5G/mmWave Materials Assessment and Characterization Project


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1. Introduction

Circuit designers urgently need dielectric properties data for materials at millimeter-wave (mmWave) frequencies to optimize device performance of new 5G hardware and for quality assurance. Unfortunately, there are no standard reference materials or even agreed upon characterization test methods for materials at mmWaves. Without reliable mmWave materials data, manufacturers are forced to extrapolate materials data from low frequencies to high frequencies, which can lead to mistakes that have potentially devastating costs.

In response, iNEMI members organized the 5G/mmWave Materials Assessment and Characterization project to develop guidelines and best practices for a standardized measurement and test methodology that can be shared with industry and relevant standards organizations [1]. The initial focus is to benchmark current available test methods and provide pro/con analysis, identify gaps (if any) for extending test methods to 5G/mmWave frequencies, and develop reliable reference standard materials for setup and calibration. There are two tasks for the benchmarking phase. Task 1 (presented in Report 1) introduces some of industry’s ‘go-to’ measurement techniques. Task 2 (presented in this report) discusses emerging measurement techniques that will become increasingly relevant as new spectrum auctions open up additional communication bands above those in the current industry release documents for 5G mmWaves. Other impacts include materials characterization for automotive radar applications, which overlaps some 5G bands.

When it comes to dielectric materials characterization, manufacturers routinely rely on cavity perturbation for ‘right,’ ‘easy,’ and ‘fast’ measurements. However, the dimensions of the cavity are inversely proportional to the resonance frequency used to measure a material’s dielectric response. As 5G technologies push toward higher frequencies, the dimensions of the cavity and the sample size must shrink, which increases the fractional uncertainties and sensitivity to measurement error.

Each new material for mmWave 5G applications requires careful consideration to determine the best measurement methodology, fixture, sample fabrication and test instrument. There are dozens of different methodologies that could be used, but which to choose is often ambiguous. This report presents new measurement and metrology methods especially focused on next generation methods that can be useful to develop for 6G communications. [2]

2. Problem Statement

Many of the commercial implementations of mmWave technologies involve planar transmission lines and distributed element structures fabricated on high density interconnect (HDI) boards or organic package technologies. The iNEMI 5G/mmWave Materials Assessment and Characterization project addresses characterization of these materials. The geometries of these devices create unique characterization challenges that differ from more traditional ‘bulk’ characterization methods. Many of the methods described in literature utilize large 3-dimensional material samples that are capable of being precisely machined to fit into test fixtures. However, this form factor is not typically used with the materials that are the focus of this iNEMI effort.

The material sets commonly used to fabricate HDI and organic package substrates are provided as either curable liquids or curable thin film sheets. Often the materials’ electrical characteristics are dependent
on the specifics of the curing process. Because of this, large, thick, machinable material samples are generally not available and any suitable metrology has to be able to work with thin (10 um - 250 um) sheets that are frequently fragile, flexible, and brittle. Furthermore, the thickness uniformity of these samples may not be ideal and metrologies that can tolerate or incorporate non-uniformity of the samples into the measurement may have advantages.

Many of the techniques used for material characterization heavily rely on knowledge of the sample thickness. Although the typical samples available are not generally soft, they are frequently compliant under pressure. Contact thickness measurements like hand-held micrometers can lead to significant measurement errors that depend on operator technique, particularly for more compliant materials.

We recognize that as mobile communication moves into higher frequency domains in the next decade, the industry will continue to face many challenges for design, implementation, reliability and cost. Therefore, there is continuing need for industry collaboration in a pre-competitive manner to enable faster implementation of specialized solutions. This report specifically discusses the issues associated with higher frequency (>100 GHz) applications.

3. Scope

The scope of this document is to present some of the emerging techniques in research and development stages that can be commercially deployed for next generation material characterization, especially at higher frequencies than what is required at 5G frequencies. The document is not an exhaustive study. It introduces several methodologies for materials measurement and metrology that are useful and relevant as the industry moves beyond 5G communication. Other applications such as radars (automotive and space) also utilize frequencies >100 GHz.

4. Wafer-Level Measurements

4.1 Dielectric constant and loss tangent of dielectric materials

The electromagnetic properties that define microwave materials are permittivity (\( \varepsilon \)) and permeability (\( \mu \)). Permittivity of a material determines its response to the application of an electric field. A material is characterized as dielectric when it stores energy upon the application of an electric field. Permittivity of a material is expressed as a complex number. The real part is called the dielectric constant, which represents the amount of energy stored. The imaginary part represents the loss factor. The ratio of the imaginary part to the real part is defined as loss tangent. The dielectric constant and loss tangent are the two most important electrical parameters of dielectric materials.

To accurately extract the properties of the dielectric material, we suggest the following procedure:

1) Design via-less microstrip ring resonators (MRR) with calibration and de-embedding structures. This requires the use of design and modeling tools such as HFSS and ADS.

2) Design CPW and microstrip lines along with suitable calibration and de-embedding structures to confirm the extracted dielectric properties. This requires the use of design and modeling tools such as Ansoft HFSS and Keysight PathWave ADS.
3) Develop a common mask design that can be applied to different dielectric materials.

