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Material characterization and uncertainty evaluation at millimetre wave frequencies in TUBITAK UME

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ABSTRACT
The frequency response of the materials which are used in communication systems has gained increasing importance nowadays. Frequency response measurements of materials in laboratory environments do not contain sufficient information about real working environment conditions. The free space dielectric measurement method, known as one of the most frequently used material characterization methods at high frequencies, is used to give more accurate results under real operating conditions. In this study, using the TUBITAK UME's infrastructure, the measurement results in free space at millimetre wave frequency, and the uncertainty calculations of the measurements are given for some materials such as Teflon, Fr4, Komacel, and Air. Measurements were performed at two different frequency bands such as E and D bands by using KMMS software which is known to run up to 50 GHz frequency. Measurement results of materials were compared with the known low-frequency response of the materials in this study. And uncertainty calculation model was discussed in this study either.

Section: RESEARCH PAPER

Keywords: Relative dielectric; material characterization; millimetre wave; Keysight material measurement suit

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1. INTRODUCTION

Frequency response of the materials which are used in high frequency is getting important in the world nowadays. Many insulators, semiconductor materials and metals are used in system and circuits design in high frequencies. While the electronic circuits were being realized, copper-clad fibre bases are used for fixing the electronic components. Moreover Teflon-derived Radom is also used for the housing of the outdoor antennas and Teflon is used as insulator for the coating or protection of the circuits. In circuit design, simulation tools are used and generic material properties are used in this simulation tools, nowadays. Designers can tailor circuits accurately if actual frequency responses of used materials at the operating frequencies is known well.

In order to determine the frequency response of a material, it will be sufficient to measure the permittivity (ε) (dielectric coefficient) and permeability (μ) (magnetic coefficient).

Relative dielectric coefficient (ε_r) of a material is defined in equation (1) [1].

$$\kappa = \varepsilon_\mathrm{r} = \frac{\varepsilon}{\varepsilon_0} = \varepsilon_\mathrm{r}' + j\,\varepsilon_\mathrm{r}'' \,, \tag{1}$$

where, ε_0 is vacuum permittivity, and ε is frequency-dependent permittivity of the material in complex number. Relative dielectric coefficient is a dimensionless number that is in general complex-valued; real ε_r') and imaginary (ε_r'') parts are denoted in equation (1).

Relative dielectric coefficient of a medium is related to its electrical susceptibility. There are so many publications and studies on the relative permittivity measurements in literature [2]-[11].

By the development of measuring devices such as Vector Network Analysers (VNA), vector measurements of S-parameters and measurement of the relative dielectric coefficient related with S-parameters have been made possible.

Component of the complex relative dielectric coefficient contains some information for designers. The real part (ε_r') of the relative dielectric coefficient of the material gives information about the energy it can store at the operating frequency, while the imaginary part (ε_r'') gives information about the energy it will absorb.

There are so many relative dielectric measurement methods in the literature and measurement tools in the market. Relative dielectric coefficients measurement methods such as transmission lines, open-ended coaxial probes, resonator cavities, free space method (for lower and higher frequencies), parallel plates and inductance (up to 1 GHz) are commonly used at high frequencies.

Transmission line [2], [3] and free space measurement method [4], [5] are used to measure the complex dielectric coefficient and complex magnetic coefficient of materials, whereas open-ended coaxial probe kits [6] are used to measure the complex dielectric coefficient only. Although the reflection coefficients of materials are generally measured with arch measurement systems, studies are continuing for its use in electromagnetic material characterization [7].

The transmission line method is a useful technique for determining the dielectric and magnetic properties of the rectangular prism-shaped material, sized in the waveguide cross-section, in the frequency range of the waveguide used [8].

All other methods are operated under laboratory conditions and under special ambient conditions, except for the free space method **Fehler! Verweisquelle konnte nicht gefunden werden.**.

Relative permittivity and permeability measurement methods are summarized in Table 1.

Table 1. Relative permittivity and permeability measurement methods.

Measurement Method	Measurand	Environment
Open ended coaxial probe	ε	In lab
Transmission line	ε, μ	In lab
Resonator Cavity	ε	In lab
Free space	ε, μ	In / out lab
Parallel plate	ε	In lab
Inductance	ε	In lab

The free space method also allows for measurements using special heating or cooling systems or special ambient conditioning systems such as clamshell furnace.

The advantages and disadvantages of the free space measurement method are given in Table 2.

Table 2. Advantages and disadvantages of the free space measurement method.

Advantages	Disadvantages
• Preferred for high frequency measurement	• Wide and flat MUT surface is needed, if the frequency decreases
• Allows non-destructive measurement of material under test (MUT)	• Multiple reflections between antenna and surface of material.
• Allows measurements of the MUT in harsh environments	• Diffraction effects at the edge of MUT
• The magnetic and electric properties of MUT can be evaluated	• Sensitive to MUT surface roughness

Millimetre wave frequencies are getting important by 5G/6G communication systems and autonomic vehicles in recent years. In the scientific literature there are a lot of studies on the measurements of dielectric coefficients when millimetre wave frequencies are achievable [9], [10], [11].

On the other hands, dielectric coefficient measurements can be made by using some special dielectric measurement kits (SWISSto12) [12] and some special coaxial probes with specific software (Keysight Material Measurement Suits – KMMS etc.).

Some costumers of TUBITAK UME have asked the frequency response of some materials such as Teflon, Fr4 and Komacel at millimetre wave frequency ranges. One of TUBITAK UME costumer asked for Teflon's characterization at milimeter wave frequencies. This manufacturer produces a radom for the housing of the outdoor antenna at 5G frequencies. Another TUBITAK UME costumer asked Komacel's characterization and it uses the Komacel as fixed to open area test field construction. Komacel integral skin-foam sheets combine a strong, solid cover sheet and a celled core of the same material. Komacel manufacturers claim that Komacel is similarity as air for frequency response. Fr4 material is mostly preferred as circuit substrate for electronic circuit designing. Due to these kind of demands, the KMMS software in the TUBITAK UME infrastructure was operated with the free space method at millimetre frequencies and the dielectric coefficients of materials under test (MUT) such as Teflon, Fr4, Komacel, and Air were measured in this study at frequencies ranging from 67 GHz to 110 GHz. Measured values were compared with low frequency values of materials. There are not any dielectric coefficient values for Teflon, Fr4 and Komacel at millimetre frequencies in the literature. Reason of this miss information, lower frequency values of the measured materials were used as reference values for comparison in this study. Type A uncertainties of relative permittivity measurement of MUT was given and possible uncertainty component was discussed in this study either. This article is expended version of 25th IMEKO TC4 presentation [13].

