Reference-Plane Invariant Free Space Dielectric Material Characterization up to 330 GHz

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Abstract

This paper describes the process followed to implement a system to characterize the complex permittivity of materials in the 10-330 GHz frequency band. Firstly, the method used and the system's calibration process are shown, consisting of a double calibration TRL (Thru-Reflect-Line) and GRL (Gated-Reflect-Line). Subsequently, a smoothing technique is used to improve the accuracy of the results. Finally, a test is performed on quartz and glass fiber samples, showing that the results are quite reliable over the entire measured bandwidth.

1 Introduction

In the communications industry, various materials are employed to design and manufacture devices. These materials exhibit diversity and can be tailored for specific purposes such as developing radiating, absorbing [1], or reflecting devices [2], among others. However, properly utilizing these materials requires a foundational understanding of their electromagnetic properties [3]. Notably, electrical permittivity and magnetic permeability play significant roles for non-conductive materials. Both are intricate parameters that distinctly elucidate the behavior of these materials in the presence of an electromagnetic field.

Traditionally, two main characterization methods can be found in the literature. On the one hand, resonant methods [4, 5] use cavity resonances to extract material properties at specific frequencies. These methods are highly accurate, although they have the disadvantage of providing a set of solutions in a discrete domain of frequencies corresponding to those at which resonances occur within a cavity. On the other hand, non-resonant or broadband methods [6] have traditionally been based on measurements of reflection and transmission of the material in free space. A notable approach in this category is the Nicolson-RossWeir (NRW) method [7], ideally functioning across an infinite bandwidth but practically limited by the transmission system of the waveguide and antenna. The challenge with such methods lies in demanding a high degree of alignment accuracy and the application of calibration techniques to isolate sample effects concerning propagation in free space. In this context, Baker-Jarvis et al. introduced a method in 1990 enabling the extraction of material characteristics from sample measurements [8]. This method requires precise knowledge of the distance between calibration planes and the sample thickness for accurate results. In recent years, with the new 5G and 6G frequency bands, the characterization of materials in bands beyond 100 GHz has become an interesting topic for researchers [9].

Generally, techniques for extracting material properties are further divided into three categories. The first one calculates permittivity, \mathcal{E}_r^* , and permeability, μ_r^* , from equations in which the unknowns are variables to be cleared, using transmission media, cavities, or free-space transmission and reflection [10]. The second group comprises genetic, evolutionary, or optimization algorithms and analytical equations [11], which cannot be solved directly. The last group of methods proposes using optimization algorithms using electromagnetic simulators, which try to find similarities between measurements and simulations to study the characteristics of materials [12].

In this contribution, we will use a broadband method based on the measurement in free space and reference-plane invariant, which starts from the solution of analytical equations for estimating the material parameters. Section 2 describes the measurement method, calibration process, and a technique to post-process the achieved results. In Section 3, an experimental validation is carried out with quartz and glass fiber samples in the 10-330 GHz band. Finally, Section 4 details the most important conclusions drawn from the work.

2 Method Description

2.1 Measurement Scheme

The starting point of the measurement method used was proposed in [10], corresponding to a variation of the one proposed in [8]. Specifically, the authors of [10] proposed the use of an air-dielectric coaxial guide, through which a TEM mode is propagated, knowing the distance between antennas perfectly, Lair, and the thickness of the material to be characterized, L. From this premise, they proposed a system invariant to the relative position of the material with the reference planes, i.e., in which the material could not be at the center of the transmission line. The main limitation of this method is the need to have an air-filled coaxial guide in which the material samples to be characterized could be inserted. To facilitate the measurement method, this work proposed using a free space measurement scheme, a system composed of horn antennas and lenses that generate a flat wavefront, as shown in the scheme in Fig. 1. In this case, the main difficulty is accurately knowing the air gap L_{air} . For this, it is necessary to address the calibration of the system.

