Benchmarking of Current Industrial Best Practices and Emerging Techniques for the Consistent Electric and Dielectric Characterisation of Materials from Microwave to Millimetre-Wave Ranges

Malgorzata Celuch, Marzena Olszewska–Placha, Lukasz Nowicki, Janusz Rudnicki

QWED Sp. z o. o.
Warsaw, Poland
www.qwed.eu
Email: mceluch@qwed.eu

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ULTCC6G_EPac

I4Bags

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3. Thanks to Lukasz Nowicki, my co-author and Ph.D. student, for summarising the work (written paper & some slides) from a newcomer's perspective.

25 years of QWED

Outline

1. Why Measuring MATERIALS?

2. Why RESONANT Methods?

3. ROUND-ROBIN Results for Low-Loss-Substrates in iNEMI 5G Project:

   - Microwave Best Practices,

   - Emerging mmWave Techniques.

4. Modelling-Based Design of Resonator Test-Fixtures for Other Materials:

   - Metallic Foils and Resistive Films (inl. Battery Anodes),

   - Liquids (incl. Coolants).

4. Extensions to Surface Imaging.

5. Conclusions.

Why Measuring Materials?

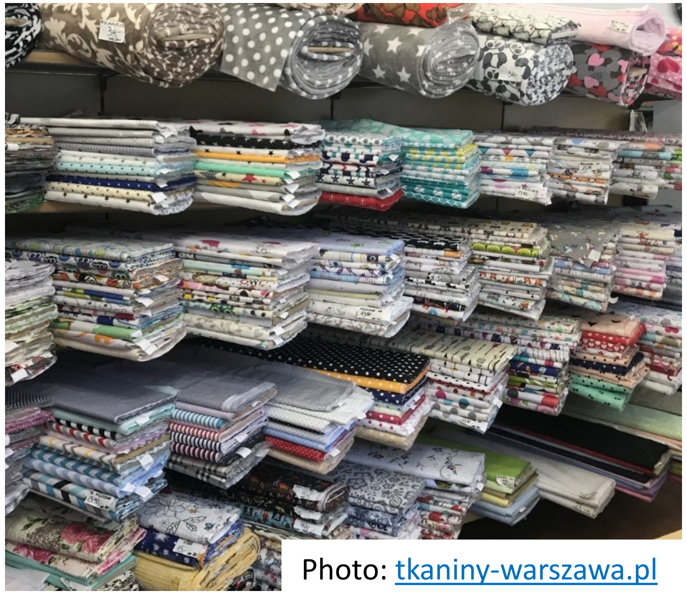


Photo: tkaniny-warszawa.pl

Premise #1: Personal Disillusion

1. my ”dream” materials are usually UNAVAILABLE
2. for available materials, characterisation data ARE usually UNAVAILABLE at my desired use conditions
3. available catalogue data is usually DIFFERENT from my test measurements
4. testing with equipment from a DIFFERENT vendor gives a DIFFERENT result

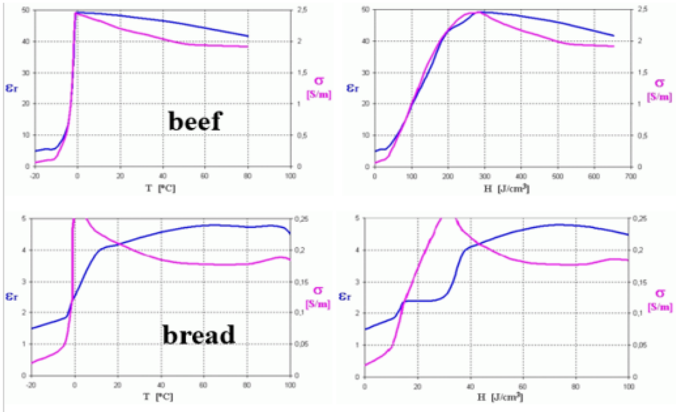
Vendor 1 → ε_r^1

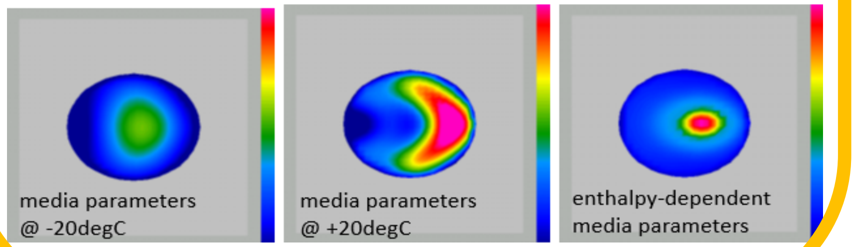
Sample

Vendor 2 → ε_r^2

what is vendor-specific,
what is method-specific,
what other criteria come into play

In some cases, nonlinear modelling is necessary to correctly design devices with tempearature-dependent materials.





source: IEEE Microwave Magazine, Dec. 2022, article by J. Grosinger announcing IEEE RWW 2023 Women in Microwaves Event

Simulation results can only be as good as the material data fed into the simulator.

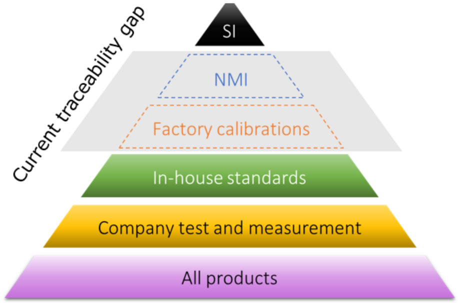
Why Measuring Materials?

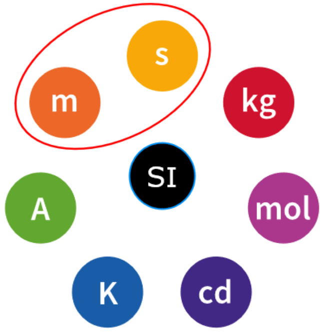
Premise #2: Industrial Demand & Treaceability Gap for 5G/mmWave Materials

1ST SPACE MICROWAVE WEEK

source: L. Enright (NIST), ICEP 2023 [17]

Currently, there is no traceable standard

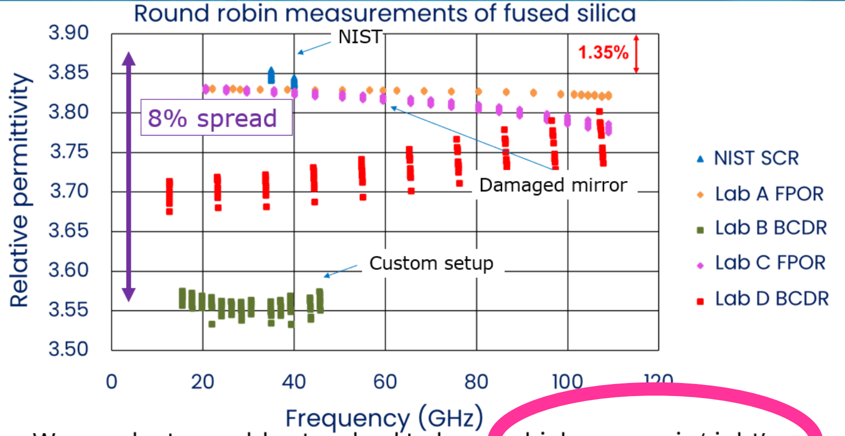




"Traceability" is an unbroken chain of measurements that end at the SI

iNEMI

The semiconductor industry needs dielectric standards



We need a traceable standard to know which answer is 'right'

iNEMI

source: M.Hill (Intel) & M.Celuch (QWED), IPC-APEX 2021
https://www.qwed.eu/nanobat/IPC_APEX_2021_5GMaterialsCharacterisation.pdf

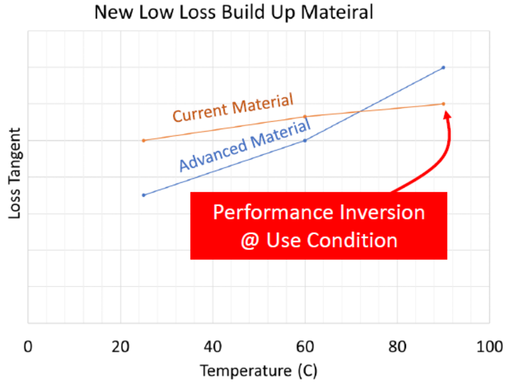
Motivation & Industry Needs

IPC BUILD ELECTRONICS BETTER

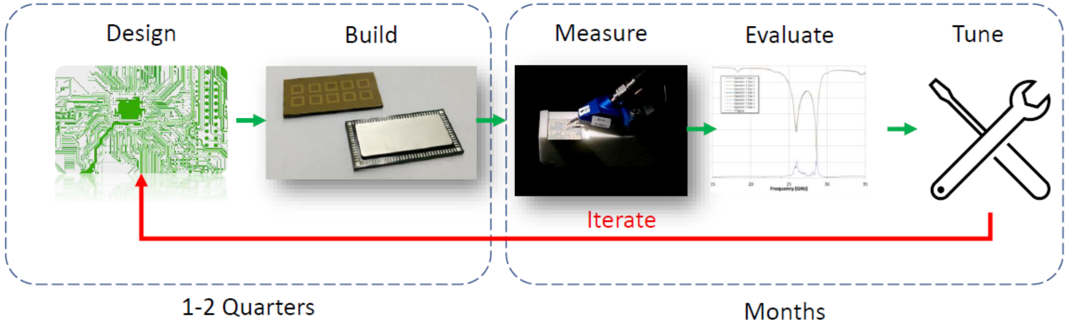
- Development of new materials requires the ability to evaluate the performance of those materials at use condition
- Errors can be very costly

Cost to switch: ~$2 per CPU substrate

x 20M units = $40M



Errors in characterization can cost many $10's of millions for a single program, or worse, induce unexpected product failures



Why Measuring Materials?

Premise #3: General perspective & European priorities

source: [ami20230.eu](ami20230.eu)

MATERIALS 2030 MANIFESTO

Systemic Approach of Advanced Materials for Prosperity – A 2030 Perspective

7 February 2022

"Materials, especially advanced materials, are the backbone and source of prosperity of an industrial society"

source: emmc.eu

"Materials are at the core of industrial processes, products and their functions and properties. "

Model development
Data and knowledge representations
Software tools
Industrial transformation
Policy frame

Why Resonance Methods?

