DIELECTRIC MATERIAL MEASUREMENTS AT HIGH TEMPERATURE

J Skatt

Cambridge Consultants, 29 Science Park, Milton Road, Cambridge, CB4 0DW

Email: johan.skatt@cambridgeconsultants.com

ABSTRACT

In this paper we present a method for measuring the permittivity of dielectric materials up to very high temperatures, 1100°C. Enabling some type of materials to be measured beyond their melting point. The proposed method uses a freespace focused beam system. Therefore, it has no limitations on the measurement frequency range, except for any limitations to the antennas used in the test fixture.

An improved numeric algorithm is proposed allowing the extraction of the permittivity correctly from thick, relative to the measurement frequency, samples without having to measure multiple samples with different thickness of the same material. The algorithm also supports de-embedding of the crucible material across temperature for measuring samples beyond melting point.

INTRODUCTION

The importance of measuring the properties of dielectric materials is becoming increasingly important with the higher operating frequencies of many modern radio systems, such as 5G and 6G. However, most commercially available systems for measuring permittivity, and permeability, are limited to room temperature measurements. For example, waveguide or resonator based systems do not provide a way to heat or cool the sample without effecting the measurement fixture itself. The proposed method in this paper uses a freespace focused beam system. This allows the sample to be heated, or cooled, across a very large temperature range without effecting the performance of the measurement system.

TEST SETUP

A free space focus beams system can be realised using a setup like the one shown in Figure 1. In this case the system includes two frequency extenders covering the frequency range 75-110GHz, two spot focus antennas, a Vector Network Analyser (VNA) compatible with the frequency extenders and a tube furnace capable of heating the sample up to 1100°C.

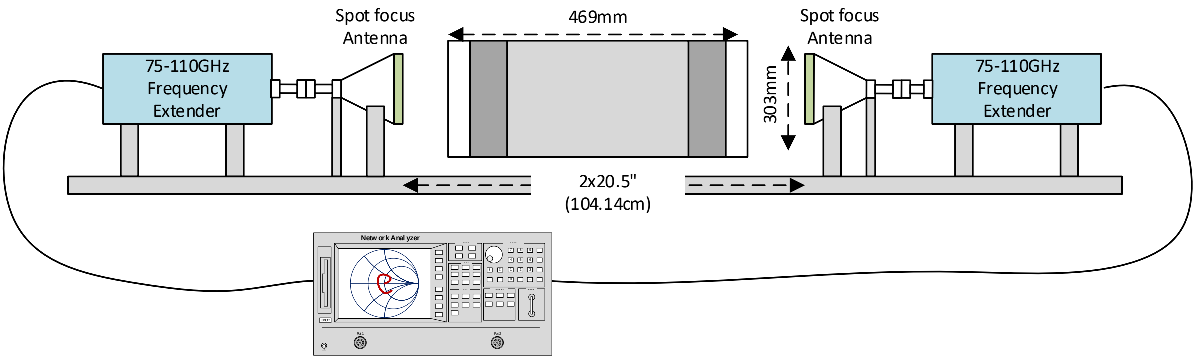


Figure 1 - Free space focused beam system with tube furnace

A spot focus antenna is a horn antenna incorporating a dielectric lens to create a focused beam at some distance, the focal length. At the focal length the 10dB beam width is normally somewhere between 0.5” and 2”, enabling samples with a small diameter to be measured.

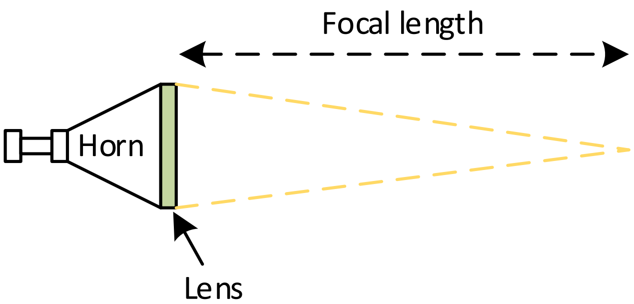


Figure 2 – Spot focus antenna

As with most dielectric measurement systems a Vector Network Analyzer (VNA) is used. The measured S-parameters can then converted to complex permittivity using either the Nicolson-Ross-Weir (NRW) algorithm, [2], or numerically using one of the algorithms proposed by James Baker-Jarvis in [1].

