



WMH-4

Emerging applications of microwave resonators to energy- and biobased-materials under extended temperature and humidity ranges

<u>Marzena Olszewska-Placha</u>, Malgorzata Celuch, Lukasz Nowicki and Janusz Rudnicki

QWED Sp. z o.o., Warsaw, Poland







Emerging applications of microwave resonators

1





Emerging applications



- Materials testing for 5G and beyond
 - Dielectric substrates
 - Conductor foils and pastes
- Light and power electronics
- Electronic and food liquids
- Energy materials





IMS 5G materials testing – where to start?



- New material for mmWave 5G applications careful consideration to determine best measurement methodology, fixture, sample fabrication and test instrument
- Dozens of different methodologies which to choose is often not obvious.
- International initiatives e.g. *International Electronics Manufacturing Initiative* (*iNEMI*)
- Associating standardization institutes, electronic industry, equipment vendors, materials producers, etc.
- Benchmarking existing materials characterisation methods
- Applicability for emerging 5G and 6G technologies
- Investigation of repeatability and reproducibility
- Known and industrial materials



iNEMI 5G Project Members



Project Team

- 3M ٠
- 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)

NIST

ITRI

CFT

MacDermid-Alpha

- Mosaic Microsystems ٠
- NIST ٠
- Nokia ٠
- Panasonic ٠
- QWED •
- Shengyi Technology Company ٠
- Sheldahl ٠
- Unimicron Technology Corp
- Zestron ٠



iNEMI 5G Project Team – **Spanning across Supply Chain**

IEEE MICROWAVE THEORY &

INEMIS FOR Project Working Group

WMH-4





3M, USA	Keysight, USA
Dupont, USA	Nokia, Finland
Intel, USA	NIS,: USA
ITEQ, Taiwan	Showa-Denko, Japan
ITRI, Taiwan	Shengyi Electric, China
QWED, Poland	

Map of available techniques vs Frequency





Benchmarking activities



INEMI 5G/mmWave Materials Assessment and Characterization project results

- Four resonant methods:
 - SPDR, SCR, BCDR, FPOR
- 3 material types
- 12 samples (6 in each of two sizes)
- •
- 10 samples kits
- 11 labs



WMH-4

• Following common thickness acquisition procedure





6



SCR





Split-Post Dielectric Resonators



Two dielectric posts

- □ Resonant mode with EM fields confined in and between posts
- □ E-field circumferential and tangential to SUT
- □ H-field is only vertical at the side wall of the enclosure
- □ Average of in-plane components of complex permittivity tensor







Field patterns remain practically unchanged but resonant frequencies and Q-factors change.

SUT of $\varepsilon_s = \varepsilon_s' - j \varepsilon_s''$ is inserted into DR:

resonant frequency changes from f_e to f_s and Q-factor changes from Q_e to Q_s .



Measurement procedure - SPDR



Connecting Minds. Exchanging Ideas.

Operation workflow – with the use of Q-Meter



General measurement scheme is common for most of resonant methods

Connecting Minds. Exchanging Ideas. Split-Post Dielectric Resonators (2) Measurement setups



Major properties of the SPDR method:

- **dielectric constant**: Dk = 1 100 (accuracy: $\delta Dk < 0.15\%$)
- **loss tangent**: Df > 2 10⁻⁵ (maximum achievable accuracy: δ Df < 2%)
- sample dimensions depend on the operating frequency
- temperature dependent measurements (standard up to 110 C deg)
- average of in-plane Dk and Df components

SPDR-compatible samples dimensions depending on the operating frequency

Nominal frequency [GHz]	Minimum sizes/diameter of sample (<i>dmin</i>) [mm]	Maximum width of sample (<i>dmax</i>) [mm]*^	Recommended sizes of sample (<i>drec</i>) [mm]**	Maximum thickness of sample [mm]
1.1	120x120 / 120	165	165	6.0
2.45 / 2.5	55x55 / 55	100	85	3.1
5 / 5.1	30x30 / 30	90	65	1.95
10	22x22 / 22	90	45	0.95
15	14x14 / 14	40	35	0.6

* Limitation for diameter of circular samples. For rectangular samples, limitation relates only to one sample dimension (the other dimension may be larger).

