

# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

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**Tymoteusz Ciuk, PhD**

Łukasiewicz Research Network



**Institute of Microelectronics and Photonics, Warsaw, Poland**



**I4BAGS Webinar**

**January 10, 2023**

# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

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Thermal stability of transport properties

Two-dimensional character   **Why graphene?**   Relatively high charge carriers mobility

Well-defined charge carriers concentration

# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

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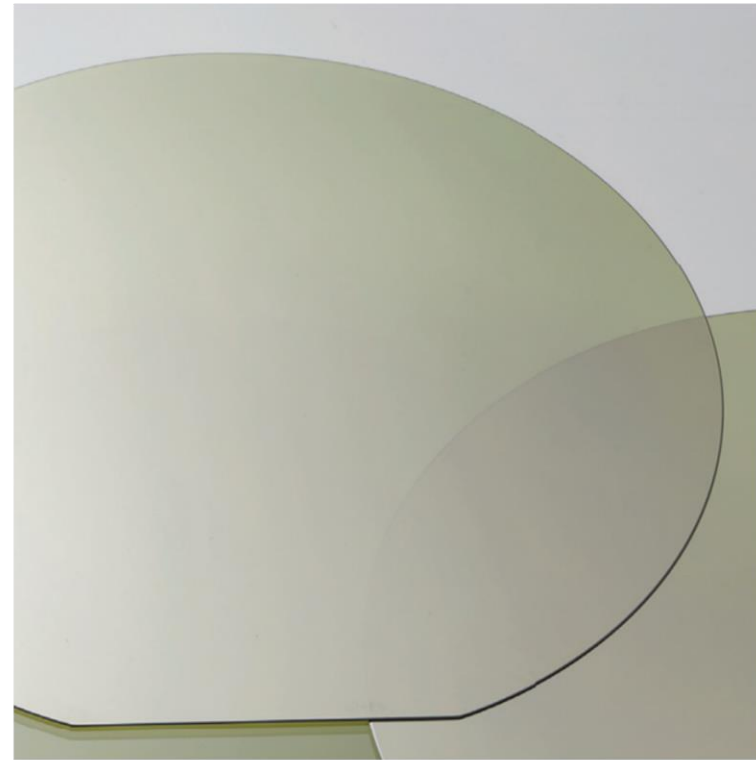
**Epitaxy:** Chemical Vapor Deposition (CVD)

**Carbon source:** methane or propane

**Substrate:** 4H-SiC(0001) or 6H-SiC(0001)

**Type:** semi-insulating on-axis

**Dimensions:** 20 mm x 20 mm



[dx.doi.org/10.1016/j.carbon.2015.06.032](https://doi.org/10.1016/j.carbon.2015.06.032) [dx.doi.org/10.1016/j.carbon.2016.01.093](https://doi.org/10.1016/j.carbon.2016.01.093)

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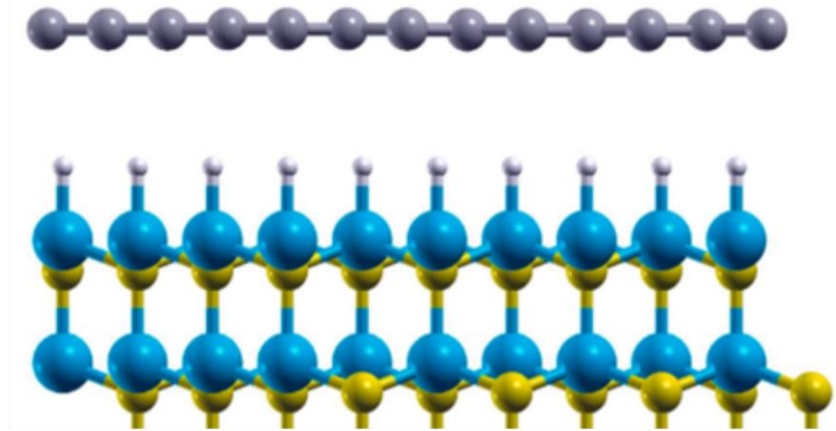
# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

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Hydrogen intercalation: quasi-free-standing graphene

On 4H-SiC(0001):  $\rho = 1.2 \text{ E}13 \text{ cm}^{-2}$

On 6H-SiC(0001):  $\rho = 7.5 \text{ E}12 \text{ cm}^{-2}$



[doi.org/10.1016/j.apsusc.2020.148668](https://doi.org/10.1016/j.apsusc.2020.148668)

# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

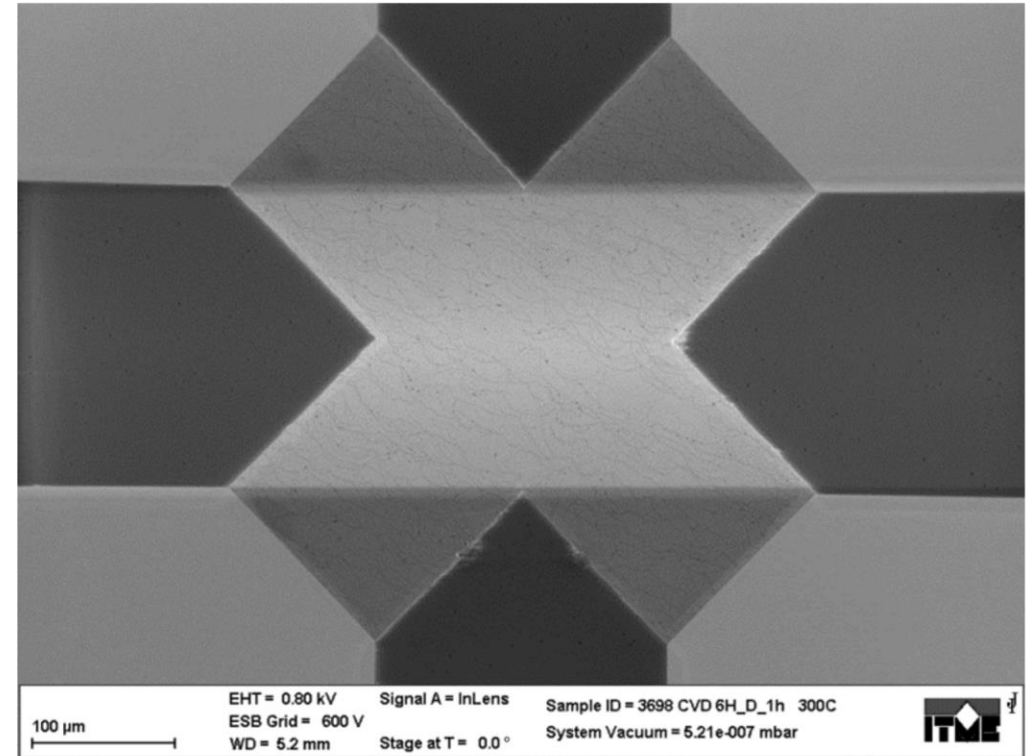
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Principle of operation: classical Hall effect

Configuration: van der Pauw

Active area: equal-arm cross 100  $\mu\text{m}$  x 300  $\mu\text{m}$

Total dimensions: 1.4 mm x 1.4 mm



[doi.org/10.1016/j.carbon.2018.07.049](https://doi.org/10.1016/j.carbon.2018.07.049)

# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

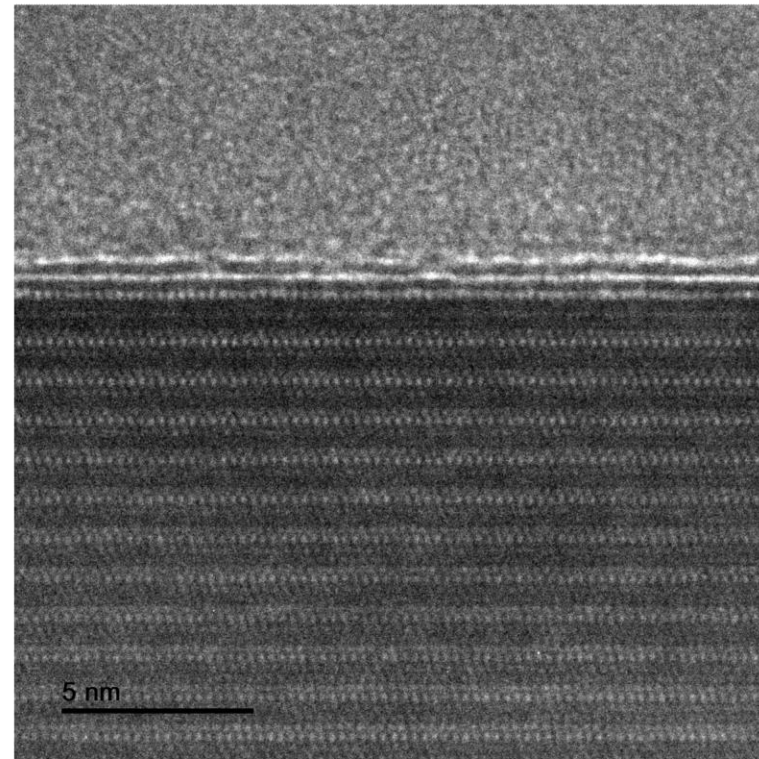
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**Passivation:** aluminum oxide

**Process:** atomic layer deposition

**Precursors:** TMA and DI

**Purpose:** environmental protection



[doi.org/10.1016/j.physe.2022.115264](https://doi.org/10.1016/j.physe.2022.115264)

# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

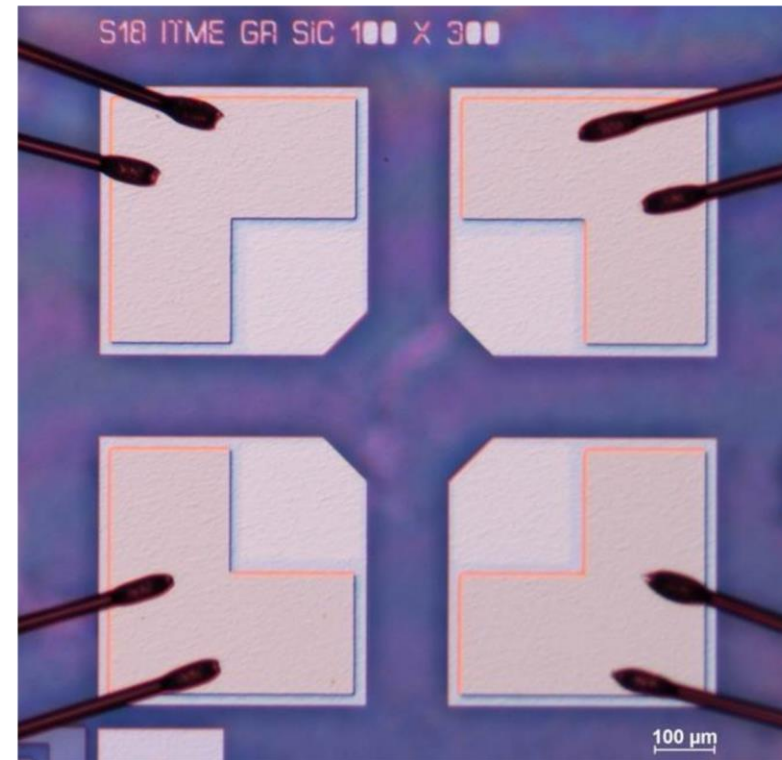
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**Mounting:** custom holders or ceramic packages

**Feed current:** < 10 mA

**Magnetic induction:** 0.55 T

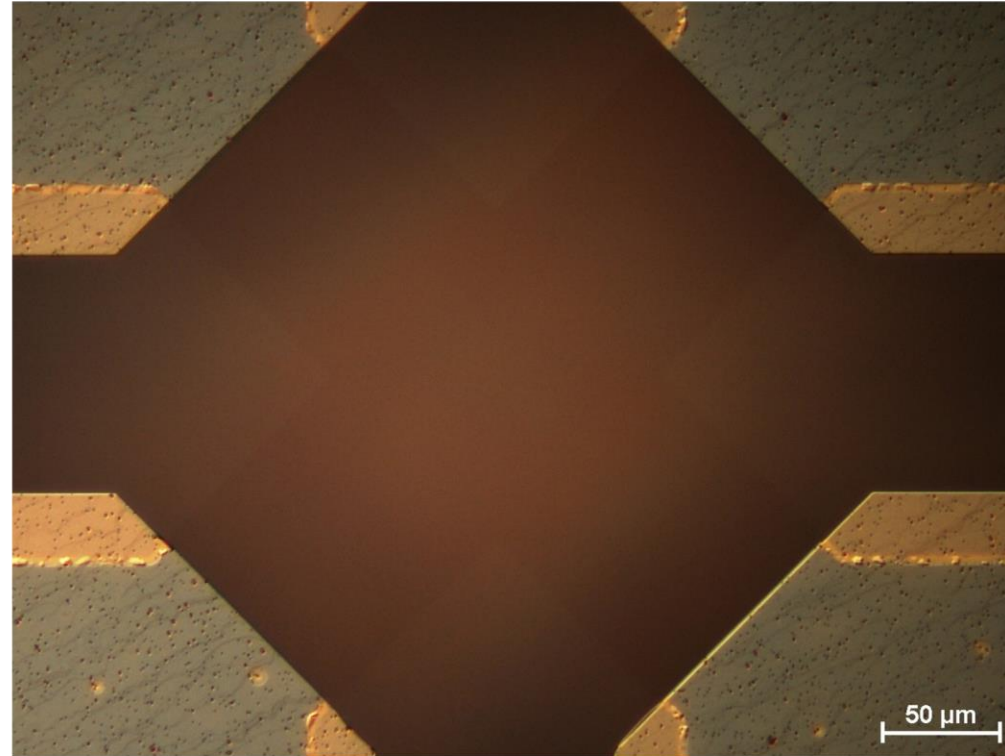
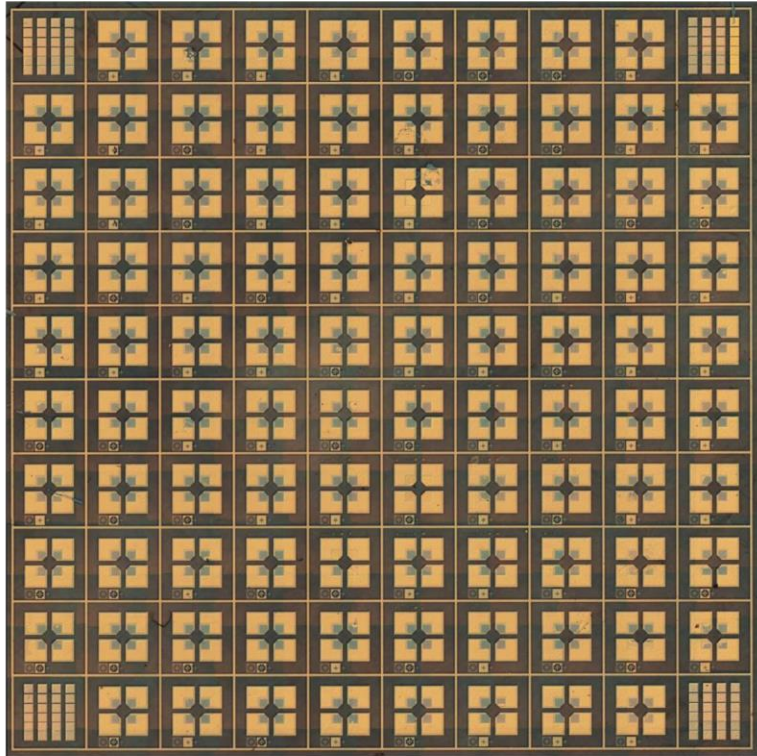
**Temperatures:** from liquid nitrogen to 500 °C



10.1109/TED.2019.2915632

# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

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[doi.org/10.1016/j.physe.2021.114853](https://doi.org/10.1016/j.physe.2021.114853)



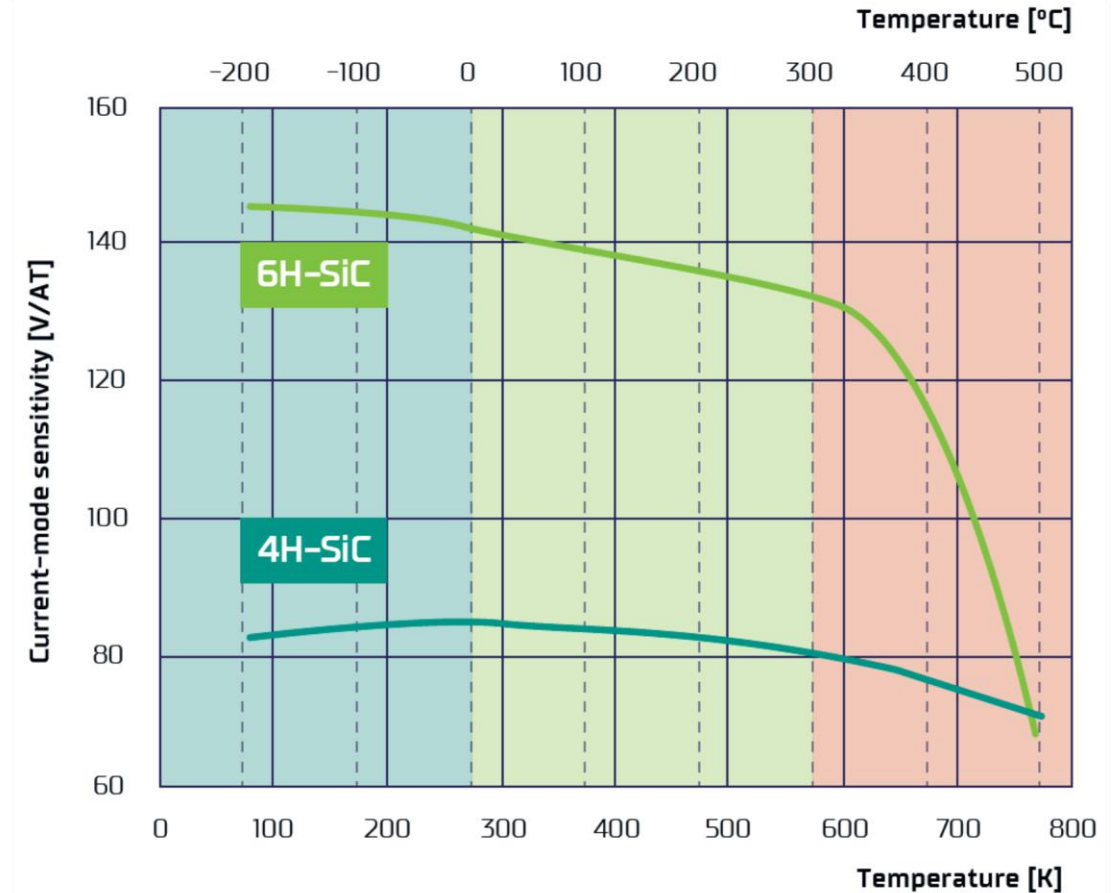
# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

