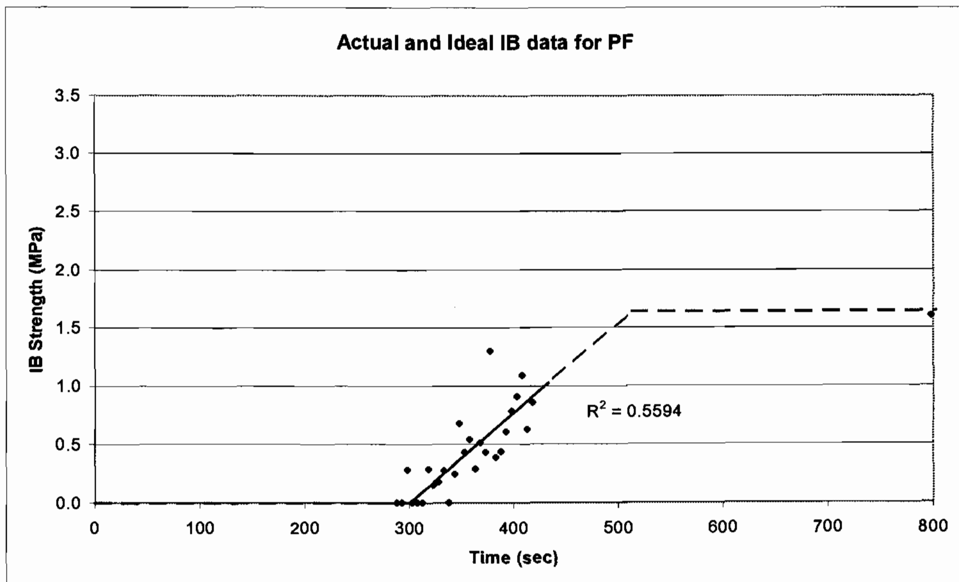

Figure 28 - Actual and ideal IB data for PF boards

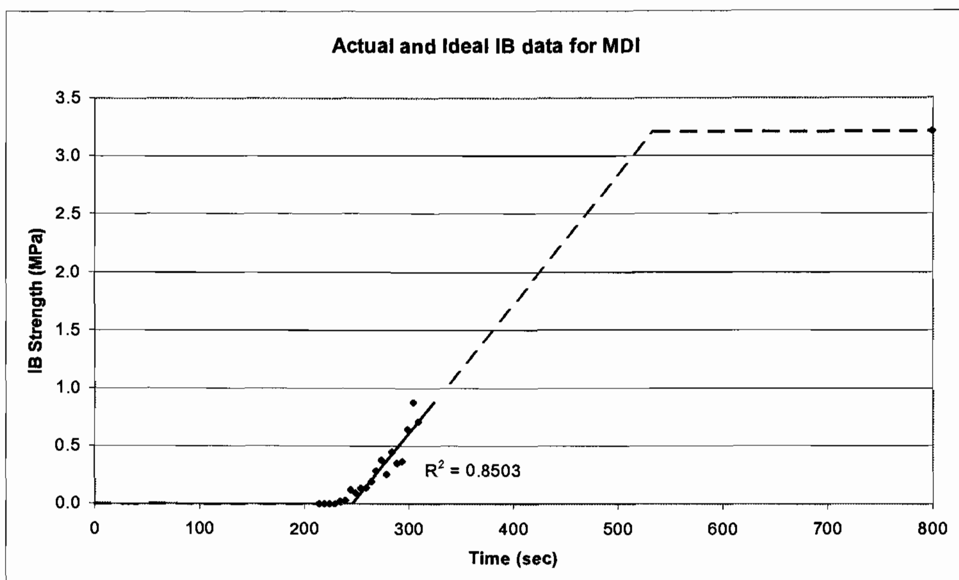

Figure 29 - Actual and ideal IB data for MDI boards

Adhesive	UF	PF	MDI
Pre-cure time (sec)	228	303	244
Duration cure time (sec)	110	212	286
Post-cure time (sec)	482	287	266
Begin-cure time (sec)	228	303	244
End-cure time (sec)	318	513	534
Maximum bond strength (MPa)	1.26	1.64	3.21

Table 5 - Adhesive cure time data

Dielectric Response Curves of the Three Adhesives

Dielectric response curves were obtained for each of the three adhesives, according to the circuit setups described earlier. All dielectric response curves stayed within range of the system and exhibited repeatable and very different characteristics attributable primarily to the fact that different chemical reactions are taking place as each adhesive cures. Each dielectric response curve shown is an average of a few boards–the UF dielectric response curve had four boards averaged, while MDI and PF had three boards averaged (Appendix B shows individual boards). Those results are displayed in Figures 30, 31 and 32.

