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Detection of Water and Ice on Bridge Structures by AC Impedance and Dielectric Relaxation Spectroscopy Phase I

Final Report

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While we originally expected that both measurements would be required to reliably determine the condition of a bridge deck surface with regard to the presence of frozen water or deicing solutions, we have found that the TDR approach is adequate for this task. This suggests that a significant reduction in both the cost of development of practicable sensors and supporting software/electronics can be realized, as well as the ultimate cost of deploying a system based on TDR alone can be realized. As such, TDR becomes the focus for the next phase of development of these sensors.

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Executive Summary

Proof of concept has been shown for the application of time domain reflectometry to the detection of air, water, ice and electrolyte in both liquid and frozen states in contact with passive metal transmission line sensors. Further work will be carried out to perfect the hardware and software required to make this economical transmission line technology practicable for deployment on bridge decks and other areas of concern, so that activation of safety signage or signaling to maintenance personnel can be automated based on the response of these sensor systems to unsafe road conditions (e.g. accumulation of ice, frost, etc.).

Chapter 1
Introduction

Our research objective, simply stated, has been to develop reliable, low cost water/ice sensors systems which can be deployed in a variety of critical locations. Our aim is to develop these technologies in configurations amenable to inclusion in remote sensing networks.

We hope to deliver a package system, but this cannot be accomplished in a one year time frame with such a modest budget. A more realistic expectation for Phase 1 is a report of definitive lab results which show proof of concept, with system refinement in Phase 2. Finally, in Phase 3 our attention will turn to and field testing, while minimizing the cost of the electronics components and modules used in the systems.

In light of the lack of affordable alternative ice/water detection systems, if we are successful, these outcomes could provide revolutionary sensing alternatives to those currently available. The benefits could be enormous in terms of decreasing loss of life, personal injury and loss of property at critical sites by virtue of making real time data available to trigger automated dispensing systems, or alert maintenance crews of immediate need for application of deicing chemicals to these critical areas. The primary benefits would accrue to the public in terms of reduced accident risk at critical traffic sites.

Chapter 2
Technical Background

Two technologies will be evaluated for ice/water sensing on road and bridge deck surfaces. Both involve non-chemically specific sensors, and rely on the measurement of the impedance of an electrode pair or array. In the first method (AC Impedance Spectroscopy), a sophisticated measurement of the low frequency (10-100KHz) impedance of a test cell comprised of these electrodes and the roadbed material will be made. This work will continue studies begun under a seed proposal awarded for the current FY. The second technology (Time Domain Reflectometry) is conceptually simpler in principle, and is more amenable to remote array detection of water and ice. This is a higher frequency measurement (1MHz – 20GHz) of the complex dielectric function of the roadbed material and the water or ice in contact with it. It involves the use of a linear sensor which is a transmission line.

AC Impedance Spectroscopy. Impedance spectroscopy typically involves the application of a small sinusoidal voltage to a test system, with concurrent monitoring of the current through that system. The test cell may be designed to evaluate transport properties of the bulk material placed between the electrodes, or interfacial phenomena at the interface between this material and the contacting electrodes. The impedance data are gathered over a frequency range consistent with the time domain of the transport and interfacial phenomena of interest. The impedance data are then plotted in an impedance or admittance plane (Nyquist) format which reveals characteristic relationships between the real and imaginary components of these data as a function of frequency. From the structure of such plots, inference with regard to charge transport and charge transfer dynamics within the test cell can be made to evaluate physical parameters of interest, such as conductivity, electron transfer kinetics, diffusion coefficients of electro-active and mobile changes carries within the bulk, kinetic dispersion of these parameters, etc. The complex impedance spectra characterized by such measurements in the 10 Hz – 1 MHz frequency range are then modeled, and the resulting extracted parameters are related to interfacial and bulk charge transfer and charge transport phenomena. This technique has been applied to wide range of solid and solution systems with considerable success [1] [2]. Of interest in the context of ice/water detection is the measurement of bulk transport rate for charge carried by ionic components of deicing systems in solution vs solid state. The electrodes which serve as the “sensors” in such an application would ideally be inert, such that for the application of interest in this work, stainless steel should suffice.