4) Fabricate and measure test vehicles. This requires probes and a suitable measurement setup based on frequency range.

5) Interpret and extract the measured results using a combination of modeling tools and design equations.

4.2 Electrical characterization using microstrip ring resonator (MRR) method

Resonant methods are well suited for characterization of low-loss materials. The MRR method is very reliable for high frequency characterization of dielectric materials. MRR with via-less conductor-backed coplanar waveguide (CBCPW) probe pads, transitions and feed lines are shown in Figure 1. The insertion loss of MRR has resonant periodic peaks as shown in Figure 2. Hence, the electrical parameters (dielectric constant and loss tangent) can be extracted at multiple frequencies from the response of MRR. Dielectric constant is extracted from the location of resonant peaks while loss tangent of the material is calculated by unloaded quality factor of the peaks.

![Figure 1. Microstrip ring resonator: (a) material stack-up and (b) MRR with feed lines and CBCPW to MS transition.](image)

MRRs are designed using Equation 1

\[ f_n = \frac{nc}{2\pi r_m \sqrt{\varepsilon_{\text{eff}}}} \quad \text{Equation 1} \]

where \( f \) corresponds to the \( n^{\text{th}} \) resonance, \( r_m \) is the mean radius of the ring, and \( c \) is speed of light. For calculating the radius for the fundamental frequency \( n = 1 \). To capture multiple resonances in the frequency range of interest, multiple MRRs with different fundamental frequencies need to be used.
4.3 Electrical characterization of transmission lines

Microstrip and co-planar waveguides (CPWs) are commonly used as planar transmission lines [3] and are, therefore, designed and measured as part of the material characterization test vehicle. The design for both types of lines is discussed in the following section and their insertion loss provides a good idea about the electrical performance of sub-sections.

4.3.1 CPW lines

CPW lines (Figure 3) are first designed using Keysight PathWave ADS and simulated in Ansys HFSS. Since the lines must be probed using ground-signal-ground GSG probes, one important factor in their design is to ensure the compatibility with the GSG pitch of probes. For example, at W-band, probes (ACP110) have a GSG pitch of 200 um so the line dimensions should comply with this pitch. Usually, higher impedance lines (more than 50 Ω) are designed to relax the fabrication procedure and ensure compatibility with the probes. In addition, as CPW lines do not require ground plane, only the top side of the stack-up is metallized. Since GSG pitch for the RF probes is different for W (200 um) and D (75 um) bands, two separate sets of lines need to be designed if both W and D frequency band characterization are required.

Figure 3. CPW line: (a) stack-up and (b) structure.
4.3.2 Microstrip lines

Microstrip lines can be designed using Keysight PathWave ADS and simulated in Ansys HFSS. In order to probe the designed microstrip lines, CPW-to-microstrip transitions are required. Via-less CPW probe pads can be designed according to the guidelines presented in [4] and [5]. Total width of the via-less CPW probe pads needs to be less than \( \lambda_g / 2 \) to avoid the parasitic parallel plate waveguide mode [2]. Higher impedance microstrip lines experience less dispersion [6] and, as a result, are designed with an impedance around 70 \( \Omega \) to avoid dispersion.

TRL (thru, reflect and line) calibration can be used to de-embed the effects of via-less CBCPW probe pads, CBPW-to-MS transitions and feed lines. TRL standards can be designed as shown in Figure 4(a). The length of the delay line standard determines the range of calibration. The range is calculated using the following equations

\[
\begin{align*}
    f_{\text{lower}} &= \frac{20c}{360 \cdot (\text{line-thru}) \cdot \sqrt{\varepsilon_{\text{eff}}}} \quad \text{Equation 2} \\
    f_{90} &= \frac{90c}{360 \cdot (\text{line-thru}) \cdot \sqrt{\varepsilon_{\text{eff}}}} \quad \text{Equation 3} \\
    f_{\text{upper}} &= \frac{160c}{360 \cdot (\text{line-thru}) \cdot \sqrt{\varepsilon_{\text{eff}}}} \quad \text{Equation 4}
\end{align*}
\]

where line is the length of line in meters, thru is the length of thru in meters, \( c \) is the speed of light in meters/second. The estimated \( \varepsilon_{\text{eff}} \) is obtained from LineCalc tool in ADS. The parameter \( f_{90} \) is the center frequency of the calibration range with \( f_{\text{upper}} \) and \( f_{\text{lower}} \) being the highest and the lowest frequencies, respectively.

