A BROADBAND SPECTROSCOPIC SENSOR PROBE

I M Woodhead[1], I Platt, J H Christie, S Krenek

[1]Lincoln Ventures Ltd, Lincoln University

Canterbury, New Zealand.

Email: Woodhead@lvl.co.nz

***Abstract-** The electrical properties of many materials are closely related to their composition and to their moisture content in particular. For sensor development, characterising the response of a particular volume of material over a broad frequency range is desirable, since separate measurements could generate errors due to spatial variability. A coaxial probe has been designed for measurement of the permittivity of smooth and flat, solid or liquid samples over the frequency range from 1 Hz to 6 GHz. Although the probe is capable of a very wide frequency range, separate instruments are generally required, and here we focus on measurements above 1MHz. We demonstrate measurements in the frequency domain using a vector network analyser, and in the time domain using a broadband oscilloscope. For switching, we employed a coaxial switch and demonstrate how that is included within the instrument calibration. Calibration of the probe used three references: an open circuit, short circuit (indium foil) and a reference liquid, ideally chosen for a permittivity similar to that of the sample. The sample complex permittivity was calculated by a numerical model which used as inputs, the measured reflection coefficient and physical measurements of the probe geometry.*

Index terms: coaxial probe, spectroscopy, calibration, broadband, network analyser.

I INTRODUCTION

The dairy, timber, wool, and many other primary processing industries demand rapid on-line measurement of product properties, particularly moisture. The chief advantage of the dielectric measuring technique is its relatively low cost compared with neutron backscatter, X-ray analysis, and magnetic resonance imaging. Dielectric techniques are also non-ionising, and offer clean, rapid, on-line measurements of bulk material, suited to many diverse products. For a particular product, there will be one or more frequency ranges where the dielectric constant has the highest sensitivity to changes in moisture content (or other measurable parameters). There are various dielectric sensors already available, but in many cases the sensitivity of the sensor is not matched to the dielectric properties of the material being measured. It is also possible that if permittivity spectra were measured for several similar samples of known composition, the parameters describing the dielectric spectra could be correlated with the sample composition. Here we describe methods to measure the dielectric properties of various materials over a broad frequency range, which may be used to investigate appropriate frequency bands to enable custom dielectric sensors to be tailored to the properties of the materials being measured.

Changes in moisture content and the composition of various components within composite materials such as dairy products, wood and masonry, alter their dielectric properties. The electrical properties of the materials are particularly sensitive to changes in moisture content particularly the low frequency, low moisture content conductivity [1] but also the high frequency permittivity due to the large difference in permittivity between free water, with a static relative permittivity of approximately 80 at 20 °C, and many other materials which are generally less than three [2].

The chosen measurement frequency for determining moisture content or composition may lie within a very broad range. Reasons for choosing a particular frequency will include sensitivity of the reading to changes in moisture content and the dependence of the reading on other parameters such as density and dielectric loss. For a particular polar molecular species, as the frequency is increased, the dielectric loss reaches a maximum and is accompanied by a reduction in the permittivity. The frequency of maximum loss is the relaxation frequency, and for free water begins to significantly affect the dielectric properties above approximately 3 GHz. In contrast, the relaxation frequency for bound water is typically 100 MHz [3]. Electrical conductivity is directly useful at low frequencies, particularly for materials with low moisture content where it provides a very useful measure of moisture content, and at high frequencies where it influences the dielectric loss. Apart from the Maxwell-Wagner effect which is caused by conducting inclusions in a dielectric, the predominant contribution to dielectric loss at frequencies below 10 MHz is from conductivity. In dielectrics without conducting inclusions, it is desirable to determine the contribution to the dielectric loss made by conductivity, and subtract this contribution from the loss at higher frequencies. To measure all the above effects, a probe suitable for a range of materials would ideally include measurements at a number of frequencies over the range from below 1 Hz to several GHz. Very low frequencies, typically less than 1Hz, provide information about processes such as curing development in concrete [4] and ion drift [5].