This approach will allow for a determination of the state of surface moisture (solid vs liquid) via this sophisticated “conductivity” measurement. A simple two electrode system employing stainless steel electrodes and impedance analysis, as has been applied to the evaluation of the effects of freeze-thaw cycles on Portland cement porosity and implications with respect to composite mechanical strength [3], will be utilized.

In the current application test cells of various geometric configurations will be examined, each of which will involve the placement of stainless steel electrodes at or near the roadbed surface. In addition to measurements involving surface conductivity on concrete and bituminous roadbeds, overlay systems such as that used in specialized overlayers (e.g. SafeLane and other epoxy sealant-based systems) will be examined using embedded electrodes. These laboratory test fixtures will be constructed on temperature controlled Peltier devices, to facilitate measurement over a temperature range of -20 to 50 C during dosing and impedance measurements.

Measurements of complex impedance will be measured using a potentiostat currently in house, modified to carry out these measurements over the frequency range of 10 Hz to 100 KHz. A block diagram of the apparatus is shown in Figure 2.1, below.

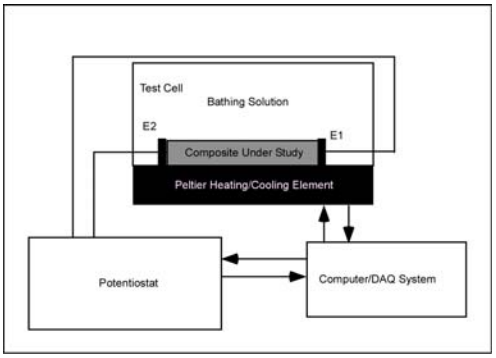


Figure 2.1. Schematic of proposed apparatus, electrodes (E1 and E2) will be fabricated from stainless steel, and the composite will be cast in a slab configuration of dimensions 8 mm wide and 13 mm long.

Aside from evaluating the retention or presence of deicing chemicals, the above arrangement will also be examined as the basis for a sensing system for determining the state of aqueous solutions (frozen vs liquid) in/on the composite.

Time Domain Reflectometry. Time domain reflectometry (TDR) is a technique used to test electrical interconnects and transmission lines in high speed circuitry (e.g. serial disk drive communication protocols, Ethernet cabling systems, etc.). Any changes in the impedance of the current path (transmission line) can be mapped, because these cause reflections at the interfaces between domains of differing impedance (as indicators of differences in dielectric constant of the medium surrounding the transmission line). The technique relies on variation in the dielectric relaxation of media in contact with the transmission line, and, furthermore, the location of discontinuities in the dielectric properties of the adjacent media can be determined. As such, simple sensing systems, which are directly amenable to remote sensing situations can be realized. This approach has been applied to a wide range of systems, although the primary application has been to soil samples, and evaluation of volumetric water content in these samples. It should be noted that frozen water in soils an electrolyte salt concentration in soils have been measured, as well. Furthermore, the sensors themselves (elements of a transmission line) are very inexpensive [3]. Examples of recent applications of this technology to sensing (as opposed to testing) include measurement of resin flow in polymer curing applications [4], water and ice content in soil systems [5] [6] [7] , and detection of ice, water and deicing solutions of aircraft wings and rotors [8]. Indeed because of the ease with which dielectric relaxation measurements can be made over such a wide frequency range (1 MHz to 20 GHz), TDR has been applied to fundamental research involving the structure of water [9] and the relaxation of water bound to silica gel [10].

Like impedance spectroscopy, TDR allows one to measure dielectric relaxation, but at a much higher frequency and bandwidth. Furthermore, in principle, changes in the complex dielectric function of material near the sensing transmission line can be resolved in terms of the positional dependence along the length of the transmission line to a resolution of a centimeter or less. The effective real part of the dielectric constant of water is approximately 80, while that of ice is 3. The TDR technique can also be employed to measure the conductivity of salt solutions, such as the deicing systems commonly in use.