Two platforms: 6H-SiC and 4H-SiC

Two levels of sensitivity: 140 V/AT, 80 V/AT

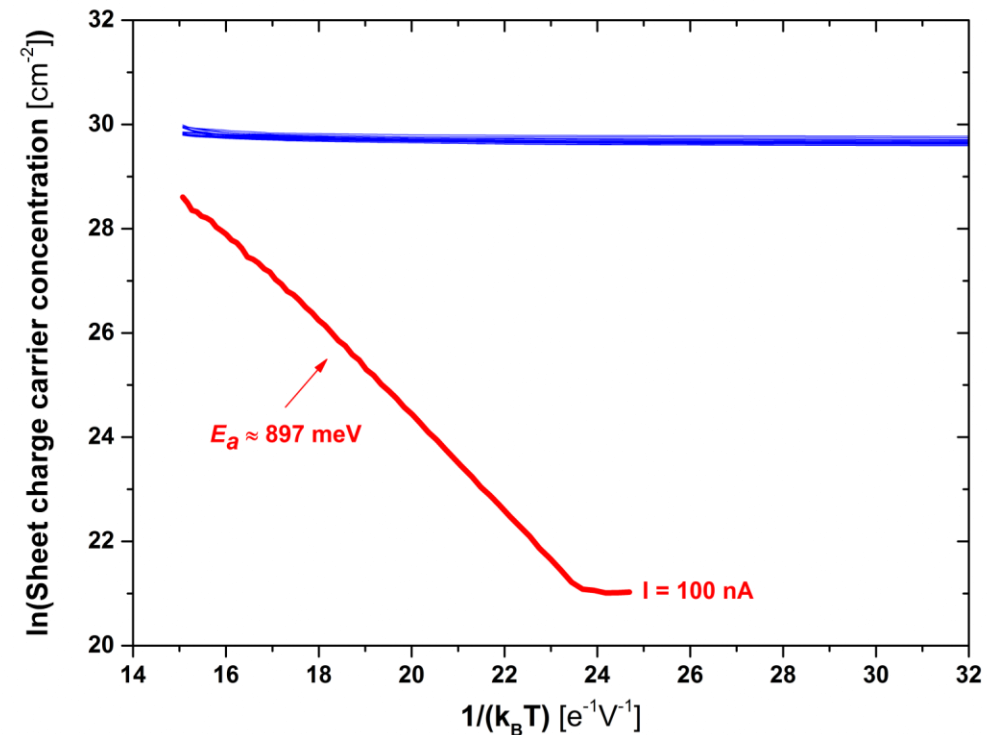
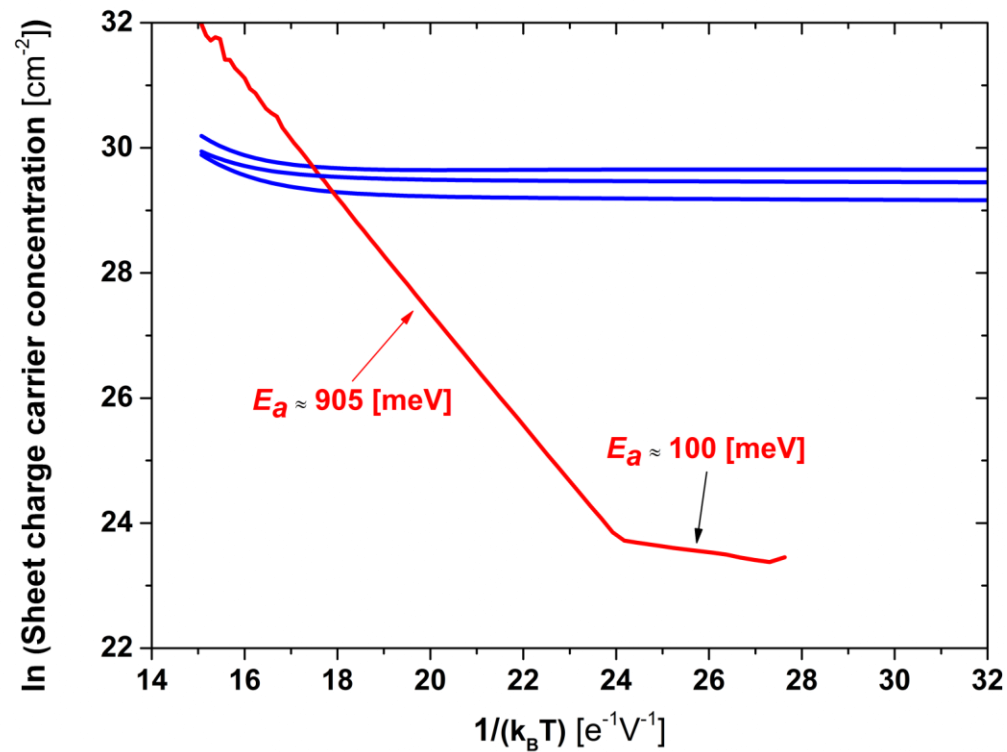
Start temperature: liquid nitrogen

End temperature: 500 °C



# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

Double-carrier transport: holes in QFS graphene and thermally-activated electrons emitted in the bulk of the semi-insulating 6H-SiC(0001) and 4H-SiC(0001)



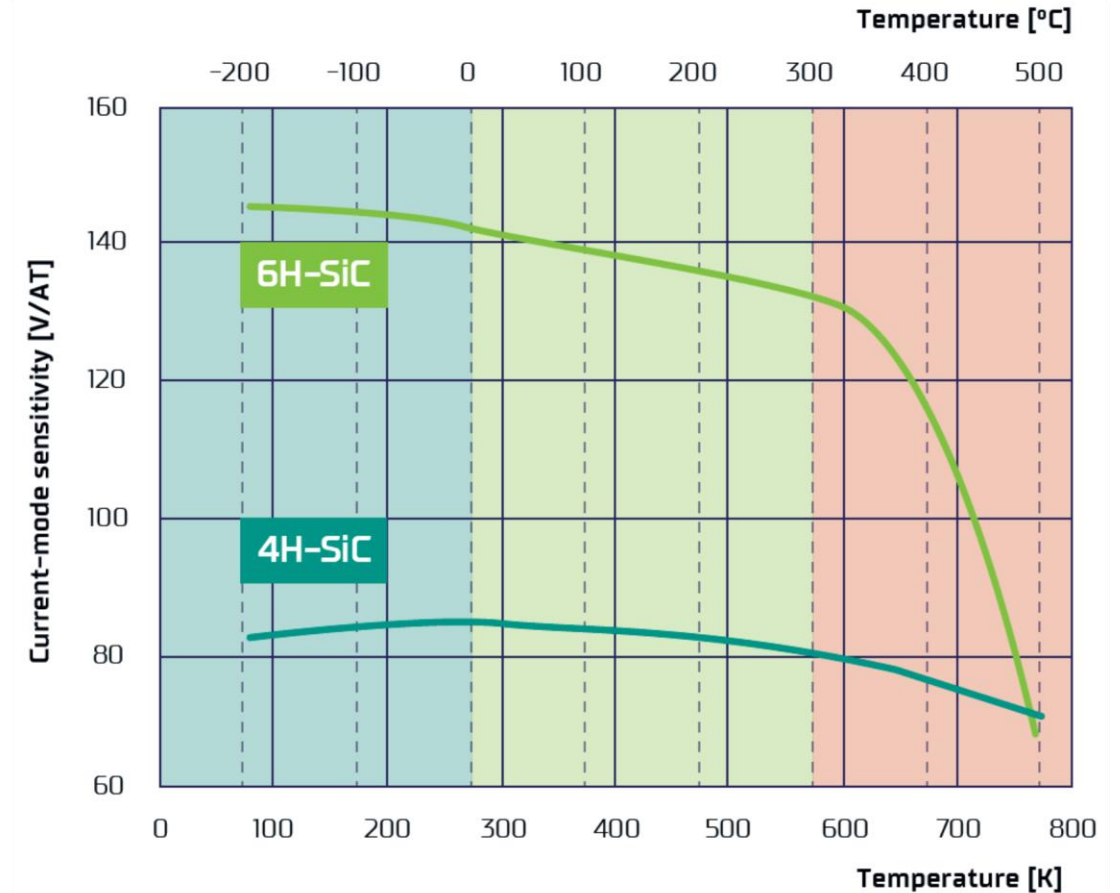
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# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

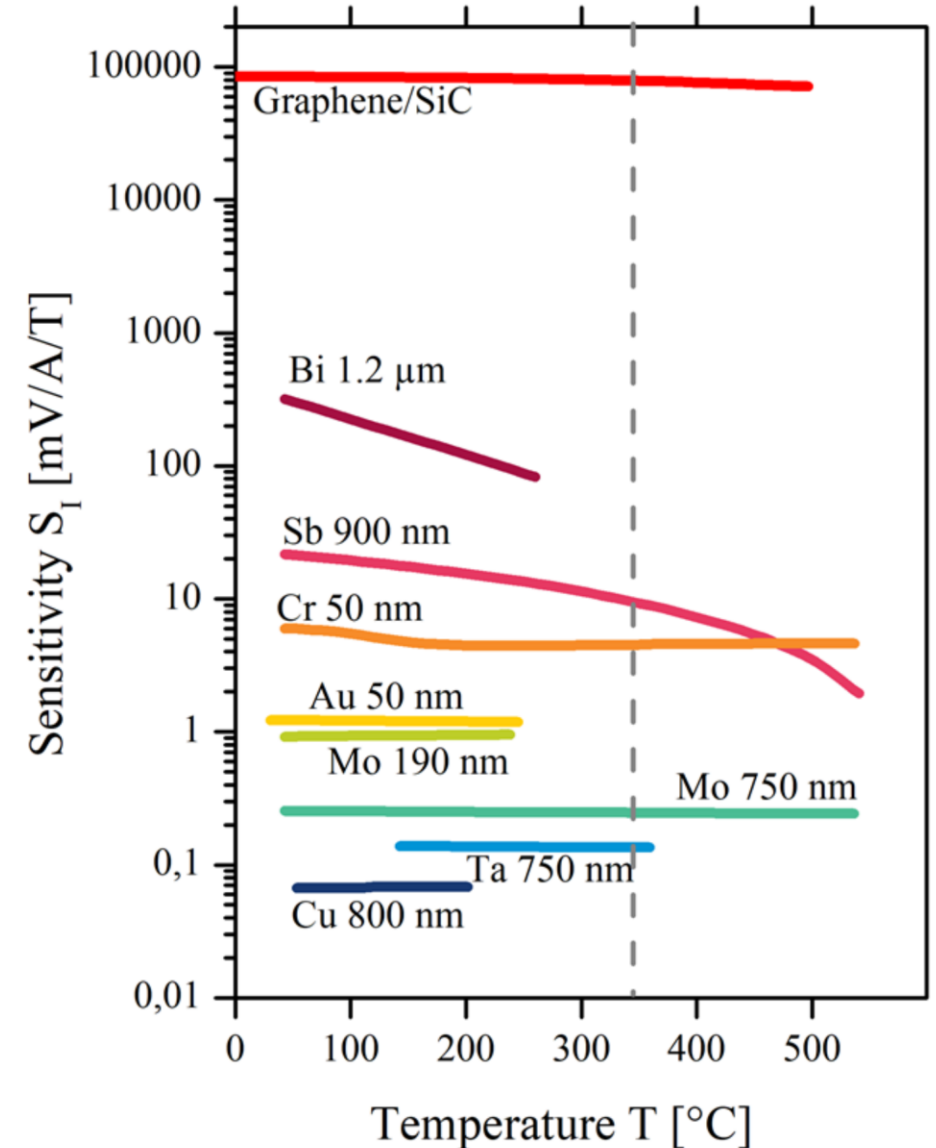
Alternative platforms: Bi, Sb, Cr, Au, Mo, Ta, Cu

Sensitivities: 0.1 mV/AT - 100 mV/AT

Start temperature: 50 °C

End temperature: 500 °C

Source: Entler S., et al., Sensors 2021, 21, 721.

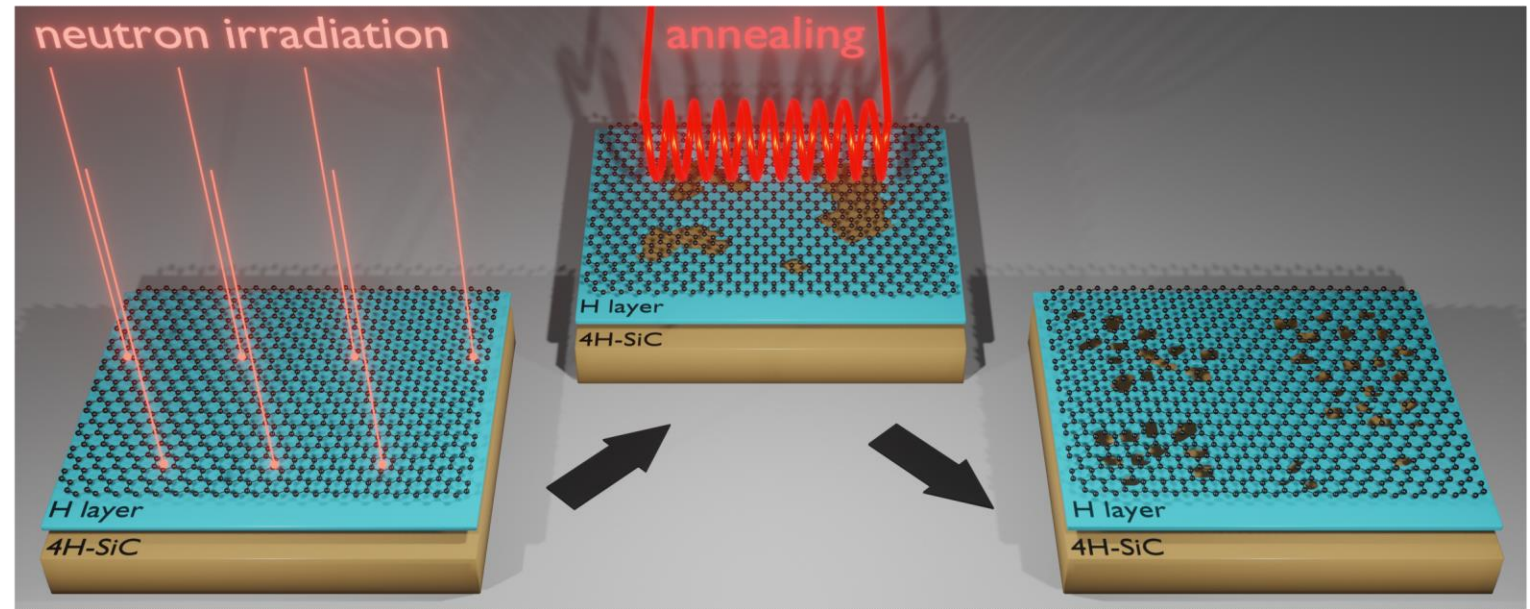
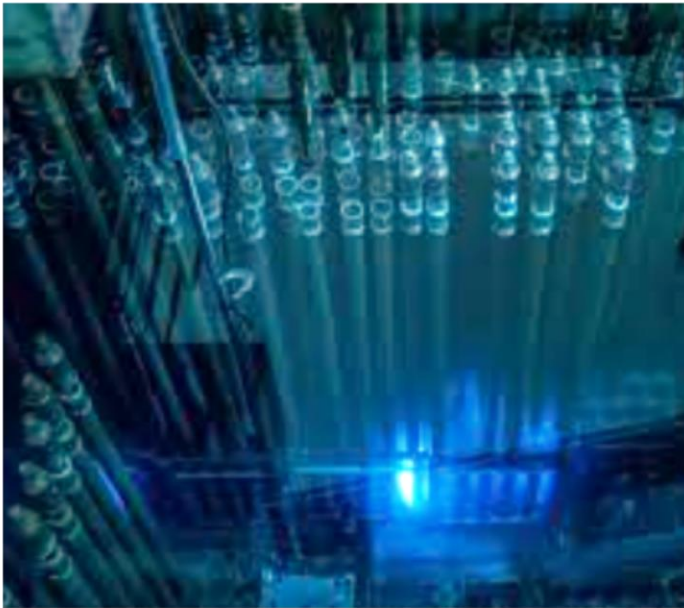


# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

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Completed experiment in MARIA reactor: neutron fluence of  $6.7 \times 10^{17} \text{ cm}^{-2}$

Estimated defect density:  $4 \times 10^{10} \text{ cm}^{-2}$  (low cross-section)



[doi.org/10.1016/j.apsusc.2022.152992](https://doi.org/10.1016/j.apsusc.2022.152992)

[doi.org/10.3390/s22145258](https://doi.org/10.3390/s22145258)

# Neutron-irradiation-resistant high-temperature graphene Hall effect sensor for advanced magnetic diagnostics

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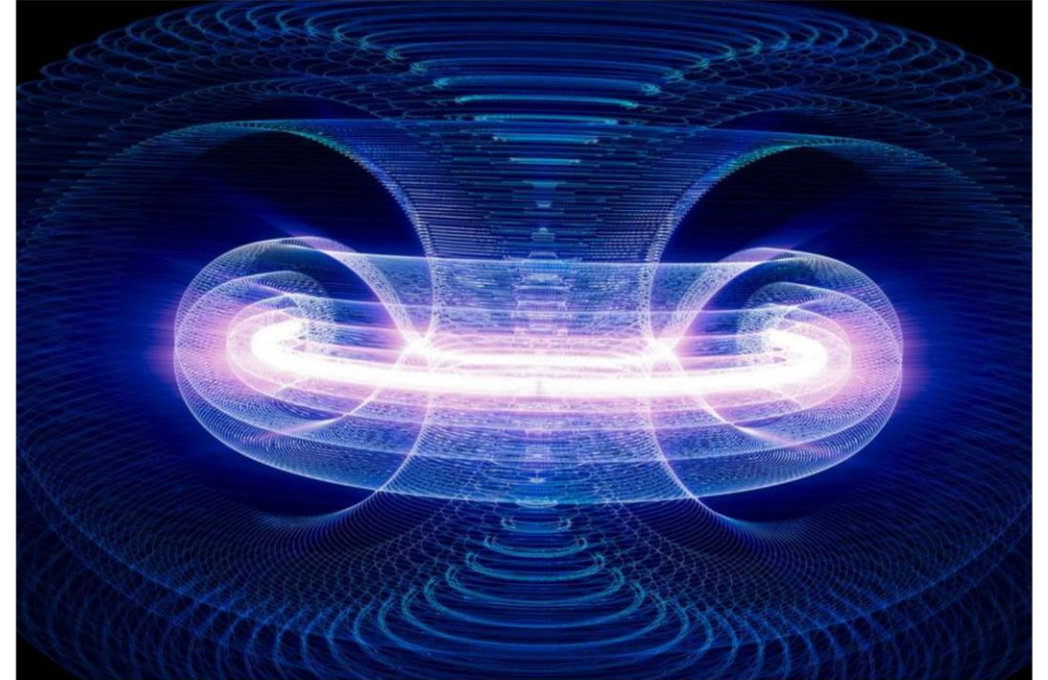
**Summary:** Al<sub>2</sub>O<sub>3</sub>/QFS-graphene/SiC(0001)

**Competitive advantages:**

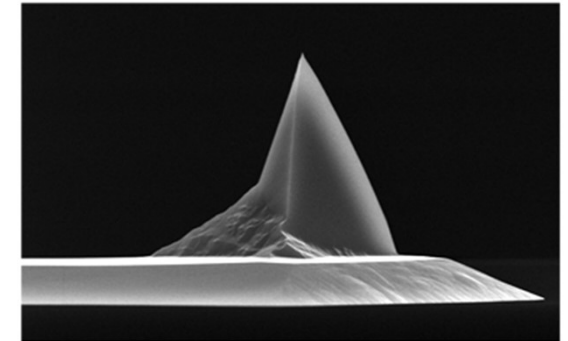
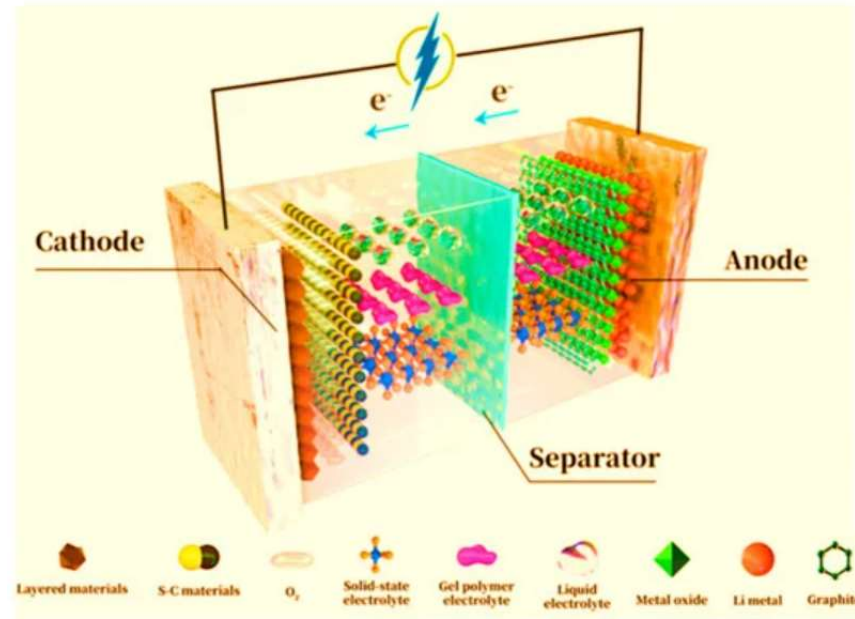
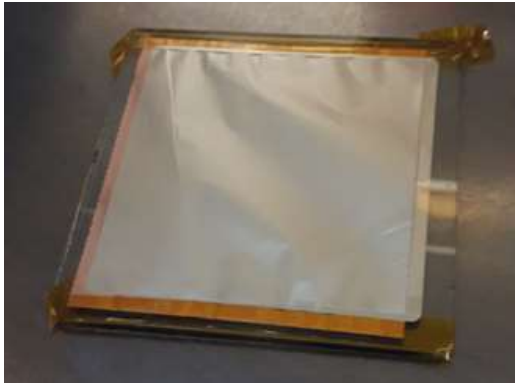
- operates at elevated temperatures
- largely resistant to neutron irradiation