Basic requirement of free space measurement techniques and dielectric coefficient calculation formulas, detailed KMMS tools and software, KMMS and free space dielectric coefficient measurement application at millimetre wave frequencies, dielectric measurement results and uncertainties of the measurement results are given following section in this paper.

2. FREE SPACE MEASUREMENT TECHNIQUES OF DIELECTRIC COEFFICIENT

Relative permittivity is determined by means of S-Parameters free space measurement. A functional measurement setup based on a VNA is given in Figure 1. Frequency extenders is required only for measurements at millimetre wave frequencies.

Material Under Test (MUT) is fixed between the antennas with a proper distance (*d*) in free space measurement method. This distance should be more than the far field region of antennas. Far-field (Fraunhofer) region is defined as that region of the field of an antenna where the angular field distribution is essentially independent of the distance from the antenna. Far-field region is commonly taken to exist at distances greater than $2\,D^2/\lambda$ from the antenna, where λ is wavelength and D is the maximum aperture of antenna [14], [15]. The far-field patterns of some antennas, such as multibeam reflector antennas, are sensitive to variations in phase over their apertures. For these antennas far field distance might be inadequate.

VNA should be calibrated for error terms determination first, before starting the permittivity measurement in free space

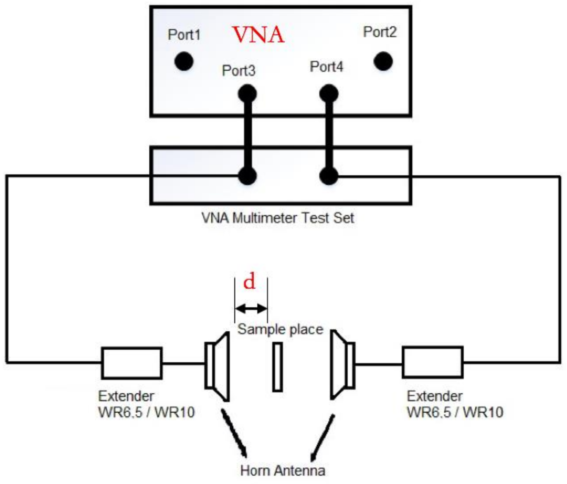

Figure 1. Basic free space measurement setup.

method. To this aim, one of the TRL (through-reflect-line), TRM (through-reflect-match) and LRL (line-reflect-line) method can be preferred.

In free space measurement process below steps are followed:

- Select the error terms determination method (TRL/TRM/LRL) for VNA and perform it at reference plane of the VNA
- Connect the antennas to the reference plane of VNA
- Place the material holder in the middle of antennas
- Measure the S-parameters of an empty material holder by VNA
- Put a fully reflecting material such as a kind of metal into the holder
- Measure the S-parameters of full signal reflection by VNA
- Put the MUT into the holder
- Measure the S-parameters of the MUT
- Remove the effect of the material holder from S-parameters of the materials with the holder to obtain the actual S-parameters of the MUT.
- Calculate the MUT dielectric coefficient by using the corrected MUT S-parameters.

Reference plane is selected the VNA ports which are used for antenna connection during the error term determination. When the measurement will be performed at millimetre wave frequency with free space method, frequency extenders' ports will be the reference plane of the measurement.

In order to get correct S-parameters in free space method, MUT surface should be larger than the antennas beam pattern. Also time domain gating should be applied to prevent multiple reflection from material. Time domain gating also eliminates the diffraction of energy from the edge of the antennas. The dielectric properties can be determined by post processing with a dedicated calculation tool or a software.

There are various approaches for obtaining of the permittivity and permeability by using S-parameters of material. Commonly used permittivity measurement method which uses the S-parameters are listed in Table 3 [16].

Nicolson-Ross-Weir (NRW) is the most commonly used method for performing direct calculation of both the permittivity and permeability from the S-parameters [17]. This method requires all reflection coefficients and transmission coefficients (S_{11}, S_{21}, S_{12}, S_{22}) or a pair (S_{11}, S_{21}) of S-parameters of the material under test to be measured.

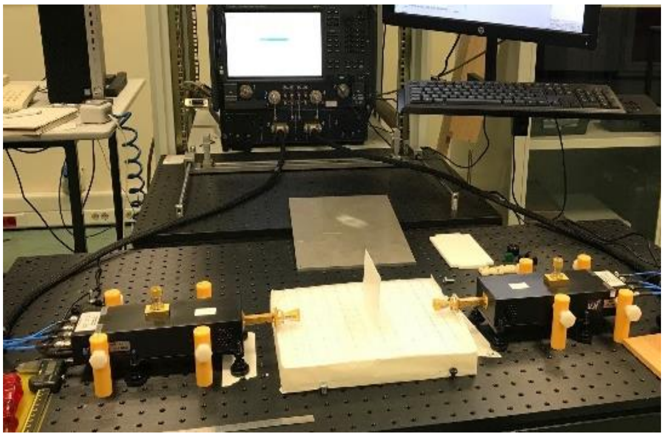

Figure 2. Free Space measurement setup at TUBITAK UME.

However, material thickness is so important for NRW measurement method. This method diverges for low loss materials at frequencies corresponding to integer multiples of one-half wavelength in the sample which is due to the phase ambiguity. So, optimum sample thickness should be an integer multiple of $\lambda/4$.