The details of the physics of TDR measurements cannot be presented in the framework of this report, so only a brief introduction will be given. A more detailed introduction can be found on the following web page: http://www.d.umn.edu/~jevans1/. The technique involves applying a very fast rise time (ps time frame) electromagnetic pulse to one end of a transmission line. The pulse travels down the transmission line at a velocity nearing the speed of light, which depends on the dielectric properties of the medium surrounding the transmission line, according to:

$$\upsilon = c / \sqrt{\varepsilon} = c / n$$

where, c is the velocity of light in vacuum, ε is the dielectric constant of the medium surrounding the transmission line and *n* is the complex refractive index of that medium.

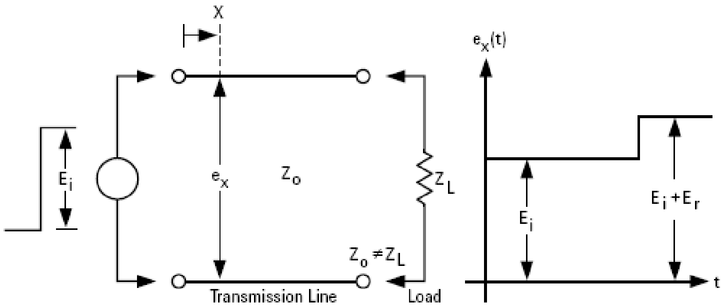


Figure 2.2. Voltage vs. time at a point on an impedance mismatched transmission line driven with a step voltage of height E_i.

As depicted above, any point along or at the terminus of a transmission line where the impedance changes, due to a change in dielectric constant of the surrounding medium, a portion of the energy in the pulse is reflected back to the source. That portion of energy not reflected or adsorbed at a discontinuity continues down the line until it is either absorbed or reflected. The physical position of the discontinuity can be determined with appropriate calibration. For example, consider a segmented system as shown in the following figure:

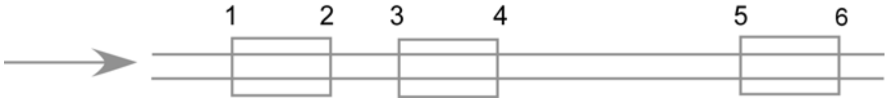


Figure 2.3. Transmission line with six dielectric discontinuities.

As the launched pulse encounters each dielectric discontinuity (points 1-6), a portion of the energy is reflected back to the source. In addition to determining the location of these dielectric discontinuities, a more detailed analysis of the differential complex impedance of that reflection can be made, and from this analysis the effective dielectric constant determined and attributed to materials for which the dielectric properties have been previously calibrated (e.g. water, ice, and electrolyte solutions).

Figure 4 shows the reflected pulse for a terminal sensing arrangement in which R and C represent the circuit equivalence impedances associated with the conductivity and dielectric loss associated with a salt solution. Here the solution properties of the electrolyte (real part of the dielectric constant and conductance) may be extracted for the measured values of R and C.

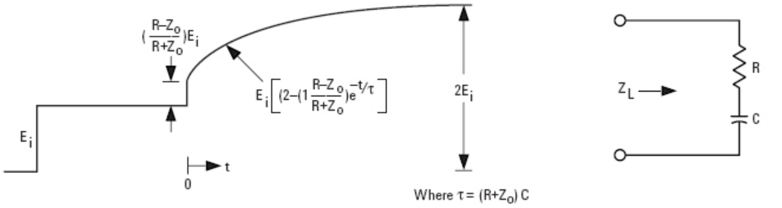


Figure 2.4. Idealized TDR trace for a series RC terminated transmission line.

Due to the significant difference in the real and imaginary components of the impedance of ice, water and deicing solutions, these boundaries are easily discerned [9]. With respect to the spatial sensitivity and other design issues, reasonable care must be exercised in the physical and electrical design of the transmission line. Care must also be taken to impedance match the line to the source impedance of the TDR pulse generator, to preserve optimal sensitivity and spatial resolution. Given that transmission line theory is well established, such optimizations should be relatively straightforward. During the first year of this study the p[primary focus of the experimental work will be on the design of transmission lines in terms of geometry and materials employed, with consideration of segmented or continuous lengths of transmission lines also under consideration.