1[ST] SPACE MICROWAVE WEEK

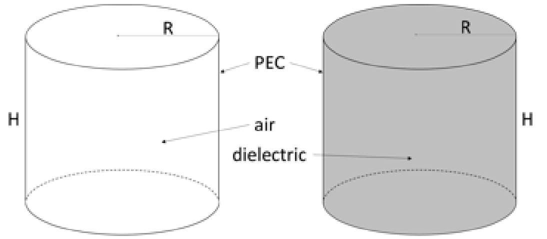
Premise #1: Well-Controlled Sensitivity to Material properties

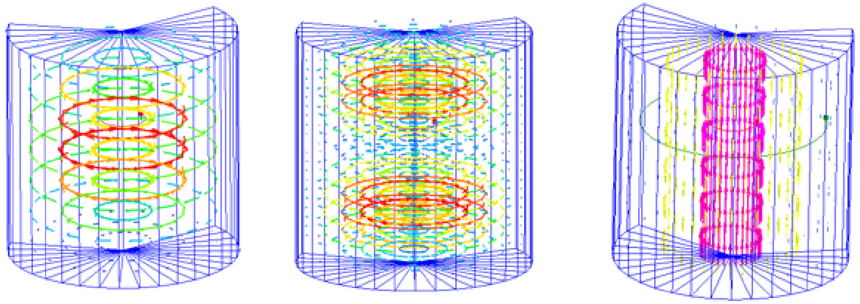
Resonance in theory: non-zero electromagnetic fields exist in isolated structures (no excitation).
Field properties are well-defined and **linked to material properties**.

E.g. for cylindrical cavities:

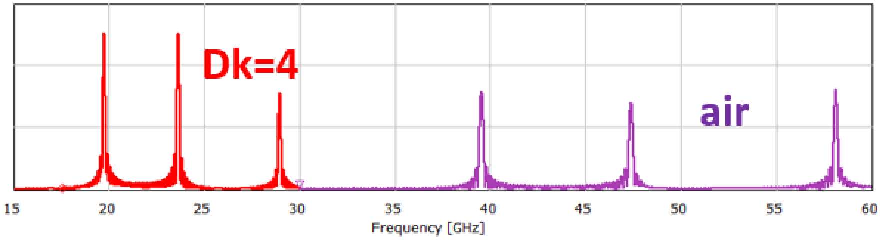
$$f_{r,mnp} = \frac{c}{\sqrt{Dk}} \sqrt{\left(\frac{\kappa^{(,)}_{mn}}{\pi R}\right)^2 + \left(\frac{p}{H}\right)^2}$$

in non-magnetic low-loss dielectrics





TE011 TE012 TM010

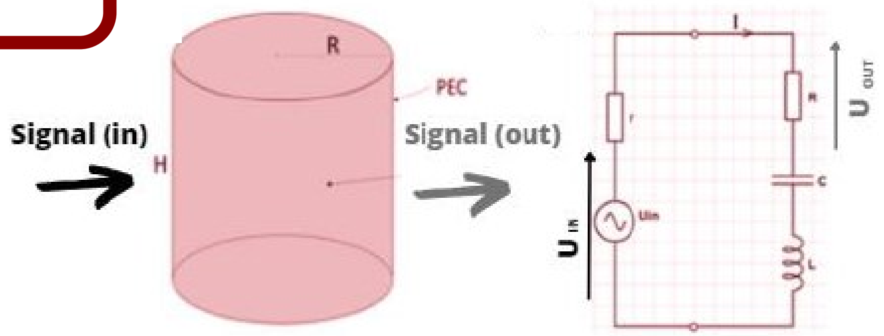


For filled cavities of any shape:

$$f_{r,mnp} = \frac{c}{\sqrt{Dk}} \, K(\text{modal\_index, cavity dimensions})$$

For inhomogenous filled resonators, the ralations are not so simple, but can be resolved with computational modelling.

Lumped circuit analogue for a single resonance:



Why Resonance Methods?

Premise #2: Sensitivity to Ultra-Small Losses (increasingly relevant to 5G & above)

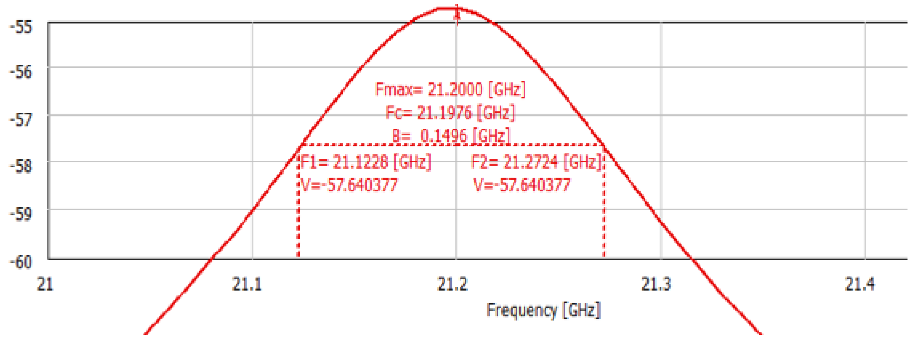
1ST SPACE MICROWAVE WEEK

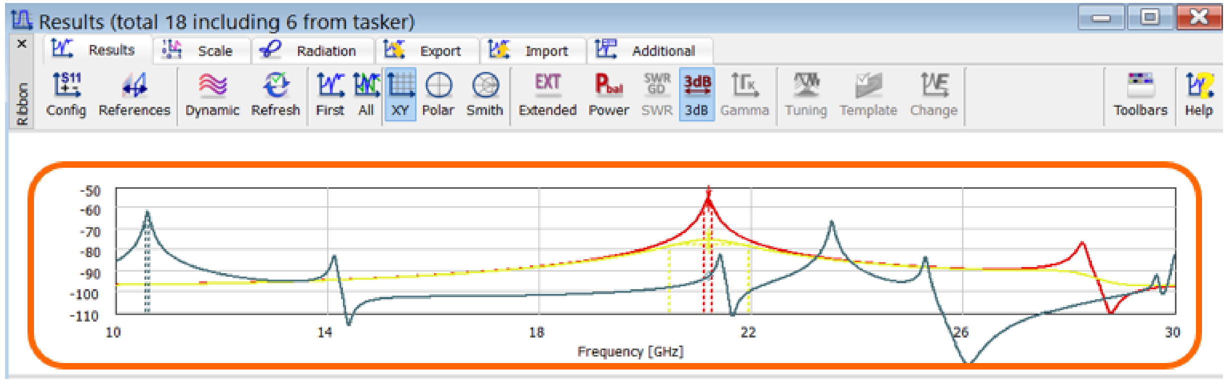
in non-magnetic low-loss dielectrics

2* electric_energy_stored

$$Q = 2\pi \frac{\iiint_V \varepsilon \vec{E} \cdot \vec{E}^* dv}{T \iiint_V \sigma \vec{E} \cdot \vec{E}^* dv} = \frac{\omega\varepsilon}{\sigma} = \frac{1}{Df} \approx \frac{fres}{\Delta f} = (\tan\delta)^{-1}$$

electric_dissipated_power





ε_r=1 σ=0.00833 S/m
@21.2GHz:
tanδ=0.071
Q_{SUT}= 1 / 0.0071 = 141
Q_{S21}=21.2/0/1496= 141

ε_r=1 σ=0.0833 S/m

ε_r=4 σ=0.0166 S/m

Typical 5G materials as measured in iNEMI round-robin [2][16]: tan δ ~ 10^{-4}

Specifically challenging measurements of ultra-low losses (at cryogenic tempreratures): Q ~ 10^{8}
(J. Krupka et al. IEEE Trans. Microwave Theory Tech., vol 47, pp. 752-759, June 1999.)

iNEMI 5G Project Overview

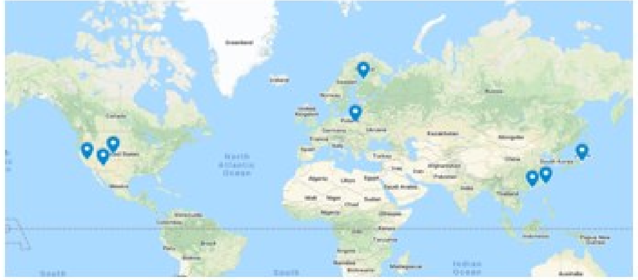
[1] ”5G/mmWave Materials Assessment and Characterization” Project

https://www.inemi.org/article_content.asp?adminkey=5cc4f4100ebf2ba1f3e6fd6294749139&article=161

1ST SPACE MICROWAVE WEEK

Our project: iNEMI

- 3M
- AGC-Nelco
- Ajinomoto USA
- AT&S
- Centro Ricerche FIAT-FCA
- Dell
- Dupont
- EMD Electronics (Co-Chair)
- Flex
- Georgia Tech
- Showa Denko Materials
- IBIDEN Co Ltd
- IBM
- Intel
- Isola
- ITRI (Co-Chair)
- Keysight (Co-Chair)
- MacDermid-Alpha
- Mosaic Microsystems
- NIST
- Nokia
- Panasonic
- QWED
- Shengyi Technology Company
- Sheldahl
- Unimicron Technology Corp
- Zestron



Sample Material Requirements

- Stable, Low loss
- Low moisture absorption / temperature dependency
- Isotropic
- Good mechanical & handling properties