NRW method uses the following formula for determining the relative dielectric coefficient.

$$S_{11} = \frac{\Gamma(1 - T^2)}{(1 - \Gamma^2\, T^2)} \tag{2}$$

$$S_{21} = \frac{T(1 - \Gamma^2)}{(1 - \Gamma^2\, T^2)}\,, \tag{3}$$

where

$$\Gamma = X \pm \sqrt{X^2 - 1}\,. \tag{4}$$

By solving of equation (4), equation (5) can be obtained.

$$X = \frac{S_{11}^2 - S_{21}^2 + 1}{2\, S_{11}} \tag{5}$$

$$T = \frac{S_{11}^2 + S_{21}^2 - \Gamma}{1 - (S_{11} + S_{21})\,\Gamma}\,. \tag{6}$$

Relative permittivity and relative permeability can be calculated from equation (7) and equation (8).

$$\varepsilon_\mathrm{r} = \frac{\lambda_0^2}{\mu_\mathrm{r}}\left(\frac{1}{\lambda^2} - \left[\frac{1}{2\,\pi\, L}\ln\left(\frac{1}{T}\right)\right]^2\right) \tag{7}$$

where

$$\mu_\mathrm{r} = \frac{1 + \Gamma}{\Lambda\,(1 - \Gamma)\sqrt{\frac{1}{\lambda_0^2} - \frac{1}{\lambda_\mathrm{C}^2}}} \tag{8}$$

$$\frac{1}{\Lambda^2} = \left(\frac{\varepsilon_\mathrm{r} \cdot \mu_\mathrm{r}}{\lambda_0^2} - \frac{1}{\lambda_\mathrm{C}^2}\right) = -\left(\frac{1}{2\,\pi\, L}\ln\left(\frac{1}{T}\right)\right), \tag{9}$$

where λ_0 is centre frequency and λ_C is cut off frequency.

3. KMMS SOFTWARE AND KMMS CAPABILITIES

Keysight Material Measurement Suite (KMMS) is a commercial software [18], [19]. It has many special tools for permittivity and permeability measurement. By using KMMS, measurements can be performed from 200 MHz to 50 GHz and in a temperature range from -40 ºC to 200 ºC with the different probes such as high temperature and different type of open-ended coaxial probes. With KMMS tools, paramagnetic materials, flat, isotropic and homogeneous materials can be measured. KMMS has also NRW calculation method for permittivity and permeability.

KMMS can run with multiport VNA also and it calculates permittivity and permeability coefficients by using the S-parameter measurement with VNA. The permittivity and permeability model obtained with KMMS is valid for higher frequencies.

In this study, KMMS software was also used at millimetre wave frequencies with NRW method, assuming with the dielectric calculation model would be the same at millimetre wave frequencies. During the measurement, discontinuities in the results appeared where thickness was not an integer multiple of $\lambda/4$. For this reason, “Reflection/Transmission Epsilon Fast Model” was chosen in KMMS software. All measurements were performed using “Reflection/Transmission Epsilon Fast Model”.

4. FREE SPACE MEASUREMENT AT MILLIMETRE WAVE FREQUENCIES WITH KMMS

Relative dielectric coefficient measurements were performed in TUBITAK UME RF and Microwave laboratory at two different millimetre wave frequency bands in this study. E band (from 67 GHz to 115 GHz) and D band (from 110 GHz to170 GHz) are selected as frequency ranges. VNA calibration for error term calculation was performed by using WR10 and WR6.5 calibration kits.

Functional block diagram of free space dielectric measurement setup in TUBITAK UME is similar as Figure 1 and setup picture is given in Figure 2.

In order to obtain the millimetre wave frequencies with VNA, some suitable special frequency extenders were used for these frequency ranges. In this study, WR10 and WR6.5 compatible frequency extenders were used to reach for each millimetre wave frequency bands.

The equipment list which is used at the measurement setup in Figure 1 is given as below;

- Network Analyser: Keysight N5247B PNA-X
- VNA Test Set: Keysight N5292A
- Frequency extender: VDI WR6.5 110-170 GHz VNAX
- Frequency extender: VDI WR10 67-115 GHz VNAX
- Calibration Kit: VDI 6.5 VNAX N5262AC06
- Calibration Kit: VDI 10.0 VNAX N5262AC10
- Horn antenna pairs for 65-115 GHz frequency band
- Horn antenna pairs for 110-170 GHz frequency band

Table 3. Permittivity measurement method by using S- parameters.

Method Name	**Used parameters**	**Properties**
Nicholson-Ross-Weir Method	S_{11}, S_{21}, S_{12}, S_{22} or S_{11}, S_{21}	ε_r, μ_r
NIST Iterative Method	S_{11}, S_{21}, S_{12}, S_{22} or S_{11}, S_{21}	ε_r, $\mu_\mathrm{r} = 1$
New non-iterative Method	S_{11}, S_{21}, S_{12}, S_{22} or S_{11}, S_{21}	ε_r, $\mu_\mathrm{r} = 1$
Short circuit line (SCL) Method	S_{11}	ε_r

Thickness of the fully reflective material which is used for KMMS calibration is 5.0 mm at 67-115 GHz frequency (E) band and 1.0 mm at 110-170 GHz frequency (D) band.

1.0 mm and 6.0 mm thickness Teflon samples, 1.2 mm thickness Fr4 and 1.9 mm thickness Komacel was selected as MUTs. Given below parameters were accepted for the measurements in this study.

- 10 repetitive measurements for each frequency step with 2 seconds wait.
- Number of points were selected 1201 at 110-170 GHz band and 961 at 67-115 GHz band for VNA calibration and KMMS application.
- Nominal VNA extender’s power was arranged less than 10 dBm for both frequency bands.
- Distance between antenna and MUT was arranged as 76.6 mm for 67-115 GHz frequency band.
- Distance between antenna and MUT was arranged as 35 mm for 110-170 GHz frequency band.

