5G mmWave Materials Characterization Project
Project Close-out Review

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iNEMI: Urmi Ray

Watch the webinar recording:

YouTube
https://youtu.be/KNOClJxhN6U

Other video
http://thor.inemi.org/webdownload/2022/Projects/5G_characterization_EOP.mp4
5G mmWave Materials Characterization Project

Agenda:

- Introduction, Project Goals, Timeline – Urmi
- Tasks 1 and 2: Marzena/Say
- Task 3: Mike
- Task 4: Lucas/Nate
- Summary and next steps
Thank You Project Team!

- 3M
- AGC-Nelco
- AT&S
- Centro Ricerche FIAT-FCA
- Dell
- Dupont
- EMD Electronics (Co-Chair)
- Flex
- Georgia Tech
- IBIDEN Co Ltd
- IBM
- Intel
- Isola
- ITRI (Co-Chair)
- Keysight (Co-Chair)
- MacDermid-Alpha
- Mosaic Microsystems
- NIST
- Nokia
- Panasonic
- QWED
- Shengyi Technology Company
- Sheldahl
- ShowaDenko Materials
- Unimicron Technology Corp
- Zestron
‘5G’ extends beyond wireless applications, including automotive and industrial radar – many forward-looking wired applications need material data spanning DC to 100+GHz

Selection of package materials may involve measurements of 50 or more test materials

- Errors in characterization limit accuracy of modeling resulting in time-consuming iterations
- Traditional methods of microwave trimming & tuning are difficult to tolerate in today’s environment
- Development of new materials requires the ability to evaluate the performance of those materials at use condition

Semiconductor products, equipment suppliers, material manufacturers have significant exposure if characterizations are incorrect

Cost to switch: ~$2 per CPU substrate \( \times 20M \) units = $40M

Source: Intel
Industry Collaboration for Solving Materials Measurement Challenges at High Frequencies: Brought Together by iNEMI

www.inemi.org
Problem Statement:

• Next-generation 5G communications solutions require ultra-low loss laminate materials and PCBs/substrates for efficient design and manufacturing.

• However, these materials pose challenges. For example, there is no consistent methodology for measuring transmission loss or Df/Dk, especially for higher frequencies (e.g., >30 GHz).

• Many different approaches are currently used, requiring different fixtures and test methods, sample preparation, and/or data analysis/extraction.
5G mmWave Materials Characterization Project: Tasks

- **Task 1 – Benchmark current industry best practices for low loss measurements**
  - Paper Study
  - **Output:** Report outlining current measurement methods, reference standards, sample preparation, fixture and outlining tradeoffs between different measurement techniques. This report will provide recommendations for Task 3

- **Task 2 – Benchmark research areas to identify new, emerging methods for low loss measurements**
  - Note: This task will be undertaken in parallel with Task 1.
  - Paper Study
  - **Output:** Report outlining emerging measurement methods, reference standards, and other areas in the research realm that can serve as reference for next generation high frequency materials characterization

- **Task 3: Development of Best Practices for Dk <5 measurement 5 – 100GHz**
  - Note: agreed measurement protocols for each technique
  - Round Robin Evaluations
  - Output: Report and test method recommendations

- **Task 4: Round Robin of Commercial Candidate materials**
  - Electrical Targets:
    - Dk – 2-5 @ 5-100GHz
    - Df – <= 0.004 @ 5-100GHz
  - Output: Test report

- **Task 5: End of Project Webinar**
### Project SOW

**Project kick off**

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<th>Quarter</th>
<th>Q1</th>
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<th>Q3</th>
<th>Q4</th>
<th>Q5</th>
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<th>Q7</th>
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<td>Task 1: Benchmark current measurement methods</td>
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<td>Task 2: Benchmark emerging measurement methods</td>
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<td>Task 4: Commercial material testing</td>
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We are Here
Task 1 – Benchmarking permittivity methods, potential reference materials
Task 2 – Emerging technologies / 100GHz & beyond
Task 3 – Multi Lab Round Robin Reference Experiment
Task 4 – Extension to advanced substrate materials
Tasks 1 and 2
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 not obvious.

iNEMI Report focuses on the following resonant measurement techniques: split-post dielectric resonator (SPDR), split cylinder resonator (SCR), balanced circular disk resonator (BCDR), and Fabry-Perot open resonator (FPOR).