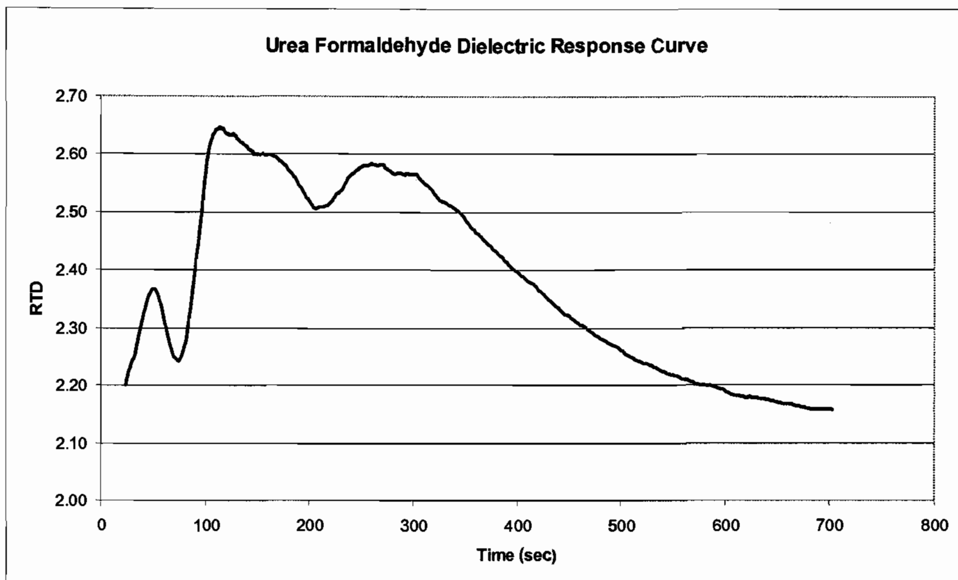

Figure 30 - Dielectric response curve for UF boards

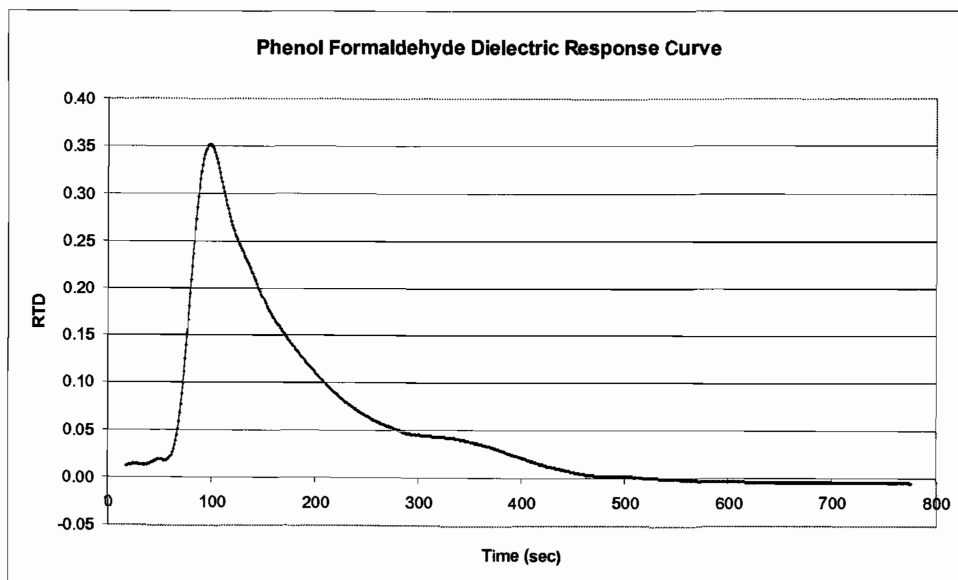

Figure 31 - Dielectric response curve for PF boards

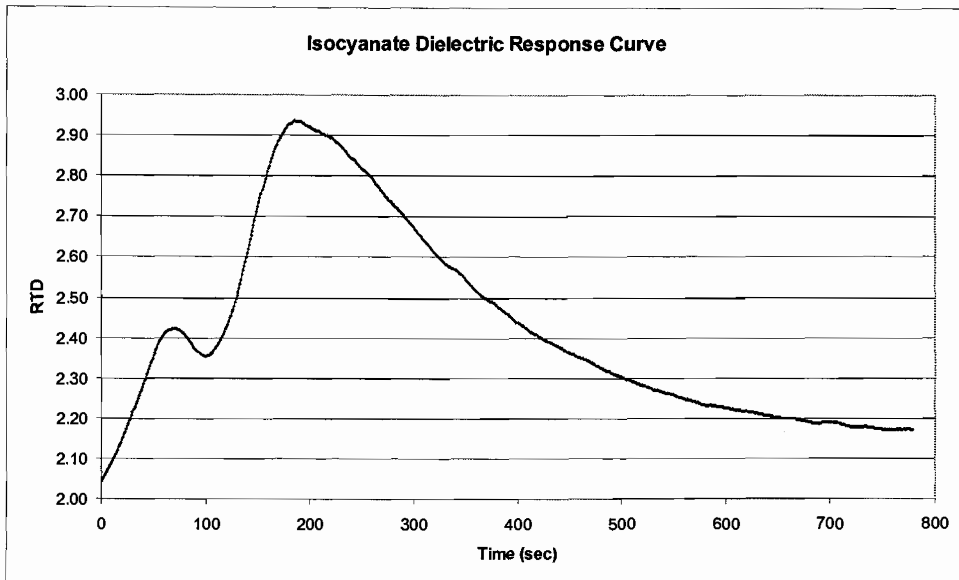

Figure 32 - Dielectric response curve for MDI boards

DISCUSSION

Compilation of Dielectric and IB Data

Having shown that data was obtained for dielectric properties and IB strength over time for each adhesive, it remains to combine them to begin to determine whether they mean anything. Ideally, the inflection points (begin and end-cure points) of each IB graph would correspond to specific characteristics in their respective dielectric response curve. This sounds simple, but in reality is quite complex. For example, the beginning cure point on each of the IB cure charts is close but technically not the very beginning of the cure process. It is rather the point at which the furnish at the center of the board has developed enough strength to overcome the internal vapor pressure of the steam still inside when the press opens. Adhesive cure begins somewhat before that point. This should be reflected in the dielectric response curves.

Knowing what each characteristic of every dielectric response curve means chemically would be an enormous benefit. We could then state with certainty when cure had occurred, moisture flashed into steam, etc. As it was not possible to monitor at a cellular level the chemical dynamics of what was happening in the boards as they cured, we must rather rely on known properties of the adhesives and gather knowledge on the physical chemistry

behind what is happening. Then we can begin to piece together a hypothesis as to what each of the dielectric response curves is revealing at any one point in time. At this point it will be easier to address the adhesives individually rather than treat them all in one generalization.

Physical Chemistry in Dielectric Response curves

Urea Formaldehyde

First, it is important to display the dielectric data and the IB data superimposed on a common time line. This is given in Figure 33. The most obvious explanation of a characteristic in the dielectric curve is the tallest

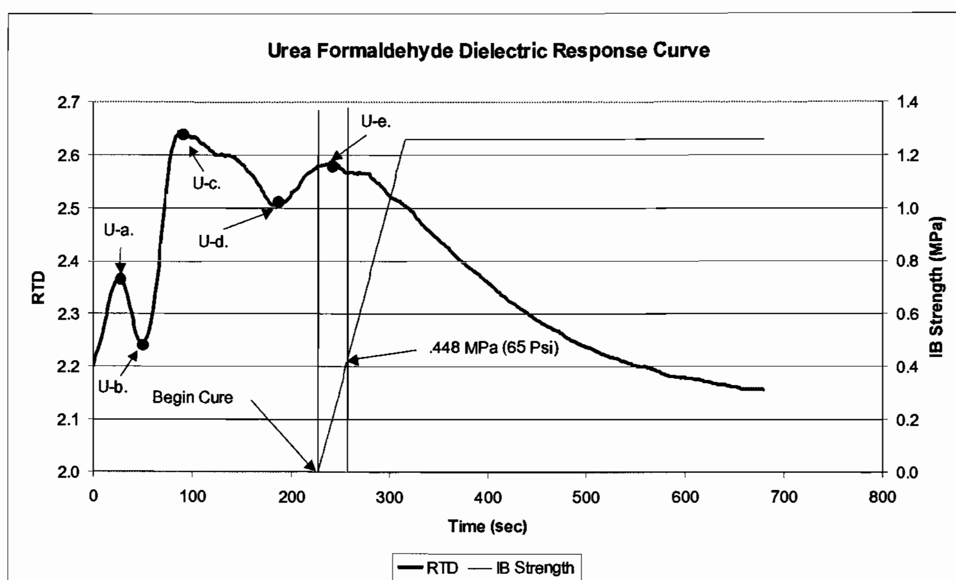

Figure 33 - Dielectric and IB data for UF

peak (U-c). It corresponds with the peak in the dielectric response curve of the moisture boards as described earlier (Figure 26). It is likely the water flashing into vapor accounts for that peak. There is also a smaller peak (U-a) and valley (U-b) in the first 100 seconds of the moisture dielectric response curve that shows up greatly amplified in the adhesive dielectric response curve. It is yet unclear what this can be attributed to. It is possible that the UF reaches a glass transition point, which would affect the dielectric reading.

UF undergoes a condensation reaction, releasing water as the adhesive crosslinks. It seems likely that this occurs at U-d, the dielectric response curve beginning to rise again as a result of the introduction of more liquid water. It then turns into steam and the dielectric response curve starts falling again (U-e), leveling out over time to the usual RTD value of $\geq$ 2.16. Analysis of the graph shows a promising relationship between the top of the second peak (U-e) and the begin-cure point of the IB strength curve. The beginning of the condensation reaction occurs just prior to a readable bond strength.

UF is unique among the three adhesives studied in that it is the only one that utilizes an ionic catalyst. The addition of extra ions in this manner over the other adhesives should increase the impedance component of the phase angle, resulting in an increase of the RTD. The extra ions could also have the effect of introducing more noise into the system than adhesives that do not use the same type of catalyst.

It is interesting to compare the values obtained for UF in this study to the values obtained by Wolcott and Rials for the same type of adhesive. The results from that are shown again in Figure 34. While the time scales are different, and it is unclear what conductivity quantifies, they exhibit similar characteristics that bear noting. They both start at one value, peak, and after time level out to a value lower than they began. The beginning of the dielectric response curve in Figure 33 shows an inflection point (U-a, U-b) that is not immediately obvious in Figure 34, but that may be due to sampling rate limitations of the Eumetrics system used by Wolcott and Rials. There is