A tube furnace is also included in this test setup. The furnace allows precise heating of the sample whilst measuring the S-parameters.

ALGORITHM

A focused beam free space system requires samples sizes with a minimum diameter of three times the beamwidth (spot size) to avoid fringe effects. A spot size around 25mm is achievable with the horn antenna used when fitted with a dielectric lens. Hence, a sample diameter >75mm is required for accurate measurements. To ensure a sample of this size does not flex or bend in the fixture it does require a thickness of a millimetre or more depending on the type of material. For mmWave frequencies that means the sample thickness may be several wavelengths.

A sample thickness of more than one wavelength causes phase wrapping, i.e., ambiguous group delay through the sample. This translates into multiple solutions for the permittivity. This is a known issue with the Nicolson-Ross-Weir algorithm due to the infinite number of roots as explained below. However, looking at Equation 1 it is not obvious that the numeric iterative NIST algorithm also suffer from multiple solutions with thick samples. A novel approach to overcome this issue for isotropic materials for the NIST algorithm is described below.

Nicolson-Ross-Weir algorithm

The NRW algorithm provides a direct conversion from S-parameters to permittivity and permeability. However, it has one major drawback in that the term $ln\frac{1}{T}$ in the equation has an infinite number of roots since the imaginary part of it is $j(\theta + 2\pi n)$ where $n = 0, \pm 1, \pm 2, ...$ For thin samples, with a thickness $< \lambda$ at the measurement frequency, this is not an issue as $n = 0$. For thick samples we must estimate the group delay through the sampled to identify the correct value of n. This can be done by measuring two identical samples with different thicknesses. Note, the thickness difference between the two samples needs to be $< \lambda$ to ensure a single value of n can be identified.

In cases where it is difficult, or not possible, to produce two samples with identical material properties the NRW algorithm becomes unsuitable for thick samples with unknown group delay, and an iterative algorithm offers a better option.

NIST algorithm

Two iterative algorithms were proposed in [1] by James Baker-Jarvis. One suitable for through measurements of S-parameters and the second one considering the use of a reflective plate behind the sample for a reflective measurement.

The latter is limited to measuring permittivity or permeability as it only uses S_{11} for the measurement. It also requires the use of an electromagnetic reflective surface, e.g., a metal plate, fitted behind the sample. The use of a metal plate makes it difficult to use at high temperatures where the metal may become soft or its properties change.

The through method has a number of advantages when measuring samples across temperature.

a) It is possible to measure a full set of S-parameters. Hence, it is possible to extract both permittivity and permeability if required.
b) It is possible to correct for any curvature in the sample by averaging S_{21} and S_{12}
c) Only S_{21} and S_{12} measurement is required to extract the permittivity.
d) It does not require a reflective plate behind the sample.
e) Very accurate calibration of the VNA can be completed. Either only through calibration if only S_{21} and S_{12} is required. Or Through, Reflect and Line (TRL) calibration in the case were a complete set of S-parameters is required.

The relationship between S_{21} and S_{12} and the permittivity for a free space measurement system can be expressed as Equation 1, see [1] and [3].

$$f(\varepsilon_r) = (1 - \Gamma^2 T^2)\frac{S_{21} + S_{12}}{2} - T(1 - \Gamma^2)$$

Equation 1 - Permittivity and S-parameter relationship

Where Γ is the reflection coefficient and the T is the transmission coefficient, defined as:

$$\Gamma = \frac{\sqrt{\frac{\mu_r}{\varepsilon_r}} - 1}{\sqrt{\frac{\mu_r}{\varepsilon_r}} + 1}$$

$$T = e^{-jk_0 t\sqrt{\mu_r \varepsilon_r}}$$

Equation 2 – Relation between reflection, transmission coefficients and permittivity and permeability

Combining Equation 1 and Equation 2 we can solve the roots to Equation 1 from $f(\varepsilon_r) = 0$. This is a very difficult equation to solve analytically but it is straight forward to do numerically using the Newton-Raphson method. However, there are two issues with using Newton-Raphson when solving Equation 1 for ε_r in thick samples.

a) Multiple roots – As discussed previously a thick sample will have multiple roots.
b) Stability and Convergence – Newton-Raphson requires a starting value. If the chosen starting value is poorly selected, or the permittivity of the material is completely unknown, the solution maybe converge to the incorrect root or not converge at all.