2.2 Calibration

Two methods are proposed to be used together to perform the system calibration. First, a TRL calibration is performed at the end of the waveguide feeding the antenna. For this purpose, an aluminum kit has been designed for the WR-75, WR-51, WR-34, WR-22, and WR-15 standards, consisting of a short circuit, which will act as a reflect, and a 2 mm long line. The rest of the bands are calibrated using the millimeter-wave converter calibration kits, made by Rohde & Schwarz. This method places the reference planes just at the horns' entrance, specifically at the waveguidehorn transition. After the measurement, a free space GRL calibration is performed. Initially proposed in [13], this calibration consists of taking two measures: (i) of the empty sample holder and (ii) of the sample holder holding a metal plate, in which total reflection of the plane wavefront is assumed. A process similar to that followed in the TRL calibration is performed from both measurements. The main difference is that a time-gating process, a convenient technique used in both antenna and communications communities [14], is carried out to isolate the effects of propagation to the sample holder. This gating includes the effects of the antennas and lenses used to form the plane wavefront. After calibration, the reference planes are assumed to be in the material on which the normal incidence of a plane wave is occurring.

2.3 Post-processing

The measurement results are highly affected by spurious signals that can affect the estimation of the material parameters. In this sense, several techniques for smoothing the results to make them more reliable can be found in the literature. One of the most outstanding techniques is the one proposed in [15], using Savitzky-Golay filters. In this case, it has been demonstrated that using these filters is equivalent to, or even more efficient than, using other techniques, such as time-gating, when it comes to reducing possible signal interferences, such as reflections in the measurement system. That is why we have used this type of filter for this contribution, with polynomials of order two and 51-point windows for processing the results.

3 Validation

An experiment was carried out in the 10 to 330 GHz band to validate the proposed methodology. For this purpose, a setup consisting of the Keysight N5247B network analyzer, a set of transitions or frequency extenders, and horn antennas was mounted. In addition, two Greenlight lenses were positioned, with which a flat wavefront was created for frequencies below 50 GHz. Finally, a 0.94 mm thick quartz sample and a 1.22 mm thick glass fiber sample were placed in the center of the optical table without considering that they had to be located at the midpoint between the antennas. Fig. 2 shows a picture of the complete measurement scheme. The measurement process starts by setting the frequency limits of the analyzer and establishing 2001 measurement points with +5 dBm power at the ports and an intermediate frequency bandwidth of 1 kHz. Subsequently, a TRL calibration is performed on the analyzer with the kit mentioned in section 2.2. Once this is done, the antennas are connected and correctly aligned. Finally, placing the sample holder and taking measurements of the same vacuum, with a metal plate and the material to be characterized, is necessary. Once this process is finished, the GRL calibration is performed in MATLAB, allowing isolating the effects of the sample and the empty sample holder. The measured S-parameters of the two samples are shown in Fig. 3. As can be seen, the measurements are quite clean after performing the calibration. In addition, there is good continuity between adjacent bands, which shows that the measurement and calibration process has been completed correctly.

After performing both calibrations, it is time to apply the characterization method. At this point, it is important to note that the thickness of the metal plate used for calibration is slightly greater than that of the material to be measured ($L_{air} = 2 \text{ mm}$), so we proceed to include this data in the characterization method. The results of the complex relative permittivity are shown in Figs. 4 and 5. As can be seen, they are similar to those expected from these materials. The real part of the permittivity obtained is quite flat and is only affected at points where the sample length coincides with a multiple of the wavelength and resonances that occur. This effect is common in this type of broadband method. The imaginary part, on the other hand, suffers more significant variations due to the position of the sample. When small misalignments occur, or normal incidence is not guaran-



Figure 1. Measurement scheme used for characterization of materials in free space.



Figure 2. Photograph of the setup used for material characterization.



Figure 3. Measured S-parameters of the quartz (blue) and glass fiber (orange) samples, after the calibration process.

teed, the imaginary part, directly related to the losses, is seriously affected, so the estimation deteriorates considerably.



Figure 4. Estimated real part of the effective relative permittivity (ϵ'_r) of the quartz (blue) and glass fiber (orange) samples.



Figure 5. Estimated imaginary part of the effective relative permittivity (ε_r'') of the quartz (blue) and glass fiber (orange) samples.

4 Conclusions

In this work, we have proposed a setup for characterizing materials' complex permittivity from 10 to 330 GHz. To achieve this, we employed the classical NRW method and the Baker-Jarvis variant to implement a broadband approach invariant to the material's position during the measurement process. Following this, we explored the system's calibration to obtain the estimation. Furthermore, we applied a processing technique to enhance the response at frequency points affected by external factors, such as reflections on the optical table. Lastly, an experimental validation was performed using quartz and glass fiber samples. The results were verified to be accurate across the entire bandwidth.