As an example, the dimensions of thru, line and microstrip used for W-band measurements on glass/ABF test vehicles are shown in Figure 4. With \( \varepsilon_{\text{eff}} \) of 3.3 the lower, center and upper frequencies for the range of calibration are calculated as 26.2 GHz, 117.96 GHz and 209.70 GHz, respectively. A test vehicle was measured in the range of 75 GHz to 110 GHz. As shown in Figure 4(b), after performing TRL calibration the thru standard varies around 0.01dB only which implies that the effect of probes pads, transition and feed lines have been de-embedded.
4.4 Common mask design

A test vehicle comprising CPWs, microstrips and MRRs is required for characterization of each dielectric, as shown in Figure 5. The dimensions of CBCPW probe pads are dictated by the probes and, hence, are the same for all materials. The Georgia Tech Packaging Research Center (GT-PRC) has designed a common mask consisting of the test structures with dimensions easily achievable using typical fabrication processes. The mask can be used to fabricate the material characterization test vehicles on different substrates. The results can be post processed after measurements by simply renormalizing the impedance based on the dimensions in the mask (width of microstrip line) and the dielectric constant of the material. This common mask has been used to fabricate test vehicles on different materials at GT-PRC.
4.5 Measurement equipment and probes

The scattering parameters of the designed test structures can be measured using a vector network analyzer (VNA). Two different measurement setups need to be used to record the measurements from 1 GHz to 170 GHz due to the nature of the equipment required.

4.5.1 Broadband millimeter wave setup (100 MHz to 110 GHz)

Anritsu VNA (ME7808) and frequency extenders 3742A-EW are used to measure all the scattering parameters from 100 MHz to 110 GHz. Cascade MicroTech ACP-110-GSG-200 probes are used to probe the samples. LRRM calibration can be performed using standard calibration substrate provided by Cascade and WinCal XE software. The broadband measurement setup is shown in Figure 6.

4.5.2 D-band (110 GHz to 170 GHz) measurement setup

Agilent’s E8361C VNA, along with millimeter wave controller and extenders (V06VNA2), can be used to measure D-band (110-170 GHz) CPWs. The probes used for D-band are 170-S-GSG-75-BT by Cascade. LRRM (line-reflect-reflect-match) calibration can be performed using WinCal XE software to remove the
losses from cables, test head and probes. The measurement setup is shown in Figure 7.

![Measurement Setup](image)

**Figure 7.** D-band measurement setup: (a) VNA with extenders and probes and (b) Cascade Infinity Probe with 75 um GSG pitch.

### 4.6 Surface profile measurements for D-band samples

D-band probes are very sensitive to the non-planarity of the pads hence, the surface profile measurements at the probe pads are crucial for D-band samples. When D-band samples were probed the team observed that probe scratch marks were uneven for some samples (Figure 8a, b). In some of the samples, buckling of some traces was observed. Usually these problems occur due to non-planarity of the probes. There are planarization knobs on the probe station which are adjusted to ensure that probe tips are horizontal. To know the reason for uneven scratch marks, the probe pads can be examined using a profilometer. Since D-band pads are the smallest features in the panel, they experience the most variation in copper thickness while electroplating. As a result, instead of being planar (as shown in Figure 8c) the pads were non-planar (Figure 8d). The samples in the center of the panel were relatively planar while samples toward the edges of the panel had more copper thickness variation. The tolerance for non-planarity of the RF infinity probes (75 um pitch) is 1 um. Pads with average variation of more than 1 um can be probed in normal cases but it reduces the life of the probe. Particularly in this stack-up, the probes were highly sensitive to the non-planarity of the pads because of the hard surface of the glass under a very thin polymer layer. There is not enough room for buffering and hence, the samples with non-planar pads tend to be destroyed. Figures 8c and 8d show the results of z-profiling of two different samples in the same panel. Some samples were good (Figure 8e) while some samples had too much variation and hence, they are not fit for probing. Planarity of all the pads need to be checked before probing in order to ensure good contact with the pads and safety of the probes.
Figure 8. D-band probe pads: (a) uneven probe contact, (b) probe pads with lifted ground pad, (c) planar pad, (d) non-planar pad, (e) probe pad with 0.127 um variation and (f) probe pad with 3.5 um variation.

4.7 Measurement procedure

This section describes the measurement details for the transmission lines and MRRs fabricated on glass/polymer stack-up (as an example) [8].

4.7.1 CPW lines (40 GHz to 170 GHz)

LRRM calibration was performed on Wincal XE. Since CPWs do not have a backside metallization as shown in Figure 9, it is assumed that the fields don’t escape the dielectric. For conducting measurements, the substrate is placed on a metal chuck. To avoid any reflections from the metal chuck, a 1 mm thick FR4 sheet was placed between the CPW samples and chuck. The scattering parameters of different samples of 5 mm long CPW lines were measured from 40 GHz to 110 GHz using broadband millimeter wave setup. The D-band CPW lines were 4 mm long and they were measured using D-band measurement setup. The scattering parameters of the CPW lines are shown in Figure 10. The average insertion loss for CPW lines at 40 GHz, 110 GHz and 140 GHz was measured to be 0.085dB/mm, 0.21dB/mm and 0.25dB/mm.

Figure 9. CPW structures: (a) panel and (b) CPW line examined by Zeta Optical Profilometer.
4.7.2 Microstrip lines (75 GHz to 110 GHz)

The fabricated microstrip line and TRL structures shown in Figure 11 were first measured under LRRM calibration. The measured insertion loss for thru structure, line and microstrip line can be seen in Figure 12. These measurements include the effect of via-less CBCPW probe pads, transition and feedlines. To extract the loss of microstrip line the reference plane needs to be shifted using TRL calibration.