**Potential application:**

- **magnetic diagnostics in fusion reactors**



# I4BAGS – Technical kick off meeting January 10<sup>th</sup> 2023



## Ion implantation for monitoring material properties in thin film solid state battery

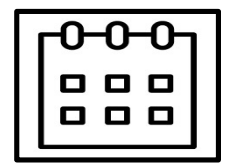
# Outline

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- Presentation of Materia Nova
- Battery development
- Action Plans
- Equipment
- Collaborative activities



# Presentation of Materia Nova



20 years



Turn over: 8 Million €



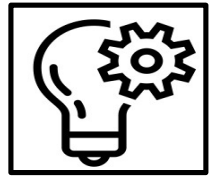
80 Experts  
(with UMONS -285 Experts)



Equipments  
(> 300 analytical measurements)



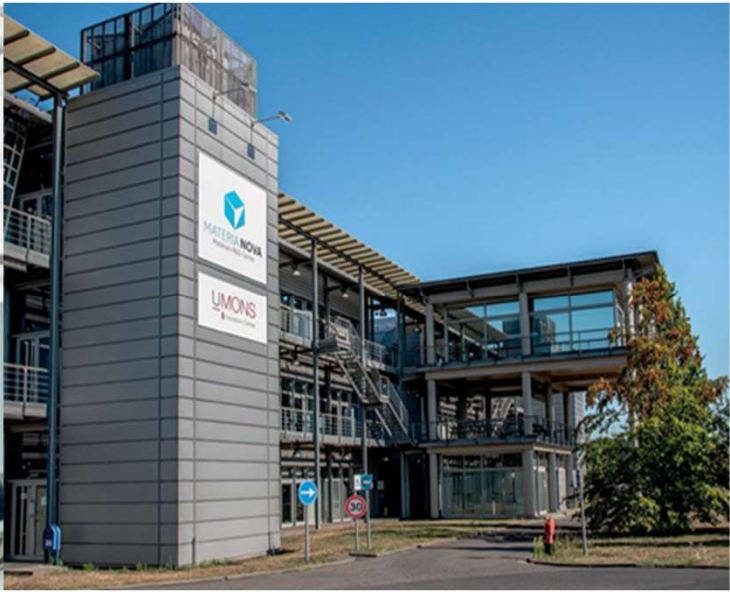
International network



Projects  
Services  
Patents



THE TECHNOLOGICAL  
ACCELERATOR OF  
RESPONSIBLE INNOVATIONS  
IN MATERIALS AND  
PROCESSES



THE TECHNOLOGICAL ACCELERATOR OF RESPONSIBLE INNOVATIONS IN MATERIALS AND PROCESSES

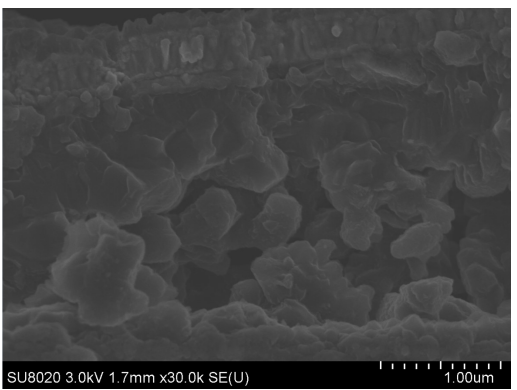
[www.materianova.be](http://www.materianova.be)

# Battery developments at Materia Nova

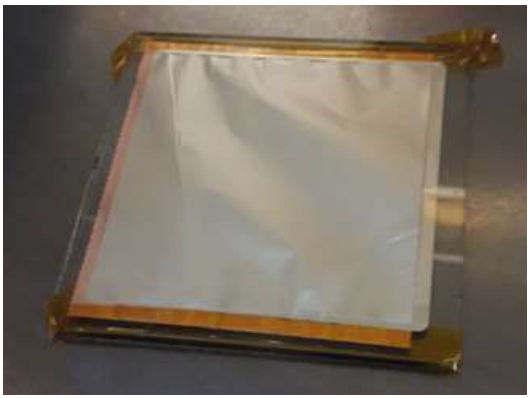
## Materia Nova : validator off innovative material and processing solution in thin film battery

Background and current activities:

- Li layer (up to 10  $\mu\text{m}$ ) deposited by PVD
- Deposition of thin film anode by PVD for low weight solid state batteries
- Characterization of materials thin films towards (semi-)battery systems

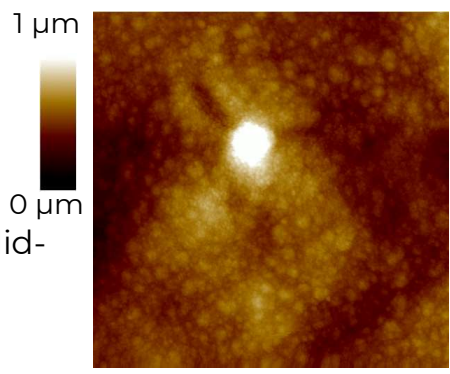


Cross-sectional topography of Li thin film by SEM



Li film on 5x5 cm<sup>2</sup> glass (left) and 10x10 cm<sup>2</sup> Cu substrates

15x15  $\mu\text{m}^2$  TM-AFM of Li film



Impedance spectroscopy of solid-state devices

# Equipment for thin film deposition and ion implantation

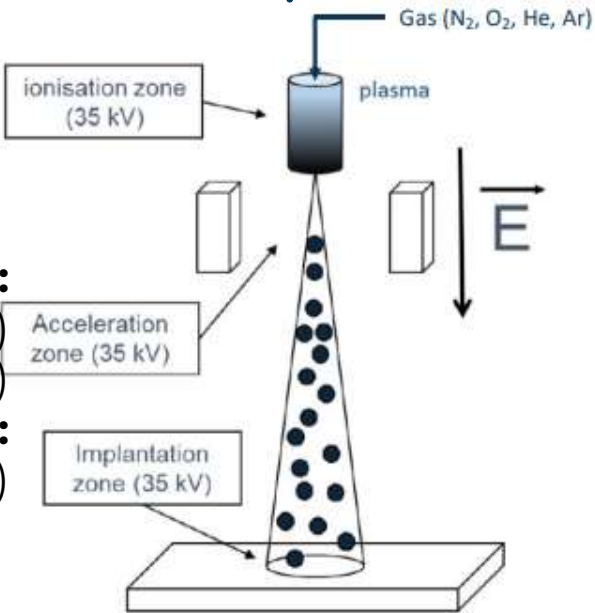
## Deposition methods



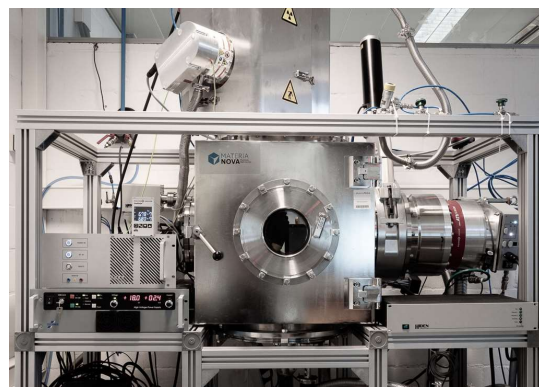
Spectros™150 from K. J. Lesker  
Connected to a Glovebox Jacomex  
conditioned in inert atmosphere (Ar)

**3 lab-scale ion implanter for:**  
Flat samples (up to 40x40cm<sup>2</sup>)  
Small 3d shape and powder (up to 100cm<sup>3</sup>)  
**2 semi-industrial ion implanter (Ionics) for:**  
large dimensions (1.6x1.6 m<sup>2</sup>; R2R)

## Ion Implantation



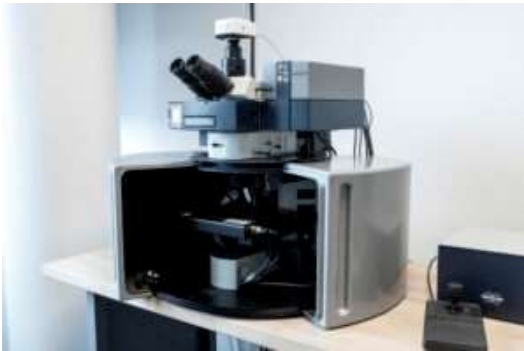
Magnetron sputtering and PECVD  
chambers  
To be equipped with ion gun for direct  
implantation



# Equipment for characterization of materials and devices

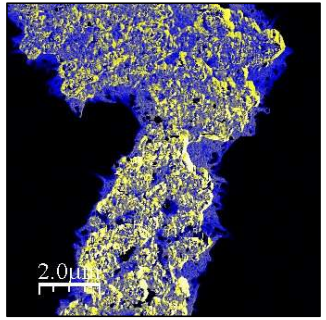
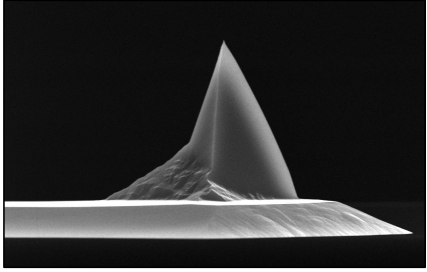
## Chemical analysis

XPS  
(Omicron)



Raman spectrometer  
(Bruker Senterra)

## Topographical and electrical analysis



Atomic Force Microscope with electric modules  
Low T to RT Hall effect characterization platform



## Device conditioning

MSK-115 Vacuum Sealing Machine  
Conditioned in inert atmosphere (Ar)

PEC Corp multichannel cell tester  
PEC ACT0505 Table Top  
Cell Tester. 10 Ch. 5A, 5V Incl LifeTest



# Low energy and low coast ion implantation activities at Materia Nova and Ionics

## R&D activities

Implanted species: all kind of gaseous atomic or molecular compound

Operating environment: high vacuum ( $\sim 10^{-5}$  Torr)

Energy range: 10 keV to 40 keV

Dose (fluence):  $10^{14}$  cm<sup>-2</sup> to  $10^{18}$  cm<sup>-2</sup>

Near surface implantation: up to 500 nm

Simple process → Industrialization

- Any solid materials: metals - polymers - glasses...
- Any shapes: flat - powders - small 3D objects, wire...
- No adherence issues

Monitoring mechanical properties

- Surface hardening
- Improved corrosion and HT oxidation resistance
- Wettability modification
- Adhesion improvement at interfaces
- Enhancing barrier properties (polymers)
- Doping and allowing (oxides, nitride...)

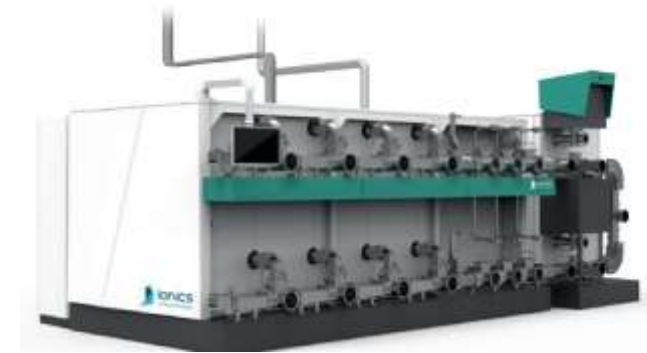


## Spin off from Materia Nova

Design, conception and fabrication of ion implanter

Large dimension implantation equipment

Versatile application: automotive, health...



THE TECHNOLOGICAL ACCELERATOR OF RESPONSIBLE INNOVATIONS IN MATERIALS AND PROCESSES

[www.materianova.be](http://www.materianova.be)

# Action plan at Matera Nova

## 2023 **Graphite electrodes:**

- Purchasing of graphite thick electrode
  - Carbon thin film (50 nm) deposited by magnetron sputtering
  - Ion implantation
  - Non-reactive implantation: Ne, Ar for monitoring vacancies in graphite
  - Reactive implantation: N for doping
- Morphological, chemical and electrical characterization

### **Solid state electrolytes:**

Materials and fabrication methods: Li-PEO (Wet X) and LIPON (PVD)

Non-reactive ion implantation for monitoring ionic transport: Species: Ar, N, He, Ne

Electrical characterization with impedance spectroscopy (Biologic, Modulab)

## 2024 **Li electrode:**

Deposition of Li layer ( $\mu\text{m}$ ) and additional interfacial thin film (LiF,  $\text{MoS}_2$ )

Transfer issue to be solved to prevent contamination (C, N, and O)

Ion implantation for monitoring stability upon cycling

Non-reactive implantation of multilayer vs. Reactive implantation ( $\text{H}_2\text{S}$ ,  $\text{CF}_4$ ) of Li layer

**Characterization of layers in half cells:** capacity & energy, (de)charging time, stability in cycling

# Collaborative actions

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## **From Materia Nova to QWED:**

- Fabrication on low loss substrate (quartz, borosilicate glass) of reference (thin) films (graphite, solid electrolyte, Li) ion implanted upon selected protocols to be sent to QWED
  - Electrical characterization (determination of permittivity) of reference samples with microwave dielectric resonator
  - Model supply to describe electrical properties and transport (electrons, ions) behavior
- First samples to be sent: ion implanted carbon and graphite thin films on quartz samples  
What are the maximum dimensions of the sample to be sent?

## **From L-IMIF to Materia Nova**

- Determination of ion implantation protocol for SiC/graphene reference structures and devices (sensors)
  - Reference graphene/SiC reference structures to be sent to Materia Nova for ion implantation, then to be returned for comparative testing
- What are the dimensions of the reference samples to be received



# Bridging the gaps between microwave modelling and materials measurements *and between women and engineering*

Malgorzata Celuch  
QWED Sp. z o.o., Poland



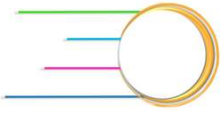
to my Father,  
MSc in engineering with PhD in economics,  
*Sybirak - survivor of Soviet deportation to Siberia*

with an appeal for a stronger response  
to Russia's invasion of Ukraine  
*to prevent Siberia happening to my children*





# Outline



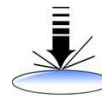
1. From research on Electromagnetic Modelling to its exploitation by QWED
2. QWED expanding into Material Measurements
3. Exploring the synergies between EM Modelling and Material Measurements
4. Validation of resonators in 5G/mmWave iNEMI project



# 25 years in a Nutshell

presented annually at IEEE IMS Show

## R&D projects



**FP6 SOCOT** – development and validation of an optimal methodology for overlay control in semiconductor industry, for the 32 nm technology node and beyond.



**FP6 CHISMACOMB** – development, modelling, and applications of chiral materials → EM validation of mixing rules



**Eureka E! 2602 MICRODEFROST MODEL** – innovative software-based product development tool for simulating and optimising heating and defrosting processes in microwave ovens



**FP7 HIRF SE** (High Intensity Radiated Field Synthetic Environment) - numerical modelling framework for aeronautic industry



**Eureka FOODWASTE** – developing new microwave treatment system for high water content waste



**ERA-NET MNT NACOPAN** – applications and modelling of nano-conductive polymer composites



**NGAM2** – designing an industrial device for thermal bonding of bituminous surfaces with the aid of microwave heating



**MMAMA** (Microwave Microscopy for Advanced and Efficient Materials Analysis and Production) – EM modelling & characterisation for the development of high efficiency solar cells



**NanoBat** - developing a novel nanotechnology toolbox for quality testing of Li-ion and beyond Lithium batteries with the potential to redefine battery production in Europe and worldwide.



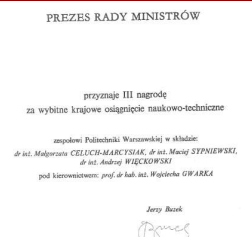
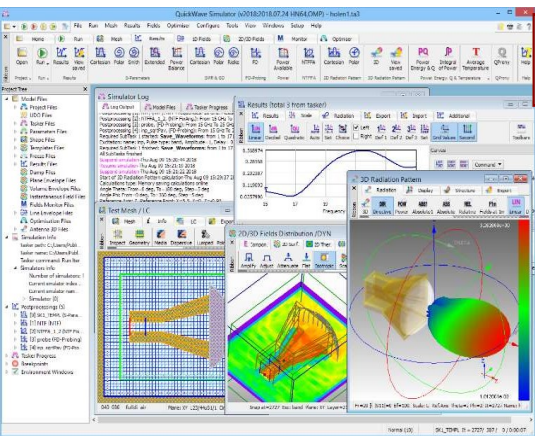
**ULTCC6G\_Epac** – development & application of novel ceramics for 5G & beyond

**I4BAGS** – modelling & characterisation of ion-implanted battery & graphene-enabled devices

## Electromagnetic simulation & design software, 3D & BOR 2D tools

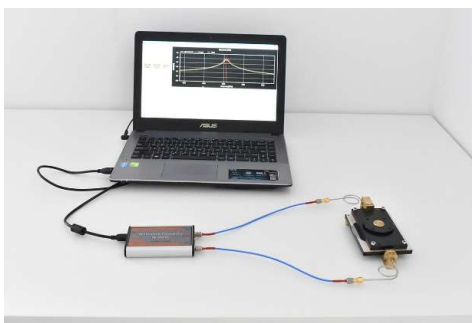
based on 300+ publications by: prof.W.Gwarek, IEEE Fellow, DML, Pioneer Award

dr.M.Celuch, President of QWED



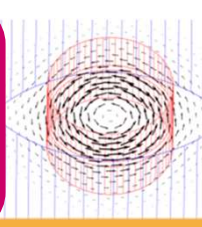
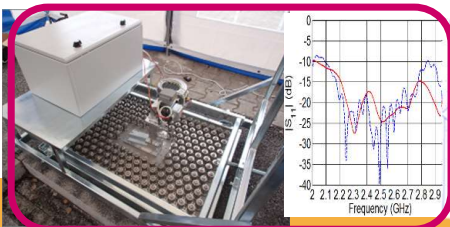
## Instruments for precise material measurements

based on 300+ publications by prof.J.Krupka, IEEE Fellow



## Consultancy & design services based on EM expertise & tools

team of 10+engineers, 4 PhDs, 2 Profs  
key areas: MW power appliances, customised resonators, antennas & feeds



# Origins of QWED Computer Modelling



since 1980s...

IEEE- awarded research of Prof. Wojciech Gwarek on 2D FDTD modelling (with novel conformal meshing)

Fellow,

Pioneer Award,

DML



... by early 2000s:

QWED commercialises & continues the development licences for QuickWave-3D by QWED used worldwide industrial applications from RF to optical bands



IEEE TRANSACTIONS ON MICROWAVE THEORY AND TECHNIQUES, VOL. MTT-33, NO. 10, OCTOBER 1985 1067

**Analysis of an Arbitrarily-Shaped Planar Circuit—A Time-Domain Approach**

WOJCIECH K. GWAREK  
(Invited Paper)

$$\nabla V(x, y, t) = -L_s \frac{\partial J(x, y, t)}{\partial t}$$

$$\nabla \cdot J(x, y, t) = -C_s \frac{\partial V(x, y, t)}{\partial t}$$

IEEE TRANSACTIONS ON MICROWAVE THEORY AND TECHNIQUES, VOL. 36, NO. 2, FEBRUARY 1988

**Computer-Aided Analysis of Arbitrarily Shaped Coaxial Discontinuities**

WOJCIECH K. GWAREK

IEEE TRANSACTIONS ON MICROWAVE THEORY AND TECHNIQUES, VOL. 36, NO. 4, APRIL 1988

**Analysis of Arbitrarily Shaped Two-Dimensional Microwave Circuits by Finite-Difference Time-Domain Method**

WOJCIECH K. GWAREK

**Industrial Design of Axisymmetrical Devices Using a Customized FDTD Solver from RF to Optical Frequency Bands**

■ Malgorzata Celuch and Wojciech K. Gwarek

Figure 1. Axisymmetric resonator with dielectric rod in air.

Figure 2. Frequency response of the resonator of Figure 1.

Figure 3. Mesh and conformal boundaries of the resonator of Figure 1.