By analogy to the reported sensitivity to location and state of water on aircraft airfoil and airframe surfaces, we propose to examine the use of TDR to examine the state of water on various pavement and overlay surfaces [9]. As in the case of the lower frequency impedance measurements, various fixtures will be examined to evaluate optimal geometry and placement of the sensor (transmission) line relative to the surface under test. The effectiveness and tradeoffs of long runs of transmission line sensor (TLS) will also be examined, to ascertain whether or not arrays and networks can be used to map the state of water across large areas of roadbed or bridge deck surface. In principle a single TLS should be useful over a 150 m distance, so long as the dielectric in contact with it is not overly lossy, permitting spatial resolution of approximately 1 cm. One of the most promising aspects of this technology is the ability to sample various segments of a single transmission line, multiple transmission lines or a combination of both using a single set of TDR electronics in conjunction with commercially available network switches. As such, it is possible that commercially available TDR systems used for soil water analysis in agricultural applications (e.g. Trase system with a model 6020 multiplexer from Soilmoisture Equipment Corp., Santa Barbara, CA) could provide the electronics package for a 256 TLS array system covering an entire bridge, at a cost of approximately $20K. Not only would this provide a significant advantage over light based technologies which sample a single spot, but at a considerably lower cost per sensor site ($20K/256 = $78/sensor). Furthermore, many of these lower cost systems are configurable for remote sensing applications (wireless modems, Bluetooth modules and battery powered electronics) which are particularly attractive attributes for this application.

Chapter 3
Development of a Temperature-Controlled Test Stand

Temperature control experiments were conducted in an insulated container with both a model AC-194 peltier air cooler/heater and model CP-200TT cold plate made by TE Technology Inc. The air cooler/heater was mounted in the lid of the container and the cold plate up through the bottom of the container with the surface just above flush with the bottom interior surface. Power and temperature control was provided and accomplished by power sources and temperature controllers also from TE Technology Inc. A block diagram of the test system is shown in Figure 3.1. A metal pan used as a solution container was placed on top of the cold plate and held the solution that was being tested. The TDR probe was mounted to the pan through a hole cut in the side of it by piercing the three probe rods through a plastic rectangle and fixing the plastic to the side of the pan with silicone adhesive. A temperature probe purchased from Vernier was also placed in the solution container away from the TDR probes to provide temperature readings of the solution as it was cooled or heated. In all experiments involving liquid testing, 1L of the liquid was placed in the container at or slightly below room temperature. Tap water was used for testing and for making salt solutions. Diamond Crystal® Solar Salt of the extra course variety was used for salt solutions and has a purity of up to 99.6 percent. This salt and water were chosen because they are more likely to be similar to the water and salt found on a bridge surface. Waveform spectra of air, water, and 1.0 molar sodium chloride solutions were taken at room temperature and also as the test cell was being cooled. Temperatures well below freezing were obtainable when given proper time and dry ice was often placed around the metal pan to speed the process.

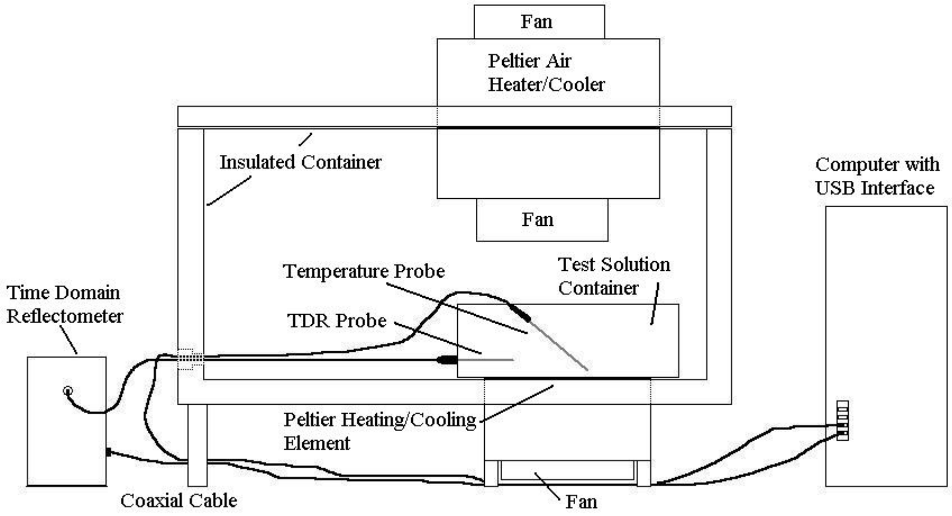


Figure 3.1. Block diagram of temperature controlled test cell.