Figure 4-10. Scattering parameters for CPW lines: (a) return loss for 40 GHz to 110 GHz, (b) return loss for 110 GHz to 170 GHz, (c) insertion loss of 40 GHz to 110 GHz and (d) insertion loss for 110 GHz to 170 GHz.

Figure 11. Microstrip line: (a) panel and (b) microstrip line examined by Zeta Optical Profilometer.
Figure 12. Measured scattering parameters for thru and microstrip line structures under LRRM calibration.

The extracted loss of microstrip line (shown in Figure 13) exhibits an insertion loss of 0.23 dB/mm at 110 GHz.

Figure 13. Extracted insertion loss of microstrip line after TRL calibration.

4.7.3 Microstrip ring resonators

Multiple samples need to be fabricated and measured for both designed rings (10 GHz and 15 GHz) to avoid any error in characterization due to fabrication and measurement discrepancies. Measurements are done using TRL calibration to de-embed CPW probe pads, transitions and feed lines. Measured responses of the designed microstrip ring resonators are shown in Figure 14. The responses are close to one another which implies that these responses can be used for accurate extraction of Df and Dk of the glass stack-up.
Figure 14. Measured insertion loss of MRRs after TRL calibration: (a) 10 GHz MRR and (b) 15 GHz MRR.

Two models can be used to extract the relative permittivity of the glass/ABF stack-up from the responses of microstrip ring resonators. Effective permittivity of the stack-up can be estimated from the location of the resonance which can serve as input to both models for calculating relative permittivity. The first model that can be used is a quasi-static model [9]. The equations used for extraction of dielectric constant are shown below.

\[ \varepsilon_{rel}(f) = \frac{2\varepsilon_{eff(o)} + M_t - 1}{M_t + 1} \quad \text{Equation 5} \]

\[ M_t = F\left(\frac{W}{h}\right) - \frac{2\varepsilon_{eff}(f)}{4.6\sqrt{W/h}} \quad \text{Equation 6} \]

\[ F\left(\frac{W}{h}\right) = \begin{cases} \left(1 + 12\left(\frac{h}{W}\right)\right)^{1/2} + 0.04 \left(1 - \frac{W}{h}\right)^2 \frac{W}{h} < 1 \\ \left(1 + 12\left(\frac{h}{W}\right)\right)^{1/2} \frac{W}{h} > 1 \end{cases} \quad \text{Equation 7} \]

This model provides a good estimate of the dielectric constant of the material but does not include the dispersion effect. Since dispersion in microstrip lines is very prominent at high frequencies, a dispersive model [10] can be used to obtain more reliable results. The equations used for extraction of dielectric constant using the dispersive model are given below.
Figure 15 shows results of the extracted electrical properties of the glass/ABF stack-up. Dielectric constant of glass/ABF stack-up remains stable around 4.6 in the entire frequency range (75 GHz to 110 GHz) according to the dispersive model.

The unloaded quality factor of the resonant peak for the ring resonator is a function of the total loss. This total loss is a combination of conductor loss, dielectric loss and radiation loss. It has been reported in [7] that ring resonator can eliminate radiation losses when the ratio of the line width to mean radius is much less than 0.2. In order to estimate loss tangent of the material, conductor loss is theoretically calculated and subtracted from the total loss, using the equations below.

$$\varepsilon_{\text{eff}}(f) = \frac{\varepsilon_{\text{rel}}(f) - \varepsilon_{\text{eff}(0)}}{1 + P(f)}$$  \hspace{1cm} \text{Equation 8}

$$P(f) = P_1P_2[(0.1844 + P_3P_4)10fh]^{1.5763}$$  \hspace{1cm} \text{Equation 9}

$$P_1 = 0.27488 + \left[0.6315 + \frac{0.525}{(157fh)^{20}}\right]u - 0.065683e^{-8.7513u}$$  \hspace{1cm} \text{Equation 10}

$$P_2 = 0.33622(1 - \exp[-0.03442\varepsilon_{\text{rel}}(f)])^{20}$$  \hspace{1cm} \text{Equation 11}

$$P_3 = 0.0363(-4.6u)(1 - \exp[-\frac{fh}{3.87}])^{4.97}$$  \hspace{1cm} \text{Equation 12}

$$P_4 = 1 + 2.751(1 - \exp[-\varepsilon_{\text{rel}}(f)/(15.916)^8])$$  \hspace{1cm} \text{Equation 13}

$$\tan(\delta) = \frac{a_d a_b \sqrt{\varepsilon_{\text{eff}}(\varepsilon_{r}^{-1})}}{\pi \varepsilon_{r}(\varepsilon_{\text{eff}}^{-1})}$$  \hspace{1cm} \text{Equation 14}

$$a_d = a_{\text{total}} - a_c$$  \hspace{1cm} \text{Equation 15}

$$Q_L = F_{\text{min}}/BW_{3dB}$$  \hspace{1cm} \text{Equation 16}

$$Q_u = Q_L/(1 - 10^{s_{21}/20})$$  \hspace{1cm} \text{Equation 17}

$$a_{\text{total}} = \pi/Q_u \lambda_g$$  \hspace{1cm} \text{Equation 18}
5. Test Methods: Time Domain Techniques for Dielectric Measurements

5.1 Introduction of time domain measurements

Since the early 1990s, time domain techniques have been investigated for dielectric characterization. One example is COMITS (coherent microwave transient spectroscopy) [11] developed at IBM. It enables the characterization of complex dielectric materials in the 15-140 GHz range. This method uses freely propagating electromagnetic pulses radiated and received by broadband antennas which are integrated with high-speed optoelectronic devices. Ultrashort optical pulses with a frequency spectrum up to 150 GHz are transmitted through a sample, the received waveform is photo-conductively sampled, waveforms are recorded for two different sample thicknesses, the time signal is Fourier transformed, and the measured waveforms from the two samples are divided to remove any effects due to sample surfaces from which the complex dielectric constant of the material is computed.