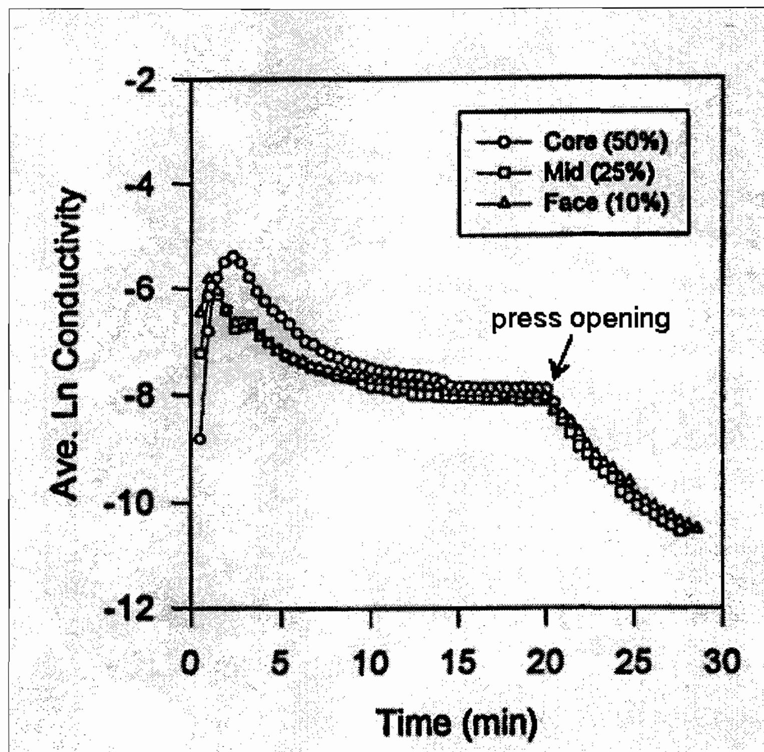

Figure 34 - Dielectric data recorded by Wolcott and Rials for UF (Wolcott and Rials, 1995b)

another peak in Figure 33 (U-e) that does not appear in Figure 34. A possible reason for that is the frequency used to make the reading was not specified. If it was a frequency other than 100 Hz, the phenomena may not have been readable, showing up only in scans made at 100 Hz. Wolcott and Rials used five excitation frequencies from 10^{-1} to 10^{3} Hz, but it was unclear how that was accomplished and reported in a single conductivity reading.

Isocyanate

Addressed next is the isocyanate dielectric response curve. It too was superimposed with the respective IB strength values, the results being shown in Figure 35. The similarity to the UF dielectric response curve is striking. The same inflection point (I-a, I-b) is exhibited only slightly earlier and with a lower amplitude. It also could be the adhesive reaching the glass transition point and softening, resulting in a lower RTD for a period of time. This inflection point does not appear in the board with only moisture in it. Wolcott and Rials report a region (shown on a time-temperature superposition graph, thus difficult to compare to these figures) of the dielectric data recorded that is a result of adhesive and wood fiber softening (Wolcott and Rials, 1995a).

MDI displays a prominent peak like the UF and moisture dielectric response curves (I-c), about 100 seconds later than in the UF. Again, this peak is likely due to the liquid water in the system turning to steam, and the peak is the point where the steam component overtakes the liquid component

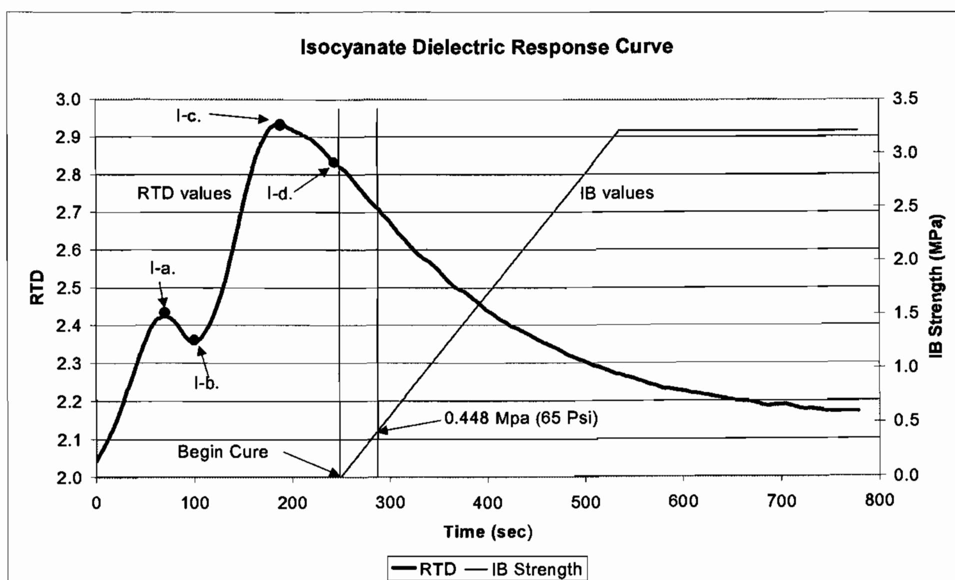

Figure 35 - Dielectric and IB data for MDI

of the dielectric response curve. Conspicuously absent is the second large peak (U-e) found in the UF dielectric response curve (Figure 33). As MDI does not experience a condensation reaction (instead it consumes some water), this makes sense. This curve, like the UF, approaches an RTD value of about 2.16 asymptotically.

A relationship between IB strength development and response curve characteristics is subtle, but apparent. At the exact point where IB strength starts there is a change in the slope of the dielectric reading, possibly indicating polymerization. As mentioned before, curing begins to develop at the panel surfaces first and is not measurable in the IB samples taken for this experiment until the adhesive in the panel core has cured enough to

overcome the internal pressure of the water vapor. This would be manifested in that a dielectric response should show up before the first IB data point registers. Regressing the IB data back to y=0 should predict that point.

Phenol Formaldehyde

While the dielectric response curves of PF for both moisture and adhesive are different in shape from those measured with the UF dielectric setup, they have similar characteristics. All adhesive dielectric response curves have at least one large peak that is most likely attributed to moisture flashing into steam. The PF dielectric response curve is no exception (Figure 36). There is one very sharp peak (P-b) rising to an RTD of about 0.35. Surprisingly, there is a much less defined inflection point (P-a) in the adhesive dielectric response curve prior to the highest peak similar to UF (U-a, U-b) and MDI (I-a, I-b). In the moisture reading (Figure 24), the RTD value starts increasing almost immediately after the press closes. It apparently does not in the PF adhesive dielectric response curve, possibly due to a cancellation of factors (i.e., polymer glass transition lowering effect equal and opposite to moisture-steam effect).

The IB strength values begin-cure point is just after the 300 second mark, and attempting to discern a correlating characteristic in the dielectric

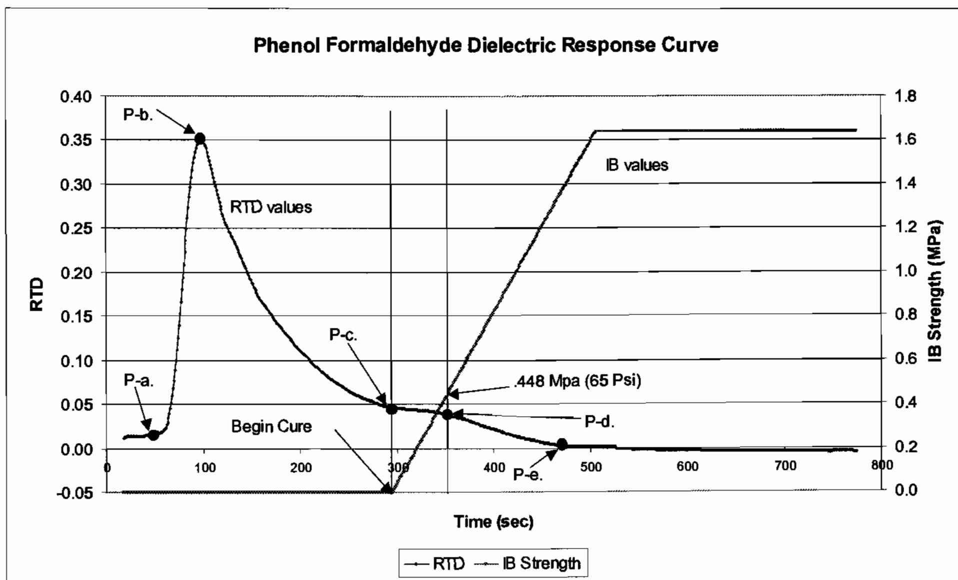

Figure 36 - Dielectric and IB data for PF

response curve is difficult at first. However, if you magnify the scale for the RTD axis, the inflection point in that region is much more pronounced and appears to be strongly related to IB cure (P-c, Figure 37). The dielectric response curve levels out for 50-60 seconds (P-d) and then resumes its declining trend until about the 460 second mark (P-e). It then levels out again for about 40 seconds, and declines (P-f) to an RTD value of about -0.005.