The Figure maps these available techniques against frequency.
<table>
<thead>
<tr>
<th>Technique</th>
<th>Frequency</th>
<th>Features</th>
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</thead>
</table>
| Split-post dielectric resonator (SPDR) | Discrete frequency points from 1 GHz up to 15 GHz | ● High measurement precision  
● Easy to use  
● Insensitive to many user errors  
● In-plane component of permittivity  
● Typically extrapolated to 5G mmWaves  
● Typical sample thicknesses less than 1 mm  
● Support temperature sweep measurement  
● IEC 61189-2-721:2015  
| Split cylinder resonator (SCR)     | Discrete frequency points from 10 GHz up to 80 GHz | ● High measurement precision  
● Can be sensitive to user errors  
● Typically interpolated to 5G mmWaves  
● In-plane component of permittivity  
● Typical sample thicknesses around 100 um  
● Support temperature sweep measurement  
● IPC-TM-650 2.5.5.13  
| Balanced-type circular disk resonator (BCDR) | Multiple discrete frequency points from 10 GHz up to 120 GHz | • High measurement precision  
• Requires full 2-port calibration (mechanical to 110 GHz or electrical to 67 GHz)  
• Out-of-plane component of permittivity (typically)  
• Typical sample thicknesses less than 1 mm  
• IEC 63185  
| --- | --- | --- |
| Fabry-Perot open resonator (FPOR, also called open-cavity) | Multiple discrete frequencies between 20 GHz up to 110 GHz | • High measurement precision  
• Can be sensitive to user errors  
• In-plane components of permittivity  
• Some solutions allow for distinguishing in-plane anisotropy  
• Solutions up to 330 GHz available  
• JIS R1660-2  
• [https://www.qwed.com.pl/resonators.html#ResonatorFPOR](https://www.qwed.com.pl/resonators.html#ResonatorFPOR)  
## 5G Initiative Report – Standard Reference Material Development

<table>
<thead>
<tr>
<th>Material type</th>
<th>Approximate thickness</th>
<th>Structure</th>
<th>Approx. $\varepsilon_r$, Tan $\delta$</th>
<th>Important notes</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyclo olefin polymer (COP/ Zeonex®)</td>
<td>100 µm</td>
<td>Isotropic, homogeneous</td>
<td>2.3, 5e-4</td>
<td>Can degrade with finger oil - avoid handling unless using gloves</td>
</tr>
<tr>
<td>Cross linked polystyrene (Rexolite®)</td>
<td>700 µm, 250 µm</td>
<td>Isotropic, homogeneous</td>
<td>2.534, 4.6e-4</td>
<td>Easy to machine, difficult to make flat, stable with temperature and humidity</td>
</tr>
<tr>
<td>PTFE / Teflon®</td>
<td>100 µm, 200 µm</td>
<td>Isotropic, homogeneous</td>
<td>2.1, 2e-4</td>
<td>Easy to machine</td>
</tr>
<tr>
<td>Fused silica</td>
<td>500 µm</td>
<td></td>
<td>3.8 / 1e-4</td>
<td>Easy to machine, preferred by project team</td>
</tr>
<tr>
<td>Sapphire</td>
<td>365 µm</td>
<td>Anisotropic (c-plane)</td>
<td>9.4 / 11.6, 5e-5</td>
<td>Hard to machine</td>
</tr>
<tr>
<td>Technique</td>
<td>Sample dimensions</td>
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<tr>
<td><strong>Split cylinder resonator (SCR)</strong></td>
<td>Thickness: 20 um ~ 300 um (best for 100 um), Lateral: 34 mm x 45 mm &gt; 20G</td>
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<tr>
<td><strong>Balanced-type circular disk resonator (BCDR)</strong></td>
<td>Thickness: 0.1 mm ~ 1 mm, Best for 0.2~0.5 mm, Lateral: 50 mmΦ x 2 each</td>
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<tr>
<td><strong>Fabry-Perot open resonator</strong> (also called open cavity)</td>
<td>Thickness: 0.050 ~ 3 mm, Lateral: min. diameter: 75 mm, max diameter: 150 mm</td>
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<tr>
<td><strong>Split-post dielectric resonator (SPDR)</strong></td>
<td>Thickness: max 0.6 mm, Lateral: min. 15 mm x 15 mm, max 40 mm x 40 mm @15G</td>
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*Common sample size, for cross reference between all devices/techniques, is a challenge*
Benchmarking Emerging Measurement and Characterization Methods