** For easy sample handling, diameter *d* of circular sample is recommended to be *drec<d<dmax*. For rectangular samples of size *a x b*, the recommendation is *a>drec* while *dmin<b<dmax*. ^ For non-standard sample sizes contact QWED team.



WMH-4



Laboratory-scale VNA







Portable Microwave Q-Meter

Fabry-Perot Open Resonator

Gaussian

10

Bridging the gap between classical resonant

methods and free space methods





Connecting Minds. Exchanging Ideas.

Major properties of the FPOR system:

- Single device for: 20-130 GHz
- Frequency resolution: ca. 1.5 GHz
- Dk accuracy: $\Delta \varepsilon / \varepsilon < 0.5 \%$
- Df range: $10^{-5} < tan \delta < 10^{-2}$
- Sample diameter: > 3 inches
- Sample thickness: < 2 mm
- Fully automated measurement: typically 3 minutes in 20-130GHz

WMH-4



Gaussian **TEM00q** modes



Electric field distribution - simulation model in QuickWave software

Fabry-Perot Open Resonator (2)



FPOR measurement setup

Advantages:

- broadband and accurate characterization of dielectric sheets in a single fixture
- high repeatability

Connecting Minds. Exchanging Ideas.

- software-controlled and fully automated
- easy insertion of the sample
- non-destructive measurement

*for more details, see: https://www.qwed.eu/resonators fpor.html





Anisotropic dielectric materials: X-cut quartz; th=492 \pm 1 μ m







Benchmarking results



INEMI 5G/mmWave Materials Assessment and Characterization project results

Repeatability studies



Each measurement repeated 16 times

Repeatability bounds: $\pm 3\sigma$

12

M. Celuch, M.J. Hill, T. Karpisz, M. Olszewska-Placha, S. Phommakesone, U. Ray, B. Salski, "Benchmarking of GHz resonator techniques for the characterisation of 5G/mmWave materials", 51st European Microwave Conference April 2021, pp. 568-571.





Benchmarking results



INEMI 5G/mmWave Materials Assessment and Characterization project results

Over 1000 measurement points in total



Round 1

Spread of Dk: $\pm 2\%$

13

WMH-4

EEE MICROWAVE THEORY &



Benchmarking results







Observations and conclusions



- Two sample size sufficient to cover all considered test methods
- Accurate thickness evaluation is of high importance
- Thickness variation and sample flatness determine uncertainty of Dk extraction
- Results variation across the labs of $\pm 2\%$
- Standard reference materials are of high interest

WMH-4

• Traceable standard – dielectric parameters certified by NIST

- Calibration of 5G & 6G material characterization fixtures
- Targeting 0.2% uncertainty in SRM in round robin results



New activities



- mmWave Permittivity Reference Material Development project
- Developing traceability path for material characterisation methods (NIST)
- Certification of SRMs (NIST)
- Support with Round Robin testing of SRM candidates (8 labs)

• 5G/6G mmWave Materials and Electrical Test Technology Roadmap (5G/6G MAESTRO) project

16



SRM candidate testing



INEMI *mmWave Permittivity Reference Material Development* project



In-plane measurements of RR1

Agreement within 1.6%

WMH-4

Industry Workshop IWWE6 mmWave Permittivity Standard **Reference Material Development**

> Wednesday 1:30 pm Location: 29D





New materials for 5G technologies









Materials for 5G and beyond



- LTCC and ULTCC materials for electronic substrates
 - LTCC sintering temperature: ca. 800 $900^{\circ}C$
 - ULTCC sintering temperature: ca. 400-700 $^{\circ}\mathrm{C}$
- Lower energy consumption
- Precise characterisation needed
- Point-wise & surface-wise (important for high density packaging at single substrate)

 Direct conductor testing – evaluation and optimization on the signal loss in final devices

19





LTCC/ULTCC fabrication scheme



LTCC/ULTCC substrate fabrication scheme



Composites characterisation before substrate fabrication





21

🗾 Fraunhofer









- □ Sample under test serves as dielectric resonator
- \Box Df as low as 5 10⁻⁷
- Accuracy:

Connecting Minds. Exchanging Ideas.