1st Project Stage

- Precision Teflon
- Cyclo Olefin Polymer

2nd Project Stage

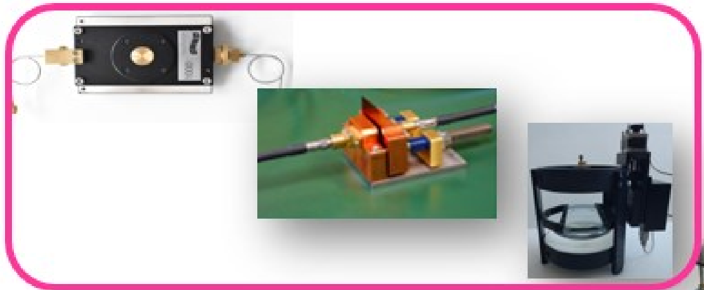
- Rexolite
- Fused Silica

Industrial

- Automotive

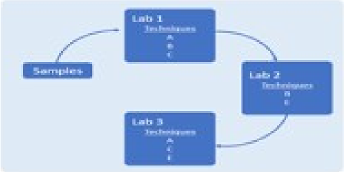
Techniques Included

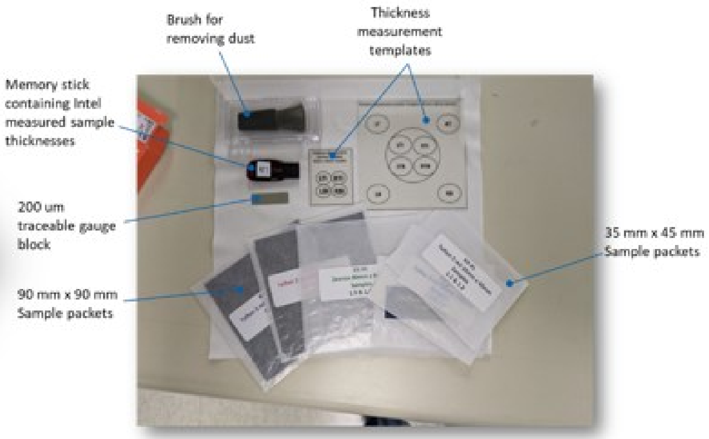
- Split Post Dielectric Resonator
- Split Cavity Resonator
- Fabry-Perot
- Balanced Circular Disk Resonator



→ Frequency Span : 10GHz – 100GHz with overlaps

10 Laboratory Round Robin



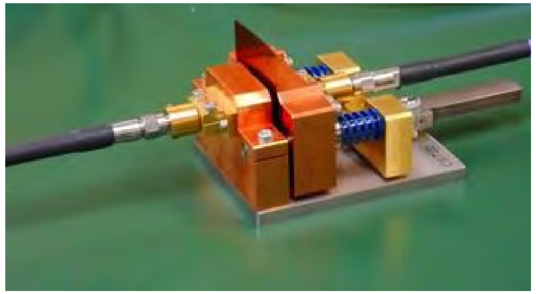


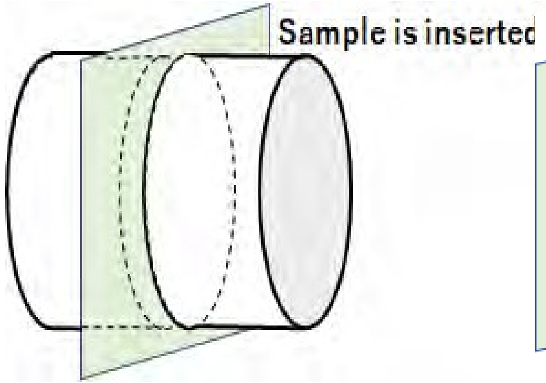
10 Sample Kits Created

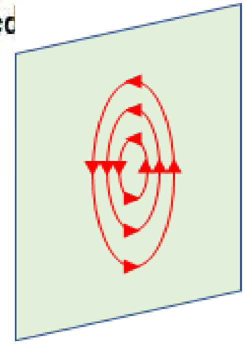
- Sample sizes 35 mm x 45 mm, 90 mm x 90 mm
- circulated between 10 labs

Operating Modes of SPDR, SCR, FPOR

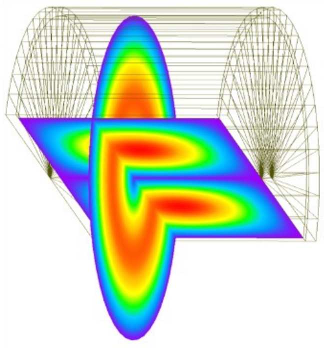
SCR





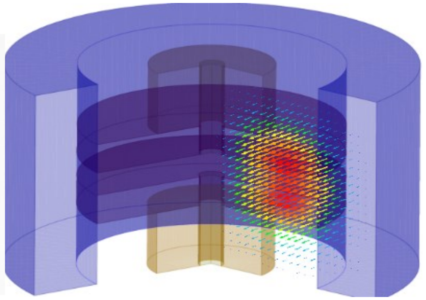


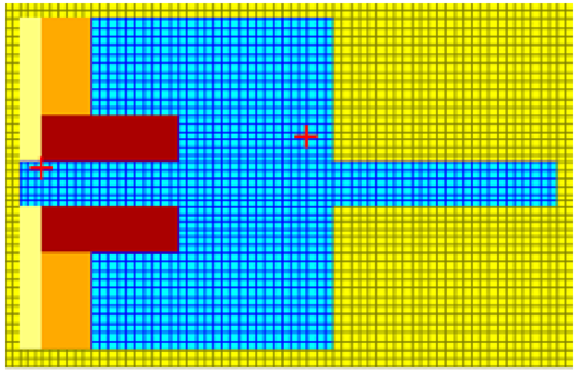
In-plane Electric field is applied to Sample

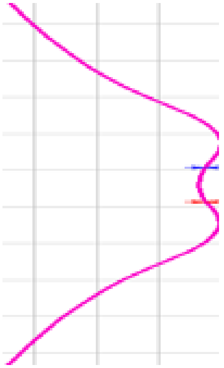


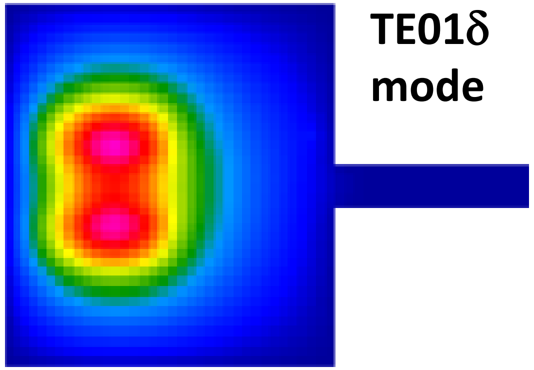
TE011 mode

SPDR



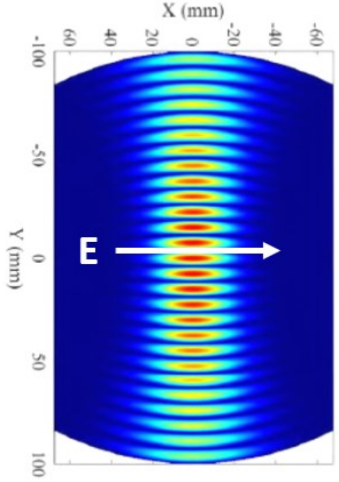




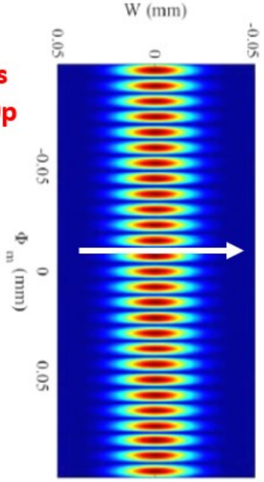


TE01δ mode

FPOR



modes TEM00p



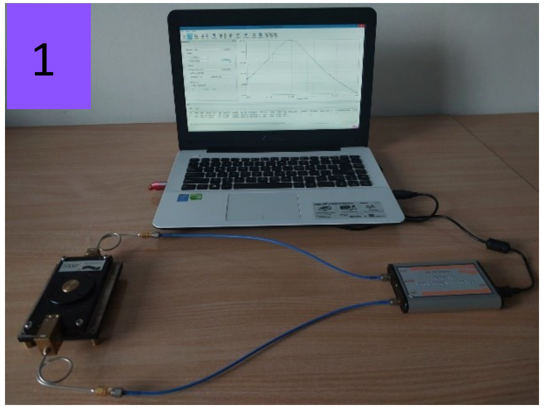
All 3 resonators are designed for in-plane permittivity measurements.

Example Operating Procedure of SPDR

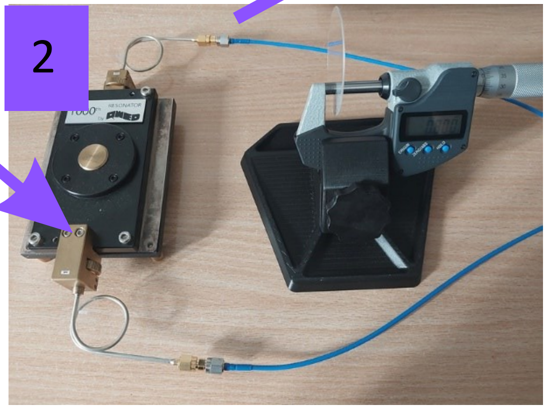
measurement time < 1 minute – and professional VNA is not needed

0. Connect the SPDR to Q-Meter using SMA cables.

Connect Q-Meter to PC using USB cable.