**Transmission Line Method**
- Material assumptions:
  - Sample fits fixture cross section
  - No air gaps at fixture walls
  - Smooth, flat faces, perpendicular to long axis
  - Homogeneous
- Method features:
  - Broadband - low end limited by practical sample length
  - Limited low loss resolution (depends on sample length)
  - Measures magnetic materials
  - Anisotropic materials can be measured in waveguide

**Free Space Method**

(a) simulation in frequency range of 20 GHz to 50 GHz
(b) measurements of MRRs in D-band 110 GHz to 170 GHz.

**Microstrip Ring Resonator**

(a) Feed Lines
(b) CBCPW to MS Transitions

(a) Microstrip Ring Resonator
(b) Free Space Method
(c) Transmission Line Method
Time Domain Spectroscopy: for THz Solutions

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.

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 as 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 the Figure, this measurement system works well over a range of frequencies. If the frequency is too low, however, the errors can increase.
Task 3: Round Robin Experiment

Round Robin Test Labs

3M: USA
Dupont: USA
Intel: USA
ITEQ: Taiwan
ITRI: Taiwan
QWED: Poland

Keysight: USA
Nokia: Finland
NIST: USA
Showa-Denko: Japan
Shengyi Electric: China

SCR
(Split Cavity Resonator)

SPDR
(Split Post Dielectric Resonator)

BCDR
(Balanced Circular Disk Resonator)

FPOR
(Fabry-Perot Open Resonator)
Material Samples

• Two Materials Tested
  • DuPont™ Teflon® FEP Type A
  • Zeonex® Cyclo Olefin Polymer ZF14-188

• Both known & demonstrated to:
  • Have little or no moisture absorption
  • Very low loss
  • Low frequency dependence
  • Available in optically clear, appropriate thickness sheets, good thickness uniformity
  • Isotropic & homogeneous

• Represent ‘best case’ substrate sample

Results from the experiment represent the best we can do today using almost ideal samples
Overview

• Round Robin Experiment
  • ~2200 Data points in data set
  • Spanning 10 labs, 4 methods, 10-110GHz
  • 2 Materials: COP, Teflon
  • Detailed, specific procedure

• Goals
  • High level – how much agreement is there in results
  • Thickness – how closely do labs assess thickness
  • Understand sample quality, handling and sizing / compatibility
  • Understand practicality of techniques
  • Look for obvious biases between equipment types
  • Look for frequency dependency at higher frequencies
  • Do we need traceable standards?
Each color represents a different lab

Each symbol represents a different measurement type
Almost ‘ideal’ samples:
- $\pm 2\%$ Range for $\varepsilon_r$
- $\pm 0.0002$ to $\pm 0.0004$ for TanD
- Unknown accuracy
Is this good?

- Better than many expected
  - Lots of potential lab variations yet results are very good for ‘perfect’ samples
  - Even within a lab ±2% can be challenging for complicated metrologies

- But – for many applications this is not good enough
  - ±2% error could cause design cycle – customer & supplier could differ by 4%
  - Offset mean values can impact HVM distribution tails & DPMs
  - Intel example – this would cause noticeable miscorrelation with design
  - Real, imperfect samples worse

- Reproducibility does not ensure accuracy
  - Certified standard references still needed
  - Accuracy is very important; we have no way to assess it today

A Year of effort to do this study. If SRM was available, labs would have been able to validate measurement tools independently
Goals – how did we do?