5.2 Commercial aspects

In the early 2000s, time domain techniques for dielectric characterization evolved further through use of laser-based setups to extend the capability into the Terahertz frequency range. This measurement technique has led to a commercially available setup from Advantest. [12] [13]. These methods use time domain spectroscopy to obtain the DK (dielectric constant) and DF (dissipation factor) of dielectric materials. The setup is shown in Figure 16. It uses a laser to create ultra-short laser pulses of 10-100 fs duration. These are then used to excite a THz emitter which creates a narrow THz pulse. The pulse is then transmitted through the sample and received by a photo detector. A reference measurement is also conducted without the sample along with a separate path for measuring the optical delay. Mirrors are used to bend the waveforms to minimize any dispersion related effects. Advantest has developed a modification to this method by introducing two lasers (as opposed to one), which improves accuracy, as shown in Figure 16.
5.3 Dk And Df measurements

The measured time domain pulses are converted into the frequency domain using Fourier Transform. Since the transmission of the signal through a medium are defined by the Fresnel Equation and Fabry-Perot effects, these are used to relate the measured electric fields with the refractive index of the material \( n(\omega) \), where \( \omega \) is the angular frequency, as shown in [11].

\[
E_{\text{Sample}}(\omega) = \frac{4\bar{n}(\omega)}{(\bar{n}(\omega) + 1)^2} e^{\left(-j\frac{2\pi\bar{n}(\omega) - 1}{c}\right)}
\]

where \( \bar{n} = n + jk \), where ‘n’ and ‘k’ are the real and imaginary parts of the refractive index.

\[
D_k = n^2 - k^2, \quad D_F = \frac{2nk}{n^2 - k^2}
\]

The extracted refractive index is then used to extract the permittivity and dissipation factor of the dielectric material using [12]. In Equation 1, \( \bar{n} = n + jk \), where ‘n’ and ‘k’ are the real and imaginary parts of the refractive index.
Figure 17. Extracted relative permittivity (Dk) and dielectric loss (Df/Dk; Df – dissipation factor).

5.4 Advantages

- Dielectric properties can be extracted as a continuous function of frequency. This is in contrast with a frequency domain method (using resonators) where the properties can only be extracted at discrete frequencies.
- Do not have to explicitly satisfy Kramer-Kronig relationship since the complex parameters are extracted using a single measurement.
- Dielectric properties over a broad frequency range can be supported (30 GHz – 2 THz).
- Can directly measure the properties of the dielectric sample without the need for any conductive structures.

5.5 Potential sources of error

- Need to precisely account for scattering effects; otherwise, this approach can lead to larger loss tangents than the true values.
- Need precise thickness measurement (d) of the sample since as shown in [11], this parameter is used for extracting the refractive index.
- Around 30dB SNR (signal to noise ratio) may be required to keep errors low. As shown in Figure 18, this measurement system works well over a range of frequencies. If the frequency is too low, however, the errors can increase.
6. Extended Summary of Existing Test Methods

6.1 Solution mapping summary

The iNEMI report, “5G Materials Characterization Project Report 1: Benchmark Current Industry Best Practices for Low Loss Measurements,” summarizes test methods commonly used in the industry. Refer to that document for more details and a comprehensive benchmarking. A representative solution mapping is shown in Figure 19.

6.2 Transmission line method

The sample is placed in a guided transmission line, such as a coaxial airline or waveguide straight section, or suspended in free space between two antennae. Several algorithms to calculate permittivity and permeability from S-parameter measurements are available to choose from. Typical accuracy is 1 to 2%
in a frequency range of 100 MHz to 1.1 THz. The transmission line method works best for materials that can be precisely machined to fit inside the sample holder such as coaxial airlines or rectangular wave guide (Figure 20).

![Figure 20. Illustration of typical physical arrangements for transmission line methods.](image)

### 6.3 Free space method

The free space method works best for large, flat solid materials, but granular and powdered materials can also be measured in a fixture. It is very useful for many applications such as non-destructive testing, measuring materials that must be heated to very high temperatures, or measuring a large area of material that is non-uniform such as a honeycomb or a composite.

The materials are placed between two antennae for a non-contacting measurement (Figure 21).