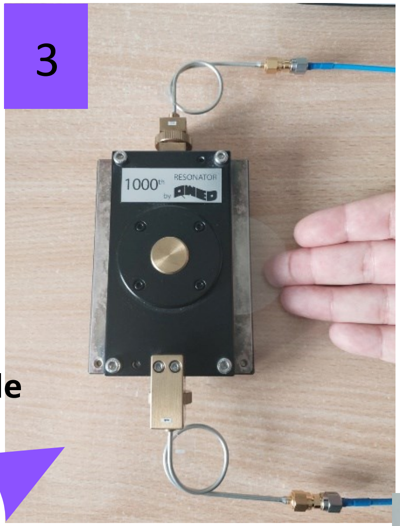


1. **Measure "empty" SPDR - app invoked measurement.**

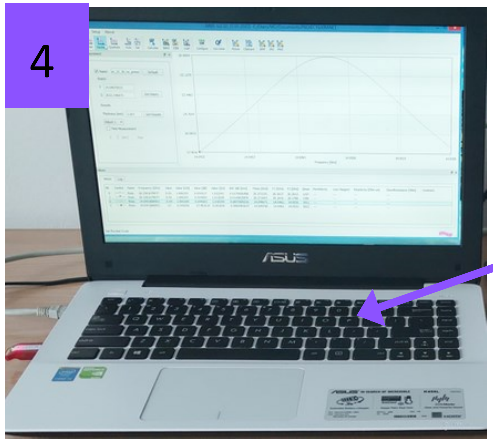


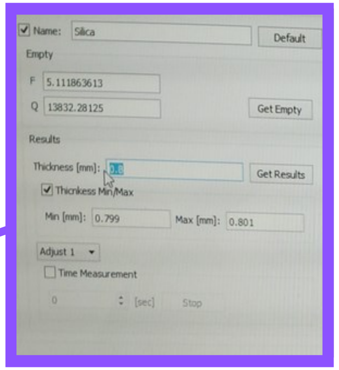
2. Measure thickness of the sample

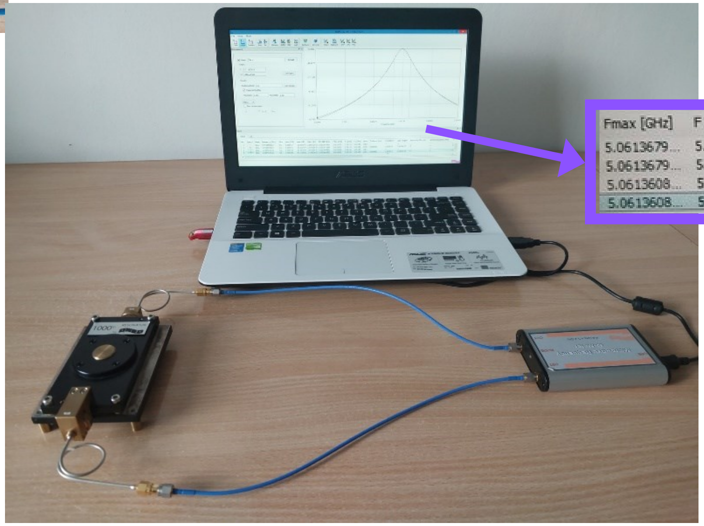
3. Insert the sample into SPDR

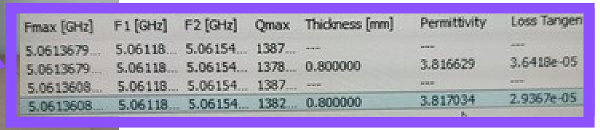


4. Insert the sample thickness into the PC app





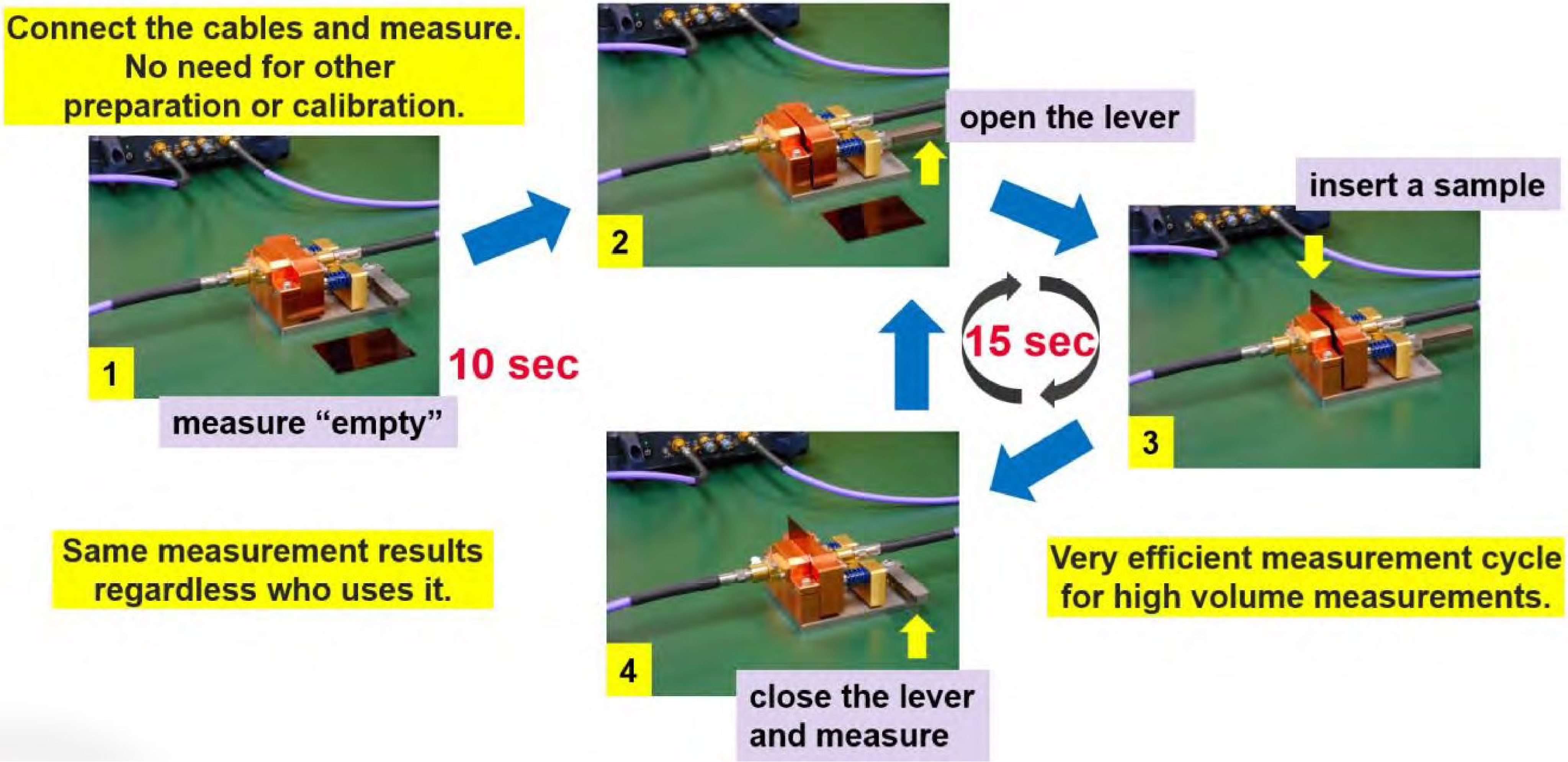




Fmax [GHz]	F1 [GHz]	F2 [GHz]	Qmax	Thickness [mm]	Permittivity	Loss Tangen
5.0613679...	5.06118...	5.06154...	1387...	---	---	---
5.0613679...	5.06118...	5.06154...	1378...	0.800000	3.816629	3.6418e-05
5.0613608...	5.06118...	5.06154...	1387...	---	---	---
5.0613608...	5.06118...	5.06154...	1382...	0.800000	3.817034	2.9367e-05

5. Material parameters are extracted automatically

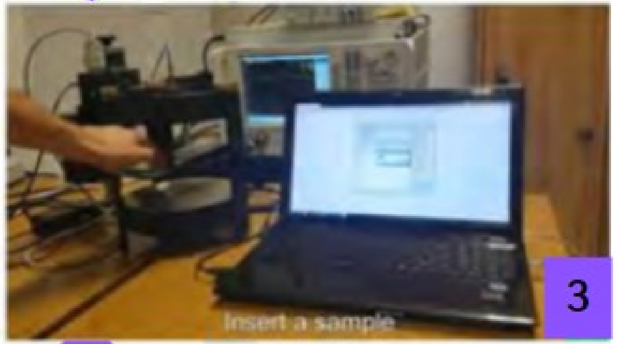
Operating Procedure of SCR



Operating Procedure of FPOR

1. Connect the FPOR to VNA and PC with control app.

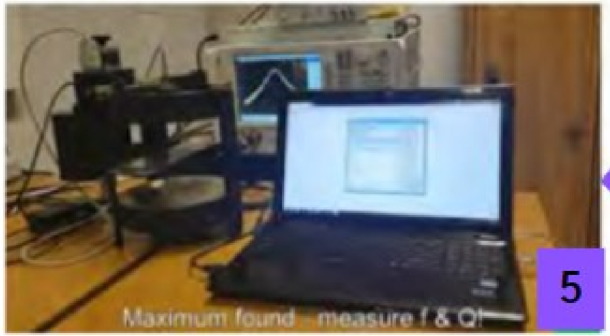
2. Measure "empty FPOR" (resonant frequency and Q-factor at M..N modes)



3. Insert the sample into FPOR.

4. Automatic procedure finds M..N modes of sample-loaded FPOR.

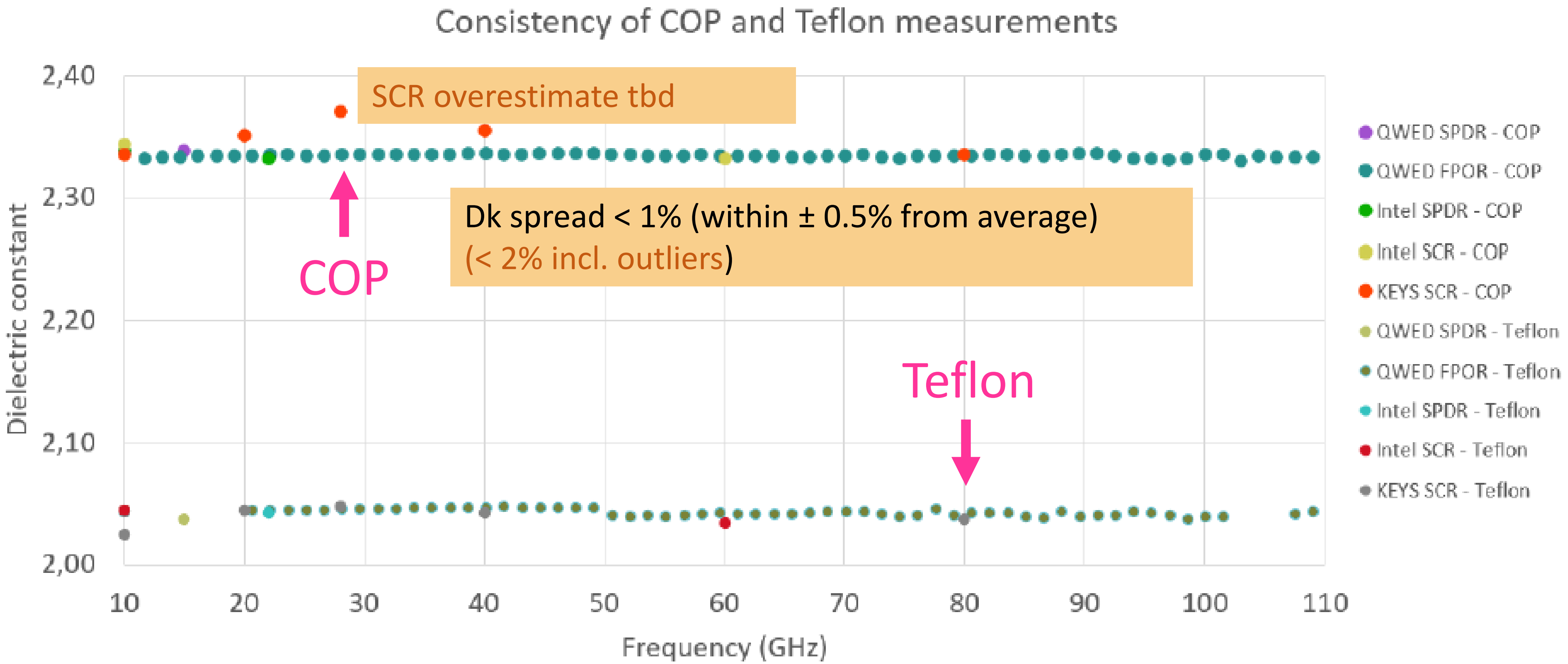
5. Material parameters at consecutive frequencies (modes) are extracted automatically.