The approach used here has been to employ a coaxial probe and to incorporate a coaxial switch to allow two measurement means, an impedance analyser or automatic bridge for frequencies up to approximately 1 MHz, and a vector network analyser or a time domain reflectometer for higher frequencies. At high frequencies, the complex reflection coefficient from the open-ended coaxial probe depends on the electrical properties of the impedance at the end of the probe. In this case, the sample material terminates the line, and its properties are mirrored in the reflection coefficient. Commonly, measurements are made at a number of discrete frequencies within the desired frequency range, and then after some mathematical processing (e.g. [6]), the complex permittivity of the sample material is obtained.

At lower frequencies, the probe and connecting cables are short in comparison with the wavelength, so the probe characteristics are also influenced by those of the connecting cable. A convenient change-over frequency between the two instruments is 1MHz since this typically represents the upper frequency limit of an automatic bridge that could be used to measure low frequency conductivity and permittivity. For measurement at low moisture content, e.g. below fibre saturation in textiles, the conductivity is very small so specialist techniques such as guarding are usually required.

In this paper, we describe the probe design and how measurements may be obtained in the frequency or time domain, and show measurement results.

II EXPERIMENTAL DETAILS

The probe was fabricated from MPC 14 mm rigid coaxial airline (Maury Microwave Corporation, Ontario, California, USA) rather than using a solid dielectric, to avoid thermal effects induced by the difference in thermal expansion coefficient between teflon and brass (or copper). Use of a teflon dielectric creates difficulties in obtaining a sufficiently flat face at the tip of the probe [7]. The design of the probe is shown in Figure 1 where the inner and outer diameters of the insulator are 6.204 mm and 14.29 mm. The radial dimensions of the transmission line dictate that the transverse electric (TE) and transverse magnetic (TM) transmission modes are not sustained within the line (the cutoff frequency for the TM_{01} and TE_{01} modes is 26 GHz. A ground, stainless steel flange was soldered to the outer (gold over copper) conductor. The 60 mm diameter of the flange was chosen in accordance with the recommendations of [8] and [9] so that it was a sufficiently accurate approximation to an infinite ground plane. An insulating collar made from cross-linked polystyrene was used to locate the centre conductor, and provide a measurement plane between the conductors. This material was chosen for its stability, machineability, low loss, well known dielectric constant of 2.530, and compatibility with acrylic adhesives. The plastic collar was sufficiently long (15 mm) that the higher order modes induced at the air-plastic interface inside the coaxial cable decayed (by a factor $\approx 10^8$) before the probe end, yet was still less than a half wavelength at 6 GHz. The collar was a press fit, and was glued into place using Loctite 406 cyanoacrylate adhesive to ensure a stable, waterproof ground plane. Finally, the ground plane was machined flat.

Figure 1. Coaxial probe fabricated from air line.

The reflection coefficient of the probe was measured between 1 MHz and 6 GHz using an HP8753D vector network analyser (VNA). Calibration of the VNA and probe is a two-step process. In previous work, we calibrated the VNA at the beginning of the probe using a coaxial open, short and 50 Ohm coaxial load, then calibrated the probe using the process described by [6]. Here we show that calibration of the VNA may be accomplished at the probe tip by using an open, a short in the form of Indium foil pressed firmly against the probe tip, and thirdly a bespoke calibration board. The board comprised a circuit board disc with concentric conductors that matched those of the probe, with much of the fibreglass substrate removed between the two electrodes, and incorporating two radially positioned 100 Ohm 1206 size SMD resistors to form a 50 Ohm load. Ideally, the resistors would be thin film microwave components, but here we report on results using standard SMD components.

Within the semi-rigid coaxial line that connected the probe to the VNA, we included a Teledyne coaxial relay CCR-33 (50 Ohm SMA, DC-18GHz) as shown in **Figure 2**. This switch enabled the coaxial probe to be alternatively connected to another instrument for measuring the low frequency (sub 1MHz) impedance of the probe, and the switch was included in the instrument calibration, which took place at the end of the probe. Reflection coefficients were measured at 201 frequencies equi-spaced on a logarithmic scale, with each reading being the arithmetic mean of five measurements. The intermediate frequency band width on the HP8753D was set to 1000 Hz.