These two issues are independent and can be resolved separately.

Stability and Convergence

Several ways have been investigated in the literature to improve the stability and convergence of the Newton-Raphson algorithm. One of the most common ways is to introduce a damping factor a to the iterative equation.

$$x_{n+1} = x_n + a\frac{f(x)}{f'(x)}$$

With $a < 1$ the convergence improves, however the smaller the value of a the slower convergence is. One way of optimising a is to used backtracking. If we consider $|f(x_{n+1})| < |f(x_n)|$ as a requirement for convergence, we can use the following algorithm to set a.

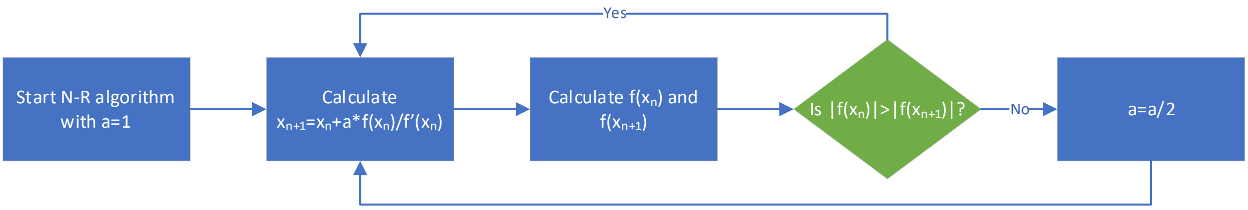


Figure 3 – Newton-Raphson with backtracking

The introduction of back tracking ensures stability as the algorithm cannot diverge since we require $f(x_{n+1})| < |f(x_n)|$. However, back tracking does not eliminate the problem of converging to an invalid root if the starting value of ε_r is chosen incorrectly.

Root convergence

Due to the sample thickness a number of solutions to $f(\varepsilon_r) = 0$ are possible, and convergence is heavily dependent of the starting value of ε_r used for Newton-Raphson. To identify the correct root for convergence an algorithm suitable for non-magnetic isotropic materials was developed.

As mentioned previously a thick, for the measurement frequency, sample will have a number of roots to $f(\varepsilon_r)$. For the test setup described above the S-parameters were measured for an 8 mm thick piece of glass and the real part of $f(\varepsilon_r)$ at 100 GHz for ε_r' from 1 to 10 and $\varepsilon_r'' = 0$ plotted in Figure 4. As can be seen there are twelve solutions to $\Re[f(\varepsilon_r)]$. In comparison a 1.6mm thick piece of FR4 only have two solutions to $\Re[f(\varepsilon_r)]$.