**FDTD for Nanoscale and Optical Problems**

Bartłomiej Salski, Malgorzata Celuch, and Wojciech Gwarek

IEEE microwave magazine

# QWED started 1997

Founders: A.Wieckowski, M.Sypniewski, M.Celuch, W.Gwarek



Prof. Jerzy Buzek awarding QWED team in 1998  
Prime Minister of Poland 1997-2002  
President of the European Parliament 2009-2012

# celebrating 25 years



**Dr. Malgorzata Celuch**  
President since 2017, VP 1997-2017

- 35 y experience in mathematical, 25 y in management
- Awards for excellence from e.g. Prime Minister of Poland, Rector of WarsawUnivTech



**Janusz Rudnicki, MS,**  
VP for IT

- 22 years of experience in simulation software development



**Dr. Marzena Olszewska-Placha,**  
VP for R&D

- 15 y of experience in simulation-based MHz to THz design and consultancy
- 4 y experience in research management



**Dr. Andrzej Więckowski**  
Senior in CAD

- 48 years of experience in computer-aided electronic engineering and engineering software development



**Prof. Wojciech Gwarek,**  
President 1997-2017

- 22 years of experience in simulation software development



**Dr. Maciej Sypniewski**  
Senior in CAE

- 35 years of experience in engineering software development and GHz measurements

# 10

people employed

# 7

consultants cooperating

# 50%

female

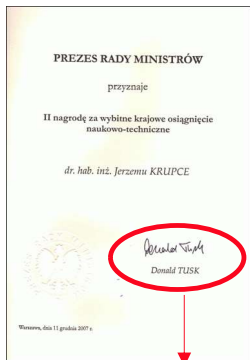
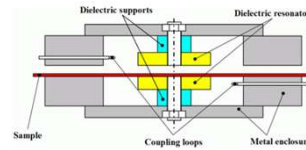




# Origins of QWED Material Measurements

since 1980s...

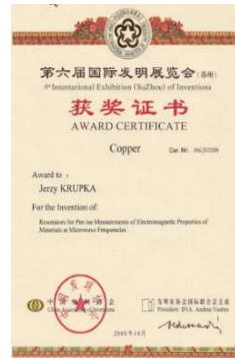
awarded research of **Prof. Jerzy Krupka** (IEEE Fellow)  
on dielectric resonators (best known: Split-Post Dielectric Resonator)



by Donald Tusk

Prime Minister of Poland 2007-2014

President of the European Council 2014-2019



Agilent Both  
IEEE IMS 2006, San Francisco, CA



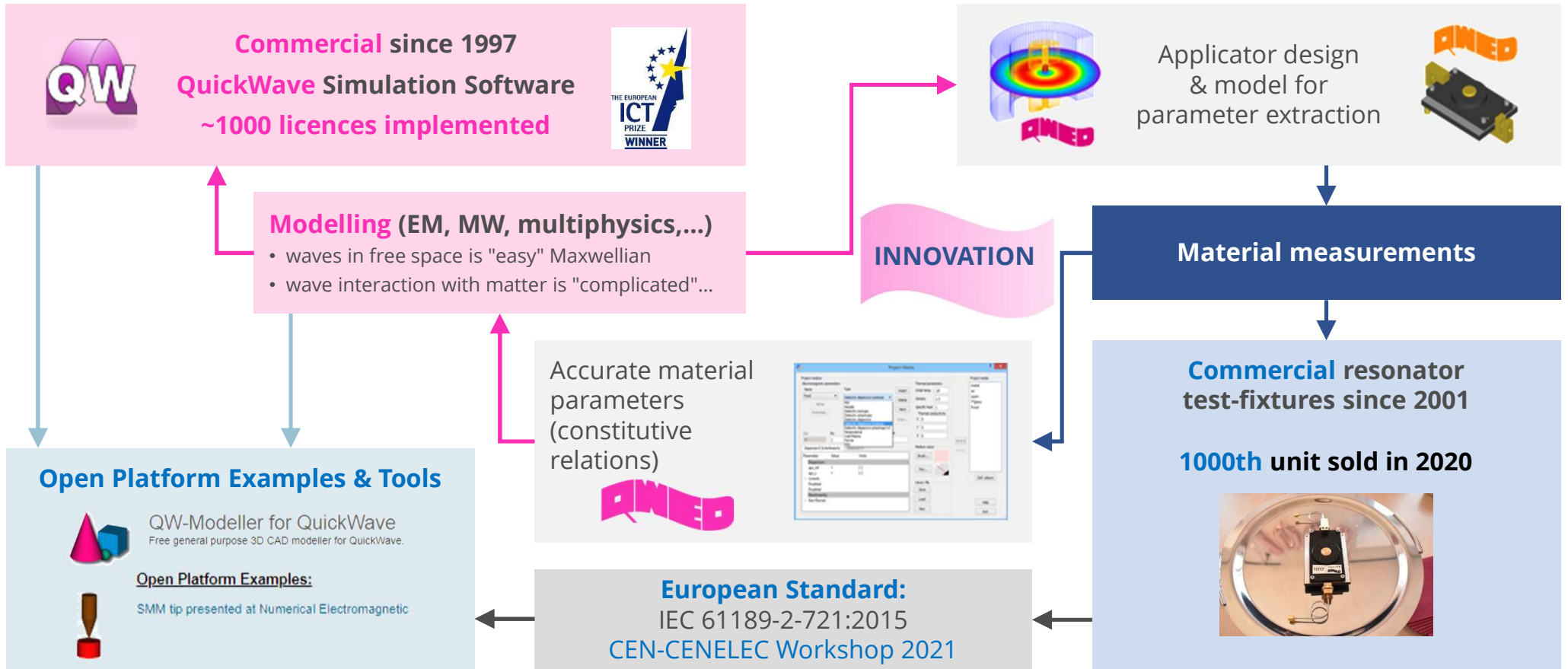
MMA-2010, Warsaw PL  
co-organised by QWED & Warsaw Univ.Tech.

... by early 2000s:

QWED commercialises the SPDRs  
endorsement by Agilent / Keysight  
publication of standard IEC 61189-2-721:2015



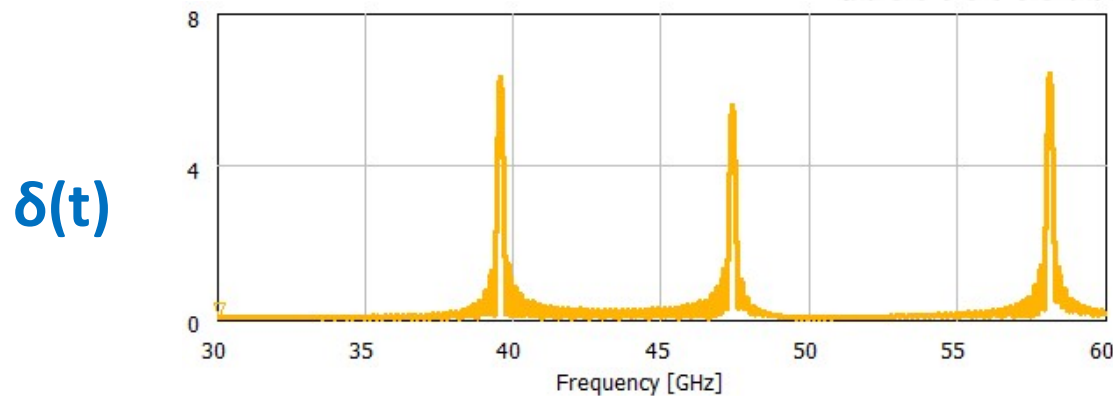
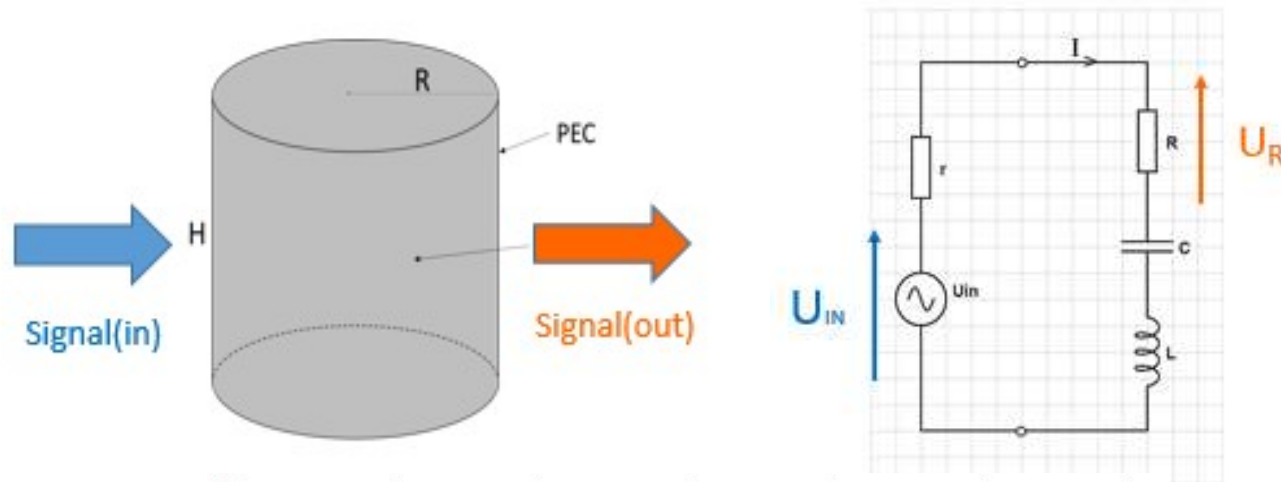
# Current Work: Bridging Computer Modelling with Material Measurements





# Resonator methods – motivation and background (1)

**Resonance in practice:** given fixed strength of **Signal(in)**, at resonance **Signal (out)** is strongest



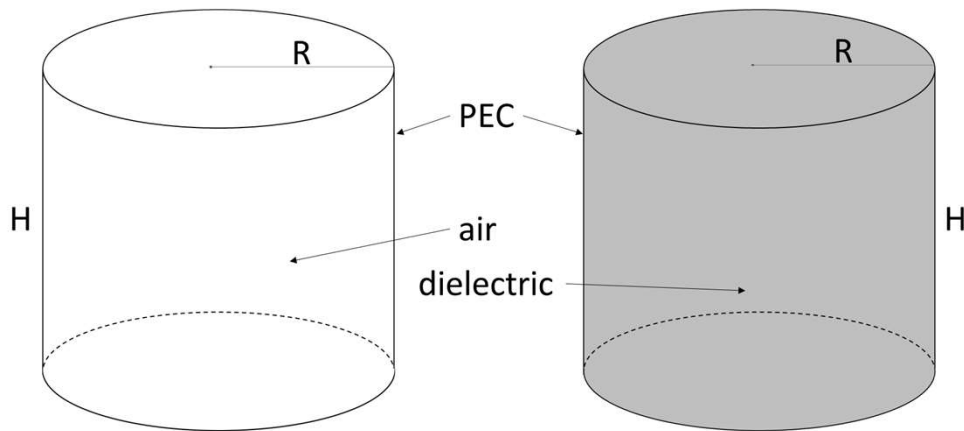


# Resonator methods – motivation and background (2)

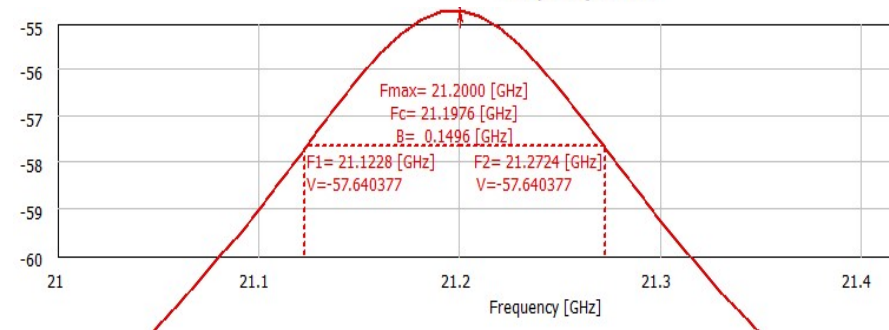
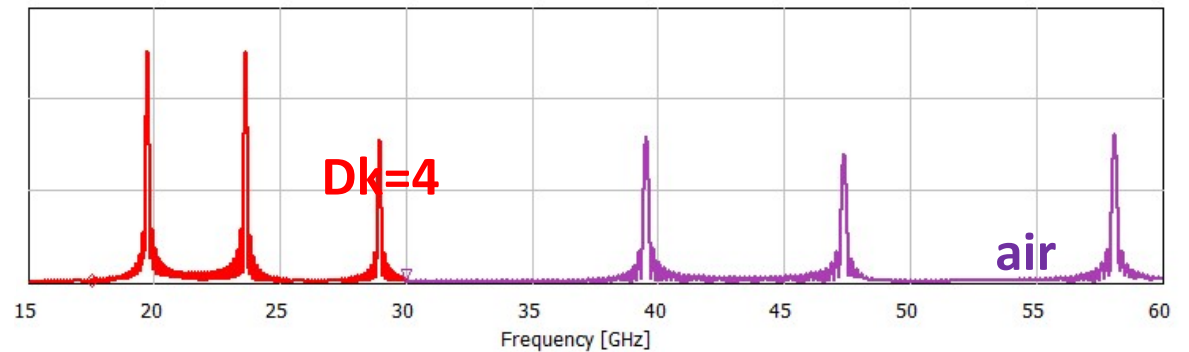
**Resonance in theory:** non-zero electromagnetic fields exist in isolated structures (no excitation).

Field properties are well-defined and **linked to material properties**.

E.g. for **cylindrical** cavities:



$$f_{r,mnp} = \frac{c}{\sqrt{Dk}} \sqrt{\left(\frac{\kappa_{mn}^{(l)}}{\pi R}\right)^2 + \left(\frac{p}{H}\right)^2} \quad \text{in non-magnetic low-loss dielectrics}$$



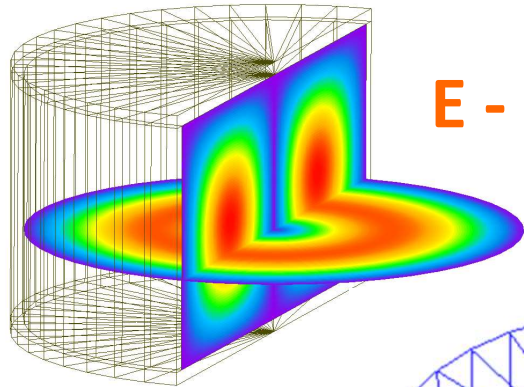
$$Q = 2\pi \frac{\iiint_V \epsilon \vec{E} \cdot \vec{E}^* dv}{T \iiint_V \sigma \vec{E} \cdot \vec{E}^* dv} = \frac{\omega \epsilon}{\sigma} = \frac{1}{Df} \approx \frac{f_{res}}{\Delta f}$$



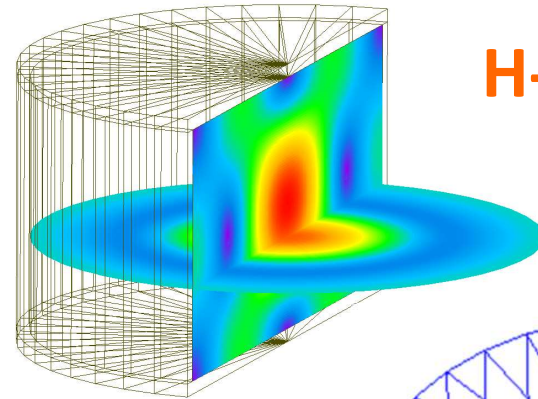




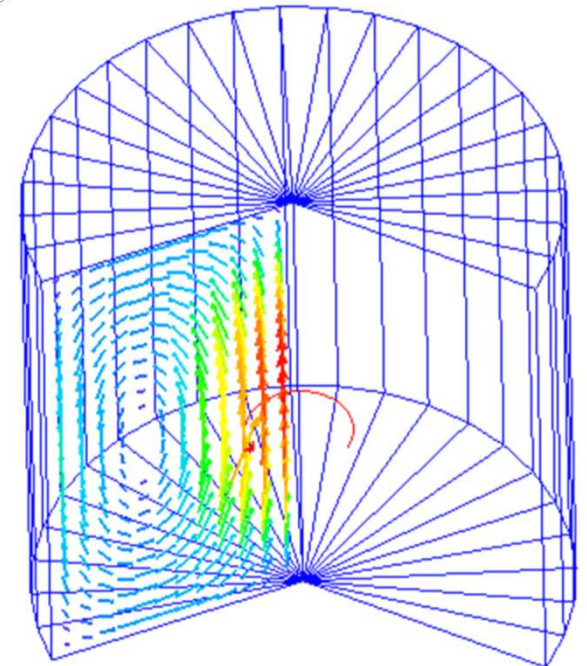
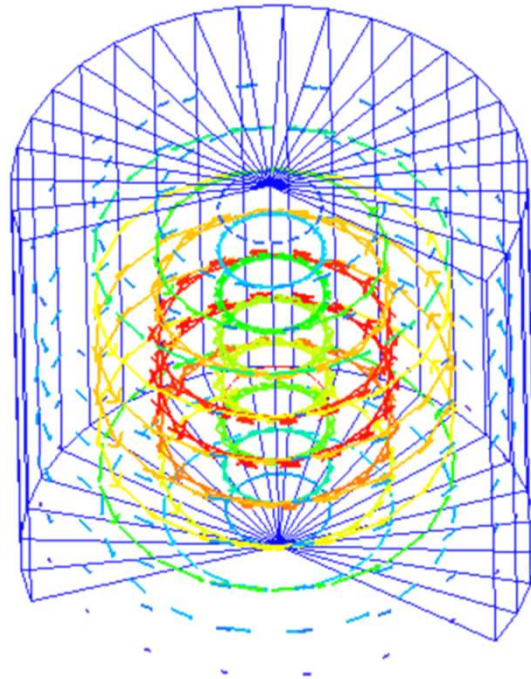
# Cylindrical resonator: TE<sub>011</sub> mode



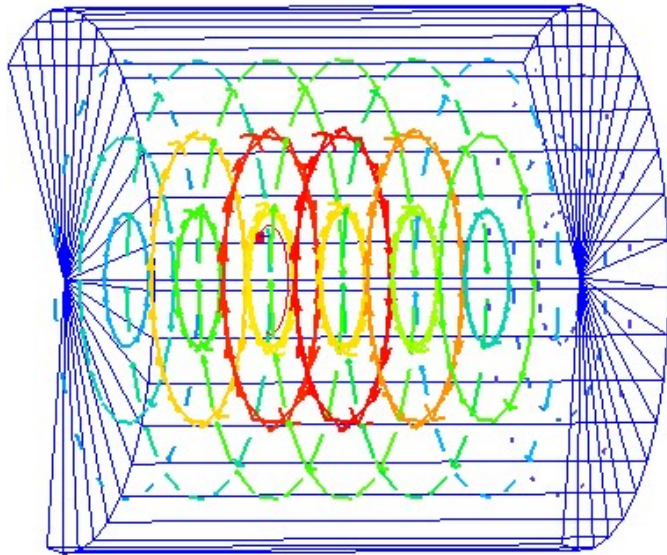
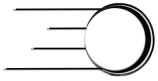
E - field



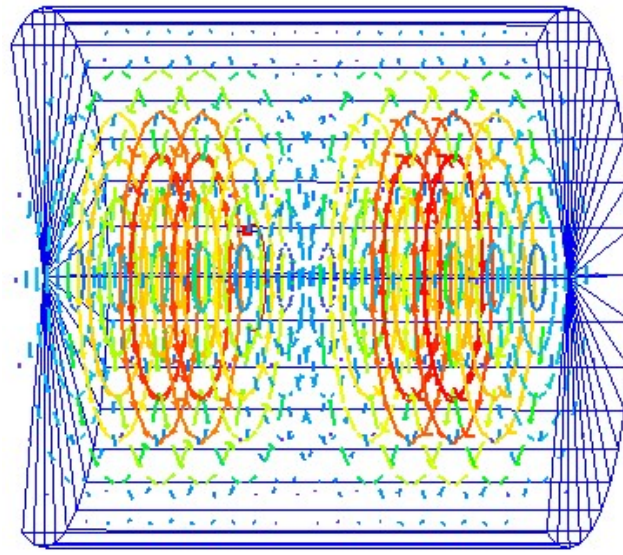
H-field



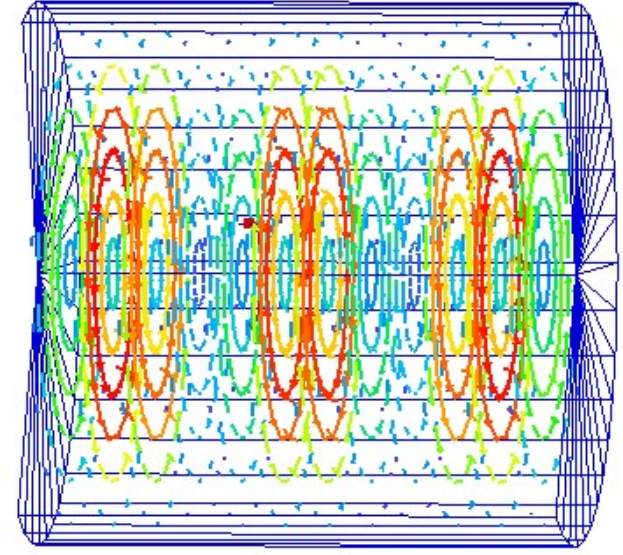
# Cylindrical resonator: single-mode versus multi-mode operation



**TE011 @ 29.43 GHz**



**TE012 @ 47.25 GHz**



**TE013 @ 57.95 GHz**

Resonators are **multimode** devices.