High level – how much agreement is there in results?
Best case samples – don't expect better than ±2%, real samples worse

Thickness – how closely do labs assess thickness?
Labs using similar micrometers and averaging yields ~0.2-0.5% differences in numerical results

Understand sample quality, handling and sizing / compatibility
Sample quality, TTV & flatness is a key limiter, equipment compatibility is challenging

Understand practicality of techniques
Limited experience / skills & speed – SCR & SPDR, benefits of FPOR, BCDR require more experience

Look for obvious biases between equipment types
No obvious biases with given sample sizes, except in industrial materials where anisotropy is visible

Look for frequency dependency at higher frequencies
COP & Teflon appear to be very constant at mmWave frequencies, however, without traceability the confidence in this statement is not clear and we have to rely heavily on BCDR and FPOR results for this claim

Do we need traceable standards?
Yes – Further progress in this space is difficult without a high confidence, known reference
Sample Quality Continues to be Measurement Challenge

- Samples have to be thin (typically <250um) – 1% variation only 2.5um
- Commonly available sample thickness variation is often unknown but this order
- Surface topology can be a factor
  - Difficult to assess
  - Easily on the order of thickness variation – potentially similar effects on results
- Best common micrometers have accuracy $\sim \pm 0.5$um with operator reproducibility of several um

Sample surface quality and thickness variation is important
Traceable, uniform & smooth SRM needed
Additional Complications – Realistic samples

- High precision micrometers tend to have limited throat depth
- Can prevent thickness measurement of exact region placed in cavity for measurement
- Some cavities are sensitive to sample bow
- Not always easy to flatten samples sufficiently
- Moving forward, advancements needed to address some of these ‘less interesting’ issues that plague these measurements
Is COP or Teflon a good candidate for SRM?

• Probably not
  • Challenges in getting sub-micron TTV samples of these materials
  • Soft, easy to scratch leads to potential challenges with traceable dimensions
  • COP – degrades with oil contact leading to difficulty in ensuring stability over time
  • Inability to metalize for T-Line structures – important for other applications

• Commonly used today as non-traceable reference
  • Good options until a traceable standard is available

• Also, likely useful for internal ‘transfer standards’ after SRM is available
  • Ability to easily cut to size is useful for compatibility with other test devices
  • Easy to handle, and not breakable – good for routine lab use, quality checks
Summary – Task 3

• Expect >±2% limitation in Er measurement reproducibility of ‘good’ samples (accuracy unknown)
  • For the most part results seem independent of resonator type

• Several contributions to this – none dominant
  • Sample surface and thickness variation
  • Ability to measure thickness in reproducible way
  • Equipment & calibration variations

• 35mm x 45mm & 90mm x 90mm sample sizes fit most equipment sets
  • Thickness requirements vary by Er and specific device but 100-250um is commonly useful for many organic dielectrics
  • Recommend these sample sizes if specific measurement equipment is not known

• Need for traceable standards & improved thickness methods
Task 4
Overview and Objectives of Task 4

Task 3: “Reference materials and measurement method recommendation”
   Materials were deliberately “golden units”
   • Teflon & COP – used by many as reference
   • Ultra low loss

Task 4: “Commercial material testing”
   Materials are real, practical substrate materials submitted by industry representatives in the iNEMI group
   Also included are Reference samples: Fused Silica and Rexolite

Task 4 seeks to validate the findings of Task 3 by showing that these same measurements are effective on real materials from multiple sectors of industry.
Here is what the electronics sample kit looks like

Electronics sample kit: 34 samples from 7 sources

- Thicknesses: 60 um – 300 um
- Dielectric constants: 2.2 - 5
- Isotropic and anisotropic samples

The other kit included some more challenging samples...
And the automotive sample kit