Described measurement process in section 2 was applied and after the measurements, complex relative permittivity values were obtained by KMMS. NRW method was used for calculation of the complex relative permittivity at KMMS software. Measurement and calculation results were given in Table 4 as magnitude, real and imaginary components of relative dielectric coefficient for Air ($\varepsilon_\mathrm{r\_A}$), Fr4 ($\varepsilon_\mathrm{r\_F}$), Teflon ($\varepsilon_\mathrm{r\_T}$), and Komacel ($\varepsilon_\mathrm{r\_K}$) in E and D millimetre frequency bands. Only relative permittivity was measured. Permeability were not measured in this study. Selected dielectric calculation method does not permit the permeability measurement either.

KMMS software gives the real and imaginary component of relative permittivity. Relative dielectric coefficient was calculated from real and imaginary component of the measurements for each material in this study. Relative dielectric coefficient was given in Figure 3 in two frequency bands with reference magnitude values. Ref_A is air, Ref_F is Fr4, Ref_T is Teflon, Ref_K is Komacel reference values in Figure 3. These reference values were obtained out from older studies performed at lower frequencies [20], [21], [22].

Imaginary component values of relative permittivity of the material are less than zero in some frequencies in Table 4. Values below zero are assumed as numerical calculation error of the calculation of the NRW model.

Due to the measurements performed using two different calibration kits (WR10 and WR6.5), differences in the relative permittivity were observed at the adjacent frequencies of the two different frequency bands (E and D band).

Loss tangent ($\mathrm{tg}\,\delta$) values are also calculated by using equation (10)

$$tgloss = \mathrm{tg}\,\delta = \frac{\varepsilon_\mathrm{r}''}{\varepsilon_\mathrm{r}'} \tag{10}$$

Loss tangent values of the materials are given in Table 5 in this study. Differences between measured and reference relative dielectric coefficient are given in Table 6.

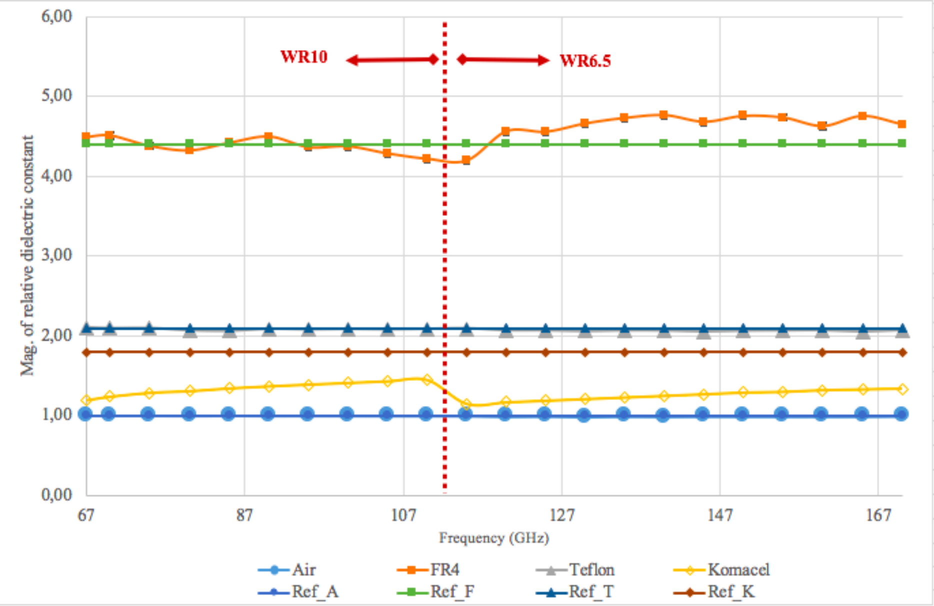

Figure 3. Relative dielectric coefficient of Air, Fr4, Teflon and Komacel with known lower frequency reference values at 67 to 115 GHz frequency band and 110 to 170 GHz frequency band.

5. UNCERTAINTY CALCULATION OF RELATIVE DIELECTRIC MEASUREMENTS AT MILLIMETRE WAVE FREQUENCIES

The uncertainty of a measurement is an expression that shows how reliable the measurement is from an error band, depending on the factors affecting a measured quantity. Uncertainty is an absolute value and it can be calculated by two type components as Type A and Type B.

While calibration services are provided, the calculated measurement repeatability is one of the inputs of the measurement uncertainty calculation. The uncertainty component (u_X), called Type A, from repeated measurements, is calculated as in equation (11) [23].

$$u_X = \sqrt{\frac{\frac{1}{N-1} \cdot \sum_1^N \left(x_i - x_{\mathrm{Aveg}}\right)^2}{N}}\,, \tag{11}$$

where x_i is the instant measurement data, x_{Aveg} is the mean of the repeated measurements, and N is the number of measurements.

Table 4. Relative dielectric constant values for Air, FR4, Teflon and Komacel from 67 to 115 GHz and from 115 to 170 GHz frequency bands.