- $\Delta \epsilon / \epsilon = T^* \Delta h / h + (2 T)^* \Delta d / d$, for 0 < T < 2 $\Delta \tan \delta = \pm 2 \times 10^{-6} \text{ or } \pm 0.03 \times \tan \delta$
- **Precisely machined samples**
- Sample: 0.25 0.6 of the cavity size
- \Box Family of TE01 δ cavities:
 - D= 12mm (ca. 38GHz), D= 18mm (ca. 23GHz), D= 24mm (ca. 19GHz), D= 32mm (ca. 15GHz), D = 60 mm (ca. 7.5 GHz)



WMH-4





22









Temperature coefficient of resonant frequency (TCF)





ULTCC bulk composites (2)



TCF of ULTCC with TE01 δ cavity

Measurement setup

VNA



PC with control app

Climatic chamber with 32mm TE01 δ cavity

ULTCC bulk composite samples





 $D{\approx}\,8mm$, $H{\approx}\,4mm$



ULTCC bulk composites (3)



Temperature and humidity dependent measurements

ULTCC6G_EPac project activities

	Sample	Dk	Df	Humidity [%]	TC Dk [ppm] (25→85°C)	TCF [ppm] (25→85°C)
ULTCC_C3 ULTCC_C2	ULTCC_C1 Sample 1	5.80	0.000288	35	114	-58.6
6.05	ULTCC_C1 Sample 2	5.84	0.000296	35	120	-61.2
6	ULTCC_C1 Sample 3	5.959	0.000359	35	114	-58.6
5.95	ULTCC_C1 Sample 3	5.96	0.000592	65		
5.9 ULTCC_C1 Sample 2 ULTCC_C1 Sample 1	ULTCC_C2 Sample 1	4.05	0.004945	35	213	-106
5.83	ULTCC_C2 Sample 2	3.96	0.005534	35	210	-104
5.75	ULTCC_C3 Sample 1	13.91	0.002227	35	64.7	-34.6
Temperature [deg C]	ULTCC_C3 Sample 2	14.08	0.002201	35	63.9	-34.3

24



ULTCC bulk composites (3)



Temperature and humidity dependent measurements

ULTCC6G_EPac project activities

	Sample	Dk	Df	Humidity [%]	TC Dk [ppm] (25→85°C)	TCF [ppm] (25→85°C)
ULTCC_C3 ULTCC_C2	ULTCC_C1 Sample 1	5.80	0.000288	35	114	-58.6
6.05	ULTCC_C1 Sample 2	5.84	0.000296	35	120	-61.2
6	ULTCC_C1 Sample 3	5.959	0.000359	35	114	-58.6
5.95	ULTCC_C1 Sample 3	5.96	0.000592	65		
5.9	ULTCC_C2 Sample 1	4.05	0.004945	35	213	-106
5.83	ULTCC_C2 Sample 2	3.96	0.005534	35	210	-104
5.75	ULTCC_C3 Sample 1	13.91	0.002227	35	64.7	-34.6
Temperature [deg C]	ULTCC_C3 Sample 2	14.08	0.002201	35	63.9	-34.3

25

LTCC and ULTCC substrates (1)

Ultra-Low Temperature Co-fired Ceramics for 6th Generation Electronic Packaging (ULTCC6G_EPac) project



WMH-4

Consistent within uncertainty bounds – related to thickness variation



• Sample 1 FPOR Sample 2 SPDR • Sample 3 SPDR

Family of SPDR fixtures



26



Connecting Minds. Exchanging Ideas.