Hence formally, material measurement can be performed at **many frequencies** in the same resonator.

However, **some modes provide highest accuracy** of material characterization. Some are difficult to excite.

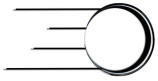
Software provided with the resonator is compatible only with modes pre-selected by the vendor.

Software provided with the resonator is compatible only with modes pre-selected by the vendor.

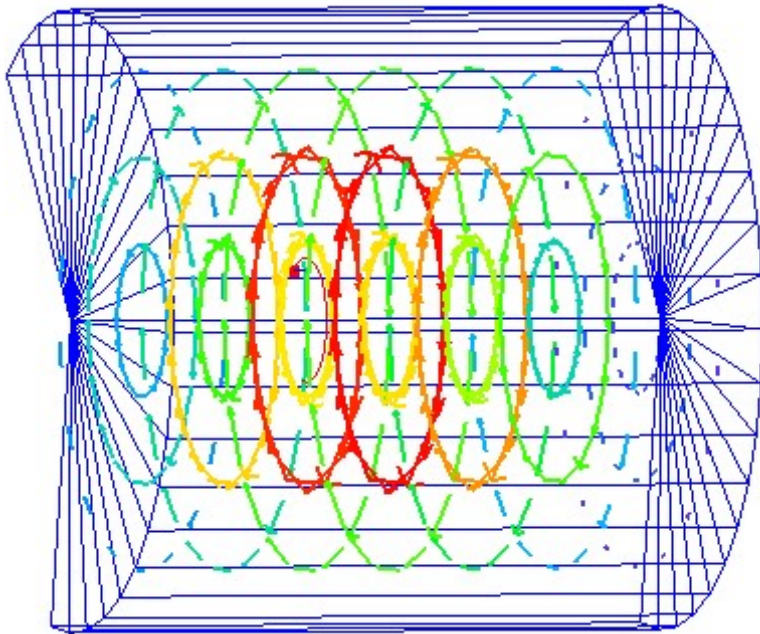
Among the popularly available resonators, **BCDR and FPOR work as multi-modal**.



# Resonator methods – motivation and background (3)



**TE010**

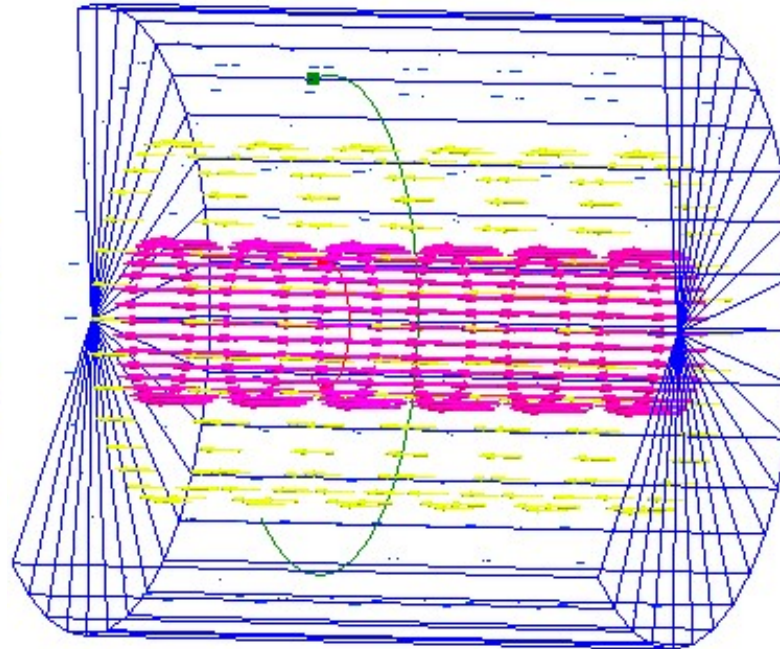


**TE modes to measure  
in-plane component of  $D_k$ ,  $D_f$**

**SCR, SPDR, FPOR**

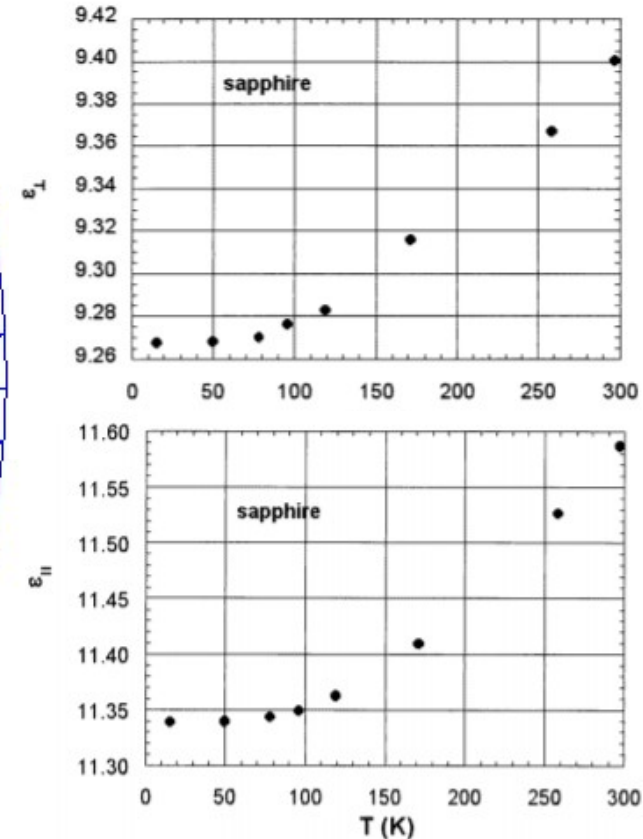
**Full characterisation of anisotropic materials (like crystals) requires both measurements.**

**TM010**



**TM modes to measure  
out-of-plane component of  $D_k$ ,  $D_f$**

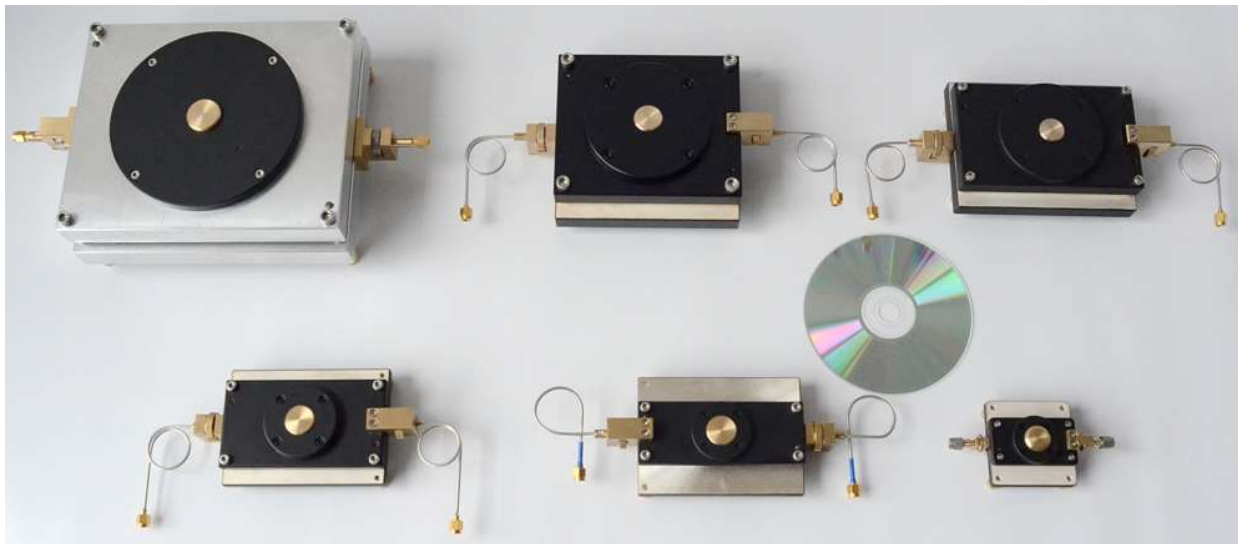
**BCDR**



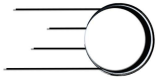
J.Krupka et al., "Complex permittivity of some ultralow loss dielectric crystals..", Meas. Sci. Technol. 10 (1999).

# Popular Dielectric Resonators by QWED

SPDRs for laminar dielectric materials  
typical units: 1.1 GHz -15 GHz



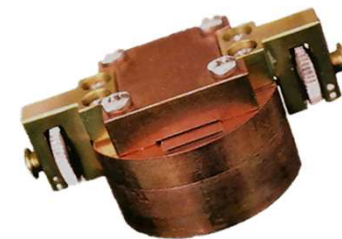
5 GHz SiPDR for resistive sheets



TE01 $\delta$  cavities, typically 1 – 10 GHz  
for bulk low-loss dielectrics

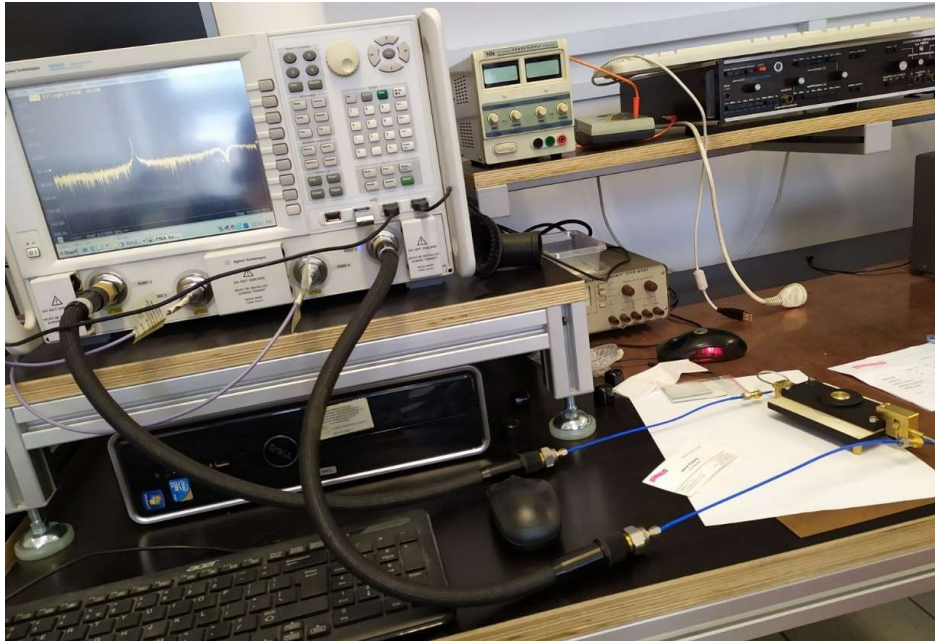


modified SiPDR for graphene





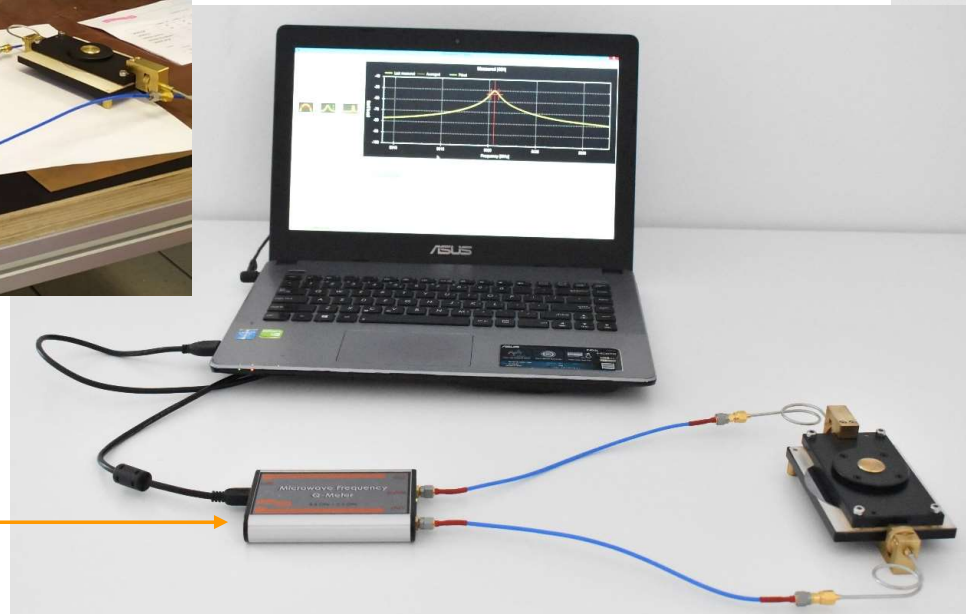
# Resonators Operating in Different Setups



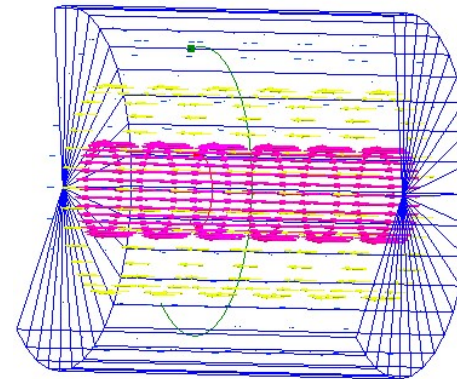
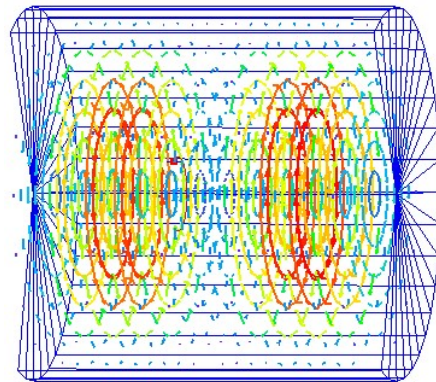
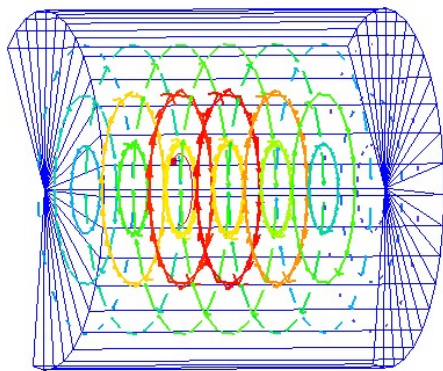
also for home-office!



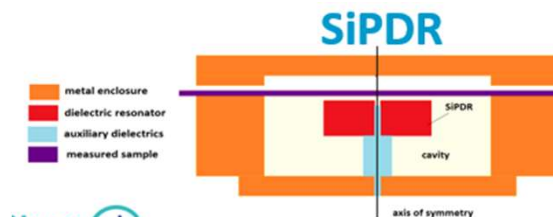
QWED Microwave Frequency Q-Meter units for 5 GHz and 10 GHz



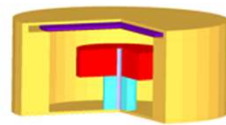
# QuickWave Modelling for Enhanced Design & Calibration of Resonators



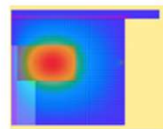
Tutorial examples on  
NanoBat Open Platform  
<https://qwed.eu/nanobat.html>



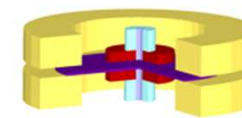
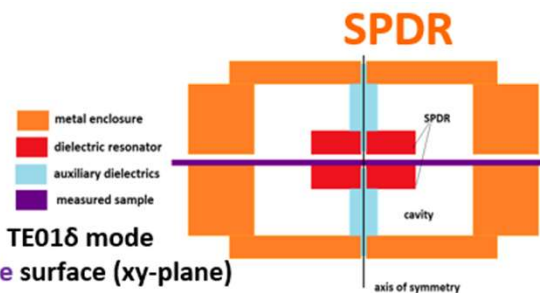
two configurations used with TE<sub>016</sub> mode  
E-field tangential (parallel) to sample surface (xy-plane)



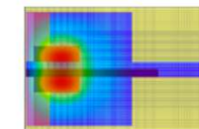
sample between the **single post dielectric** and the **ground plane**



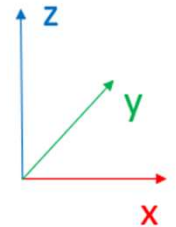
E-field distribution  
in the half cross-section



sample half-way between the **two dielectric posts** (in the "split" of the "post")



E-field distribution  
in the half cross-section





# Resonator methods considered in iNEMI 5G project

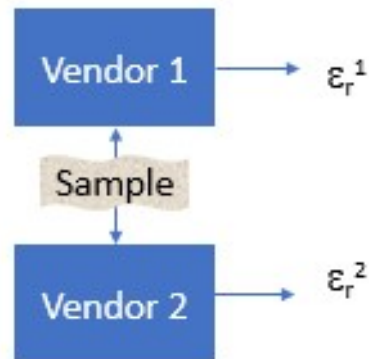
**Why** we use resonant methods

**How** these resonant methods work

*Presentation will be illustrated  
with full-wave electromagnetic modeling  
with QuickWave™ software by*



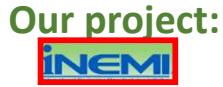
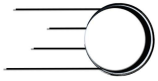
**Why** these different methods may produce different results



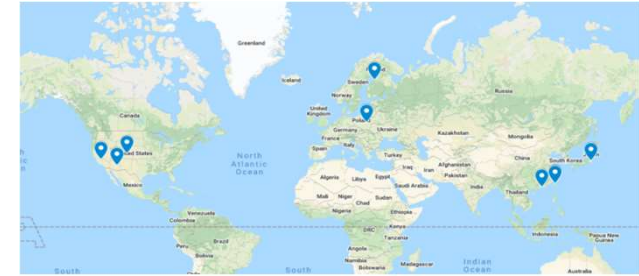
what is **vendor-specific**,  
what is **method-specific**,  
and what other criteria may come into play



# iNEMI 5G Round Robin Overview



- |  |   |   |
|--|---|---|
| <ul style="list-style-type: none"> <li>• 3M</li> <li>• AGC-Nelco</li> <li>• Ajinomoto USA</li> <li>• AT&amp;S</li> <li>• Centro Ricerche FIAT-FCA</li> <li>• Dell</li> <li>• Dupont</li> <li>• EMD Electronics (Co-Chair)</li> <li>• Flex</li> </ul> | <ul style="list-style-type: none"> <li>• Georgia Tech</li> <li>• <b>Showa Denko Materials</b></li> <li>• IBIDEN Co Ltd</li> <li>• IBM</li> <li>• <b>Intel</b></li> <li>• Isola</li> <li>• ITRI (Co-Chair)</li> <li>• <b>Keysight (Co-Chair)</b></li> <li>• MacDermid-Alpha</li> </ul> | <ul style="list-style-type: none"> <li>• Mosaic Microsystems</li> <li>• <b>NIST</b></li> <li>• Nokia</li> <li>• Panasonic</li> <li>• <b>QWED</b></li> <li>• Shengyi Technology Company</li> <li>• Sheldahl</li> <li>• Unimicron Technology Corp</li> <li>• Zestron</li> </ul> |
|--|---|---|