Example of iNEMI Round Robin Results

1ST SPACE MICROWAVE WEEK

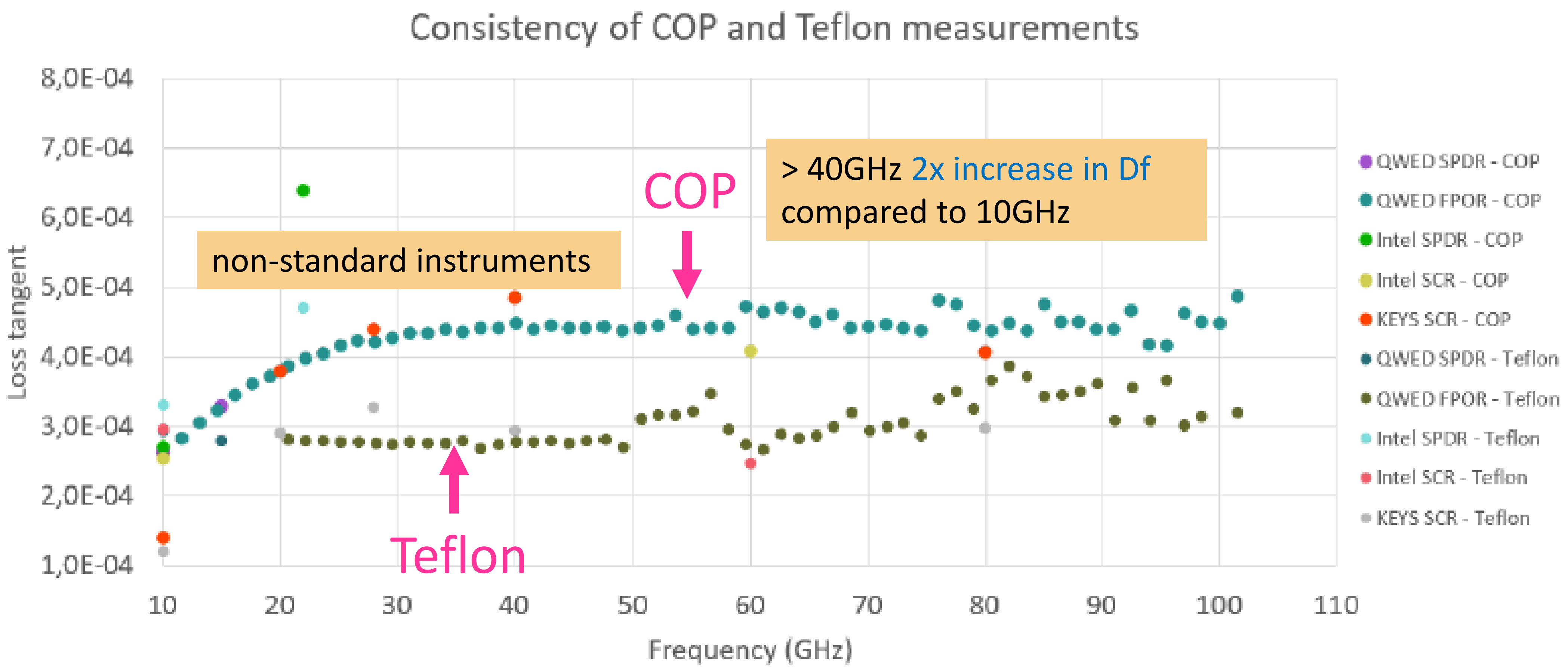
Repeatability and reproducibility studies have shown that error bars of Dk (excluding outliers) are comparable to uncertaintes in sample thickness and sample-to-sample differences.



Example results of dielectric constant measurements by SPDR, SCR and FPOR methods for Cyclo Olefin Polymer (COP) and Teflon.

Example of iNEMI Round Robin Results

1ST SPACE MICROWAVE WEEK



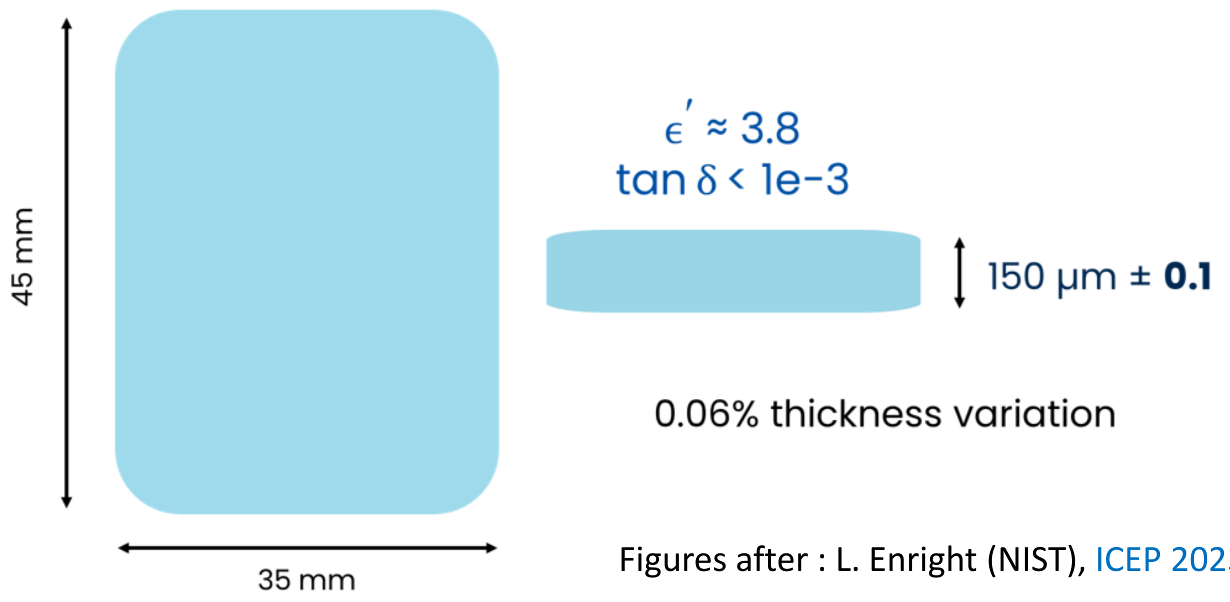
Example results of loss tangent measurements by SPDR, SCR and FPOR for COP and Teflon.

iNEMI Ongoing Work

1ST SPACE MICROWAVE WEEK

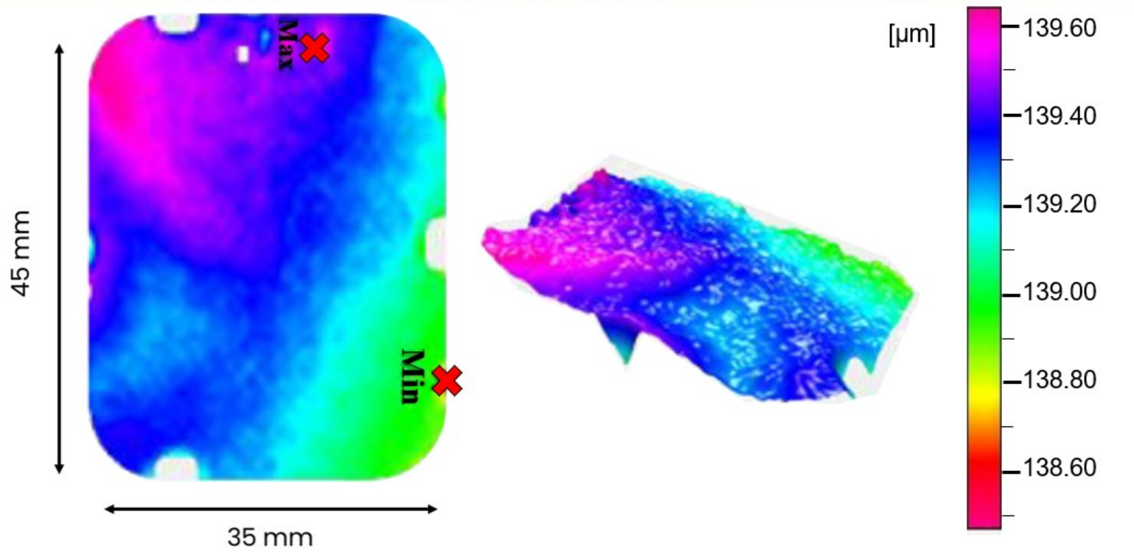
Development of Standard Reference Materials for 5G (to be offered by NIST)

High-purity fused silica with high uniformity of thickness material selected by iNEMI SRM consortium