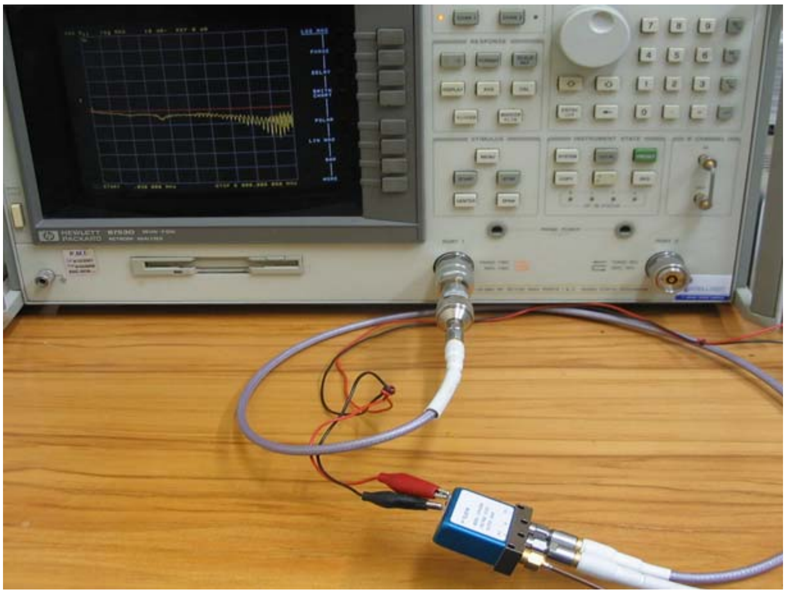

Figure 2. Coaxial relay and network analyser. The centre common connector of the relay was connected to the coaxial probe, and the short length of rigid coaxial line was terminated and connected to low frequency measuring instrument.

III ANALYSIS

The complex reflection coefficient from a terminated line is defined, e.g. [10], as

$$\Gamma = \frac{Z_t - Z_0}{Z_t + Z_0} \qquad (1)$$

where Z_t is the transmission line impedance and Z_0 is the terminating impedance. Rearranging, the terminating impedance becomes:

$$Z_t = \frac{Z_0(1+\Gamma)}{(1-\Gamma)} \tag{2}$$

Then for the case where the terminating impedance is purely capacitive (e.g. open circuit) so that $Z_t = 1/j\omega C$ where ω is the angular frequency and *C* is the terminating capacitance,

$$\varepsilon_r = \frac{(1-\Gamma)}{j\omega Z_0 C_0 (1+\Gamma)} \tag{3}$$

where ε_r is the complex relative permittivity of the material terminating the probe, and C_0 is the open circuit capacitance terminating the probe. The terminating capacitance is just that due to the fringing field that extends beyond the end of the probe. If the reflection coefficient is represented by $a+jb$, then rearranging and separating into real and imaginary parts gives

$$\varepsilon_r^{'} = \frac{-2b}{\omega C_0 Z_0 ((1+a)^2 + b^2)} \tag{4}$$

and

$$\varepsilon_r^{''} = \frac{1-a^2-b^2}{\omega C_0 Z_0 ((1+a)^2 + b^2)} \tag{5}$$

where $\varepsilon_r^{'}$ and $\varepsilon_r^{''}$ are the real and imaginary parts of the relative permittivity of the material terminating the probe.