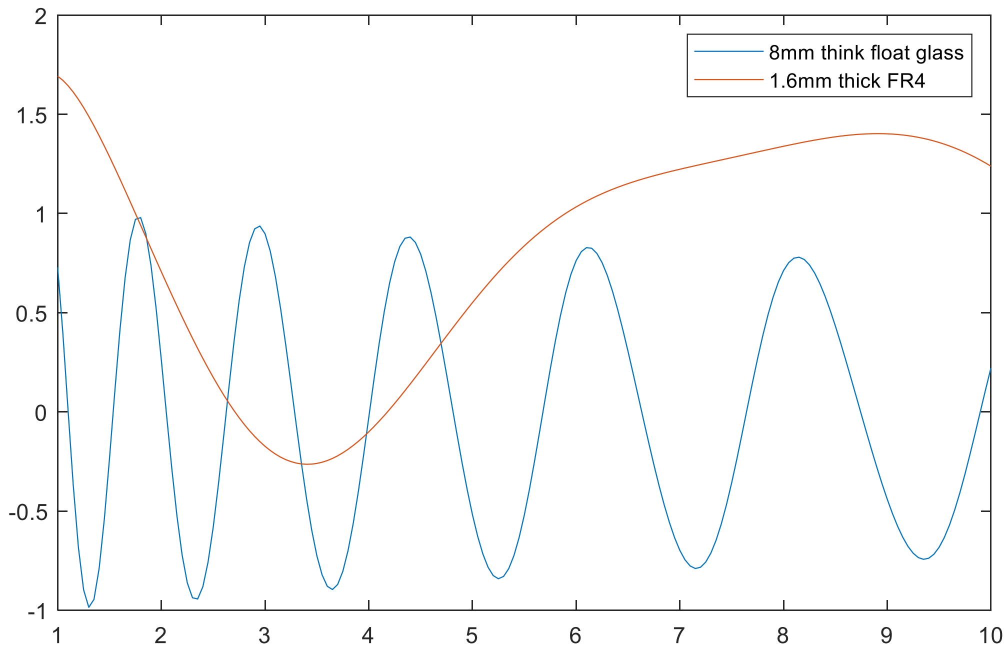


Figure 4: $f(\varepsilon_r)$ for 8mm thick float glass and 1.6mm thick FR4 PCB at 100GHz

For an isotropic material the real part of ε_r is relatively stable across a small frequency range. Note, the imaginary part, representing the loss of the material, may increase substantially across the same frequency range. With these assumptions let us consider the deviation of the real part of ε_r across frequency and its relationship to the roots of $\Re[f(\varepsilon_r)]$. If plotting the starting guess of ε_r versus the converged ε_r across the measured frequency range we can see the spread in the converged ε_r, as shown in Figure 5, versus the start guess of ε_r. For this example, the graph shows the smallest deviation for the converged ε_r with a starting ε_r between 6 and 8, with a converged $\varepsilon_r \approx 6.7$. This is in line with the data sheet suggested range for $\varepsilon_r = 6.5 - 7.5$[1].

[1] Permittivity quoted at 1MHz.

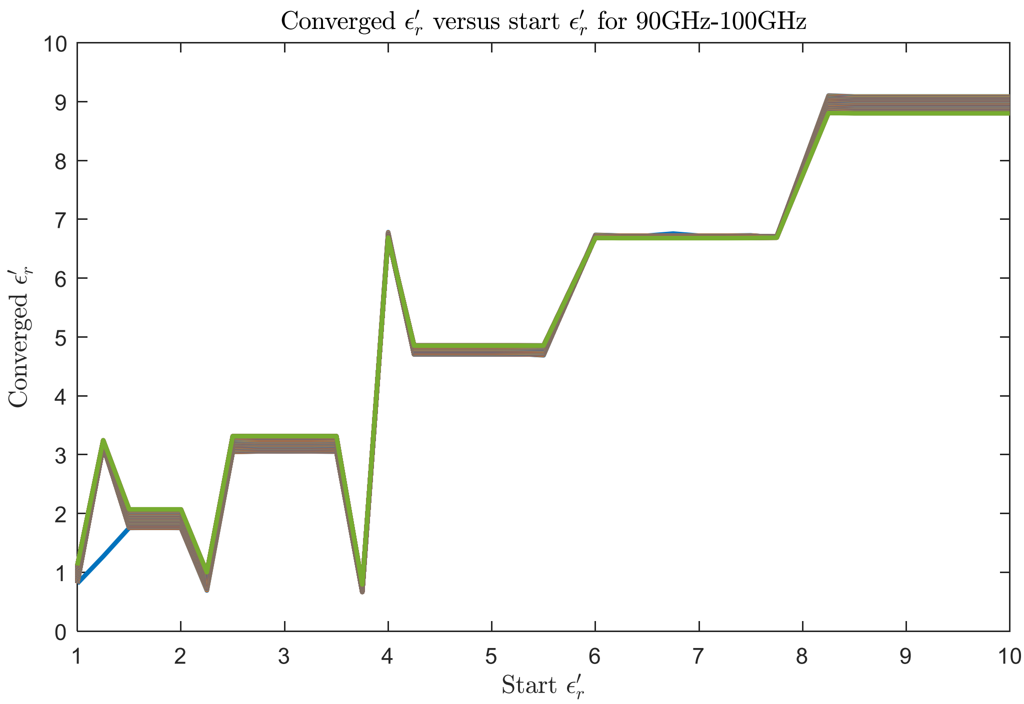


Figure 5: Deviation in converged ε_r versus start ε_r for 8 mm thick float glass at 22°C

Based on these results and assumptions we can devise an algorithm that searches for a suitable starting guess for a given range and step size of ε_r. The algorithm solves $f(\varepsilon_r) = 0$ for a set of start ε_r defined across the whole frequency range being measured. It then calculates standard deviation of the real part of the converged roots, selects the root with the smallest deviation in the real part of ε_r as the correct root, and uses this as the starting guess when calculation ε_r.