Figures after : L. Enright (NIST), ICEP 2023 [17]

An example thickness map of an earlier prototype



1 µm TTV with old fabrication process

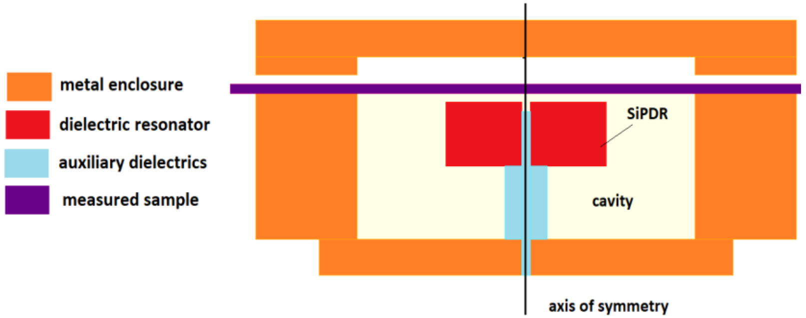
iNEMI International Electronics Manufacturing Initiative

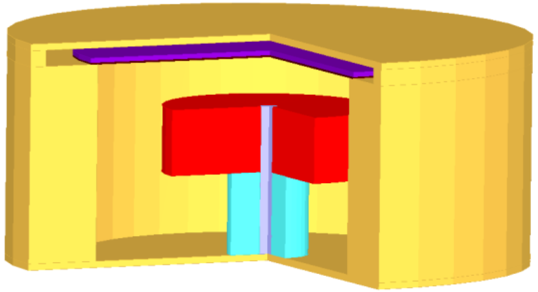
"mmWave Permittivity Reference Material Development":
https://www.inemi.org/article_content.asp?adminkey=3674fa3699682cd862a12262cfa07fd5&article=238
5G/6G mmWave Materials and Electrical Test Technology Roadmap (5G/6G MAESTRO)" , part of NIST Roadmap:
https://www.inemi.org/maestro

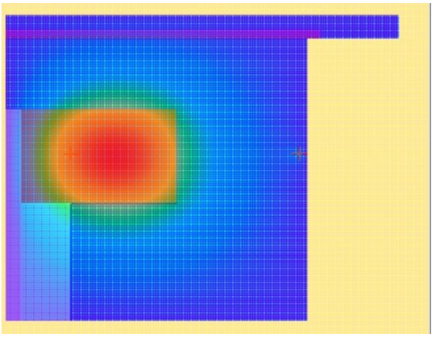
Measurements of Resistive Films: SiPDR

SiPDR

Single-Post
Dielectric
Resonator

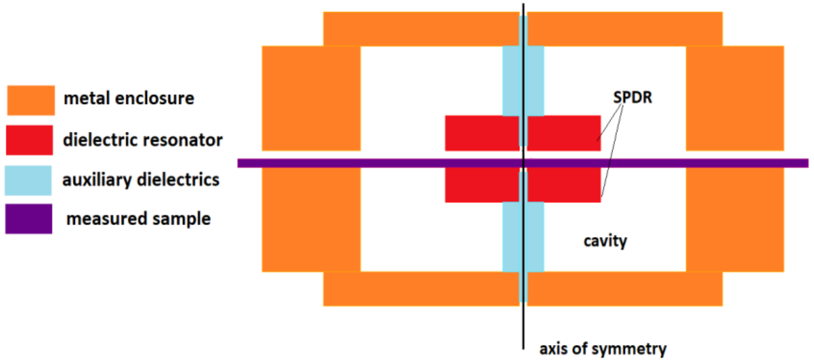


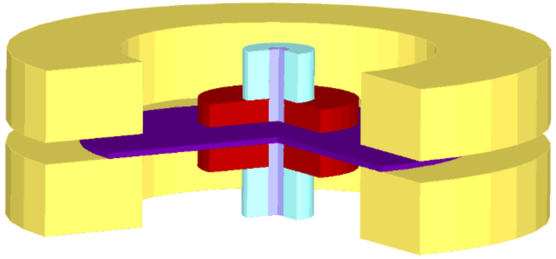


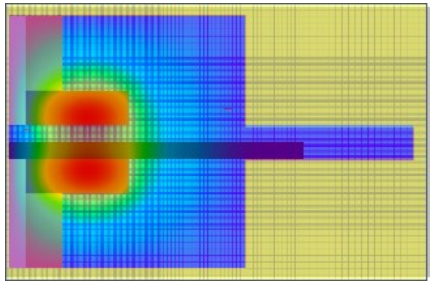


SPDR

Split-Post
Dielectric
Resonator



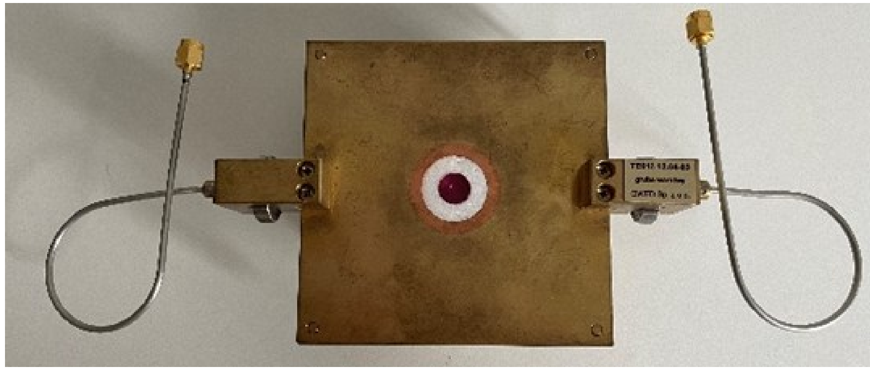




Compared to SPDR, in SiPDR the sample is placed in weak (but well defined) electric field.
This is why measuring of higher electric losses becomes feasible.

Measurements of Metal Foils: SaDR

Sapphire Dielectric Resonator shown with an open and closed cavity.

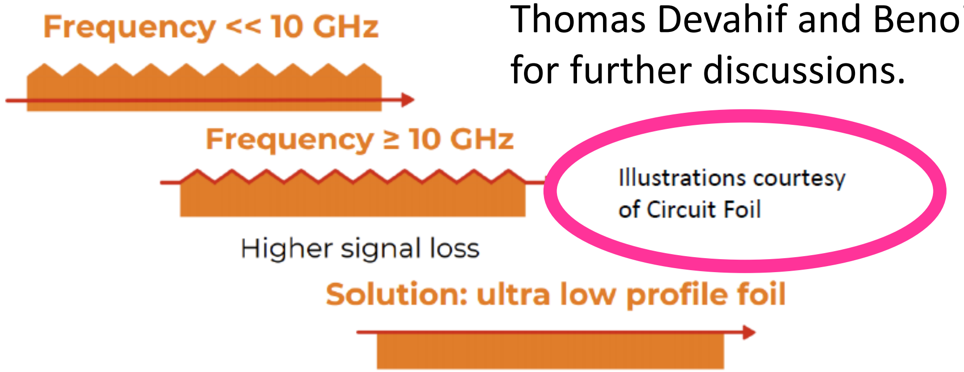


Note: sapphire resonator is very-high-Q.
Foils made of real conductors and placed on its top and bottom sides decrease the Q-factor, allowing for the measurement of metal conductivity or sheet resistance.

Example measurements results of copper foils

[1] " Reliability & Loss Properties of Copper Foils for 5G Applications" Project
https://www.inemi.org/article_content.asp?adminkey=b5202baac78313e4914809b2f481b372&article=209

Samples	*Frequency [MHz]*	*Q-factor*	*Effective Conductivity [S/m]*
HighRoughness TopSide	13902.226	5674.4	4.9920E+07
LowRoughness TopSide	13892.998	5775.2	5.1889E+07
HighRoughness BottomSide	13897.898	4636.1	3.2333E+07
LowRoughness BottomSide	13899.851	4654.4	3.2602E+07



Special thanks to Circuit Foils:
Thomas Devahif and Benoit Wittmann for further discussions.

Cavities for Measuring Ultra-Low-Loss Dielectrics

TE01δ mode Dielectric Resonator shown with an open and closed cavity.

Note: the sample-under-test forms the actual resonator within the cavity

Measurements of Liquids (incl. Coolants)

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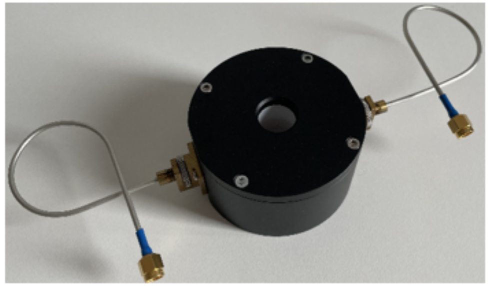
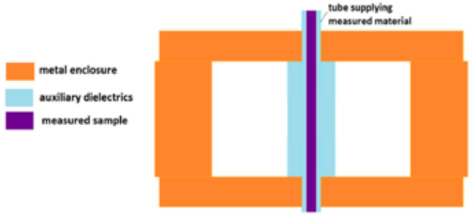
Low frequency dielectric resonator cavities



Dielectric resonator cavity at 2.5 GHz

Higher frequency cavity resonators



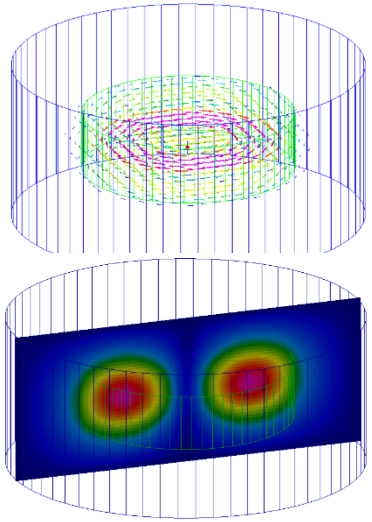
24-GHz Cavity resonator
(with fused silica tube, rubber tube and syringe)

Fabry-Perot Open Resonator

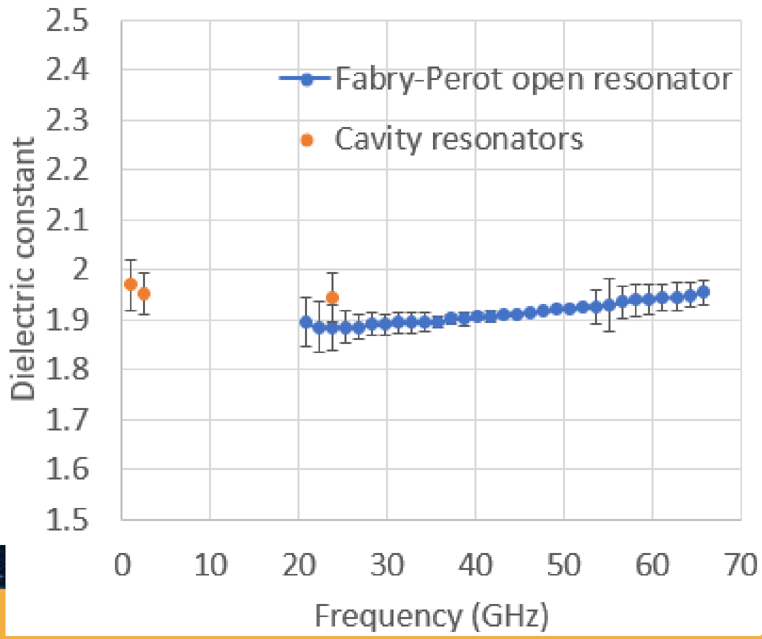
Note: FPOR with a liquid holder is still a prototype at this moment.