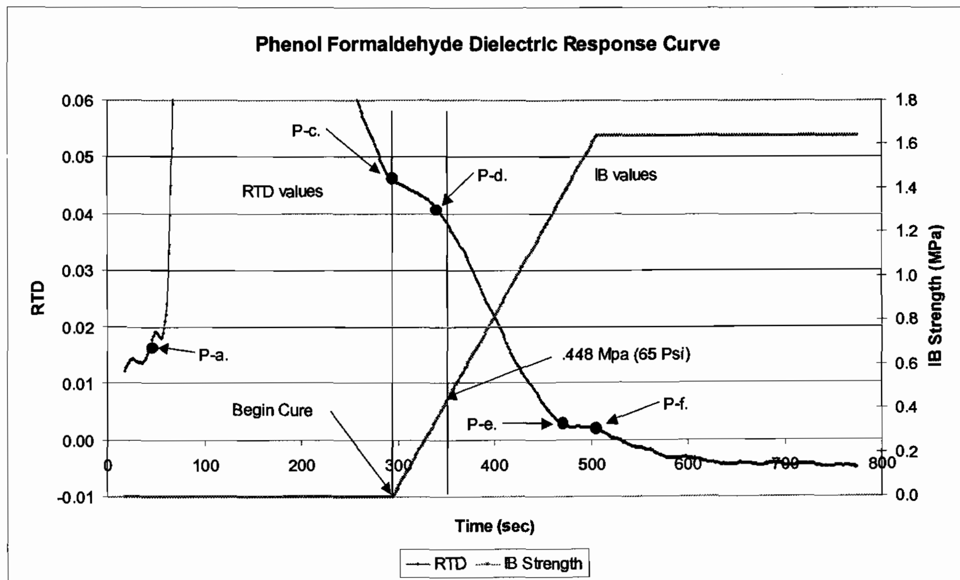

Figure 37 - Dielectric and IB data for PF, magnified scale

CONCLUSIONS

Experiment Summary

The UF adhesive dielectric response curve appeared to have a number of features in the vicinity of the points at which cure began and ended. There was a very pronounced peak (U-e, Figure 33) that occurred almost exactly at the IB begin cure point. That peak also matched up with approximately when the condensation reaction should have occurred.

MDI also had dielectric response curve characteristics that were attributable to its IB data. There was a large peak (I-c, Figure 35) in the data that was primarily a result of the moisture in the panel. Approximately 60 seconds after I-c, the slope in the decreasing curve changed a small amount. It then proceeded to decrease and level out toward the end of the pressing cycle. This change in slope may be the inflection point that could be used to monitor IB cure in a commercial system.

The last adhesive, PF, had good results too. Characteristic P-c in Figure 37 almost exactly matched up with the begin-cure point of the IB data. The following inflection point (P-d) seemed to be related to the end-cure point of IB cure, as the RTD resumed a slope similar to prior to point P-c.

All three adhesives showed very encouraging relationships between particular features in their dielectric response curves and events in IB cure data. This study showed that designing a commercial dielectric cure monitoring system for composite panel products has great future potential.

Recommendations for Future Research

This feasibility study opens the door to a wealth of subsequent studies. It would be beneficial to chemically analyze the boards that were partially cured and tested for IB strength. Changes in properties of the adhesives would substantiate claims of resulting dielectric response curve properties that occurred at the same points in time. A chemical map of what is happening at a molecular level for each adhesive would give an even larger picture and would possibly explain many characteristics in the dielectric response curves.

Running more samples for IB strength values would strengthen the inferences drawn from begin-cure and end-cure points to dielectric response curve characteristics. More data needs to be obtained along the entire pressing time, including the post-cure region. It would also be beneficial to run more panels for the entire 800 seconds, to obtain more dielectric response curves. Having more dielectric response curves from individual boards to average would increase confidence in the averaged dielectric response curves for each adhesive, and yield more information about repeatability.

It would be interesting to apply this technology to other hot-pressed composite panel products such as MDF, hardboard, OSB, plywood, LVL and more.

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APPENDICES

APPENDIX A

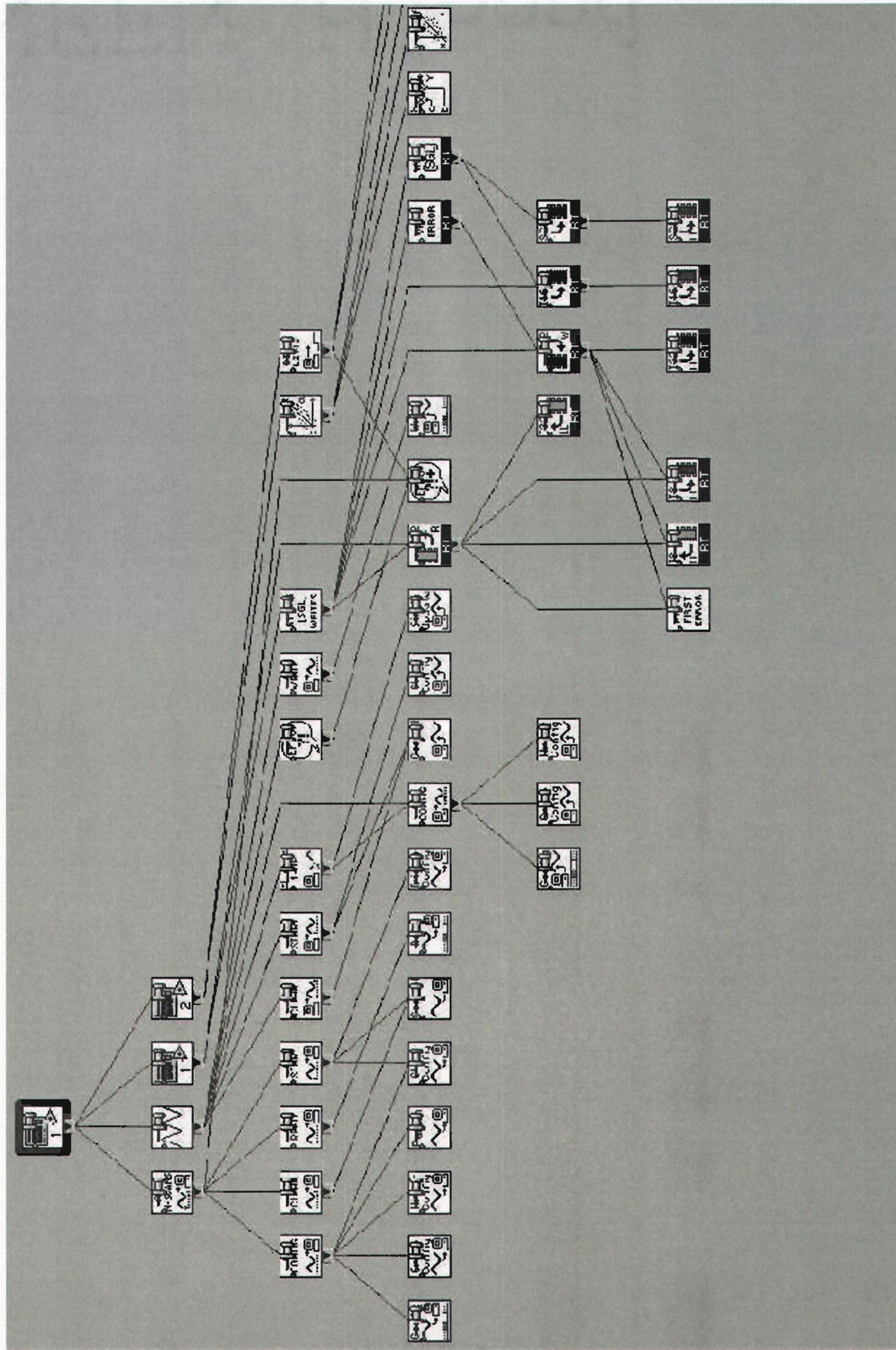

Figure 38 - Hierarchy diagram of LabVIEW VIs working together to collect and process data