## Sample Material Requirements

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

## Techniques Included

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

→ Frequency Span : 10GHz – 100GHz with overlaps

## 10 Sample Kits Created

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

## 1st Project Stage

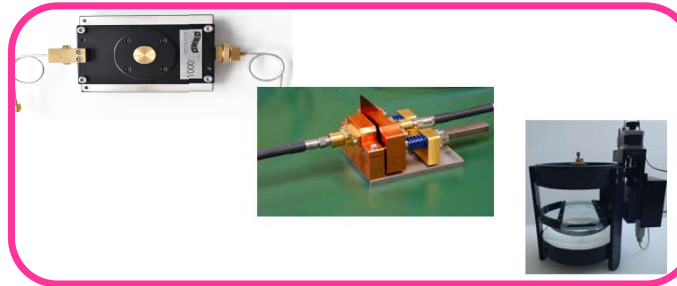
- Precision Teflon
- Cyclo Olefin Polymer

## 2nd Project Stage

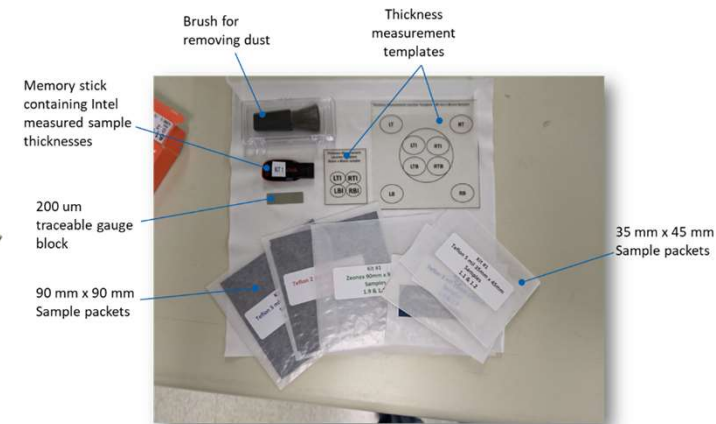
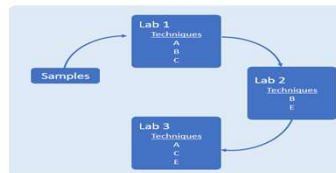
- Rexolite
- Fused Silica

## Industrial

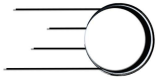
- Automotive



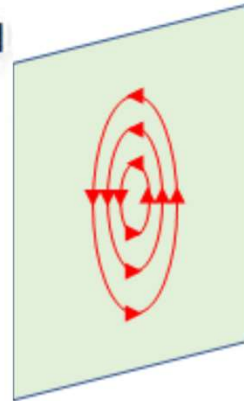
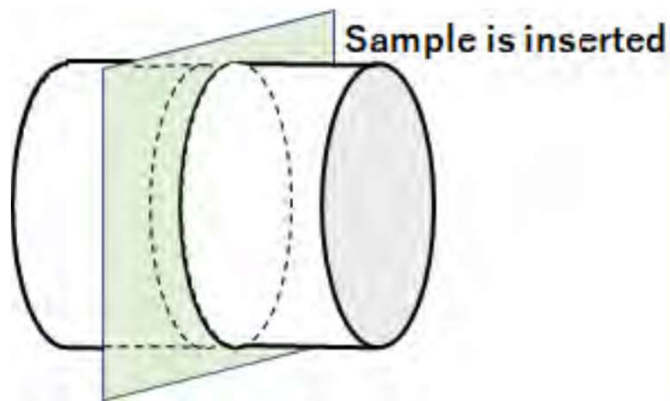
## 10 Laboratory Round Robin



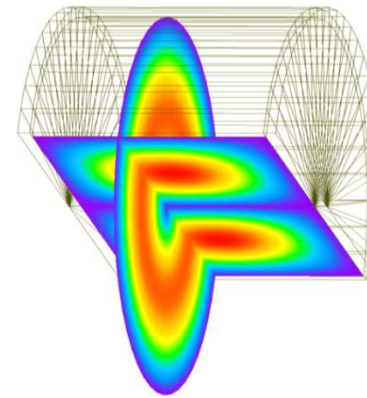




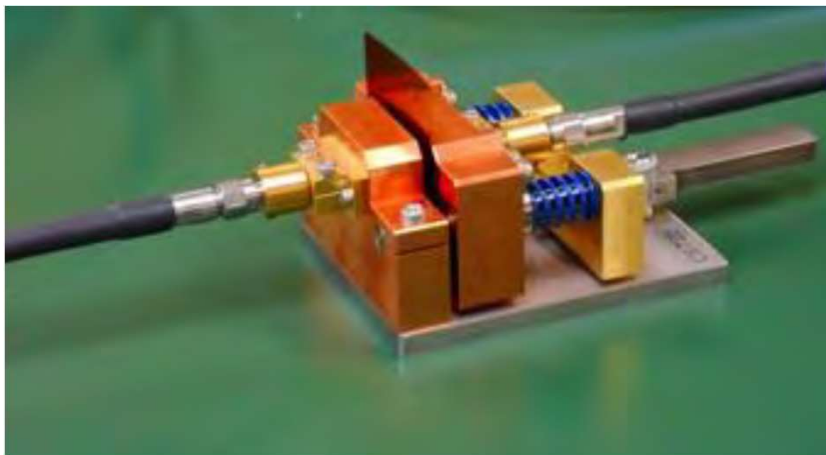
# Split Cylinder Resonator (SCR) - basics



In-plane Electric field is applied to Sample



TE011 mode

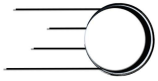


Split cylinder resonator (SCR)

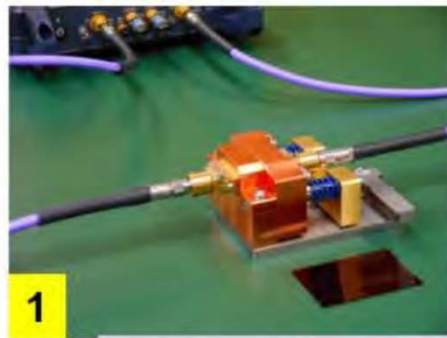
Discrete frequency points from 10 GHz up to 80 GHz

- High measurement precision
- Can be sensitive to many user errors
- Typically interpolated to 5G mmWaves
- Typically in-plane component of permittivity
- Typical sample thicknesses around 100 um
- Support temperature sweep measurement
- IPC-TM-650 2.5.5.13
- <https://www.keysight.com/us/en/assets/7018-06384/brochures/5992-3438.pdf>

# Split Cylinder Resonator (SCR) - operation



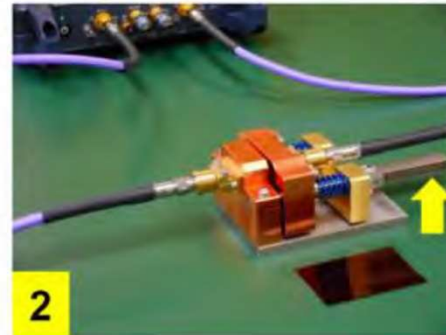
Connect the cables and measure.  
No need for other  
preparation or calibration.



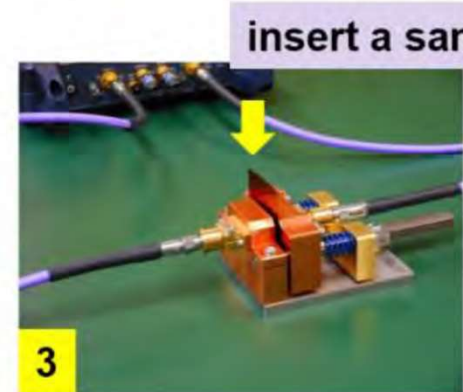
measure "empty"

10 sec

Same measurement results  
regardless who uses it.



open the lever



insert a sample

15 sec

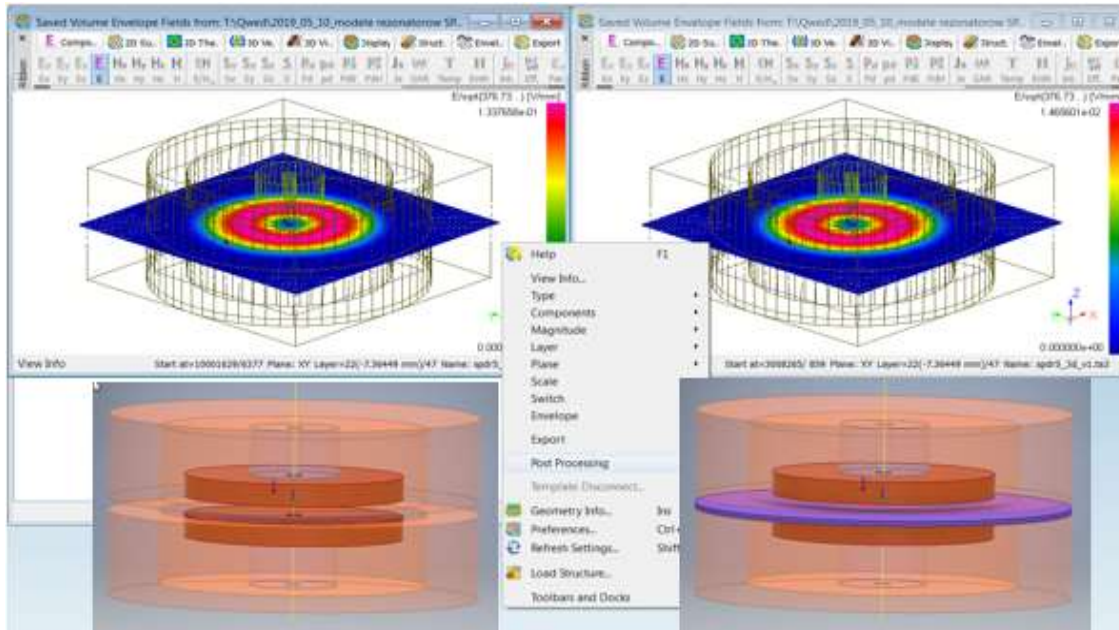
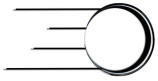
Very efficient measurement cycle  
for high volume measurements.



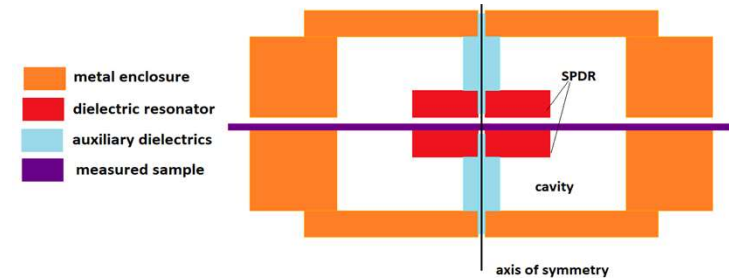
close the lever  
and measure



# Split-Post Dielectric Resonator (SPDR) - basics



- resonant mode with EM fields mostly confined in and between those ceramic posts
- minimal losses in metal enclosure
- H-field is only vertical at the side wall of the enclosure → circumferential currents
- no radiation through slot
- E-field tangential to SUT
- air slots between SUT and posts have negligible effect
- easy SUT insertion through slot, no dismounting



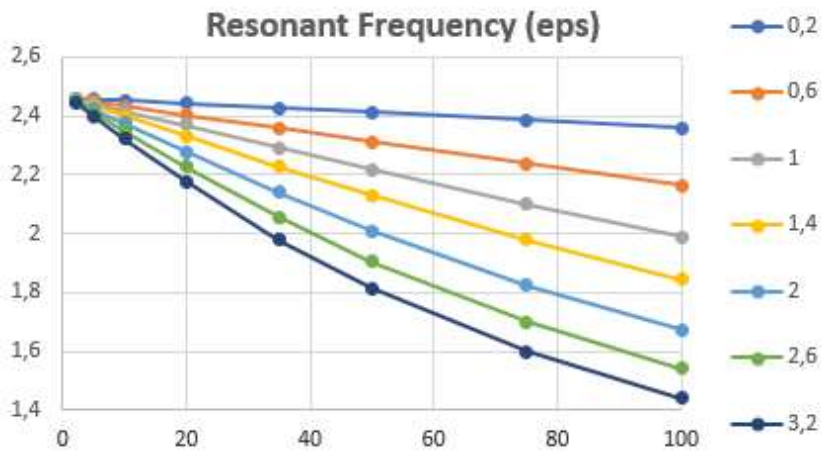
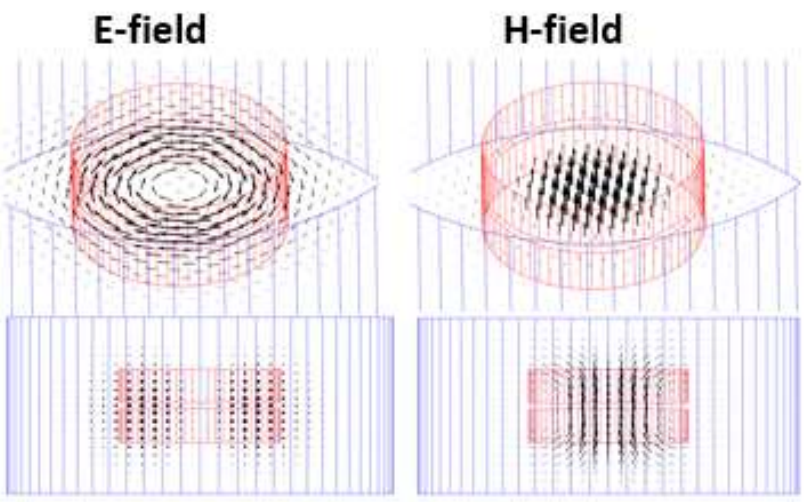
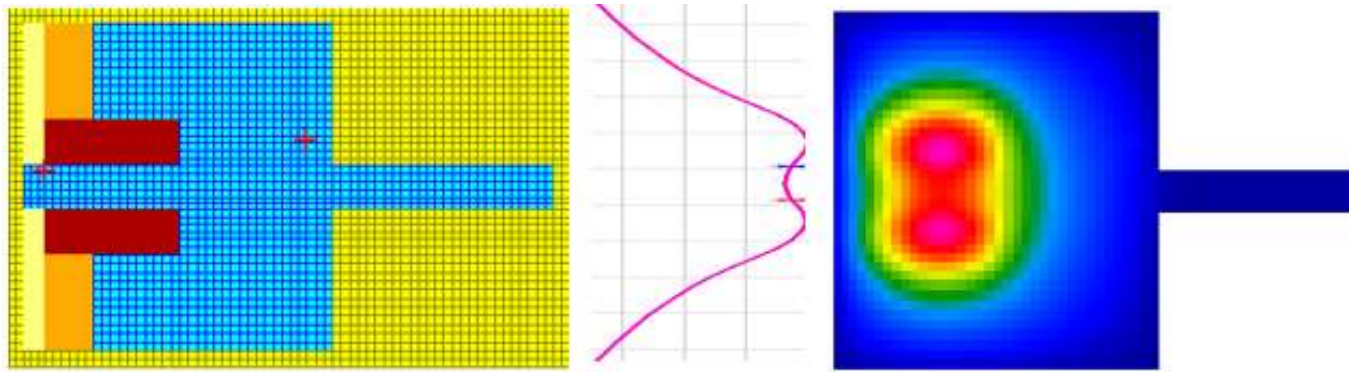
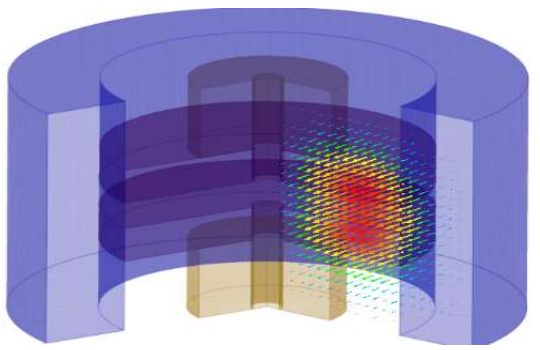
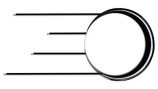
Split-post dielectric resonator (SPDR)

Discrete frequency points from 1 GHz up to 15 GHz

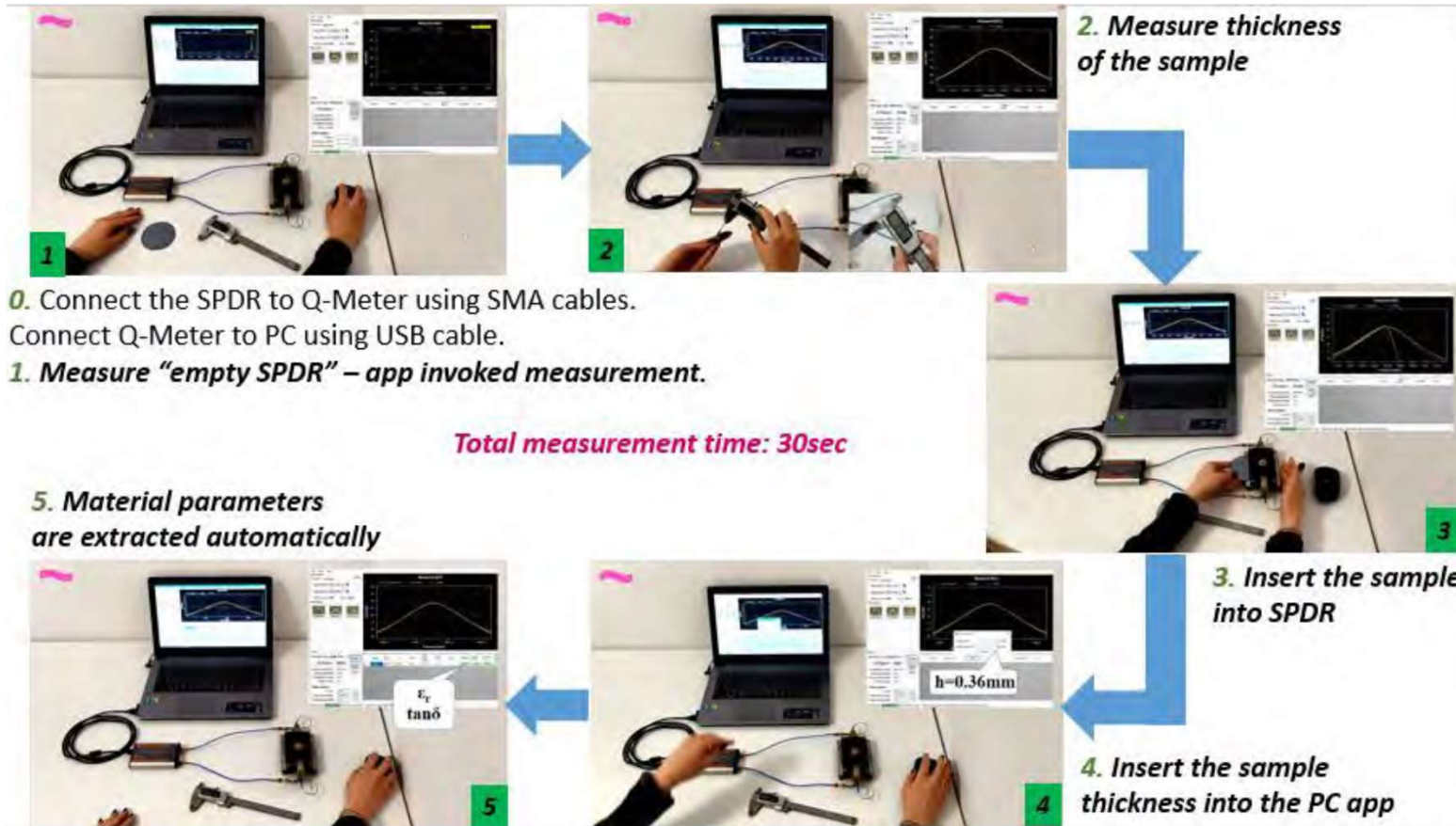
- High measurement precision
- Easy to use
- Insensitive to many user errors
- Typically in-plane component of permittivity
- Typically extrapolated to 5G mmWaves
- Typical sample thicknesses less than 1 mm
- IEC 61189-2-721:2015
- [https://www.qwed.com.pl/resonators\\_spdr.html](https://www.qwed.com.pl/resonators_spdr.html)
- <https://www.keysight.com/us/en/assets/7018-01416/application-notes/5989-5384.pdf>