![Figure 21. Illustration of free space method.](image)

### 7. Test Methods: NIST’s Wafer-Level Materials Metrology to 1 THz

The National Institute of Standards and Technology (NIST) has a long history with microwave materials. NIST’s mission has been to provide new materials test methods for industry. Perhaps the best examples are the split cylinder cavities that were inspired by the pioneering work of Gordon Kent [14]. This section describes NIST’s approach to on-wafer materials measurements and the techniques used to extend the frequency range to 1THz.
All materials at some power level have frequency dependent materials properties. Take for example sea water. At low frequencies, the real part of the permittivity (blue) does not change much with increasing frequency. As the frequency increases to the dipolar relaxation frequency in the GHz, the permittivity drops. At this frequency, the imaginary part of the permittivity (red) has a peak, where the dipoles in solution are out of phase with the driving electromagnetic field. Above the GHz regime, there are other relaxations in the infrared wavelengths due to atomic processes. Even higher in the deep UV, electronic transitions lead to yet other relaxation processes.

![Figure 22. Schematic depicting the complex relative permittivity of sea water.](image)

For convenience, the conductivity is absorbed into the complex permittivity, which appears in Figure 22 as the upturn in the imaginary part (red) at low frequencies. Sea water’s permittivity has a Debye relaxation around 15 GHz, which leads to decrease in the real part (blue) and peak in the imaginary part (red). Other relaxation processes occur at higher frequencies. In any given material, many different relaxation processes can occur across the spectrum. NIST’s on-wafer materials measurements cover 100 Hz to 1.2 THz.

In the early 1990s, Dylan Williams and Roger Marks made major advances in on-wafer measurement science that literally changed radio science to what we know today [15]. Perhaps their most controversial idea at the time was to use multiple transmission lines to build an ultrabroadband calibration [16]. This one big idea is the cornerstone of on-wafer materials characterization at NIST today.

Today’s on-wafer materials metrology at NIST follows five steps to extract the permittivity of materials (Figure 23). The first step to compute the permittivity is to correct the vector network analyzer (VNA) with a reference wafer. This reference wafer has an ensemble of devices called artifacts. Each artifact has a known circuit model. The measurement and the models are used to compute corrections to the VNA. In the complex case where a thin film is the material of interest, an ensemble of devices on both a substrate and thin film is measured. This ensemble of devices allows computation of the capacitance...
and conductance per unit length. Next, a series of finite element simulations of the cross section of the devices is performed. These simulations allow mapping from the capacitance and conductance that are measured to the permittivity. Finally, the permittivity is computed in the last step.

Figure 23. NIST’s on-wafer materials characterization protocol.

The next few paragraphs introduce the measurement techniques, followed by a discussion of the associated calibration protocols and best practices. The goal is to introduce the key ideas of how to perform materials measurements following a protocol like NIST’s. Readers are encouraged to read the complete works of Dylan Williams and Roger Marks. The goal of any on-wafer materials measurement is to provide a quantitative measurement of complex permittivity as a function of some independent variable (frequency, electric field, temperature, etc.). The resulting complex permittivity for a high-k dielectric that exhibited high tunability and low loss is shown in Figure 24.

Figure 24. NIST’s on-wafer materials characterization for a complex oxide thin-film (credit: E. Marksz, N. Dawley, D. G. Schlom).
7.1 Measurements and calibrations

Microwave calibrations is a misnomer. Rather than a true calibration to a standard reference traceable to the SI (International System of Unit measurement), anyone using a VNA is really performing data correction. Despite this distinction—most of the world calls this practice microwave calibration. The goal of any calibration is to accurately remove (or correct) everything between the VNA receivers and sources and the on-wafer measurement probes. In effect, this calibration places the reference planes at the device of interest.

Figure 25. Left is a close-up of the reference wafer with an ensemble of artifacts. Right, a schematic of a network analyzer with a stylized cable with on-wafer probes used to measure an artifact.

Everything in between the instrument and the sample to be measured impacts the data. Take, for example, a simple transmission line. The raw uncorrected data of a transmission line needs to be corrected as follows. As shown in Figure 26, the transmission is high and the reflection is low, but the device appears nonreciprocal ($S_{12} \neq S_{21}$, blue and yellow do not overlap) and the reflection coefficients exhibit different impedences ($S_{11} \neq S_{22}$, green and black do not overlap). There is also a large discontinuity where the instrument switches from a low frequency network analyzer to a high frequency network analyzer. This is incorrect.

A calibration removes these effects and allows the user to compute the true transmission and reflection of a given device (Figure 26, black and yellow lines, right). In this case, any given measurement $M$ is modified by the left ($X$) and right ($Y$) error-boxes as

$$M = XAY.$$  

A calibration simply solves for the $X$ and $Y$, and then multiplies the measurement by their inverses to find $A$, the actual properties of the device.

Designing a set of artifacts that produce a calibration like the one shown here is easy with NIST’s help. NIST’s ultrabroadband calibrations are custom matched to optimally extract the complex permittivity of test substrate or thin film over as broad a bandwidth as possible with the lowest possible uncertainties. The NIST design starts by optimizing the lengths of the transmission lines. The process uses finite
element simulations to estimate the propagation constant and then applies a Monte Carlo optimization protocol to minimize the normalized standard deviation as a function of frequency. Further details about this optimization process/method will be published in a forthcoming publication from NIST.