LTCC and ULTCC substrates (2)





Thickness variation – major issue for Dk uncertainty

WMH-4



Connecting Minds. Exchanging Ideas.



Family of SPDR fixtures



LTCC and ULTCC substrates (2)



ULTCC6G_EPac project activities



SPDR 10GHz measurement setup

PARAMETERS OF ULTCC MATERIALS MEASURED WITH 10 GHz SPDR

Sample name	Thickness [mm]	Dielectric constant	Loss tangent
Li_2WO_4 with 4 wt.% CuBi_2O_4	0.485 ± 0.015	5.40 ± 2.5%	0.00200 ± 3%
Li_2WO_4 with 4 wt.% AIF ₃ CaB ₄ O ₇	0.64 ± 0.030	6.52 ± 4%	0.00233 ± 3%
$LiBO_2$ with 4 wt.% $CuBi_2O_4$	0.6 ± 0.030	5.12 ± 4%	0.00195 ± 3%
$LiBO_2$ with 4 wt.% AIF ₃ -CaB ₄ O ₇	0.55 ± 0.020	4.48 ± 3%	0.00328 ± 3%

19 szewska-Placha, D. Szwagier czak, B. W. Swinkiewicz-Musialska, J. Rudnicki, J. Mularik, "Az Characterisation of dielectric properties of ultra 208 temperature co-fired ceramic materials for 6G systems

Connecting Minds. Exchanging Ideas.

TC for relative permittivity of LTCC and ULTCC with SPDRs



Control PC with MMS app

Handheld VNA

Climatic chamber with 10GHz SPDR

29

Up to 110 deg C in standard SPDRs configuration

Temperature dependent measurements with FPOR under development



Connecting Minds. Exchanging Ideas. Temperature dependent measurements

Temperature and humidity dependent measurements ULTCC6G_EPac project activities



Sample	Method	Dk	Df	Humidity [%]	Temperature coefficient Dk [ppm] (25→85°C)
LTCC Sample 1	SPDR 10GHz	5.61	0.000995	35	120
LTCC Sample 2	SPDR 10GHz	5.62	0.000110	35	91
ULTCC Sample 1	SPDR 10GHz	10.02	0.02673	35	711
ULTCC Sample 1	SPDR 15GHz	9.88	0.03038	35	515
ULTCC Sample 2	SPDR 15GHz	9.91	0.02136	35	494
ULTCC_c Sample 1	SPDR 15GHz	5.43	0.00160	35	170
ULTCC_c Sample 1	SPDR 15GHz	5.32	0.00155	5	300

30

LTCC Sample 1



ULTCC ULTCC_c Sample 1 Sample 1

	Sample 1						
	S. Martin						
all here							
		1					

Connecting Minds. Exchanging Ideas. Temperature dependent measurements

Temperature and humidity dependent measurements ULTCC6G_EPac project activities



Sample	Method	Dk	Df	Humidity [%]	Temperature coefficient Dk [ppm] (25→85°C)
LTCC Sample 1	SPDR 10GHz	5.61	0.000995	35	120
LTCC Sample 2	SPDR 10GHz	5.62	0.000110	35	91
ULTCC Sample 1	SPDR 10GHz	10.02	0.02673	35	711
ULTCC Sample 1	SPDR 15GHz	9.88	0.03038	35	515
ULTCC Sample 2	SPDR 15GHz	9.91	0.02136	35	494
ULTCC_c Sample 1	SPDR 15GHz	5.43	0.00160	35	170
ULTCC_c Sample 1	SPDR 15GHz	5.32	0.00155	5	300

31

LTCC Sample 1



ULTCC ULTCC_c Sample 1 Sample 1



Connecting Minds. Exchanging Ideas. 2D imaging of material parameters (1)