# Split-Post Dielectric Resonator (SPDR) – modelling results

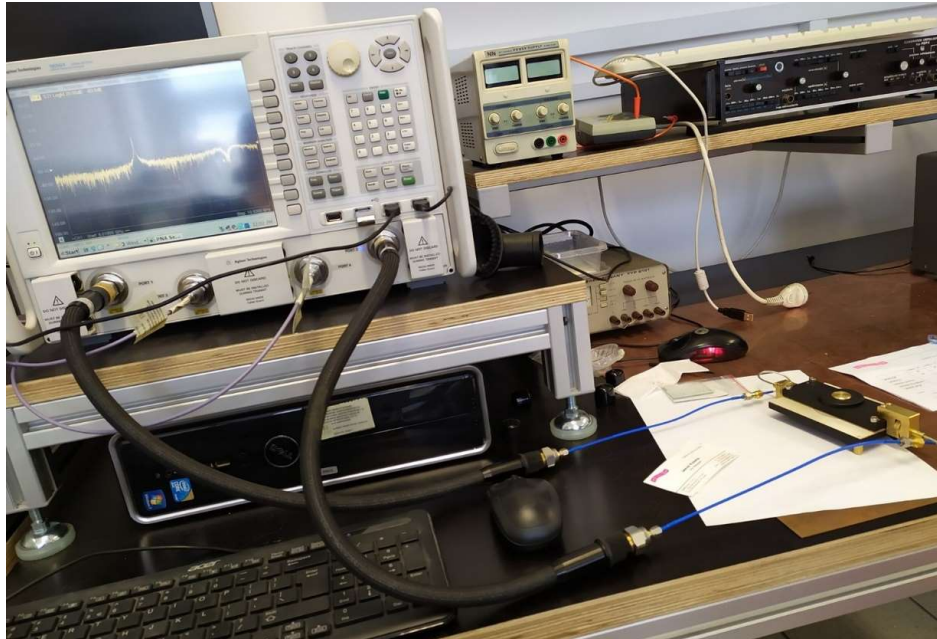


# Split-Post Dielectric Resonator (SPDR) – operation (1)





## Split-Post Dielectric Resonator (SPDR) – operation (2)

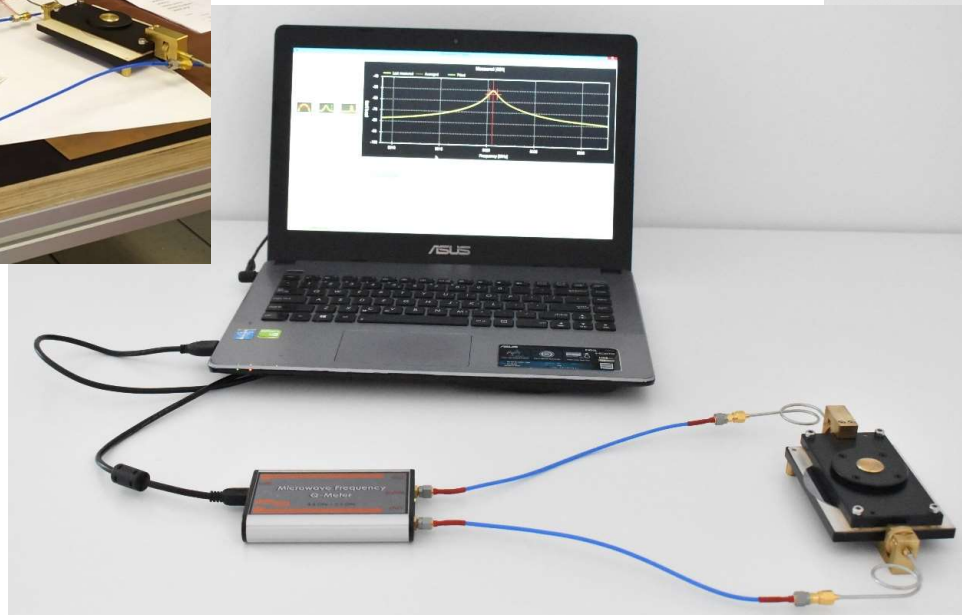


SPDR use in labs...  
...and at home



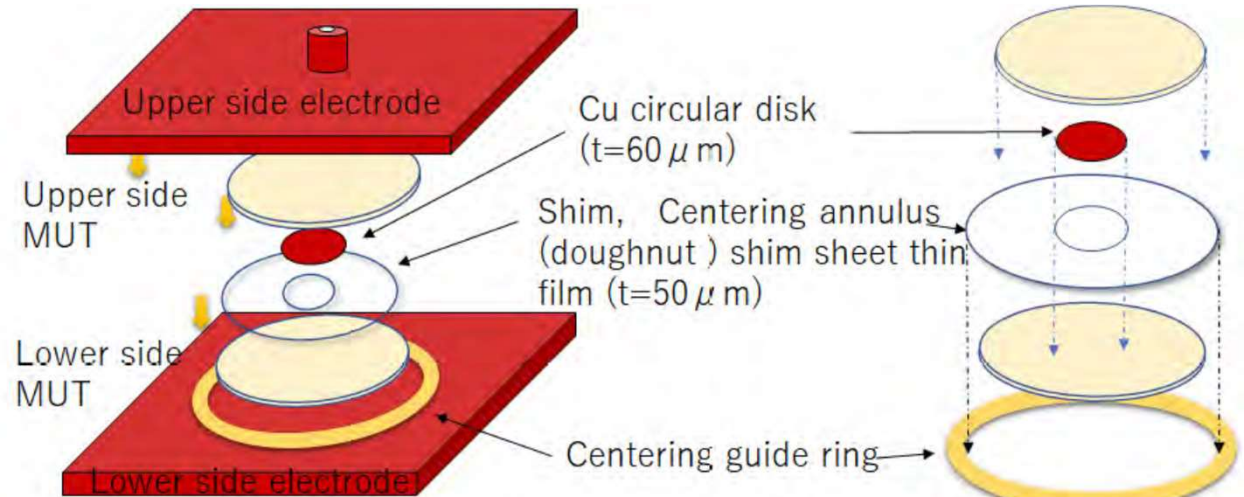
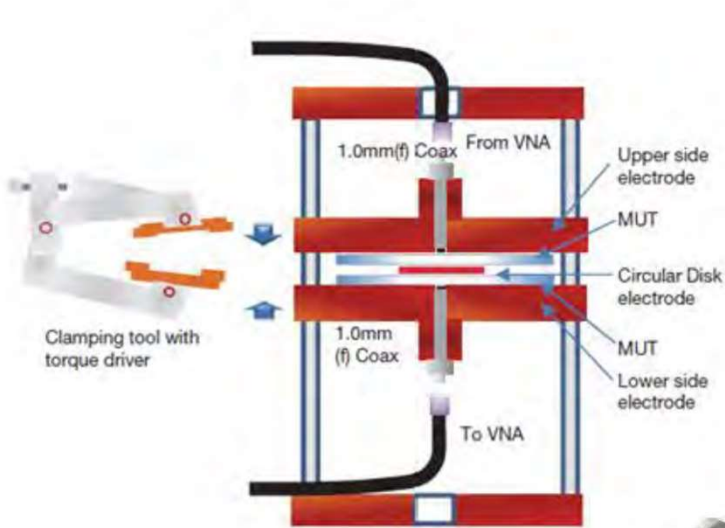
For many practical materials, measuring only abs ( $S_{21}$ ) provides appropriate accuracy.

Keysight Option N1500A uses  $S_{21}$  (amplitude & phase) which helps enhance accuracy (*under study in iNEMI project*).





# Balanced-type circular disk resonator (BCDR) - basics



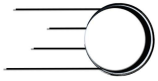
Balanced-type circular disk resonator (BCDR)

Multiple discrete frequency points from 10 GHz up to 120 GHz

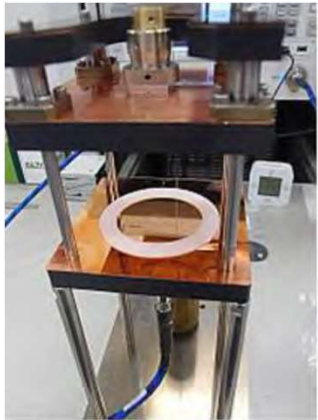
- High measurement precision
- Requires full 2-port calibration (mechanical to 110 GHz or electrical to 67 GHz)
- Typically out-of-plane component of permittivity
- Typical sample thicknesses less than 1 mm
- IEC 63185
- <https://www.keysight.com/us/en/assets/7120-1214/flyers/N1501AE11-67-Balanced-Type-Circular-Disk-Resonator-BCDR.pdf>



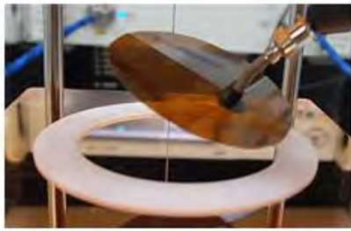
# Balanced-type circular disk resonator (BCDR) - operation



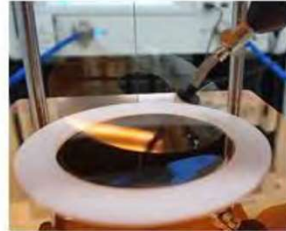
concentricity must be preserved



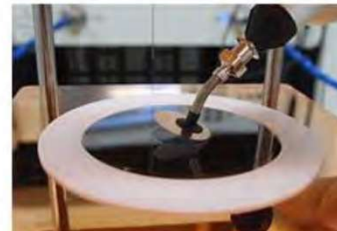
Open the resonator



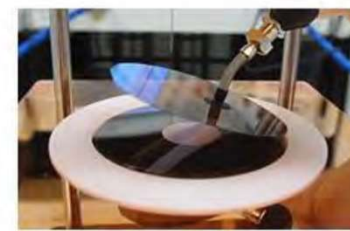
Set lower side sample



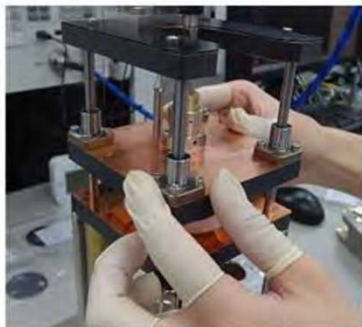
Set shim sheet



Set center electrode



Set upper side sample



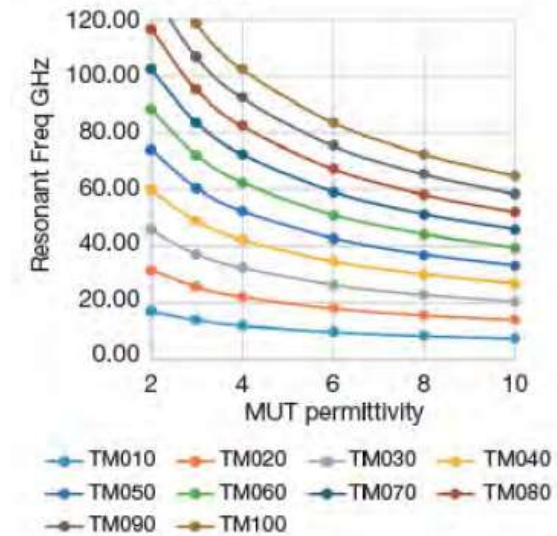
Close the resonator



Clamp and measure

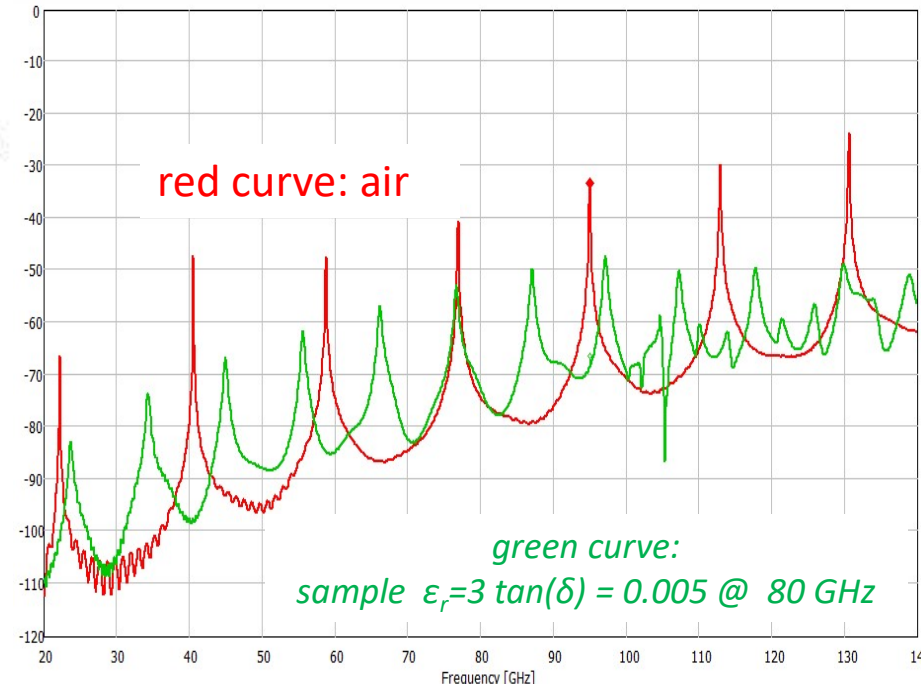
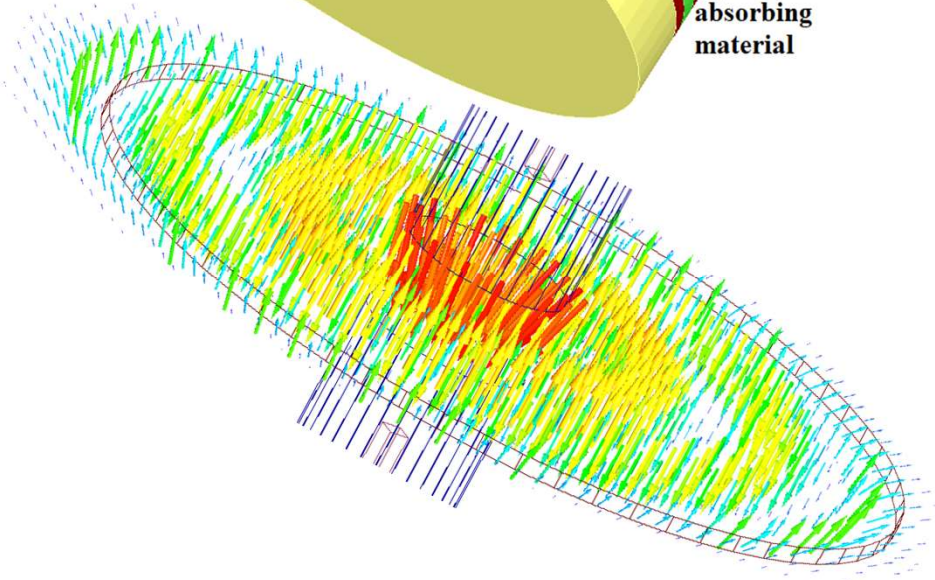
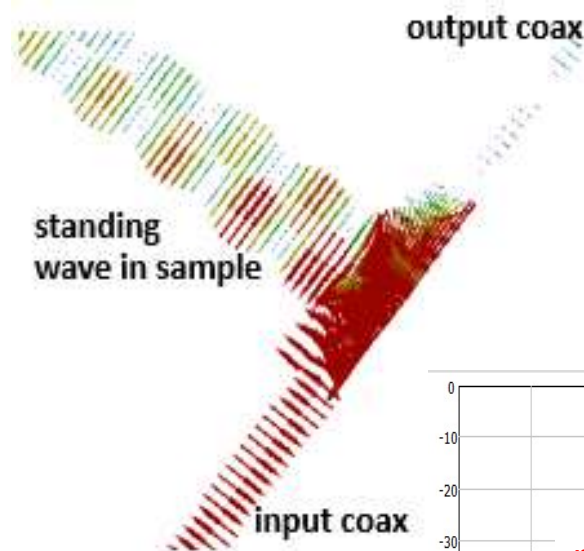
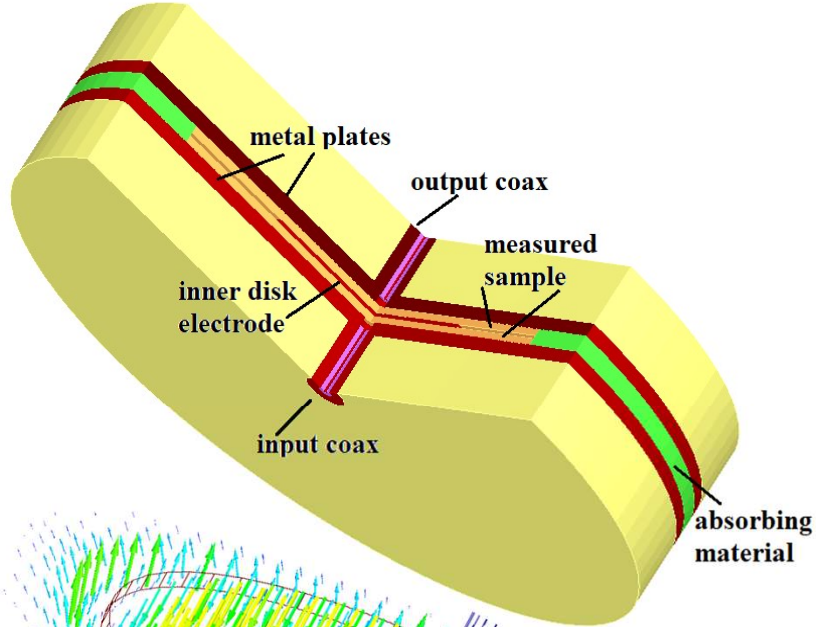


Resonant Freq. vs. Permittivity @ disc diameter 15mm

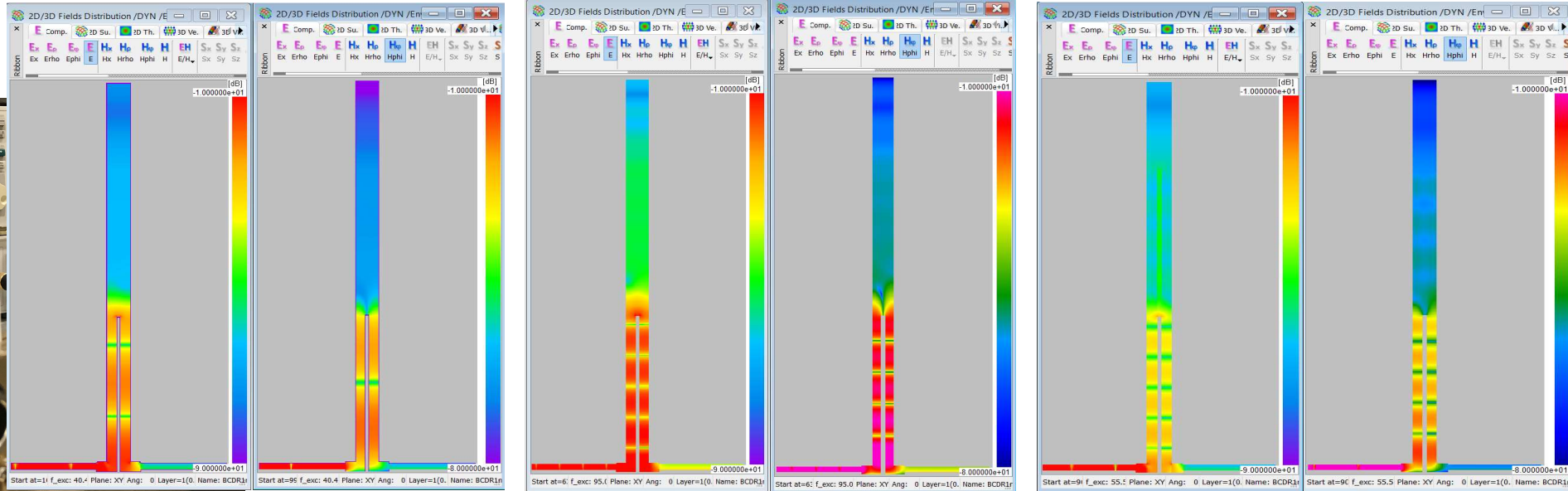
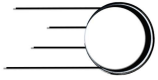