Freq. (GHz)	Band	Air			Fr4			Teflon			Komacel		
		ε_r'	ε_r''	$\lvert\varepsilon_{r\_A}\rvert$	ε_r'	ε_r''	$\lvert\varepsilon_{r\_F}\rvert$	ε_r'	ε_r''	$\lvert\varepsilon_{r\_T}\rvert$	ε_r'	ε_r''	$\lvert\varepsilon_{r\_K}\rvert$
67	E (WR10)	1.00	0.00	1.00	4.02	0.38	4.50	2.05	0.00	2.10	1.22	0.01	1.20
70		1.00	0.00	1.00	4.03	0.30	4.51	2.07	-0.01	2.09	1.24	0.00	1.24
75		1.00	0.00	1.00	3.88	-0.06	4.38	2.04	0.00	2.09	1.29	0.00	1.29
80		1.00	0.00	1.00	3.86	0.09	4.33	2.10	0.00	2.07	1.31	0.01	1.31
85		1.00	0.00	1.00	3.94	0.07	4.42	2.08	0.00	2.06	1.34	0.01	1.34
90		1.00	0.00	1.00	4.02	0.22	4.50	2.07	-0.01	2.09	1.37	0.01	1.37
95		1.00	0.00	1.00	3.90	0.13	4.37	2.08	-0.01	2.08	1.39	0.01	1.39
100		1.00	0.00	1.00	3.90	0.13	4.38	2.08	0.01	2.08	1.41	0.01	1.41
105		1.00	0.00	1.00	3.83	0.21	4.29	2.08	-0.01	2.08	1.43	0.01	1.43
110		1.00	0.00	1.00	3.76	0.09	4.22	2.07	0.01	2.09	1.44	0.01	1.45
115	D (WR6.5)	1.00	0.00	1.00	3.75	0.13	4.20	2.08	-0.01	2.09	1.15	0.01	1.15
120		1.00	0.00	1.00	4.56	0.15	4.56	2.07	-0.01	2.07	1.17	0.01	1.17
125		1.00	0.00	1.00	4.56	-0.11	4.56	2.07	-0.01	2.07	1.19	0.01	1.19
130		1.00	0.00	1.00	4.66	0.13	4.66	2.06	0.00	2.06	1.21	0.02	1.21
135		1.00	0.00	1.00	4.73	-0.02	4.73	2.07	0.00	2.07	1.23	0.02	1.23
140		1.00	0.00	1.00	4.77	0.06	4.77	2.07	0.00	2.07	1.25	0.02	1.25
145		1.00	0.00	1.00	4.68	0.10	4.69	2.06	-0.01	2.06	1.27	0.02	1.27
150		1.00	0.00	1.00	4.76	0.11	4.76	2.07	-0.01	2.06	1.29	0.02	1.29
155		1.00	0.00	1.00	4.74	0.14	4.74	2.07	0.00	2.07	1.30	0.02	1.30
160		1.00	0.00	1.00	4.63	0.18	4.63	2.07	0.00	2.07	1.32	0.02	1.32
165		1.00	0.00	1.00	4.76	0.11	4.76	2.06	0.00	2.06	1.33	0.02	1.33
170		1.00	0.00	1.00	4.65	0.24	4.65	2.07	-0.03	2.07	1.34	0.02	1.34

Uncertainty calculated from more than 10 repeated measurements is assumed to have a normal distribution.

In this study, for each material measurement, 10 repeated measurement had been performed.

Type B components of the uncertainty come mainly from VNA. Following parameters are accepted as uncertainty component in the EURAMET VNA calibration guide [24]:

For the reflection coefficient measurement;

- Calibration standards which are used for determining of VNA error terms
- Noises of VNA such as floor and trace noise
- Non-linearity of VNA non-linearity
- Drift of VNA
- Cable stability of test port for reflection
- Connector repeatability

For the transmission coefficient measurement;

- Calibration standards which are used for determining of VNA error terms
- Noises of VNA such as floor and trace noise
- Non-linearity of VNA non-linearity
- Drift of VNA
- Cable stability of test port for reflection and transmission
- Connector repeatability

Sampler holder's position between antennas and antennas' alignments were not considered as uncertainty component in this study. They can be considered in future studies.

Uncertainty evaluation for S-parameter measurements at millimetre wave frequencies is still a matter of active discussion. For example, VNA with frequency extenders has just started to analyse in the metrological application. Extenders' power linearities, extenders' power accuracies and other effect of the extenders were not considered as uncertainty component of this study. European research projects are currently active in the fiend, such as [9]. M. Sellone et all published an article on bilateral comparison for only scalar S-parameter measurement between INRIM and (Italy NMI) and NMC (A*Star Singapore NMI) at E band frequencies [25]. In this study vectorial uncertainty calculation was not considered at E band frequencies. In order to perform the measurement on dielectric coefficient, vectorial S-parameter measurement should be performed. Frequency extenders are sperate devices which are used to extend the frequency band covered by VNAs. In the future, while the S-parameter uncertainty problem at millimetre wave frequencies will be clarified, the effects of the frequency extender may be included in the uncertainty calculations. Most of the National Metrology Institutes (NMI) have calibration measurement capability (CMC) entity in the BIPM data base (www.bipm.org) for lower than D and E band frequencies. Only stronger infrastructure NMIs such as PTB (Germany), KRISS (South Korea), NMIJ (Japan), NPL (UK) and NIST (USA) have CMC entity for S-parameters at E band frequencies. CMC entities of PTB, KRISS, NPL and NMIJ were updated in second half of 2021 at BIPM database.

Due to this unsolved problem, only the type A component of the uncertainty was evaluated in this study.

Calculated Type A uncertainty of magnitude values of permittivity were given in Table 7 for two millimetre wave frequency bands as Air (u_A), Fr4 (u_F), Teflon (u_T) and Komacel (u_K). Uncertainties of MUT are given in Figure 4.

Type A uncertainty of magnitude permittivity of Fr4 is higher than the other uncertainties. Standard deviation of Fr4 measurements were also higher than the other materials. When a comparison is made for surface roughness of the MUTs, Fr4 surface roughness is higher than the other MUTs in this study. It is assumed that this higher standard deviation comes from high surface roughness of Fr4.

Table 5. Tangent Loss values for Air, FR4, Teflon and Komacel.