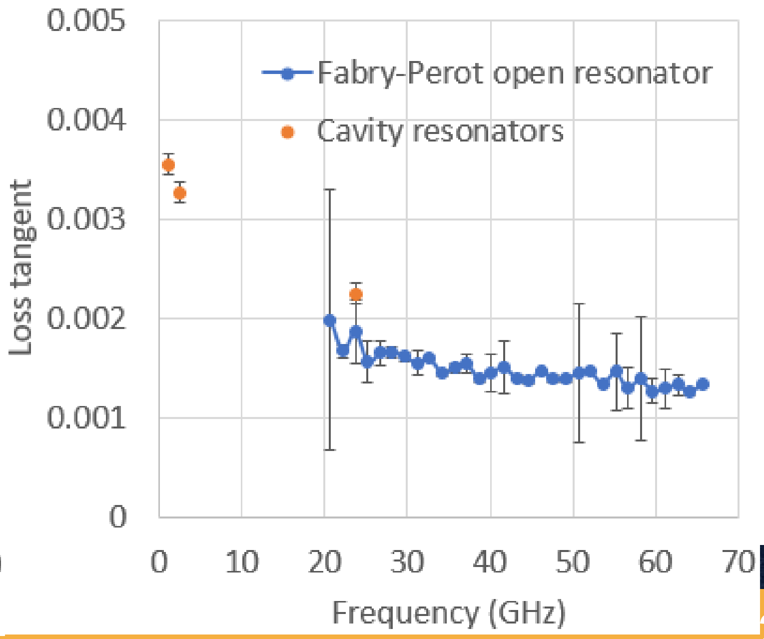
Single solution for 15-50GHz

Modelled electric field distribution:



Example measurements of a popular coolant





Extensions SPDR and SiDR Methods to Surface Imaging

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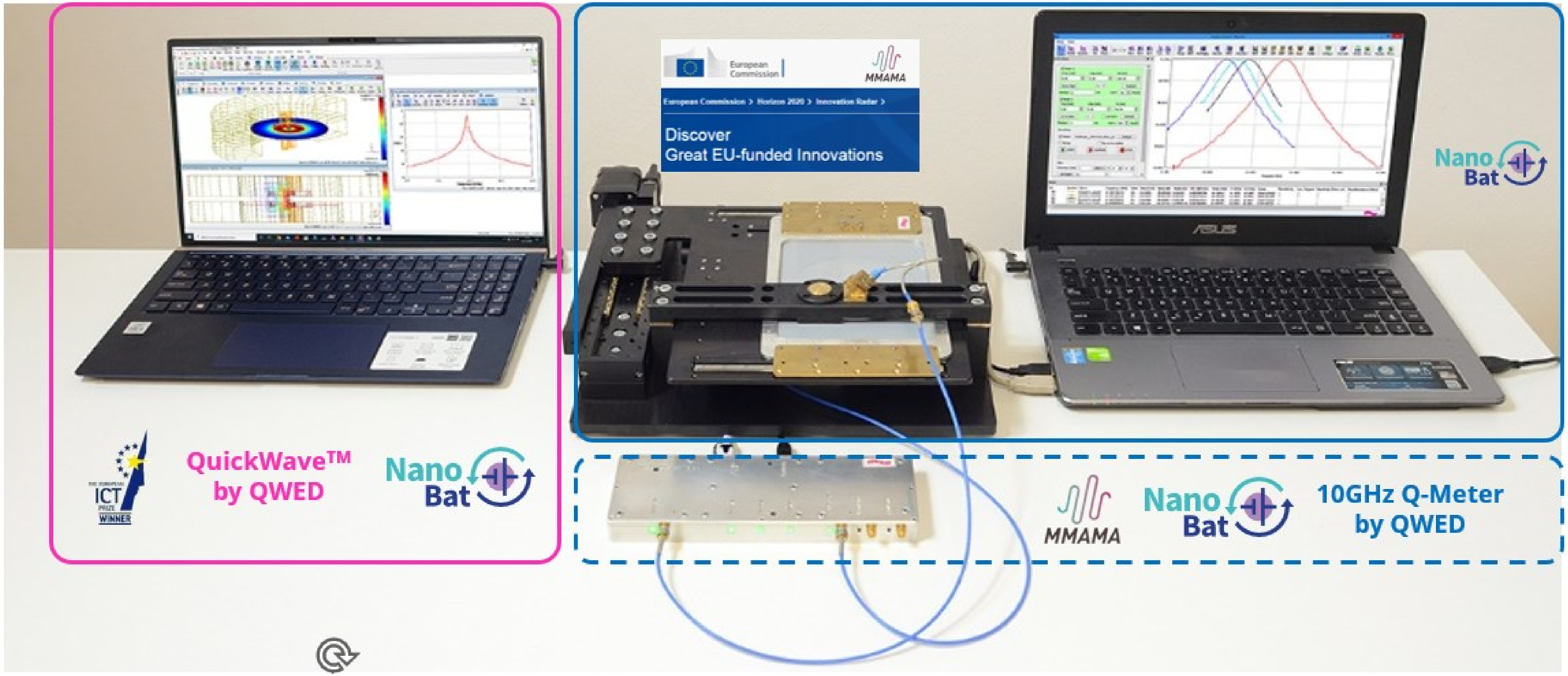

2D SPDR Scanner @10GHz: Finalist of the European Innovation Radar Prize 2021

Exploring Synergies between Materials Measurements & Computer Modelling

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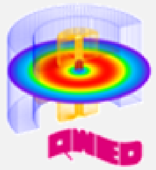
Commercial since 1997
QuickWave Simulation Software
~1000 licences implemented

THE EUROPEAN ICT PRIZE WINNER

Modelling (EM, MW, multiphysics,...)
- waves in free space is "easy" Maxwellian
- wave interaction with matter is "complicated"...

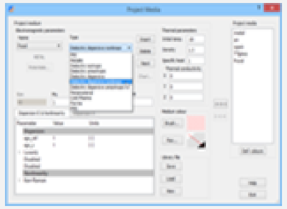


Applicator design
& model for
parameter extraction

INNOVATION

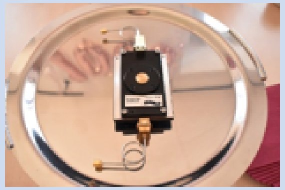
Material measurements

Accurate material parameters (constitutive relations)



Commercial resonator test-fixtures since 2001

1000th unit sold in 2020



Open Platform Examples & Tools

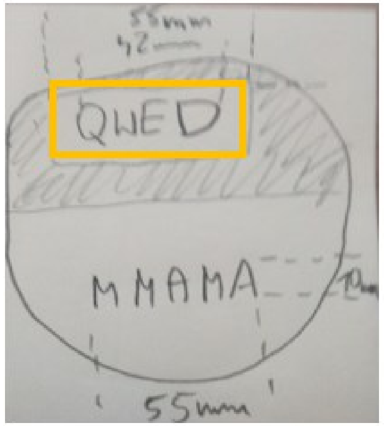
QW-Modeller for QuickWave
Free general purpose 3D CAD modeller for QuickWave.

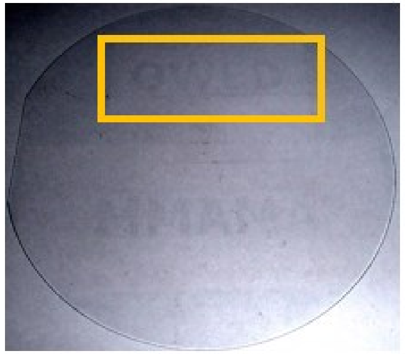
Open Platform Examples:

SMM tip presented at Numerical Electromagnetic

European Standard:
IEC 61189-2-721:2015
CEN-CENELEC Workshop 2021

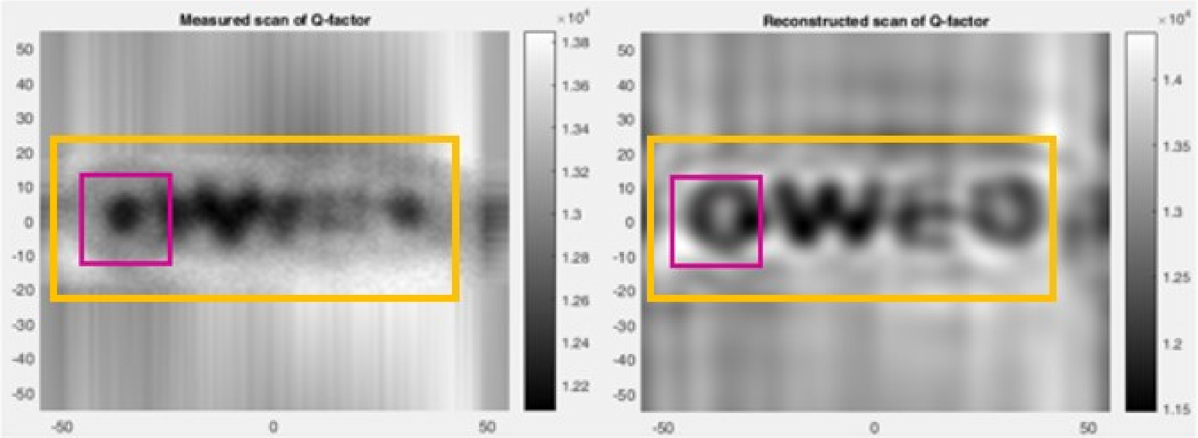
Example: 2D SPDR Imaging of Organic Semiconductors