Chapter 4
Implementation and Evaluation of AC Impedance Approach

Some preliminary programming work was conducted to set up for AC impedance experiments but no data was collected. National Instruments LabVIEW programming software was used to create a program for use with data acquisition interfaces from the same company. The basic approach was to use a swept sine perturbation applied to a planar metal electrode via a potentiostat as depicted in figure 4.1.

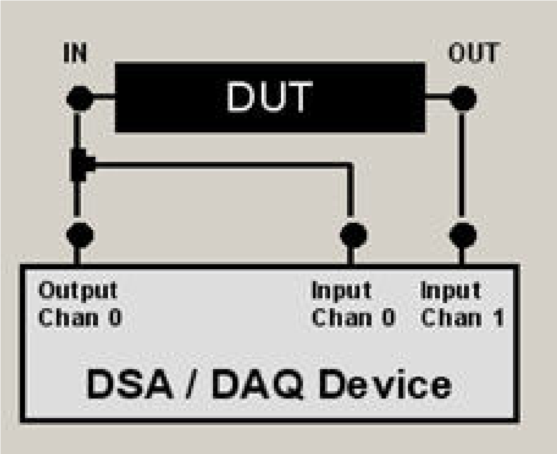


Figure. 4.1. Swept sine implementation.

A desktop computer fitted with 2 high speed data acquisition systems, running LabVIEW 8.1 was employed to generate and simultaneously acquire the sine wave signals swept between 1Hz and 250 KHz. The device under test (DUT) in this case was a commercial potentiostat (BAS, Inc.: BAS-100) connected to a three electrode cell. A Pt working electrode was used. The AC perturbations were applied atb the rest potential of the working electrode in the medium of interest (un-buffered aqueous NaCl).

While reasonable responses were obtained for both dummy loads (RC networks) and electrolyte solutions, preliminary results using TDR on electrolyte solutions as described in the following chapter, obviated the need to pursue AC impedance as a necessary component of the proposed sensing systems. That is, unique TDR responses for various concentrations of NaCl electrolyte in both the liquid and frozen state, suggested that there was no need to also acquire AC impedance data to ascertain the identity and condition of he medium (ice, water, air, electrolyte, etc.) in contact with a passive sensor. For this reason we turned our complete attention towards more detailed examination of the TDR approach.