Freq. (GHz)	Band	Air		Fr4		Teflon		Komacel	
		$\lvert\varepsilon_{r\_A}\rvert$	Tg Loss_A	$\lvert\varepsilon_{r\_F}\rvert$	Tg Loss_F	$\lvert\varepsilon_{r\_T}\rvert$	Tg Loss_T	$\lvert\varepsilon_{r\_K}\rvert$	Tg Loss_K
67	E (WR10)	1.00	0.0	4.50	0.09	2.10	0.0E+00	1.20	0.01
70		1.00	0.0	4.51	0.07	2.09	-4.8E-03	1.24	0.00
75		1.00	0.0	4.38	-0.02	2.09	0.0E+00	1.29	0.00
80		1.00	0.0	4.33	0.02	2.07	0.0E+00	1.31	0.01
85		1.00	0.0	4.42	0.02	2.06	0.0E+00	1.34	0.01
90		1.00	0.0	4.50	0.05	2.09	-4.8E-03	1.37	0.01
95		1.00	0.0	4.37	0.03	2.08	-4.8E-03	1.39	0.01
100		1.00	0.0	4.38	0.03	2.08	4.8E-03	1.41	0.01
105		1.00	0.0	4.29	0.05	2.08	-4.8E-03	1.43	0.01
110		1.00	0.0	4.22	0.02	2.09	4.8E-03	1.45	0.01
115	D (WR6.5)	1.00	0.0	4.20	0.03	2.09	-4.8E-03	1.15	0.01
120		1.00	0.0	4.56	0.03	2.07	-4.8E-03	1.17	0.01
125		1.00	0.0	4.56	-0.02	2.07	-4.8E-03	1.19	0.01
130		1.00	0.0	4.66	0.03	2.06	0.0E+00	1.21	0.02
135		1.00	0.0	4.73	0.00	2.07	0.0E+00	1.23	0.02
140		1.00	0.0	4.77	0.01	2.07	0.0E+00	1.25	0.02
145		1.00	0.0	4.69	0.02	2.06	-4.9E-03	1.27	0.02
150		1.00	0.0	4.76	0.02	2.06	-4.8E-03	1.29	0.02
155		1.00	0.0	4.74	0.03	2.07	0.0E+00	1.30	0.02
160		1.00	0.0	4.63	0.04	2.07	0.0E+00	1.32	0.02
165		1.00	0.0	4.76	0.02	2.06	0.0E+00	1.33	0.02
170		1.00	0.0	4.65	0.05	2.07	-1.4E-02	1.34	0.01

6. CONCLUSION

In this study, repeated relative dielectric coefficient measurements were performed at millimetre wave frequencies (from 67 to 115 GHz and from 110 to 170 GHz) with KMMS software by using the infrastructure of TUBITAK UME RF and Microwave Laboratory. Although, KMMS is known as a software that run up to 50 GHz with its own special probes, it was proved with this study that KMMS can be used at millimetre wave frequencies, too.

By using KMMS, complex relative dielectric coefficients were measured and tangent losses were calculated for MUTs.

Table 6. Differences between measured and reference relative dielectric coefficient values for Air, FR4, Teflon and Komacel from 67 to 115 GHz and from 115 to 170 GHz frequency bands

Freq. (GHz)	Band	Air			Fr4			Teflon			Komacel		
		Measured $\lvert\varepsilon_{\mathrm{r\_A}}\rvert$	Reference $\lvert\varepsilon_{\mathrm{r\_A}}\rvert$	Difference $\lvert\varepsilon_{\mathrm{r\_A}}\rvert$	Measured $\lvert\varepsilon_{\mathrm{r\_F}}\rvert$	Reference $\lvert\varepsilon_{\mathrm{r\_F}}\rvert$	Difference $\lvert\varepsilon_{\mathrm{r\_F}}\rvert$	Measured $\lvert\varepsilon_{\mathrm{r\_T}}\rvert$	Reference $\lvert\varepsilon_{\mathrm{r\_T}}\rvert$	Difference $\lvert\varepsilon_{\mathrm{r\_T}}\rvert$	Measured $\lvert\varepsilon_{\mathrm{r\_K}}\rvert$	Reference $\lvert\varepsilon_{\mathrm{r\_K}}\rvert$	Difference $\lvert\varepsilon_{\mathrm{r\_K}}\rvert$
67	E (WR10)	1.00	1.00	0.00	4.50	4.40	0.10	2.10	2.10	0.00	1.20	1.80	0.60
70		1.00	1.00	0.00	4.51	4.40	0.11	2.09	2.10	0.01	1.24	1.80	0.56
75		1.00	1.00	0.00	4.38	4.40	0.02	2.09	2.10	0.01	1.29	1.80	0.51
80		1.00	1.00	0.00	4.33	4.40	0.07	2.07	2.10	0.03	1.31	1.80	0.49
85		1.00	1.00	0.00	4.42	4.40	0.02	2.06	2.10	0.04	1.34	1.80	0.46
90		1.00	1.00	0.00	4.50	4.40	0.10	2.09	2.10	0.01	1.37	1.80	0.43
95		1.00	1.00	0.00	4.37	4.40	0.03	2.08	2.10	0.02	1.39	1.80	0.41
100		1.00	1.00	0.00	4.38	4.40	0.02	2.08	2.10	0.02	1.41	1.80	0.39
105		1.00	1.00	0.00	4.29	4.40	0.11	2.08	2.10	0.02	1.43	1.80	0.37
110		1.00	1.00	0.00	4.22	4.40	0.18	2.09	2.10	0.01	1.45	1.80	0.35
115	D (WR6.5)	1.00	1.00	0.00	4.20	4.40	0.20	2.09	2.10	0.01	1.15	1.80	0.65
120		1.00	1.00	0.00	4.56	4.40	0.16	2.07	2.10	0.03	1.17	1.80	0.63
125		1.00	1.00	0.00	4.56	4.40	0.16	2.07	2.10	0.03	1.19	1.80	0.61
130		1.00	1.00	0.00	4.66	4.40	0.26	2.06	2.10	0.04	1.21	1.80	0.59
135		1.00	1.00	0.00	4.73	4.40	0.33	2.07	2.10	0.03	1.23	1.80	0.57
140		1.00	1.00	0.00	4.77	4.40	0.37	2.07	2.10	0.03	1.25	1.80	0.55
145		1.00	1.00	0.00	4.69	4.40	0.29	2.06	2.10	0.04	1.27	1.80	0.53
150		1.00	1.00	0.00	4.76	4.40	0.36	2.06	2.10	0.04	1.29	1.80	0.51
155		1.00	1.00	0.00	4.74	4.40	0.34	2.07	2.10	0.03	1.30	1.80	0.50
160		1.00	1.00	0.00	4.63	4.40	0.23	2.07	2.10	0.03	1.32	1.80	0.48
165		1.00	1.00	0.00	4.76	4.40	0.36	2.06	2.10	0.04	1.33	1.80	0.47
170		1.00	1.00	0.00	4.65	4.40	0.25	2.07	2.10	0.03	1.34	1.80	0.46

Table 7. Relative dielectric constant values and Type A uncertainties for Air, Fr4, Teflon and Komacel.