WMH-4

2D SPDR scanner @ 10GHz



□ 2D SPDR-based scanner

10GHz for higher spatial resolution
 For low-loss dielectrics and high-resistivity semiconductors
 Maximum sample size: 80 x 120 mm
 2D maps of Dk, Df, and resistivity



2D surface map of dielectric constant of quartz





Connecting Minds. Exchanging Ideas. 2D imaging of material parameters (2)



Surface-wise measurements of LTCC and ULTCC materials

ULTCC6G_EPac project

WMH-4





33

Dk nonuniformities related to thickness variation











Conductor testing



- □ Industry need for testing and standards
- □ iNEMI *Reliability & Loss Properties of Copper Foils for 5G Applications* project
- □ Investigation of surface roughness
- □ Surface resistance and effective conductivity
- □ Sapphire Dielectric Resonators (SaDR)
- □ SaDR dual mode device (e.g. 13.5 GHz and 20 GHz)
- Fabry-Perot Open Resonator prototype within 20 50 GHz
- **Accuracy** \pm 2%
- Testing methods in iNEMI project

*based on the setup proposed for solid metals in the IMS 2020 paper by J.Cuper et al.: doi: 10.1109/IMS30576.2020.9224069 validated but under development for metal form

WMH-4



200 um copper foil @ 13.5 GHz: *9 um copper foil* @ 13.5 GHz:

34

 $\begin{array}{l} \sigma {=} 5.45 \cdot {10^7} \ \text{S/m} \\ \sigma {=} 4.64 \cdot {10^7} \ \text{S/m} \end{array}$

Fabry-Perot Open Resonator





Conductor testing (2)



Conceptual scheme of SaDR



Copper foil samples



	Expected Surface	Rs at	Rs at	Rank at	Rank at
Foil name	roughness -rank	13.8GHz	20.4GHz	13.8GHz	20.4GHz
Foil 1 –					
base foil	4	32,108	41,515	4	3
Foil 2	7	34,677	46,407	7	7
Foil 3	3	32,131	41,925	5	5
Foil 4	3	32,247	41,946	5	5
Foil 5	8	46,979	67,165	8	8
Foil 6	2	31,949	41,494	2	2
Foil 7	5	32,013	41,832	3	4
Foil 8	1	31,683	41,186	1	1

35

SaDR testing results of copper foils

 $\delta Rs = 1.5\% + \delta Q$

- VNA settings
- vibrations





Light and power electronics







IMS 2D imaging of material parameters (1) Connecting Minds. Exchanging Ideas.

2D SPDR scanner @ 10GHz





□ 2D SPDR-based scanner □ 10GHz for higher spatial resolution • For low-loss dielectrics and high-resistivity semiconductors □ Maximum sample size: 80 x 120 mm **2**D maps of Dk, Df, and resistivity

- High densification of semiconductor devices over a single wafer
- Surface-wise measurements of material parameters
 - Substrate

WMH-4

Final semiconductor structure/device

37

Repeatability of device parameters (e.g. LED diode)



Light and power electronics



- Quantitative and qualitative measures → usability of material
- Scanning area: 70 mm x 70 mm
- Uniform scanning step: 1 mm
- 5112 measurement points
- HR-GaN template:
 - edge ring inherent to so-called edge effect
 - ca. $2 \cdot 10^4 \Omega cm$ in the centre (dark blue),
 - ca. $5 \cdot 10^4 \Omega cm$ along the inner ring (light blue)
 - up to 1.2 3 ·10⁵ Ωcm across outer SUT's area (bluegreen).