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References

- M. Gao, Q. Chen, Y. Zheng, L. Ding, D. Liao, and Y. Fu, "Ultrabroadband absorber based on layered inkjet-printing resistive film," *IEEE Antennas and Wireless Propagation Letters*, vol. 22, no. 2, pp. 228– 232, 2023.
- [2] A. Palomares-Caballero, C. Molero, P. Padilla, M. García-Vigueras, and R. Gillard, "Wideband 3-dprinted metal-only reflectarray for controlling orthogonal linear polarizations," *IEEE Transactions on Antennas and Propagation*, vol. 71, no. 3, pp. 2247– 2258, 2023.
- [3] M. Pérez-Escribano and E. Márquez-Segura, "Parameters characterization of dielectric materials samples in microwave and millimeter-wave bands," *IEEE Transactions on Microwave Theory and Techniques*, vol. 69, no. 3, pp. 1723–1732, 2021.
- [4] C. Weil, C. Jones, Y. Kantur, and J. Grosvenor, "On rf material characterization in the stripline cavity," *IEEE Transactions on Microwave Theory and Techniques*, vol. 48, no. 2, pp. 266–275, 2000.
- [5] M. Santra and K. Limaye, "Estimation of complex permittivity of arbitrary shape and size dielectric samples using cavity measurement technique at microwave frequencies," *IEEE Transactions on Microwave Theory and Techniques*, vol. 53, no. 2, pp. 718–722, 2005.

- [6] L. A. Bronckers and A. B. Smolders, "Broadband material characterization method using a cpw with a novel calibration technique," *IEEE Antennas and Wireless Propagation Letters*, vol. 15, pp. 1763–1766, 2016.
- [7] A. M. Nicolson and G. F. Ross, "Measurement of the intrinsic properties of materials by time-domain techniques," *IEEE Transactions on Instrumentation and Measurement*, vol. 19, no. 4, pp. 377–382, 1970.
- [8] J. Baker-Jarvis, E. Vanzura, and W. Kissick, "Improved technique for determining complex permittivity with the transmission/reflection method," *IEEE Transactions on Microwave Theory and Techniques*, vol. 38, no. 8, pp. 1096–1103, 1990.
- [9] M. Shu, X. Shang, N. Ridler, A. R. Calleau, A. I. Dimitriadis, and A. Zhang, "Improvements to millimeterwave dielectric measurement using material characterization kit (mck)," *IEEE Transactions on Instrumentation and Measurement*, vol. 73, pp. 1–8, 2024.
- [10] K. Chalapat, K. Sarvala, J. Li, and G. S. Paraoanu, "Wideband reference-plane invariant method for measuring electromagnetic parameters of materials," *IEEE Transactions on Microwave Theory and Techniques*, vol. 57, no. 9, pp. 2257–2267, 2009.
- [11] J. Zhang, M. Y. Koledintseva, J. L. Drewniak, D. J. Pommerenke, R. E. DuBroff, Z. Yang, W. Cheng, K. N. Rozanov, G. Antonini, and A. Orlandi, "Reconstruction of dispersive dielectric properties for PCB substrates using a genetic algorithm," *IEEE Transactions on Electromagnetic Compatibility*, vol. 50, no. 3, pp. 704–714, 2008.
- [12] A. Hosseinbeig, S. Marathe, and D. Pommerenke, "Characterization of relative complex permittivity and permeability for magneto-dielectric sheets," *IEEE Transactions on Electromagnetic Compatibility*, vol. 60, no. 6, pp. 1786–1794, 2018.
- [13] P. Bartley and S. Begley, "Improved free-space sparameter calibration," in 2005 IEEE Instrumentation and Measurement Technology Conference Proceedings, vol. 1, 2005, pp. 372–375.
- [14] A. Ramírez-Arroyo, A. Alex-Amor, C. García-García, A. Palomares-Caballero, P. Padilla, and J. F. Valenzuela-Valdés, "Time-gating technique for recreating complex scenarios in 5G systems," *IEEE Access*, vol. 8, pp. 183 583–183 595, 2020.
- [15] D. Ma, X. Shang, N. M. Ridler, and W. Wu, "Assessing the impact of data filtering techniques on material characterization at millimeter-wave frequencies," *IEEE Transactions on Instrumentation and Measurement*, vol. 70, pp. 1–4, 2021.