Chapter 5
Implementation and Evaluation of Time Domain Reflectometry Approach

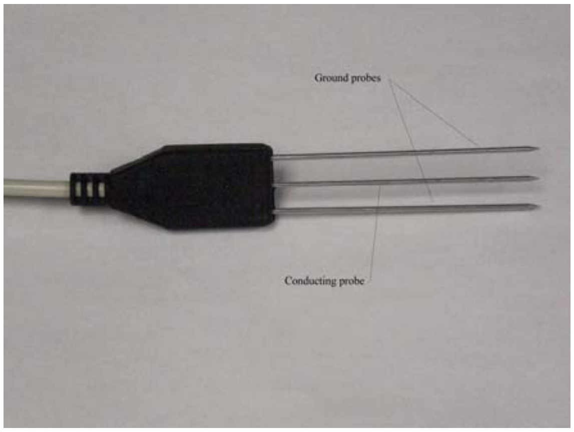


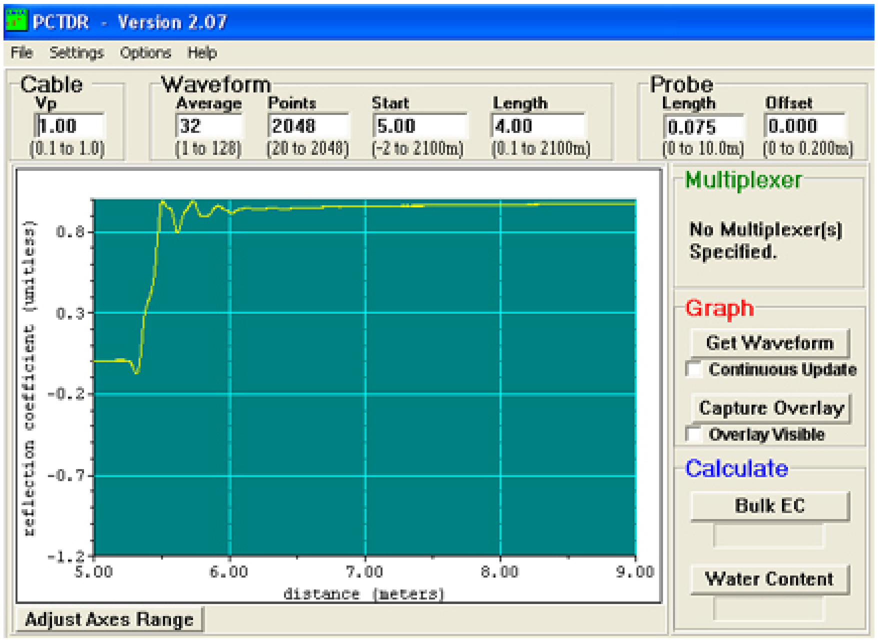
Figure 5.1. Campbell Scientific TDR probe.

TDR probes of the same basic construction as shown in Figure 5.1 were used to test responses from water, air, and sodium chloride solutions. The probes are commercially available from Campbell Scientific® and can be made in different lengths. The probes used in this work had coaxial transmission lines listed as 12 feet long and actual probe lengths of 7.5cm, 15cm, and 30cm. Table 1 shows the actual physical dimensions and part numbers of the probes and transmission lines. The TDR 100 time domain reflectometer was also purchased from Campbell Scientific®.

PCTDR software, provided by Campbell Scientific®, was used to collect the waveforms. It allowed continuous sampling and adequate control over averaging and resolution of the waveforms. Settings were established to provide consistency in the data acquisition. Figure 5.2 is a captured screen of the program interface with the parameters set as used in most experiments. The values in the fields were chosen to provide the most information about the measurement as possible. The Vp field, which is the velocity of propagation in the line, only matters for measuring the actual cable length or finding fault positions in the transmission line. In the case of this research, a value of 1.00 is adequate for determining the location of a dielectric change because values less than within 5 centimeters are unnecessary. The value of 1.00 is also good because it cancels out in calculations for water content. The start value of 5 meters was chosen because the actual Vp value for the transmission line is not 1.00 (meaning equal to the speed of light in a vacuum). The Vp values for the cables used are listed in Table 1. These Vp's are responsible for the start value of the measurement being longer than the actual cable because the apparent length of the cable is calculated as:

$$L_{apparent} = \frac{Vp}{Vp_{cable}} \times L_{actual}$$

This means that for a RG58 cable with an actual length 3.788 meters, such as in the case of probe number 1, the apparent length of the cable is 5.65 meters. As long as the actual Vp of the cable is known, however, back calculations can be done to provide location information of faults or changes in the transmission line.



Cable

Vp 1.00 (0.1 to 1.0)

Waveform

Average 32 (1 to 128)

Points 2048 (20 to 2048)

Start 5.00 (-2 to 2100m)

Length 4.00 (0.1 to 2100m)

Probe

Length 0.075 (0 to 10.0m)

Offset 0.000 (0 to 0.200m)

Multiplexer

No Multiplexer(s) Specified.

Figure 5.2. PCTDR program interface with air waveform displayed.

Vp is the cable Average is the number of averages to perform. Points is the number of samples in the waveform. Start determines the apparent distance from the TDR100 to where the displayed waveform begins. Length (under Waveform heading) specifies the width of the display window. Length (under Probe heading) is the length of the probe rods. Offset is the length of the probe rods that are encapsulated in epoxy or other stabilizing media that are not in contact with the media being measured. Get Waveform and Capture Overlay are part of the acquisition function and the Calculate features were not used as they did not matter for the measurements taken.