WMH-4

Higher range for resistivity is needed Device needs to be enhanced towards that requirement

M-ERA.NET3 I4BAGS project *samples by L-IMP, Poland

2D surface map of resistivity of semiconductor wafers [Ohm cm]







Electronic and food liquids









Measurements of liquids (1)



- Dielectric characterization of "loose" materials
 - Liquids
 - Powders
- Electronic coolants
- Food industry
- Raw materials producers
- Characterization with resonant methods
 - Cavity devices
 - Fabry-Perot Open Resonator







Measurements of liquids (2)



Resonant methods are proven to be the most accurate among microwave material characterisation methods



Higher frequency cavity resonators



Fabry-Perot Open Resonator





Dielectric resonator cavity at 1 GHz

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

41

Single solution for 20-50GHz





- Electric field mostly confined within the dielectric pill
- Circumferential electric field
 - \rightarrow no issues with galvanic connection of the lid
- Zero electric field at ρ =0

Connecting Minds. Exchanging Ideas.

 \rightarrow no risk of supressing resonance if lossy sample is inserted





Vector view * Obtained with QuickWave 3D software



WMH-4









Measurement methods (2)



- Two/three stage measurement
- Reference measurement cavity with empty container (*f*_{ref} and *Q*_{ref})
 → the inner diameter of the container needs to be precisely calibrated
- Measurement of sample-loaded cavity (*f_s* and *Q_s*)
- Scalar measurement of transmission curve (|S21|) is typically sufficient





Measurements of liquids (3)



Measurement results for a well-known electronics coolant liquid obtained with all resonant methods



Connecting Minds. Exchanging Ideas. Measurements versus temperature



Dielectric characterization versus temperature



Connecting Minds. Exchanging Ideas. Measurements versus temperature (2)

The Control Marco Under Che Reve The Control Marco Under Che Reve SAN DIEGO2023

Dielectric characterization versus temperature

coolant liquid and canola oil





Uncertainty of Dk due to variation of diameter of quartz tube @2.5 GHz - 0.1% @24GHz - 0.7%

46







Energy materials









Energy materials



- Efficient energy systems
- Well-controllable energy devices and systems
- Electrical characterization

• Semiconductor materials for photovoltaic cells (SPDR scanner)

48

• Battery testing – anodes for battery cells

• 2D imaging for qualitative and quantitative measures of parameters uniformity

Connecting Minds. Exchanging Ideas.





WMH-4





- Designed and developed for anode materials on semi-insulating substrates
- Adopted to actual anode samples anode on copper
 - Pre-cycling testing straightforward
 Post-cycling testing battery disassembly needed, protection required
- □ High control of fabrication process needed to get meaningful results for SEI presence



Substrate glass material and sample under test



2D map of Rs [Ω /sq.]



Connecting Minds. Exchanging Ideas.









M IMS

Connecting Minds. Exchanging Ideas.

2D map of Rs [Ω /sq.] Graphene-based battery anode before cycling



*samples courtesy PLEIONE Energy, Greece

Rs=85 – 160 [Ω/sq.]

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

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





Summary



- Resonant methods have wide spectrum of applications
- Existing methods are applied to new materials
- Enhancements are needed to extend material parameters range
- New dedicated resonant methods are developed depending on material type

52

• Specific challenges appear for each material type





Acknowledgement





The original designs of QWED resonators for material measurements from Prof. Jerzy Krupka, e.g.:

[1] J. Krupka, A. P. Gregory, O. C. Rochard, R. N. Clarke, B. Riddle, and J. Baker-Jarvis, "Uncertainty of complex permittivity measurements by split-post dielectric resonator technique", J. Eur. Ceramic Soc., vol. 21, pp. 2673-2676, 2001.

[2] J. Krupka and J. Mazierska, "Contactless measurements of resistivity of semiconductor wafers employing single-post and split-post dielectric-resonator techniques," IEEE Trans. Instr. Meas., vol. 56, no. 5, pp. 1839-1844, Oct. 2007.

With contribution from:

Prof. Bartlomiej Salski, Warsaw University of Technology Dr. Adam Pacewicz, Warsaw University of Technology

53

Dr. Pawel Kopyt, Warsaw University of Technology

Piotr Czekala, Warsaw University of Technology

The work presented has received funding from the

European Union's Horizon 2020 research and innovation programme













Thank you for the attention