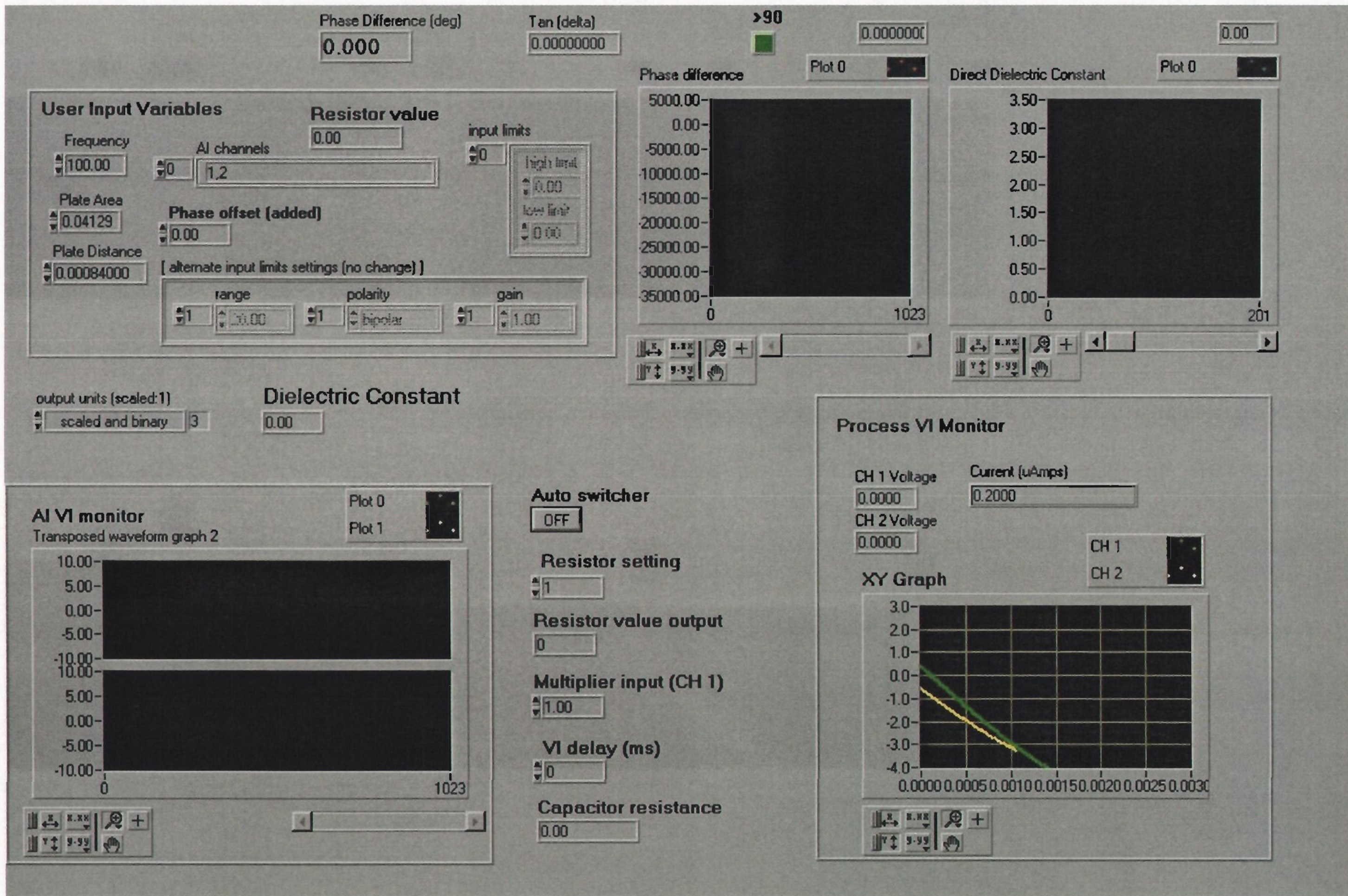

Figure 39 - Panel view of collect and send software VI

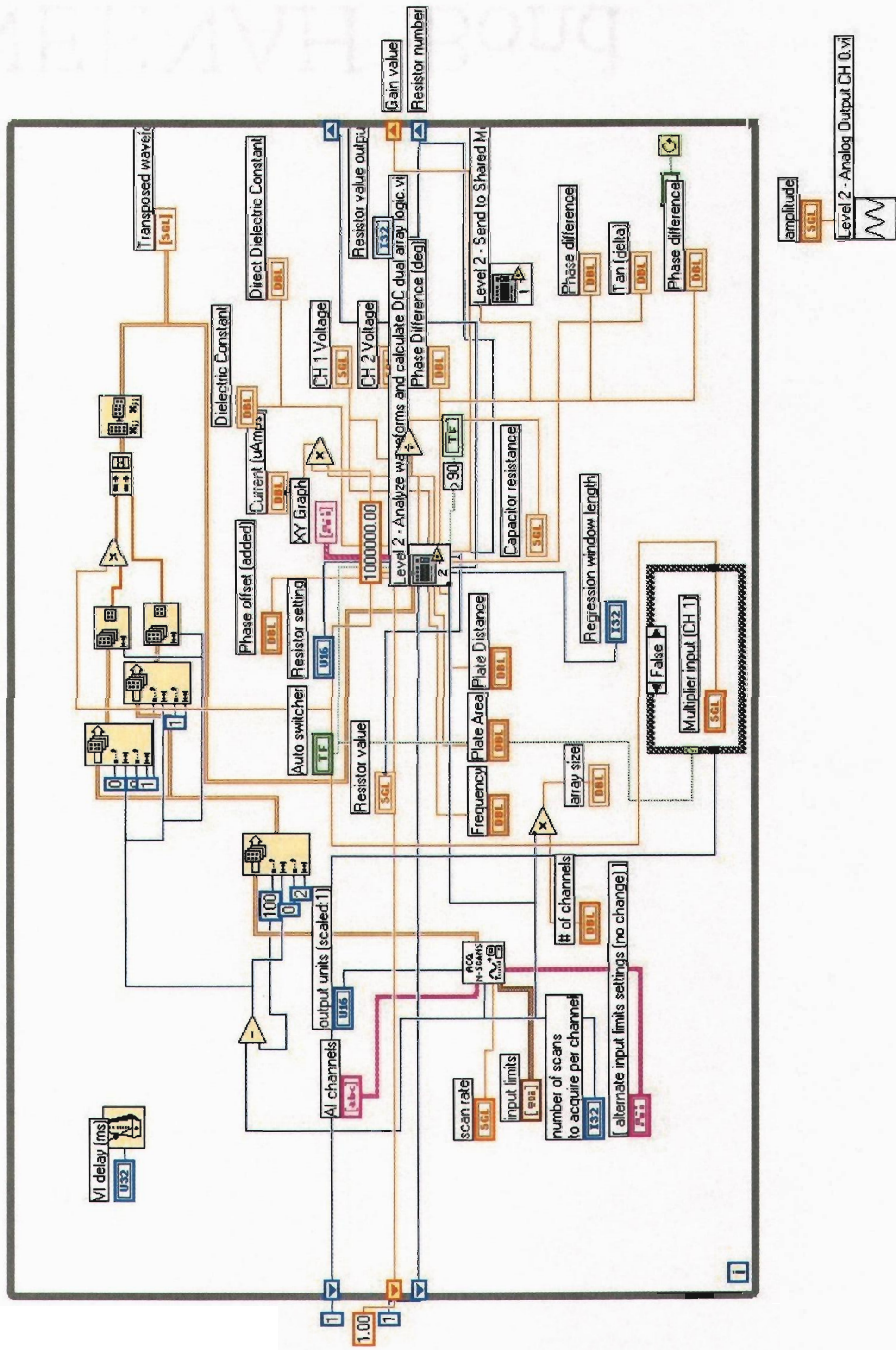

Figure 40 - Diagram view of collect and send software VI

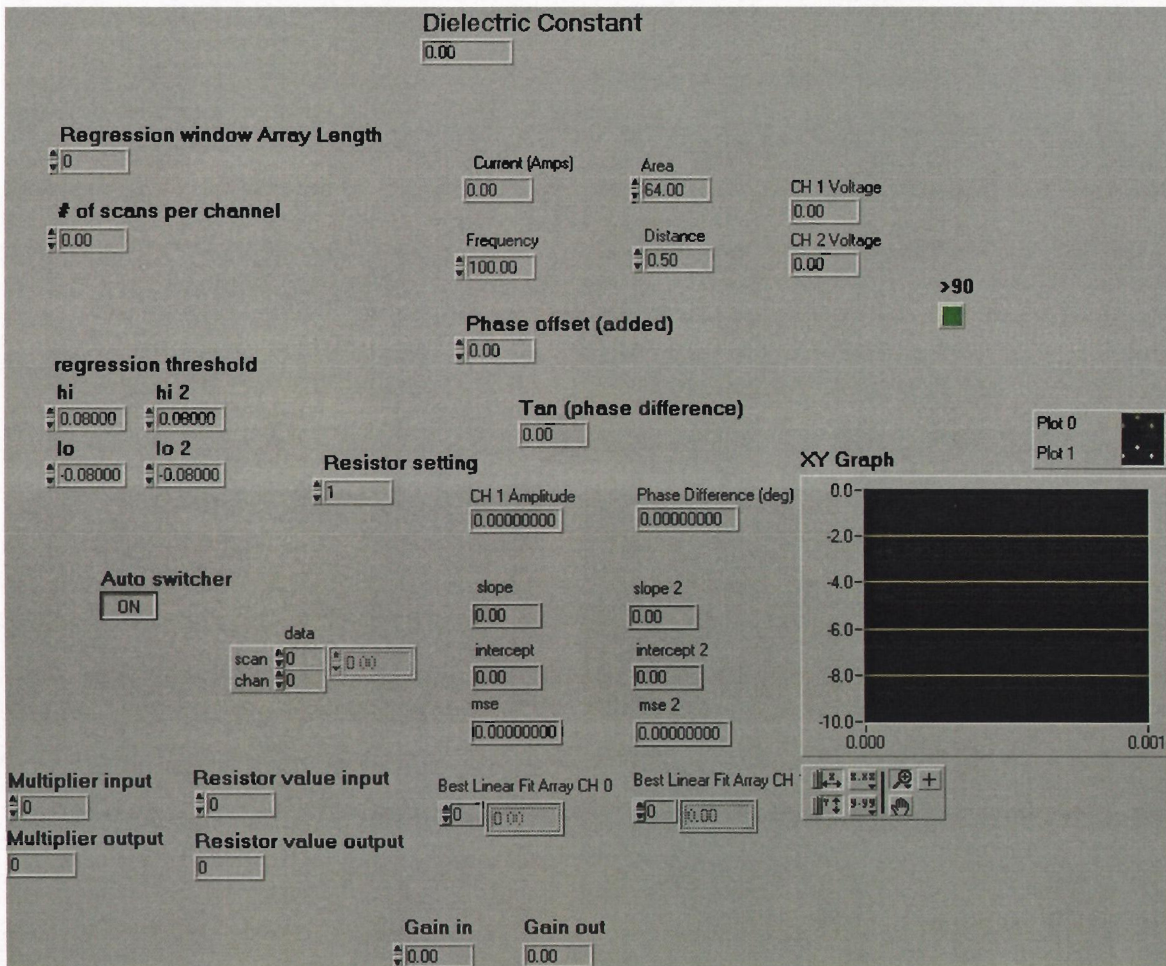