The TDR data collected was saved as a text file in ASCII format. This data was then available for analysis using Excel or programs written in LabVIEW. The software allowed the user to select a number of the text files to be used as standards in a database. Derivatives of the waveforms were calculated and used in place of the actual waveforms because they provided better discrimination of key areas that indicate the identity of the contacting medium. Standard sets are chosen by the user of the software and can be as specific as desired. For this work, standard sets of 5, 12, and 14 waveform derivatives were used. The standard sets were composed of one waveform derivative of air and multiple waveform derivatives for water and the 1.0 molar NaCl solutions.

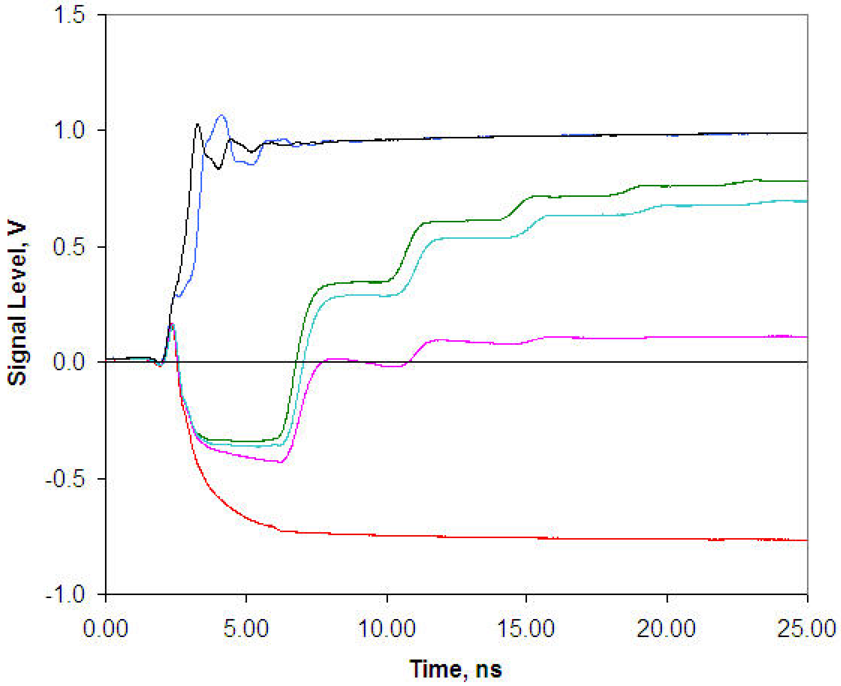


Figure 5.3. Comparison of reflected signals for commercial parallel rod probe, top to bottom: air, ice, water, 0.001 M NaCl,0.01 M NaCl and 0.10 M NaCl. All electrolytes were liquid.

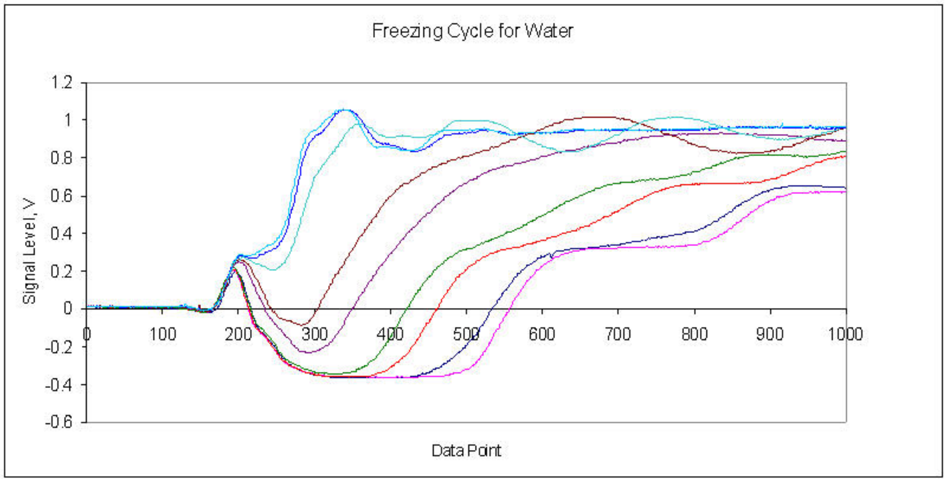


Figure 5.4. Evolution of raw signal during freezing cycle: Bottom, Water (25^o C) – Top, Ice (-5^o C).