- Thicknesses: up to 4 mm
- Composites (thin film on substrate)
- Limited measurement options
Limited Number of Test Labs for Efficiency and Schedule

- Keysight
- Intel
- NIST
- QWED
- Nokia

Samples submitted by global project members from USA, EU and Asia
Key takeaways

• Two sample sizes sufficient to cover all considered test methods (35 mm x 45 mm and 90 mm x 90 mm)

• Accurate **thickness evaluation** and low thickness variation are of high importance

• Results variation of 2% - 5%

• Standard reference materials are of high interest
Disclaimers to the Task 4 results

1. Limited sample size
   • 5 labs total, 3 labs max on one physical sample
   • Compared samples cut from the same material
   • Significant overlap in instrumentation

2. We are all measuring the same kit
   • Sample-to-sample variation is not as well captured

3. We are all using the same thickness measurements*
   • Most direct possible comparison of microwave measurements
   • Removes variation coming from different thickness measurement capabilities between labs
The electronics samples expanded range of $\varepsilon'_r$ and $\tan\delta$ values.

In just the electronics samples, we cover broad range of frequencies and materials.
This is only using Intel’s thickness measurements. What happens if the labs measure thickness?
Fused silica with lab thickness measurements

Intel measured thickness to be 99.8 um
Keysight measured thickness to be 98 um

A 1.8% difference in thickness input led to a 1.45% difference in measured permittivity

But these are just our candidate standards. What about the practical materials?
Lower boundary for thickness (~ 60 um)

Samples K1, K2, L (same material)

Out of plane!

That’s as thin as we have gone, how thick have we gone?
Upper boundary for thickness (~ 300 um)

These are also the lowest permittivity samples

Samples B, C1, C2 (same material)

The fact that these have the lowest permittivity also saves us a slide!
Upper boundary for permittivity

Samples M, P1, P2 (same material)

- Actually, we went even higher, but it was with a material that I didn’t expect to give good results.
Bonus! – Thickness test glass

This is generic glass thinned down to 150 um and 200 um to see if it was handleable.

Some weird results when permittivity, thickness, and loss are all high, especially at high frequency.

But back to the practical materials.
Known out-of-plane anisotropic materials

This one was only expected to show anisotropy when comparing in-plane to out-of-plane.
Known (in-plane) anisotropic materials

Samples A1, A2 (same material)

Out of plane!

FPOR captures the anisotropy!

And for the more challenging materials in the automotive kit...
Automotive samples – measuring 4mm thick plaques

4mm thick plastic plaque sample

These weren't the only challenging samples in the automotive kit
Automotive samples – measuring foil/thin-film composites

Foils with thin films deposited

Sample M

Sample P

Substrate material

Substrate material with thin film deposited
We are able to measure real materials as well as PTFE & COP
Results variation across labs is on average +/- 2% for real materials
Thickness measurement challenges
- 1 um error on 100 um sample leads to 1% change in measured $\varepsilon$
No unexpected challenges (handling/set up, shelf life) with thickness (60 um – 300 um) or permittivity (2 – 4)
Only 2 sample geometries needed
More challenging materials can be measured with specific measurement setups
Limitation – Lack of Standards

- No NIST traceable standards exist
- Previously available standard discontinued
- Why not redevelop previous SRM?

Currently Available for Purchase!

SRM 2387
NIST Peanut Butter

SRM 3290
Dry Cat Food

SRM 2870
Permittivity & Loss Tangent

Lack of Standards

- SRM 2387
- SRM 3290
- SRM 2870
Previous Generation NIST SRM

- Too thick for low loss mmWave methods shown below
- Required difficult to source machining to thin sample, ruining traceability
- X-Y dimensions of thinned sample still incompatible with most tools
- Certified only at 10 GHz – too low for mmWave / 5&6 G
- Today, all permittivity tool vendors are operating without traceable standards for validating tool sets
Where do we go from here?

- New iNEMI project – “5G Reference Material Development”
- Project kick-off Jun 16th
- Please join (contact Urmi Ray for further info)
Thank you

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