Figure 41 - Panel view of signal process software VI

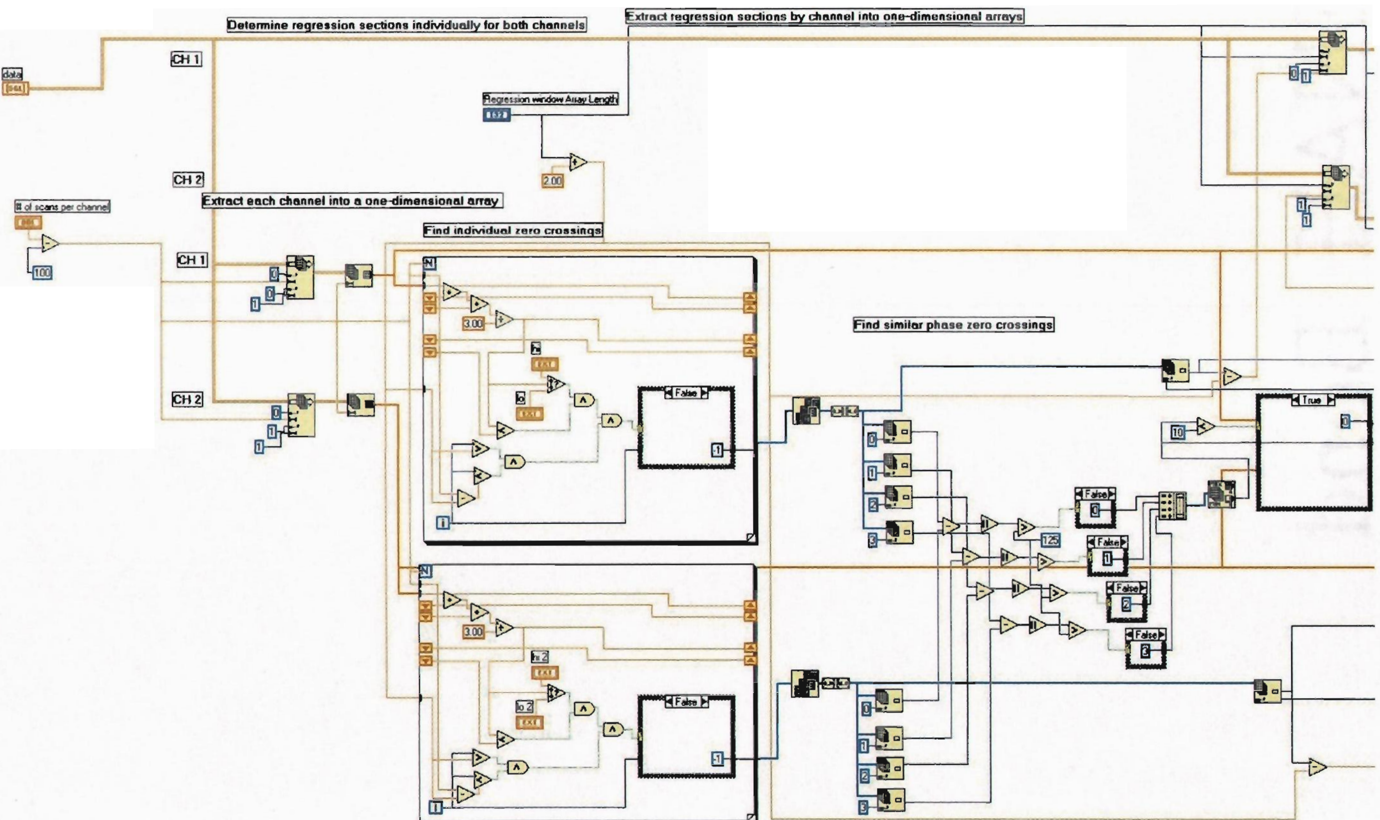

Figure 42 - Diagram view of process software VI page 1

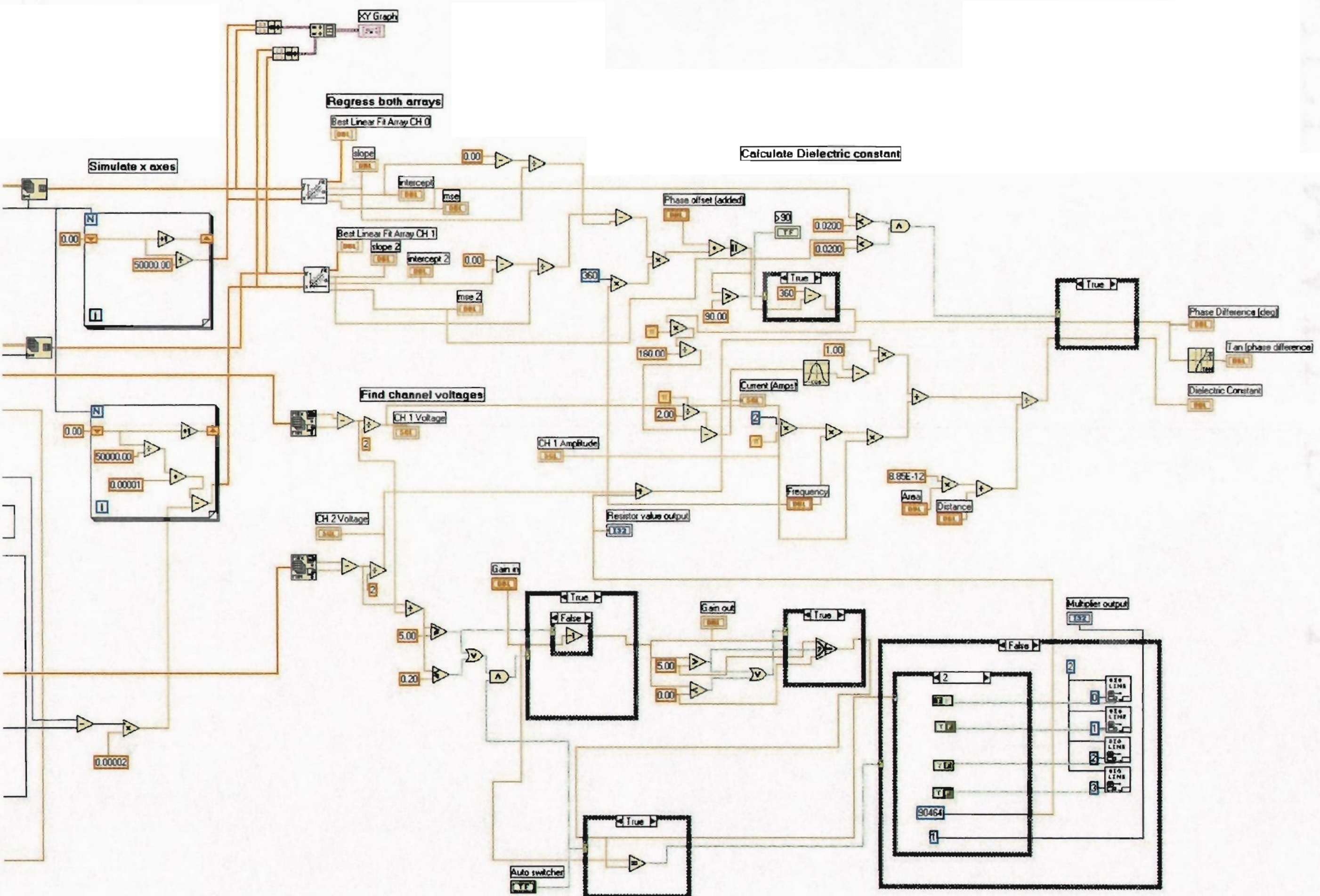

Figure 43 - Diagram view of process software VI page 2