IV RESULTS

Equation (4) was used to calculate the trace (Figure 3) of the uncalibrated real permittivity of the open circuit probe. We chose $C_0 = 0.13 pF$ to provide a mean ε_r for water (data shown later) of 80, within the frequency range 1 MHz to 1 GHz. The variation in permittivity of the open-circuit probe below 100 MHz was due to small errors of the order of 0.1% in $\varepsilon_r^{''}$, when its value was close to zero - the quoted uncertainty in magnitude for reflection coefficients measured with the HP8753D is ±0.02 (HP8753D operating manual). This highlights the difficulty of accurately measuring zero loss materials, although for practical measurements this situation does not normally arise. The decrease in the value of $\varepsilon_r^{''}$ with increasing frequency is due to a combination of the capacitance of the 50 Ohm calibration board and loss by radiation, a mechanism normally accounted for by calibration of the coaxial probe.

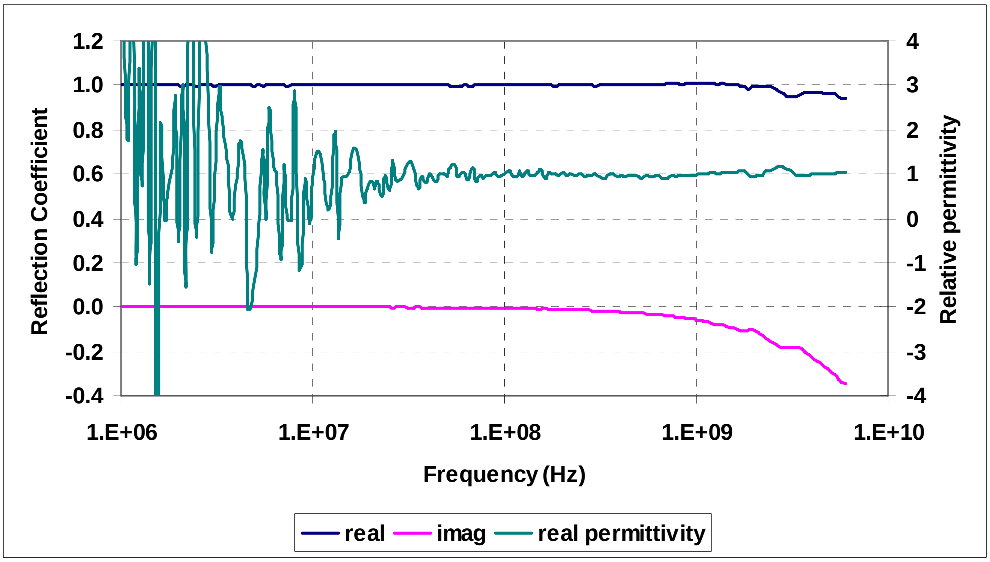

Figure 3. Reflection coefficient and uncalibrated relative permittivity of the open circuit coaxial probe obtained using (5).

Figure 4 shows a typical locus of complex reflection coefficient for water. The result shows close alignment with previous measurements up to 3 GHz. We attribute the deviation beyond 3 GHZ to the capacitance of the resistors used in the 50 Ohm calibration board. Nevertheless, the issue is relatively unimportant for establishing the response and regions of interest for a broadband spectroscopic sensor for industrial measurements.