![Figure 26. Left — uncorrected data of a transmission line. Right — corrected data of a transmission line.](image)

7.2 Substrates and thin films

The approach largely employed by NIST is a two-tiered technique. The first tier corrects the measurements of the second tier and sets the reference impedance to 50 Ω. The second-tier measurements produce two key features. At high frequencies, the second-tier measurements produce propagation constants and error boxes. These measurements primarily use a type of transmission line called a coplanar waveguide. At lower frequencies, interdigitated capacitors are used. In both cases for a dielectric material, the capacitance and conductance per unit length are extracted, as shown in Figure 27.
Figure 27. Broadband permittivity of the high-\(k\) thin film that used interdigitated capacitors at low frequency and coplanar waveguides at high frequency.

For substrates and thin films, the same set of devices is fabricated on both, depositing the metal layers at the same time and keeping the lithographic processes as similar as possible.

Figure 28. Left is a close-up of a coplanar waveguide. Right shows cross sections of the coplanar waveguide with and without a thin film.

Figure 28 shows a top view of a coplanar waveguide of some length \(\ell\). An electromagnetic wave propagates down the signal and grounds. A cross section of a coplanar waveguide is shown on the right for the substrate and thin film case.

### 7.3 Extract circuit parameters

In the high frequency case, the key measurand is the propagation constant. The propagation constant parameterizes how an electrical signal propagates down a transmission line and is given as

\[
\gamma = \alpha + i\beta = \sqrt{(R + i\omega L)(G + i\omega C)},
\]

where \(R\), \(L\), \(C\), and \(G\) are distributed circuit parameters. To a good approximation, the complex permittivity affects \(C\) and \(G\), while the complex permeability affects \(R\) and \(L\).
There are many ways to extract the propagation constant; however, one of the most analytical approaches with the fewest assumptions is very same multiline TRL technique used to correct the first-tier data. In this second-tier analysis, the simple trick developed by Marks [3] is used again. In this approach, two transmission lines with different lengths ($M_i$ and $M_j$) are measured. Using the transmission matrix representation, the following expression is computed

$$M_iM_j^{-1} = XA_iA_j^{-1}X^{-1}$$

The eigenvalues of this expression are computed and results are related to the propagation constant times the difference in the lengths. This value is computed for all the line pairs and then a weighted linear regression allows the complex propagation constant to be computed. All this is achievable with uncorrected data.

Corrected data is often used because that sets the reference impedance on either side of the second-tier error boxes. If the reference planes are translated to the edges of the probe tips, then it is possible to model the error boxes as an impedance transform and estimate the characteristic impedance [17]. In practice, this impedance measurement is almost always imperfect and convolves crosstalk, moding, and pad effects into the impedance estimation. While useful, a series of finite-element simulations is often performed to compute distributed circuit parameters, which validate with the earlier approach. Then we take the computed circuit parameters and use the propagation constant from multiline TRL, which is less sensitive to many of the effects that can confound the other technique.

At this point, we computed the high frequency capacitance per unit length. We now need to compute the low frequency capacitance per unit length. In this regime, we use interdigitated capacitors where we vary the length of the interdigitation. This changing length lets us compute the total capacitance and conductance of the device from an admittance matrix. We then perform a linear regression of the total capacitance and conductance as a function of length. The slope of the regression is the per unit length quantities that we need to compute the low frequency permittivity.

For a substrate, we now have the low and high frequency capacitance and conductance per unit length and likewise for a thin film. However, if the material under test is a thin film we need to subtract off the contribution of the substrate, which is detailed elsewhere [18], [19].

**7.4 Finite-element simulations**

The last step is to simulate the cross section of the device under test and compute the relationship between the measured capacitance and permittivity. This simulation requires a measurement of the cross section. By varying the permittivity in the simulation, it is possible to compute the capacitance.

Figure 29 shows the electric field lines for a coplanar waveguide with a thin film. The top left shows the top view of the coplanar waveguide. The same simulations are performed with the interdigitated capacitor cross-sectional geometry, which has a similar relationship.
Figure 29. A simulation of the electromagnetic fields in a coplanar waveguide. The magnitude of the electric field is the color of the arrow and the direction of the field line follows the arrow’s direction.

7.5 Map to dielectric constant

Finally, the mapping function is computed. There are analytical expressions that relate the capacitance and conductance per unit length to the complex permittivity. These expressions have some limitations, and finite-element simulations have been found to have fewer limitations. In any case, it is possible to compute a linear regression (solid line shown in Figure 30). This regression allows to linearly map from the circuit parameters measured to the permittivity desired.

Fig. 7.9 The resulting mapping function for the simulation of an interdigitated capacitor where we define the permittivity in the simulation and compute the capacitance.

The axes are then swapped and a linear regression of the permittivity versus the capacitance to obtain a linear mapping function is performed. Finally, the measured capacitance is inserted into the mapping function and final permittivity is obtained.
8. Practical Problems Impacting Reproducibility

Much of the published literature on high frequency and mmWave dielectric measurements focus on the numerical stability, mathematical extraction methods, and best-case accuracy for a given method. These factors are very useful for determining the acceptable ranges of usability for a given technique; however, industrial scale use of these measurement methods adds complications that can significantly reduce the usefulness or accuracy of these techniques. It is important to consider these issues when evaluating a measurement method.