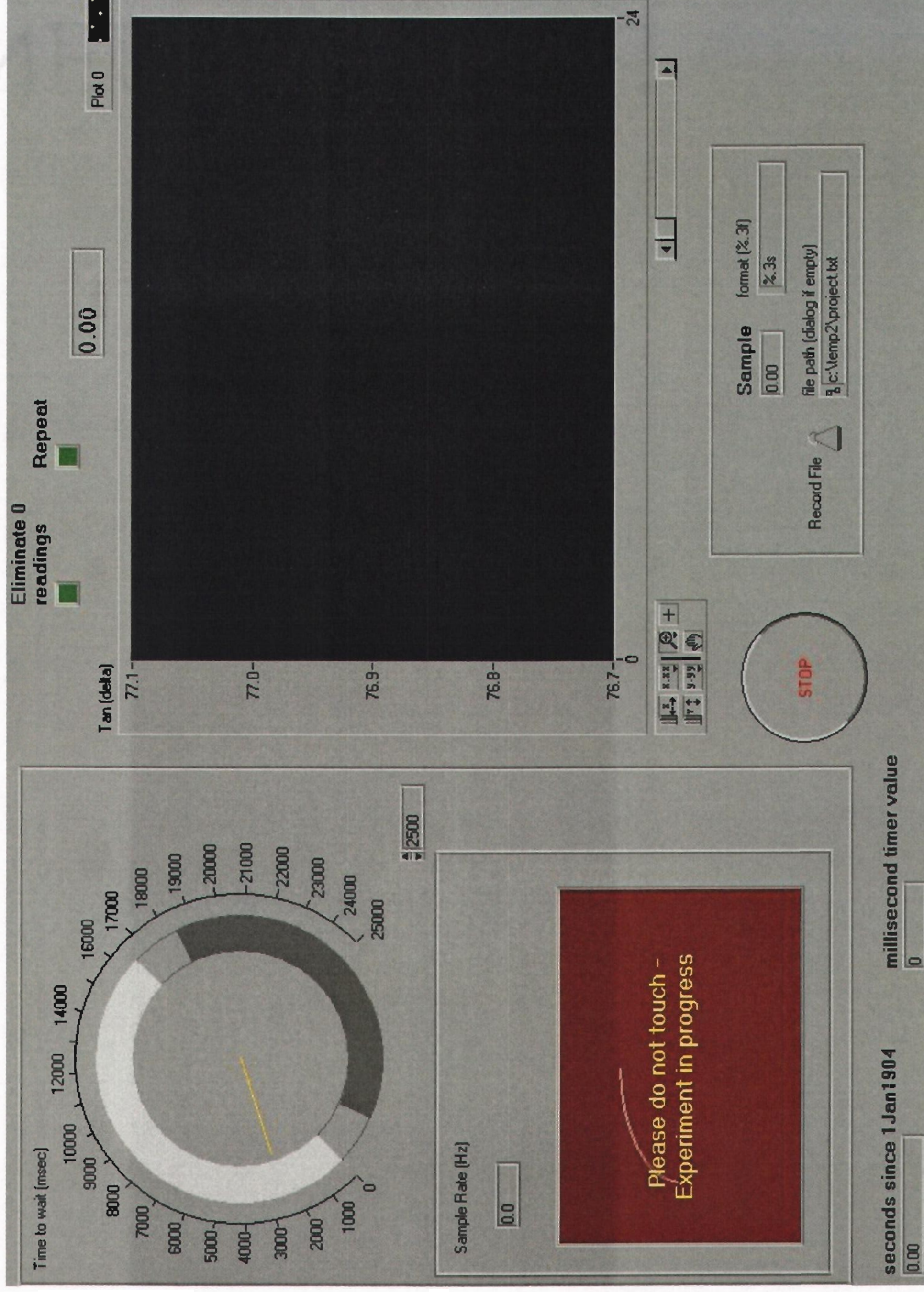

Figure 44 - Panel view of display and record software VI

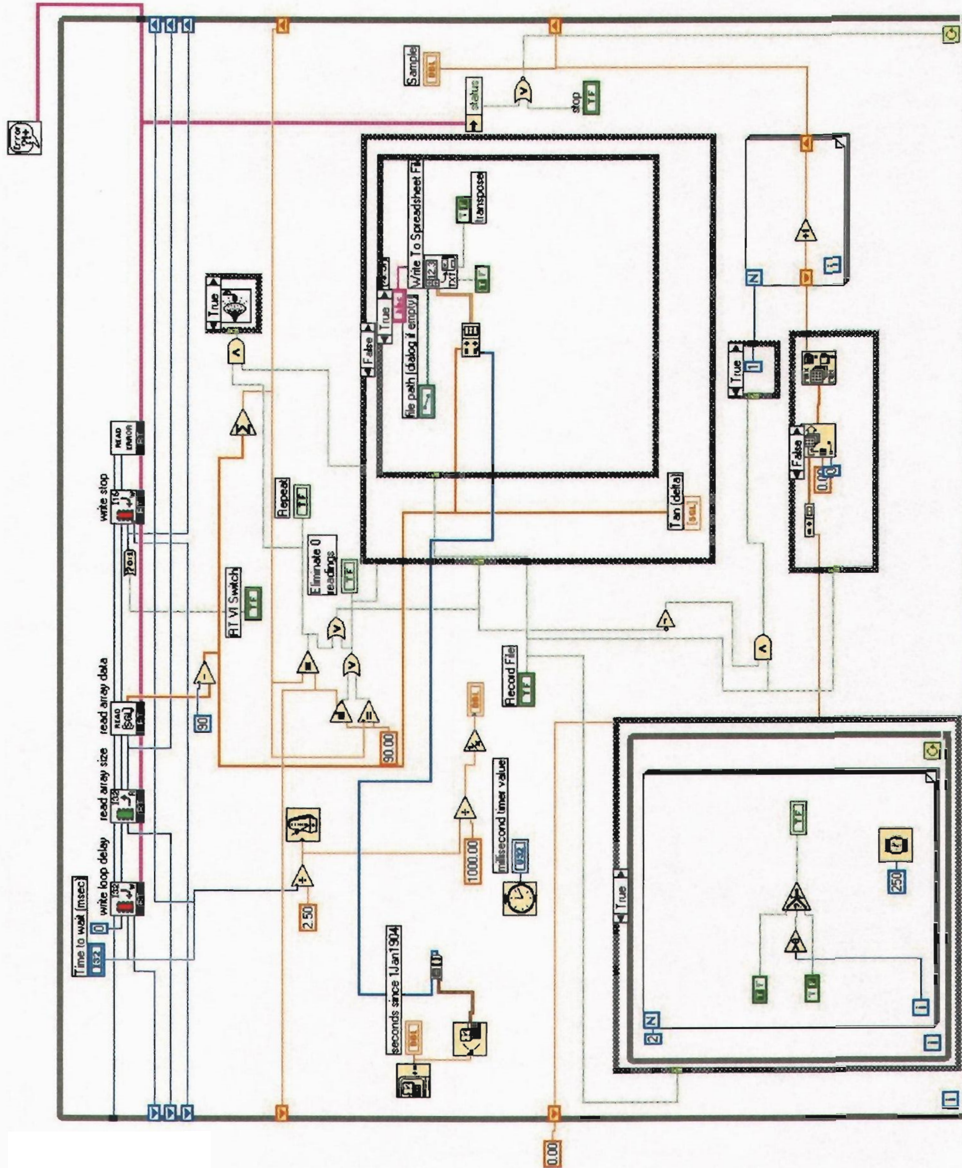

Figure 45 - Diagram view of display and record software VI

APPENDIX B

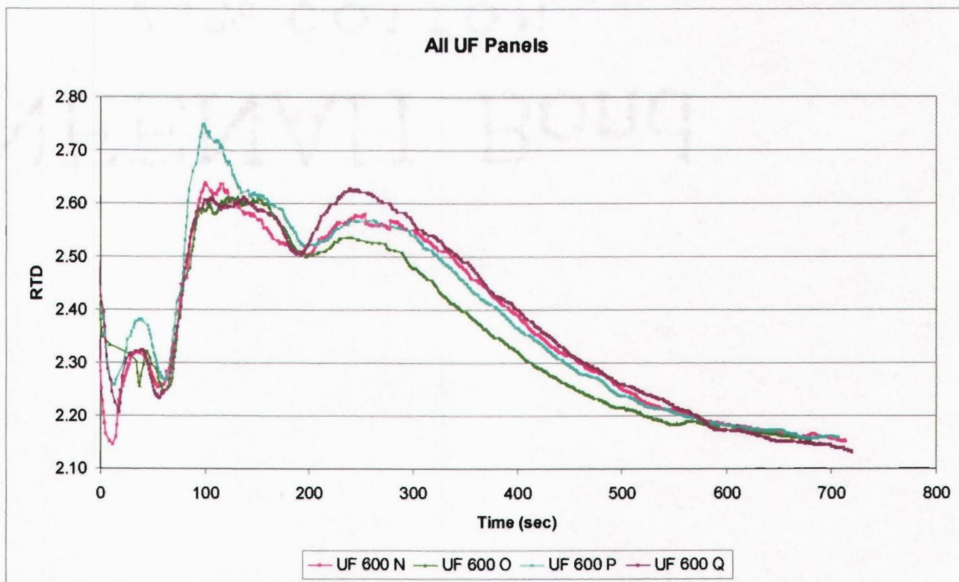