Patterned PEDOT:PSS sample
courtesy MateriaNova, Belgium

Modelling-Based Resolution Enhancement of Surface Images



raw image of sample resistivity
(measured Q-Factor)

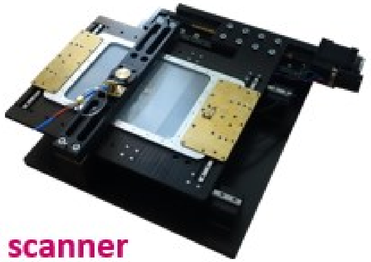
image further deconvolved
using SPDR field pattern
pre-simulated in QuickWave

MMAMA

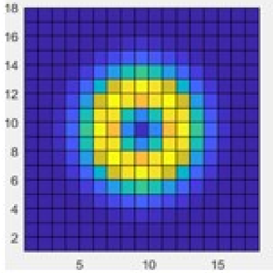
MATERIA NOVA

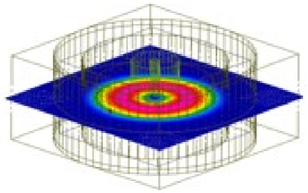
The National Centre
for Research and Development

M-ERA.NET



2D SPDR scanner



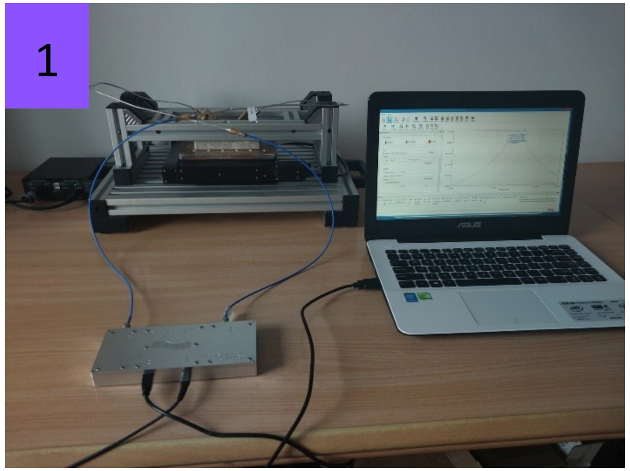


2D iSiPDR Scanner @10GHz: Operating Procedure

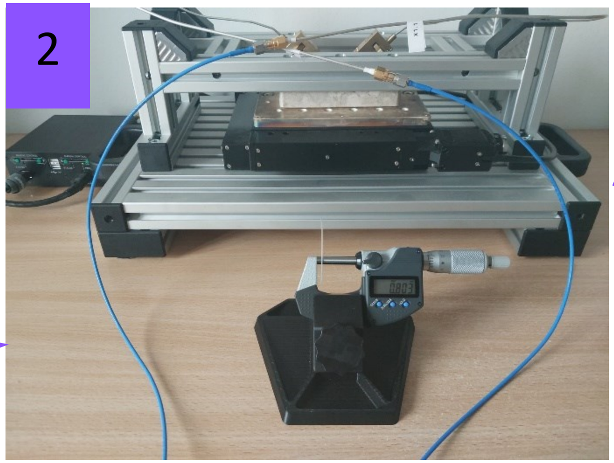
0. Connect the iSiPDR to Q-Meter using SMA cables

Connect Q-Meter and STANDA Motor to PC using USB cable.

1. Measure "empty" iSiPDR



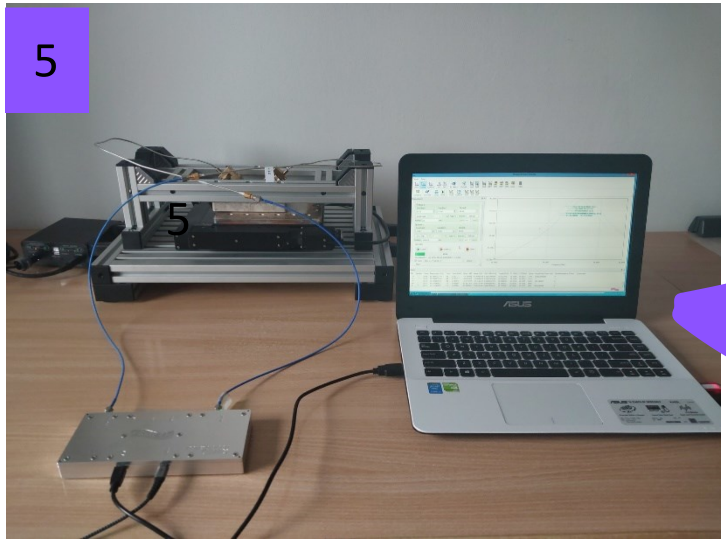
2. Measure thickness of the sample



3. Insert the sample into SPDR - by using a STANDA Move

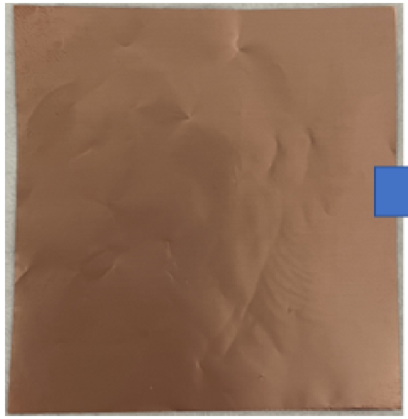
4. Insert the sample thickness into the PC app

5. Material parameters are extracted automatically with each step

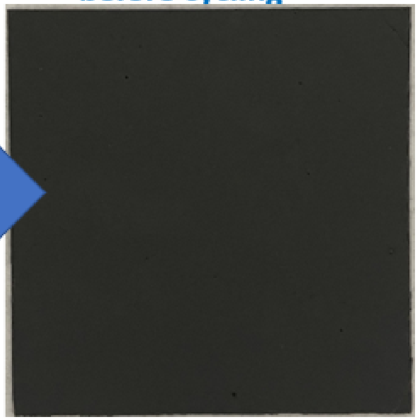


Example: 2D iSiPDR Imaging of Graphene Anodes

Copper electrode

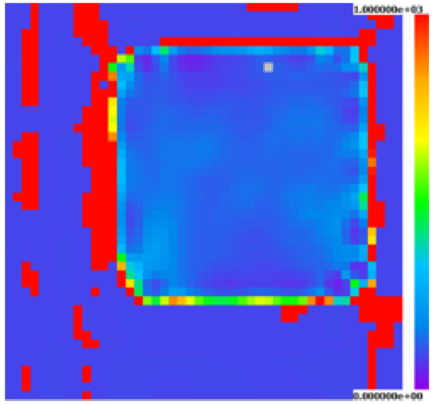


Graphene –based battery anode before cycling



Scanning range: 80 x 80 mm, scanning step: 2mm
Measurement points: 1681
Scanning time: ca. 2h

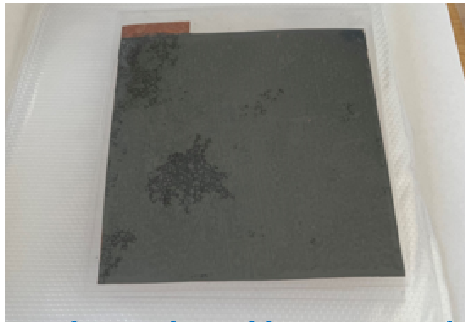
2D map of Rs [Ω/sq.]



values of Rs [Ω/sq.]

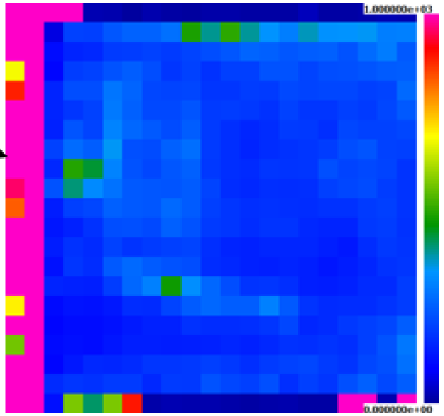
85 – 160 [Ω/sq.]

**courtesy PLEIONE Energy, Greece*



Graphene –based battery anode after battery cycling (ca. 100 cycles)

Sample edge (protecting foil)



110 – 340 [Ω/sq.]

increase indicates SEI formation

Conclusions

1ST SPACE MICROWAVE WEEK

1. Materials are key enablers - but may also be key inhibitors! – for emerging technologies, including space.
Therefore attention is required to their development and characterisation.
This is acknowledged in European initiatives (EMMC, AMI2030, Manifesto).

2. Efforts on material measurements for 5G and beyond, including:
- benchmarking of industrial best practices and emerging techniques,
- development of SRMs,
are increasing globally and coordinated e.g. by The International Electronics Manufacturing Inititive and NIST (both US).
Here, Europe seems to be lagging behind, and space sector seems underrepresented.

3. For low-loss materials, essential for space and satellite technologies, resonant methods are recommended and validated:
- instruments available from commercial vendors have been shown to deliver results in good consistency (1-2% in Dk), if applied following best practices and using samples of good thickness uniformity,
- some of the commercial instruments are more robust in use than other ones,
- non-standard instruments tend to be outliers.

4. New microwave and millimetre-wave resonators can be designed for testing specific materials, including novel electronic or energy materials, by exploring synergies with computational modelling.

5. Our team is open to new collaborations in the field!

Thank you for your attention.

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1ST SPACE MICROWAVE WEEK

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