Figure 5.3 shows the responses from a 7.5 cm commercial sensor (Fig. 5.1) used for soil moisture testing. While some of these waveforms readily allow one to distinguish the media in contact with the sensor, others (electrolyte and water) are similar enough to require more careful scrutiny. In addition, software evaluating these must provide a readily interpreted result to maintenance personnel or digital systems which would activate warning signs to alert motorist to unsafe conditions. For that reason, we have developed preliminary software to compare sensor responses to calibration runs made on these sensors. Reflection responses such as those shown in Fig. 5.3 are acquired as 1024 point files in which 32 transients are averaged requiring an acquisition time of approximately 1 min.

Figure 5.4 shows a freezing cycle for water in contact with a 7.5 cm commercial probe (Fig. 5.1). The responses at intermediate temperatures suggest that both ice and water were in contact with the probe electrodes during he transition. This strongly argues for miniaturizing the exposed part of the sensor to minimize the area from which the response arises. This should lead to sharper, more definitive transition behavior, and is focus for phase II research.

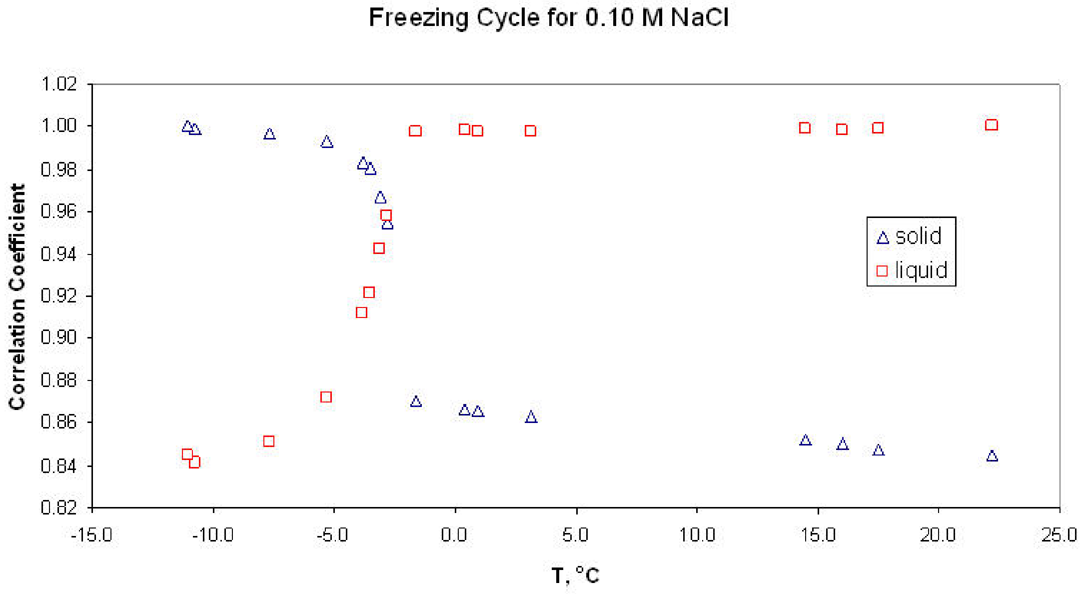


Figure 5.5. Evolution of R for 0.10M NaCl response correlated against standard signals in frozen vs. liquid state. Note that the temperature is acquired at one end of the sensor.

Nonetheless, as the data of Figure 5.5 demonstrate, a single parameter (correlation coefficient) can be used to identify the nature and state of the medium in contact with the sensor electrodes. While we are delighted to have made the progress reflected in the results reported above, we remain concerned about optimal sensor design prior to field deployment. The primary concerns are based on reducing the size of the sensors in terms of the area in contact with the medium being sampled to provide more specific responses; i.e. reduce the possibility of a mixed response leading to ambiguity regarding the state of the sensed medium, such as those observed in freezing and thawing cycles such as shown in Fig. 9. These results are for relatively large sensors with (Figs. 2 and 3). The design tradeoffs suggest that designs which maximize the length of the transmission line segment comprising the active sensor will be most effective.

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