Figure 46 - Individual UF board dielectric response curves

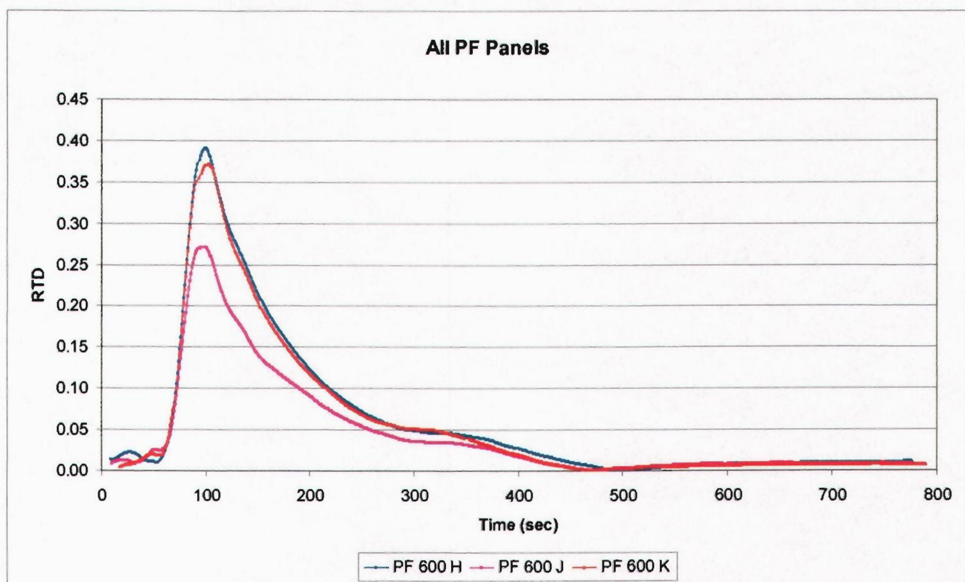

Figure 47 - Individual PF board dielectric response curves

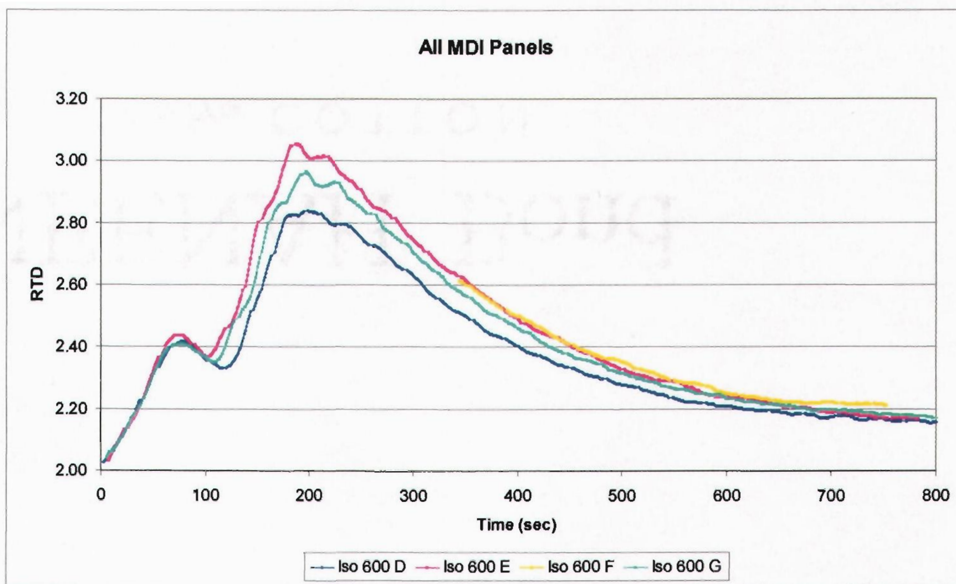

Figure 48 - Individual MDI board dielectric response curves