Freq. (GHz)	Band	Air		Fr4		Teflon		Komacel	
		$\lvert\varepsilon_{\mathrm{r\_A}}\rvert$	u_{A}	$\lvert\varepsilon_{\mathrm{r\_F}}\rvert$	u_{F}	$\lvert\varepsilon_{\mathrm{r\_T}}\rvert$	u_{T}	$\lvert\varepsilon_{\mathrm{r\_K}}\rvert$	u_{K}
67	E (WR10)	1.00	4E-05	4.50	6E-03	2.10	6E-05	1.20	6E-05
70		1.00	2E-05	4.51	7E-03	2.09	2E-04	1.24	6E-05
75		1.00	2E-05	4.38	2E-03	2.09	3E-04	1.29	7E-05
80		1.00	8E-06	4.33	2E-03	2.07	3E-04	1.31	5E-05
85		1.00	2E-05	4.42	1E-03	2.06	2E-04	1.34	5E-05
90		1.00	1E-05	4.50	1E-03	2.09	6E-04	1.37	6E-05
95		1.00	1E-05	4.37	1E-03	2.08	3E-04	1.39	6E-05
100		1.00	9E-06	4.38	1E-03	2.08	2E-04	1.41	6E-05
105		1.00	9E-06	4.29	2E-03	2.08	1E-04	1.43	6E-05
110		1.00	8E-06	4.22	3E-03	2.09	2E-04	1.45	6E-05
115	D (WR6.5)	1.00	1E-05	4.20	4E-03	2.09	3E-04	1.15	2E-04
120		1.00	1E-05	4.56	2E-03	2.07	2E-04	1.17	2E-05
125		1.00	9E-06	4.56	4E-03	2.07	2E-04	1.19	2E-05
130		1.00	8E-06	4.66	3E-03	2.06	8E-05	1.21	2E-05
135		1.00	8E-06	4.73	1E-03	2.07	1E-04	1.23	2E-05
140		1.00	9E-06	4.77	7E-04	2.07	7E-05	1.25	2E-05
145		1.00	8E-06	4.69	4E-04	2.06	1E-04	1.27	1E-05
150		1.00	7E-06	4.76	2E-04	2.06	7E-05	1.29	2E-05
155		1.00	6E-06	4.74	1E-03	2.07	2E-04	1.30	1E-05
160		1.00	7E-06	4.63	2E-03	2.07	1E-04	1.32	9E-06
165		1.00	4E-06	4.76	2E-03	2.06	1E-04	1.33	2E-05
170		1.00	8E-06	4.65	7E-03	2.07	8E-05	1.34	2E-05

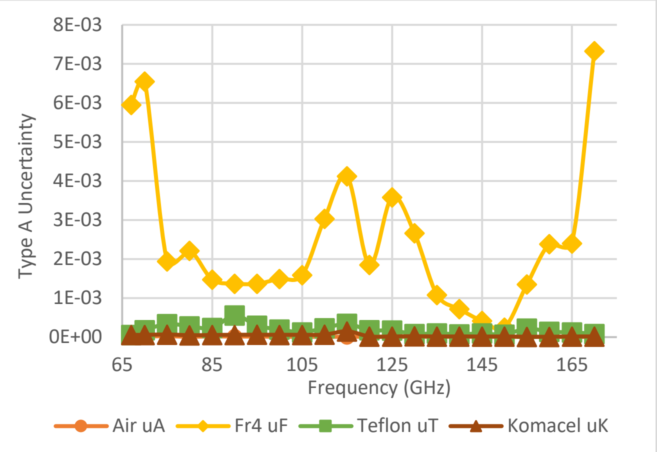

Figure 4. Type A uncertainty values of relative dielectric coefficient of Air, Fr4, Teflon and Komacel at E and D band frequencies.

From the measurement results, we can list the main influence parameters on the relative dielectric permittivity measurement accuracy at millimetre wave frequency with free space method as follow:

- Antennas should have aligned each other in mm range,
- MUT should be perpendicular to the antennas,
- MUT surface should be less rough,
- MUT size should be more than antenna beam pattern,
- Distance between MUT and antennas should be determined with the time domain,
- VNA calibration should be performed accurately with calibration kits.

It is also important that the thickness of the material at NRW method selected in this study. From the measurement results, some of imaginary component values of relative permittivity of the material resulted lower than zero at some frequencies. This values were assumed to be numerical calculation error of the model.

Known and potential uncertainty calculation components were discussed, the uncertainty component from repeated measurements was calculated and the results of the measurements were shared in this study. Type A uncertainty were less than the compared measured values. When the uncertainty calculation problem will be solved at millimetre wave frequencies, other uncertainty sources will be compared with Type A components.

With performed measurements, it has been shown that the calculation model in KMMS (Reflection/Transmission Epsilon Fast Model) can also be used at millimetre wave frequencies. With this study, it has been demonstrated that dielectric coefficient measurements can be performed at millimetre wave frequencies with the free space method in future studies in the environment where the materials are used.

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REFERENCES

[1] D. T. Paris, F. K. Hurd, Basic Electromagnetic Theory, Reflection and Refraction of Plane Waves, 1969, pp. 346–397, McGraw Hill, USA.