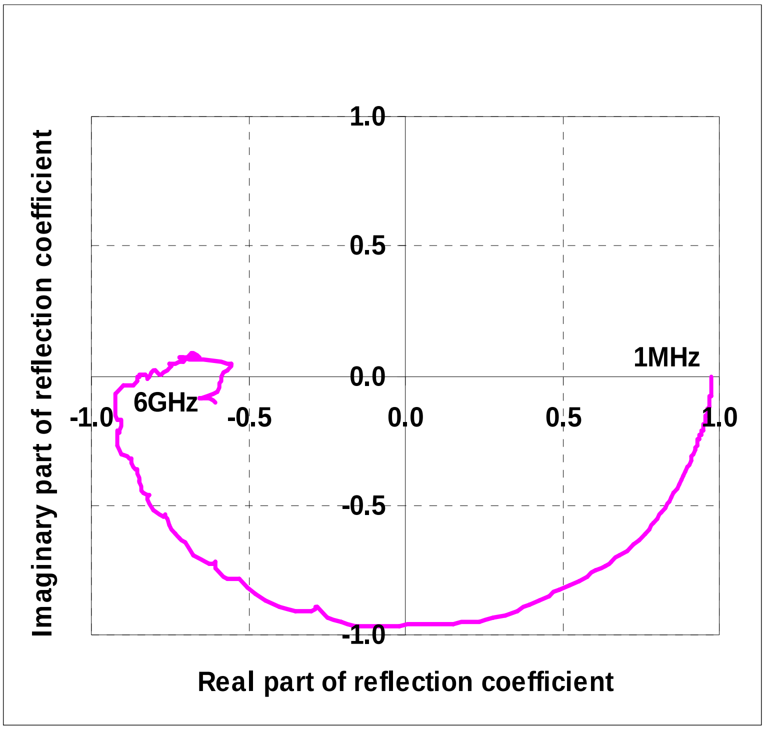

Figure 4. Uncalibrated reflection coefficient of water. Errors above 3GHz were due to capacitance of the 50 Ohm calibration board.

Figure 5 shows the same reflection coefficient data of Figure 4, but converted to permittivity using (4) and (5). As before, the probe was uncalibrated except for the empirical choice $C_0 = 0.13pF$, chosen to provide a mean ε_r for water of 80. The result shows close alignment with the expected value up to approximately 1 GHz, whereupon the capacitance of the resistors used in the 50 Ohm calibration board begins to shift the calibration reference from its ideal value of $50 + j0$ and hence adversely affect calibration of the VNA. Inaccuracies also occur at frequencies where there is resonance in either the reference liquid or the sample. For the geometry and samples used, resonances affect the spectra at frequencies above approximately 1 GHz for water.

Although accurate probe calibration such as described by [6] is appropriate for accurate measurement of complex permittivity, probe calibration for measurement of moisture content or composition is unnecessary. Instead, it is only necessary to provide an overall calibration between the desired product attributes and the broadband complex reflection coefficient.

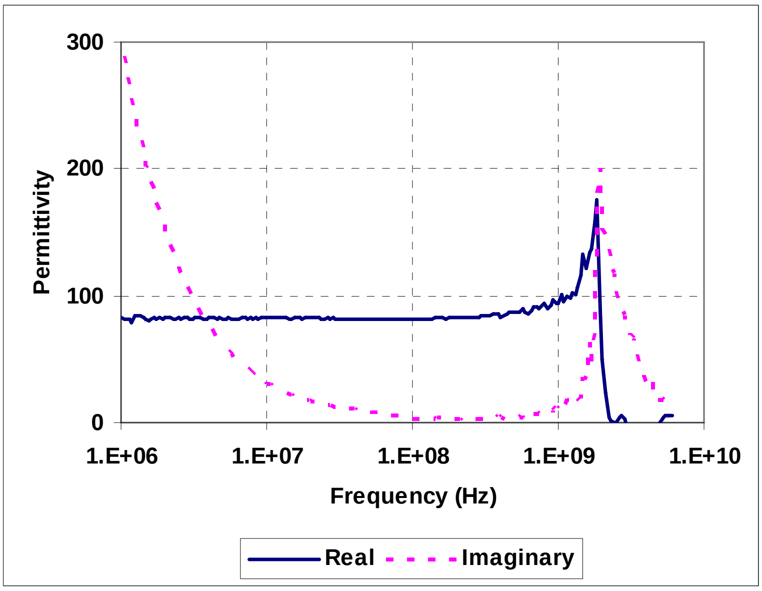

Figure 5 The reflection coefficient and uncalibrated relative permittivity of water obtained using (5).