For 5G and mmWave applications, most use cases relevant to this iNEMI effort will involve transmission-like structures fabricated with thin film dielectrics that are not typically available in large, machinable samples. This end use case practically limits the methods in this effort to techniques compatible with films or stacks of films ranging from 10 um to 500 um and places an additional hurdle on many of the techniques. Specifically, almost all the existing techniques require precise knowledge of the film sample thickness and require the samples to be smooth and uniformly thick. Measurement of this thickness necessarily requires a separate step in the measurement process, and that step often requires an operator to collect a physical measurement on the sample in a location chosen by the operator. In some cases, this step can dominate all the errors in extraction of the dielectric material parameters. Further, the compressibility of the sample material can lead to operator or tool dependence in the thickness measurement. Evaluation of a measurement method must include a study of operator independence to assess all the practical implications of a given technique.

In addition to operator independence, for many applications it is desirable to be able to characterize a material over an operating temperature range. Techniques that are inherently incompatible with data collection at reduced or elevated temperatures may be of lower interest for industry. Often material behavior under moisture absorption is a concern for high volume industrial applications. Techniques that are slow or require lengthy procedures may limit a user’s ability to evaluate materials against moisture absorption. For example, one common method for examining the impact of moisture absorption involves soaking test samples in a controlled humidity environment and then quickly collecting dielectric performance data after removing the sample from the environmental chamber. For many thin (< 100 um) substrate materials, sufficient moisture absorption or drying can occur over the course of 10 minutes to alter the performance of the material. Hence, methods that allow for samples to be quickly configured in the test equipment, with minimal alignment requirements, and speedy data collection are desirable.

9. Moving Toward 6G

Although 5G is just beginning deployment and probably will not have significant market penetration until 2025, thoughts are turning to what 6G would address. There are significant analyses by standards organizations and case studies summarized in the Heterogeneous Integration Roadmap (HIR) [2]. It is expected that 6G communication will be deployed in the 2030s and will incorporate frequencies beyond 100 GHz, necessitating new innovations in material characterization.

Figures 31 and 32 show Samsung’s perspective on 6G use cases, which include “super-enhanced mobile broadband,” “enhanced latency,” “super-precision positioning” and massive connectivity.
10. Summary

mmWave applications have historically been limited to niche and low volume products. With the expansion of 5G technologies this is no longer the case. Applications ranging from mid volume base station infrastructure to extremely high volume consumer devices are already entering the market. For engineers designing these products it is critical to start with correct electrical material models. iNEMI’s
5 G/mmWave Materials Assessment and Characterization project is working to develop guidelines/best practices for a standardized measurement and test methodology that can be shared with industry and relevant standards organizations. [1]. This report, the second of two published by the project team, discusses emerging measurement techniques that will become increasingly relevant as new spectrum auctions open up additional communication bands above those in the current industry release documents for 5G mm-Waves.

There are important new techniques summarized in this document including wafer-level measurements, as well as time domain spectroscopy-based measurement techniques extending the frequency ranges to higher than 100 GHz to 6G and next generation communication technologies. It is important for industry groups to monitor the new research being pioneered at universities and research institutions.

11. References

[1] iNEMI 5G Materials Assessment and Characterization
https://community.inemi.org/content.asp?contentid=639


https://community.inemi.org/memberfile.asp?id=474 (available to iNEMI members only)


APPENDIX 1 — Contributors

Key Contributors

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ITEQ
ITRI
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MacDermid-Alpha
Mosaic Microsystems
NIST
Nokia
QWED
Shengyi Technology Company
Sheldahl
Unimicron Technology Corp
Wistron
Zestrion
### Glossary

<table>
<thead>
<tr>
<th>Term</th>
<th>Definition</th>
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<tbody>
<tr>
<td>CBCPW</td>
<td>Conductor-backed coplanar waveguide</td>
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<tr>
<td>COMITS</td>
<td>Coherent microwave transient spectroscopy</td>
</tr>
<tr>
<td>CPW</td>
<td>Coplanar waveguide</td>
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<tr>
<td>DUT</td>
<td>Device under test</td>
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<tr>
<td>GSG</td>
<td>Ground-signal-ground</td>
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<tr>
<td>GT-PRC</td>
<td>Georgia Tech Packaging Resource Center</td>
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<tr>
<td>HIR</td>
<td>Heterogeneous Integration Roadmap</td>
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<tr>
<td>LRRM</td>
<td>Line-reflect-reflect-match</td>
</tr>
<tr>
<td>MRR</td>
<td>Microstrip ring resonator</td>
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<tr>
<td>SI</td>
<td>The SI base units are the standard units of measurement defined by the International System of Units for the seven base quantities of what is now known as the International System of Quantities. This system of measurement is based on the metric system.</td>
</tr>
<tr>
<td>SNR</td>
<td>Signal to noise ratio</td>
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<tr>
<td>TEM</td>
<td>Transmission electron microscopy</td>
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<tr>
<td>VNA</td>
<td>Vector network analysis</td>
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