[2] A. L. de Paula, M. C. Rezende, J. J. Barroso, Experimental Measurements and Numerical Simulation of Permittivity and Permeability of Teflon in X band, Journal of Aerospace Technology and Management, 3(1), 2011, pp. 59-64.
DOI: 10.5028/jatm.2011.03019410

[3] F. B. Gul, E. Danaci, N. Baydogan, Effect of the improved bulk modulus on dielectric properties of poly (methyl methacrylate)/borax hybrid composite at radar frequencies, Materials Today Communications, vol. 33, December 2022, pp. 1-10, 104960.
DOI: 10.1016/j.mtcomm.2022.104960

[4] I. Zivkovic, A. Murk, Free-Space Transmission Method for the Characterization of Dielectric and Magnetic Materials at Microwave Frequencies, Microwave Materials Characterization, 2012, pp. 1–18, Ch. 5, Eds. Costanzo, S., Intech Publications.
DOI: 10.5772/51596

[5] D. K. Ghodgaonkar, V. V. Varadan, V. K. Varadan, A Free-Space Method for Measurement of Dielectric Constants and Loss Tangents at Microwave Frequencies, IEEE Trans. Inst. And Meas., 38-3, 1989, pp.789-793.
DOI: 10.1109/19.32194

[6] Agilent Technologies, Printed Version of Agilent 85070E/85071E Dielectric Probe Kit Software's Help File, 2012.

[7] N. Dvurechenskaya, R. J. Zielinski, Advantages and disadvantages of the free-space arch method used for investigation of shielding materials at low gigahertz frequencies, Proc. of the 10th Int. Symposium on Electromagnetic Compatibility, York, UK, 26-30 September 2011, pp. 790– 795.

[8] İ. Ünal, B. Türetken, Electromagnetic Methods in Radar Absorber Material Development, Project Report, MİLTAL, Material Institute, TÜBİTAK MAM, 2013, Gebze-Kocaeli (in Turkish).

[9] X. Shang and the project team, Publishable Summary for 18SIB09 TEMMT Traceability for electrical measurements at millimeter-wave and terahertz frequencies for communications and electronics Technologies, 2019. Online [Accessed 18 September 2023]
https://www.euramet.org/research-innovation/search-research-projects/details/project/traceability-for-electrical-measurements-at-millimetre-wave-and-terahertz-frequencies-for-communicat

[10] A. Kazemipour, M. Wollnsack, J. Hoffman, M. Hudlicka, S. K. Yee, et all, Analytical Uncertainty Evaluation of Material Parameter Measurements at THz Frequencies, Int. Journal of Infrared and Millimeter Waves, 2020, 41, pp. 1199-1217.

[11] A. Kazemipour, M. Hudlička, S. -K. Yee, M. A. Salhi, D. Allal, T. Kleine-Ostmann, T. Schrader, Design and Calibration of a Compact Quasi-Optical System for Material Characterization in Millimeter/Submillimeter Wave Domain, IEEE Trans. On Instrumentation and Measurement, 2015, Vol 64, No 6.
DOI: 10.1109/TIM.2014.2376115

[12] K. B. Khadhra, A. Olk, O. Gomez, A. Fox, Permittivity estimation of rough dielectric surfaces by means of polarimetric bistatic measurements at millimeter wave frequencies16th European Radar Conference (EuRAD), Paris, France, 2-4 October 2019, pp. 369-372.

[13] E. Danaci, E. Cetin, Material Characterization at Millimeter Wave Frequencies in TUBITAK UME, 25th IMEKO TC4 Int. Symp. on Measurement of Electrical Quantities, 23rd International Workshop on ADC and DAC Modelling and Testing, Brescia, Italy, 12-14 September 2022, 6 p.
DOI: 10.21014/tc4-2022.55

[14] C. A. Balanis, Antenna Theory, John Wiley & Sons, 2016.

[15] D. M. Pozar, Microwave Engineering, John Wiley & Sons, 2011.

[16] K. C. Yaw, Measurement of Dielectric Material Properties, R&S Application Note, RAC0607-0019_1_4E, 2012.

[17] A. M Nicolson., G. F. Ross, Measurement of the Intricsic Properties of Materials by Time Domain Techniques, IEEE Trans. On Instrumentation and Measurement, 1970, Vol IM-19, No 4.
DOI: 10.1109/TIM.1970.4313932

[18] Agilent Technologies Inc., Basic of Measuring the Dielectric Properties of Materials, Agilent Application Note 5989-2589EN, , USA, April 2013.

[19] Agilent Technologies Inc., Keysight N1500A Material Measurement Suite, Technical Overview Document 5992-0263EN, USA, 2021.

[20] M. Moosazadeh, Sidelobe level reduction using Teflon for amicrowave and millimetre-wave antipodalVivaldi antenna, IET Microwaves, Antennas & PropagationVolume 14, Issue 6, May 2020, pp. 457-565.
DOI: 10.1049/iet-map.2019.0836.

[21] A. R. Djordjevic, R. M. Biljie, V. D. Likar-Smiljanic, T. K. Sarkar, Wideband frequency-domain characterization of FR-4 and time-domain causality, in IEEE Transactions on Electromagnetic Compatibility, vol. 43, no. 4, Nov. 2001, pp. 662-667.
DOI: 10.1109/15.974647

[22] Kommerling USA, Inc., Komacel integral foam sheets. Online [Accessed 18 September 2023]
https://www.professionalplastics.com/professionalplastics/content/downloads/KomaCel_Brochure.pdf

[23] BIPM, JCGM 100:2008, Evaluation of measurement data – Guide to the expression of the uncertainty in measurement, 1st ed., Bureau Int. des Poids et Measures, Sep. 2008. Online [Accessed 18 September 2023]
https://www.bipm.org/documents/20126/2071204/JCGM_100_2008_E.pdf

[24] EURAMET, Guidelines on the Evaluation of Vector Network Analysers (VNA), EURAMET Calibration Guide No. 12 Version 3.0, March 2018. Online [Accessed 18 September 2023]
https://www.euramet.org/Media/news/I-CAL-GUI-012_Calibration_Guide_No._12.web.pdf

[25] M. Sellone, L. Oberto, Y Shan., Y. S. Meng, L. Brunetti, N. Shoaib, Comparison of S-Parameter Measurements at Millimeter Wavelengths Between INRIM and NMC, IEEE Transactions on Instrumentation and Measurement, vol. 63, no. 7, July 2014, pp. 1810-1817.
DOI: 10.1109/TIM.2013.2293227