V MEASURING INSTRUMENT

The results shown above employed an HP8753D vector network analyser, but an alternative approach is to measure in the time domain using a broadband waveform such as a step function and measuring the reflected signal using an oscilloscope. The Hewlett Packard HP54121T for example, includes a synchronised step function generator with risetime <50 ps. The instrument can then be used to measure the reflection coefficient. The procedure we employed was to record waveforms of the open-circuit probe and when in contact with the target material. An automated analysis procedure then aligned the waveforms from time references, truncated the zones beyond the region of interest, and then a ramp function was used to match the amplitudes at the beginning and end of the truncated waveforms as described by [11] and then the ratio of the Fourier transforms of the measured and open waveforms was used to calculate the reflection coefficient in a manner similar to that of [12]. As with the frequency domain approach, use of the time domain method also requires careful calibration of the instrument at a reference plane that is close to the coaxial probe, to achieve repeatable results.

VI CONCLUSIONS

In this paper we have described a coaxial probe for very broadband measurement of the electrical properties of materials. Calibration of the instrument is ideally performed by reference impedances (open, short and 50 Ohms) at the measurement plane, and leads to values of complex reflection coefficient that may in turn be calibrated against moisture content or other properties such as concrete curing. The processing of signals measured in the time domain was also described to yield the complex reflection coefficient equivalent to that from frequency domain measurements. For both measurement means, calibration of the probe itself provides the means to achieve accurate measurement of complex permittivity over a broad frequency range. When measurement of a product is required, only instrument calibration is necessary since the overall transfer function between the moisture content or composition of the product and the reflection coefficient will encompass the probe calibration.

REFERENCES

[1] J. H Christie and I. M. Woodhead. "A New Model of DC Conductivity of Hygroscopic Solids. Part 1: Cellulosic Materials.", Textile Res. J. 2002, 72, pp 273-278

[2] T. Kudra, G. S. V. Raghavan, C. Akyel, R. G. Bosisio, F. R. van de Voort, H. S. Ramaswamy, "Dielectric properties of milk and its constituents at 2.45 GHz", American Society of Agricultural Engineers, June 1991, Albuquerque, paper number 913005

[3] S. Mashimo, S. Kuwabara, S. Yagihara, K. Higasi, "Dielectric relaxation time and structure of bound water in biological materials", Journal of Physical Chemistry, 1987, 91: pp 6337–8

[5] J. H. Christie, S. R. Sylvander, I. M. Woodhead, K. Irie, "The dielectric properties of humid cellulose", Journal of Non-Crystaline Solids, 2004, 341:115.

[4] S. Yagihara, M. Asano, N. Shinyashiki, "Broadband dielectric spectroscopy study on hydration of cement and some aqueous solution and dispersion systems", International Conference on Electromagnetic Wave Interaction with Water and Moist Substances, 4/15-18, 2007, Hamamatsu, Shizuoka.

[7] B. G. Colpitts, “Temperature sensitivity of coaxial probe complex permittivity measurements: experimental approach”, IEEE Transactions on Microwave Theory and Techniques 1993, 41, pp 229–33

[8] P. De Langhe, L. Martens, D. De Zutter, “Design rules for an experimental setup using an open-ended coaxial probe based on theoretical modelling”, IEEE Transactions on Instrumentation and Measurement 1994, 43, pp 810–7

[9] A. P. Gregory, R. N. Clark, T. E. Hodgets, G. T. Symm, “RF and microwave dielectric measurements upon layered materials using reflectometric coaxial sensor”, National Physical Laboratory Report, 1993, DES 125.

[6] N. I. Sheen, I. M. Woodhead, “An open-ended coaxial probe for broad-band permittivity measurement of agricultural products”, J agric Engng Res, 1999, 74, pp193-202.

[10] S. Ramo, J. R. Whinnery, T. van Duzer, “Fields and Waves in Communication Electronics, 1993, New York: Wiley

[11] A. M. Nicolson, “Forming the fast Fourier transform of a step response in time-domain metrology”, Electronics letters, 1973, 9(14), pp 317-318.

[12] W. L. Bellamy, S. O. Nelson, R. G. Leffler, R G, “Development of a time domain reflectometry system for dielectric properties measurement”, Transactions of the ASAE, 1985, 28, 4, pp 1313-1318.