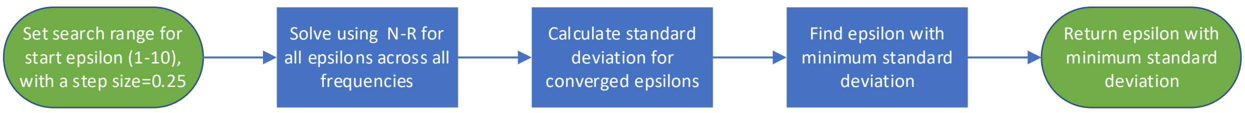


Figure 6: Root search algorithm

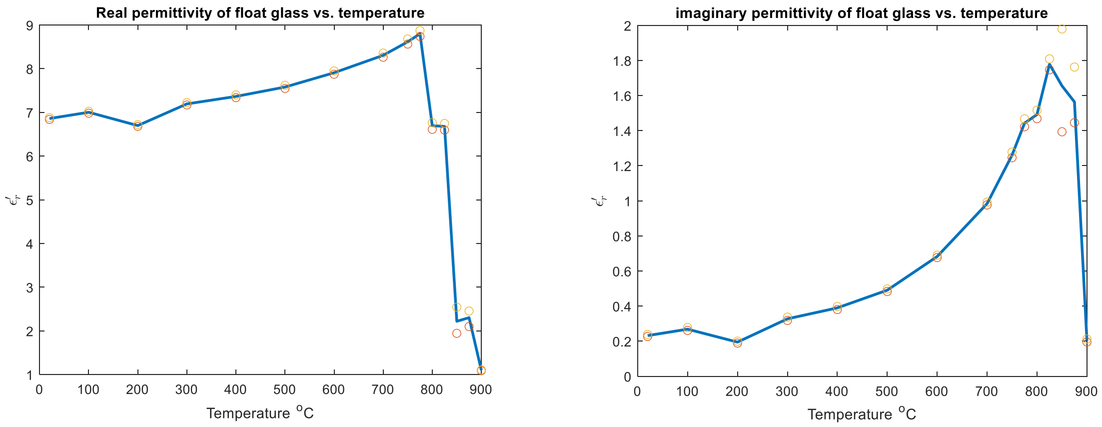
This algorithm:

- Eliminates the need for multiple identical material samples of different thickness to determine the group delay.
- If the permittivity of the material changes substantially with temperature the algorithm will search for and find the correct root at each given temperature.

RESULTS

Using the measurement method described and the algorithm developed the permittivity of float glass was measured up to and beyond the melting point (800°C). The results are shown Figure 7, as can be seen the real part of the glass increases from 6.7 at room temperature to 8.8 just before the melting point. Corresponding to a frequency response shift of more than 30%.

Over the same temperature range the change to the imaginary part is even greater going from 0.2 to 1.4. Meaning the loss tangent, $\tan\delta$, goes from 0.03 to 0.16. corresponding to an increase in loss with over 400%.



a) Real permittivity **b) Imaginary permittivity**

Figure 7: Permittivity of float glass from 22°C up to 800°C

CONCLUSIONS

Using an improved setup of the free space measurement technique for characterising the dielectric properties of materials we have shown it is possible to measure thick samples of dielectric materials up to very high temperatures accurately, all the way up to the melting point of the material.

Furthermore, we have developed a novel algorithm for extracting the correct complex permittivity from thick samples of isotropic materials without the need for group delay estimation. Enabling the characterisation of materials at high frequencies without the need to use very small or thin samples.

REFERENCES

[1]	NIST Technical Note 1341, Transmission/Reflection and Short-Circuit Line Permittivity Measurements
[2]	Nicolson-Ross-Weir method
[3]	Focused beam methods, John W. Schultz. ISBN: 1480092851