# Balanced-type circular disk resonator (BCDR) – modelling



# Balanced-type circular disk resonator (BCDR) – modelling



@ 40.49 GHz, air

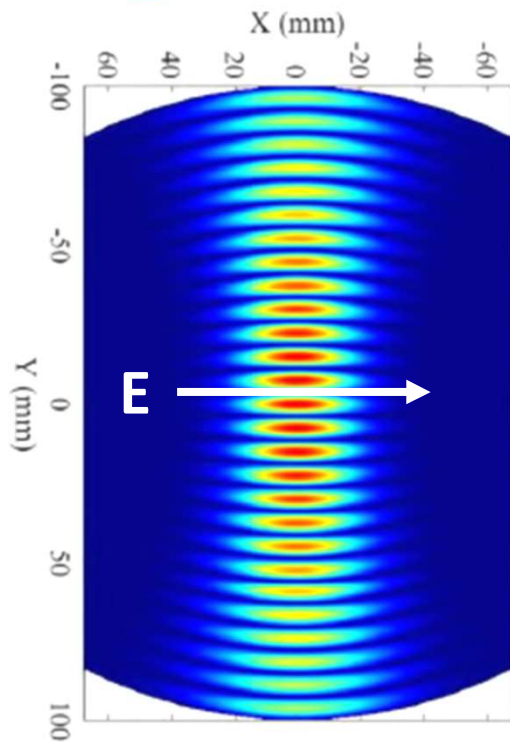
@ 95.06 GHz, air

@ 55.57 GHz, sample

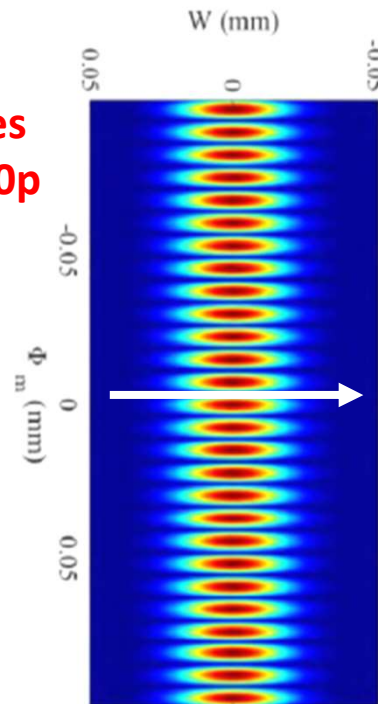
Envelope of  $|E|$  and  $H_{\phi}$  fields in log scale (-10 to -80 dB)



# Fabry-Perot Open Resonator (FPOR) – basics..



modes  
TEM<sub>00p</sub>



Fabry-Perot open resonator (FPOR, also called open-cavity)

Discrete frequencies between 20 GHz up to 110 GHz



..and modeling

- High measurement precision
- Can be sensitive to many user errors
- Uncertainty increases with increasing frequency
- Typically in-plane component of permittivity
- JIS R1660-2
- <https://www.qwed.com.pl/resonators.html#ResonatorFPOR>
- <https://www.keysight.com/main/editorial.jsp?cc=US&lc=eng&cke=y=2276755&nid=null&id=2276755>





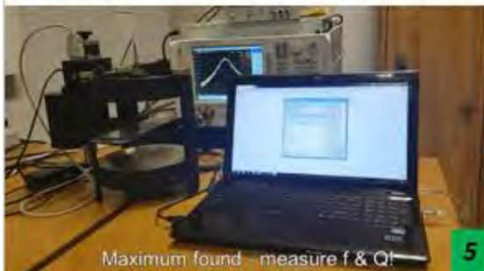
# Fabry-Perot Open Resonator (FPOR) - operation



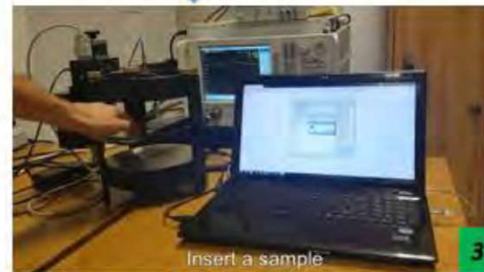
1. Connect the FPOR to VNA and PC with control app.

PC app invoked and controlled measurement – fully automatic  
Total measurement time: 10min

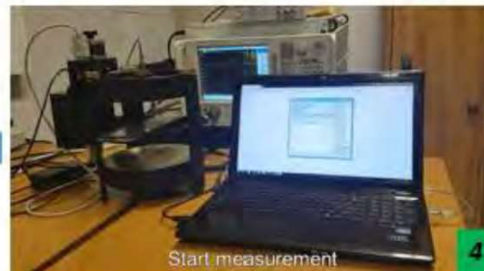
5. Material parameters at consecutive frequencies (modes) are extracted automatically



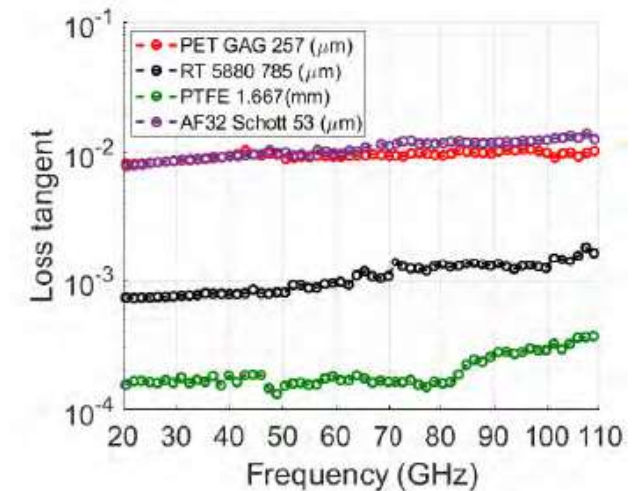
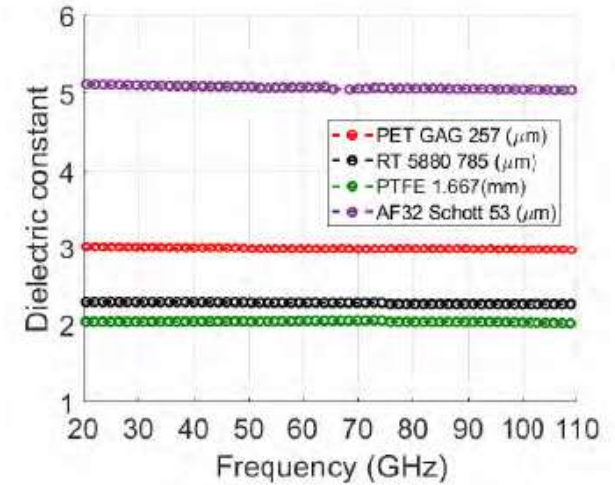
2. Measure "empty FPOR" (resonant frequency and Q-factor at M..N modes)



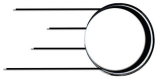
3. Insert the sample into FPOR



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

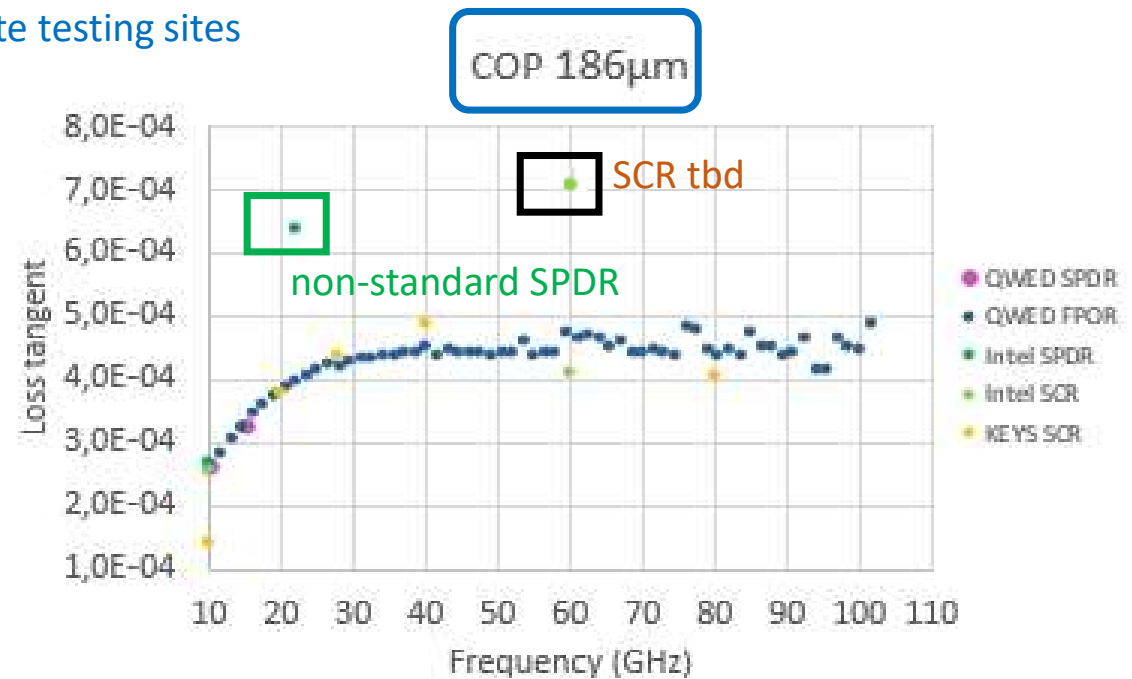
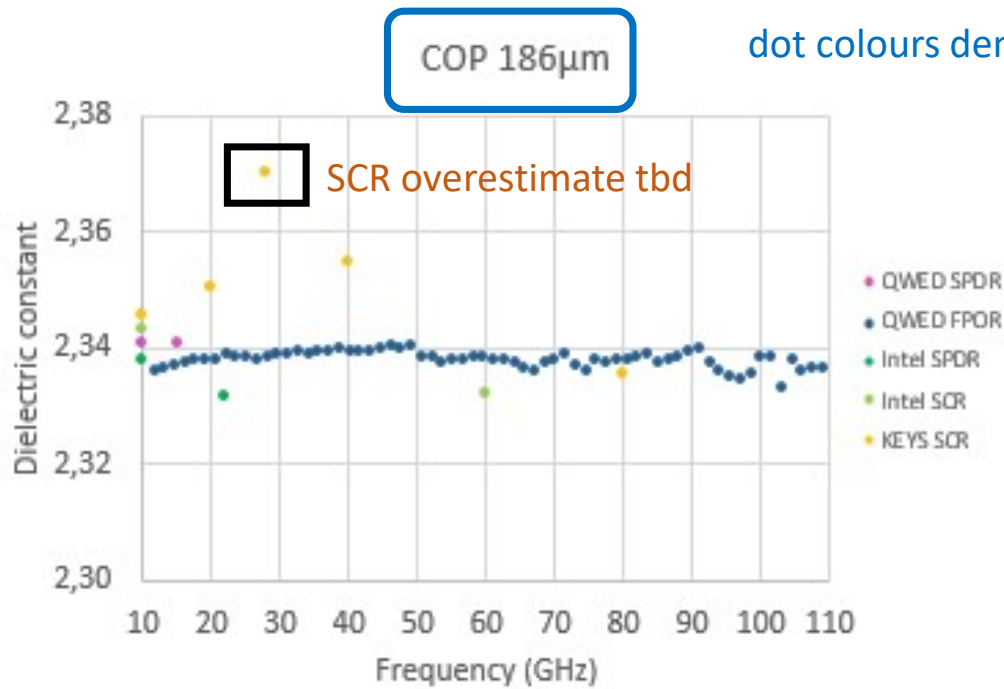


# Characterisation Results - Consistency



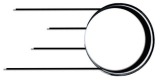
3 labs, 3 techniques, 14 laboratory setups

Intel - SCR at 10 / 60 GHz and SPDR at 10/ 20 GHz,  
 Keysight - SCR at 10 / 20 / 28 / 40 / 80 GHz  
 QWED - SPDR at 10/ 15 GHz and FPOR over 10-110GHz.



Dk spread < 1% (within  $\pm 0.5\%$  from average)  
 (< 2% incl. outliers)

> 40GHz 2x increase in Df compared to 10GHz



# Techniques selected for Task 3 if iNEMI 5G project (FPOR)

	Preferred techniques with sample dimensions			Optional
Technique	Split cylinder resonator (SCR)	Balanced-type circular disk resonator (BCDR)	Fabry-Perot open resonator (also called open cavity)	Split-post dielectric resonator (SPDR)
Sample dimensions	20 $\mu\text{m}$ ~ 300 $\mu\text{m}$ (best for 100 $\mu\text{m}$ ), 34 mm x 45 mm > 20G	0.1 mm ~ 1 mm, Best for 0.2~0.5 mm, 50 mm $\Phi$ x 2 each	0.050 – 3 mm, min. diameter: 75 mm max diameter: 150 mm	max 0.6 mm, min. 15 mm x 15 mm max 40 mm x 40 mm @15G

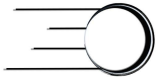
## Samples under test in Task 3:

35 mm x 45 mm

thickness: 50, 125, 188  $\mu\text{m}$

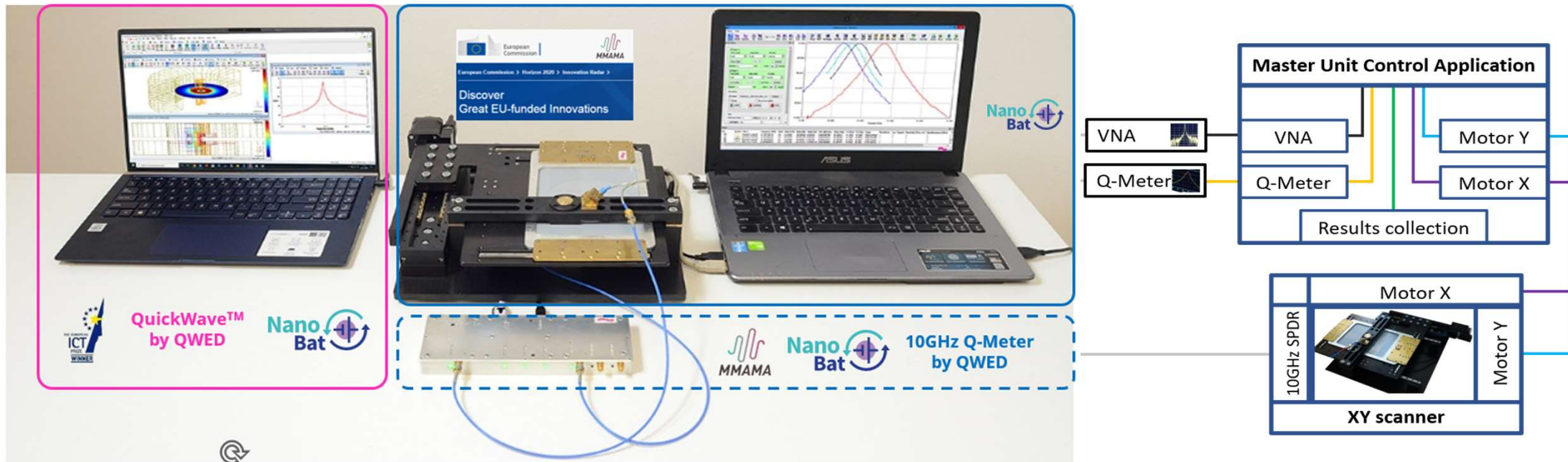
90 mm x 90 mm





# 2D Imaging of Low-Loss Dielectric Materials

2D scanner designed with a modified 10 GHz SPDR

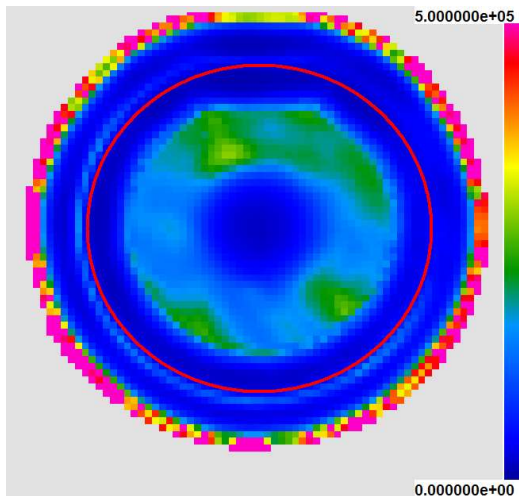
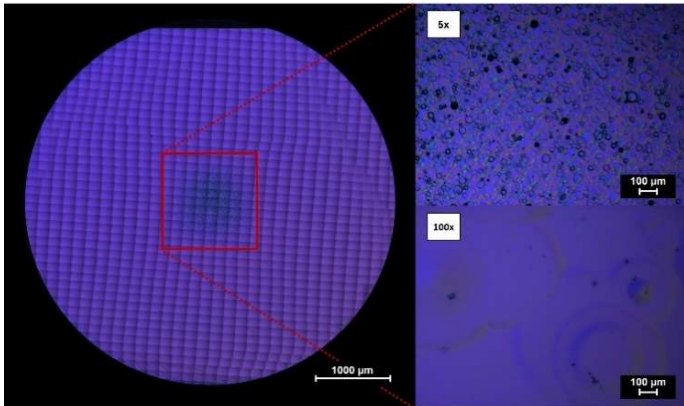


Finalist of the European Innovation Radar Prize 2021

## 2D SPDR Imaging of HR- GaN for Light & Power Electronics Devices

Optical microscopy image at L-IMiF reveals morphology inhomogeneity in the central area:

- in qualitative terms only,
- attributed to non-uniformity of the growth,
- only the central part appears useless for making devices.



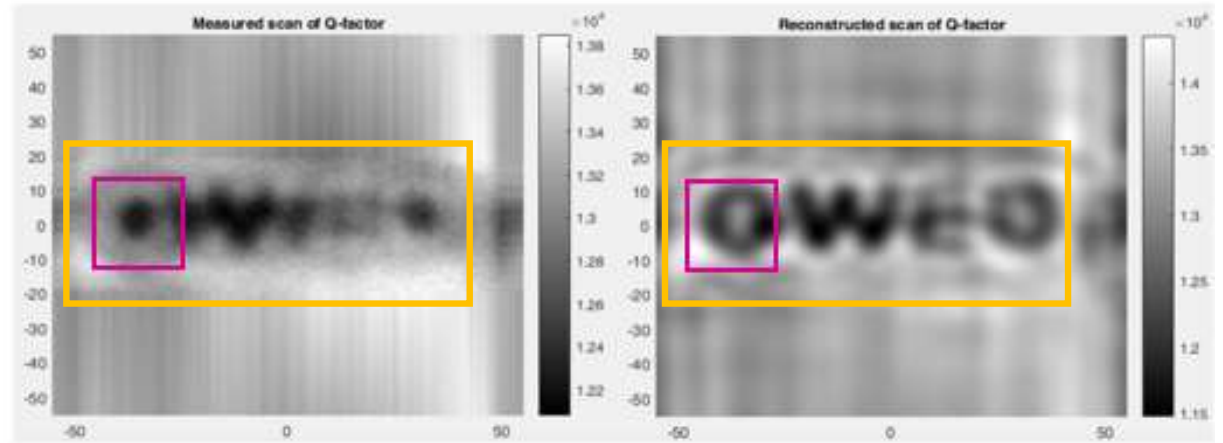
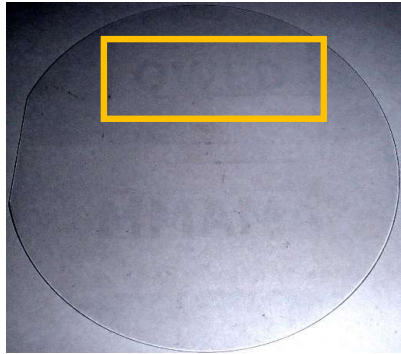
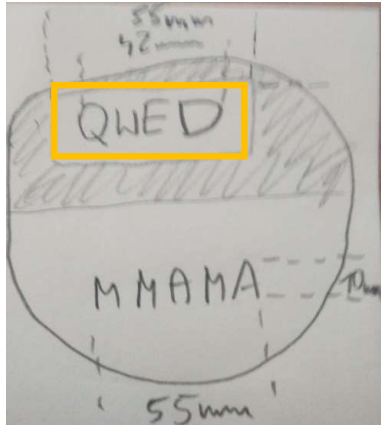
SPDR image:

- shows this whole GaN template unuseful,
- quantitative evaluation:
  - edge ring inherent to so-called edge effect,
  - ca.  $2 \cdot 10^4 \Omega\text{cm}$  in the centre (dark blue),
  - ca.  $5 \cdot 10^4 \Omega\text{cm}$  along the inner ring (light blue),
  - up to  $1.2 - 3 \cdot 10^5 \Omega\text{cm}$  across outer SUT's area (blue-green),
  - edge effect along the circumference.





# Modelling-Based Resolution Enhancement of Surface Images



raw image of sample resistivity  
(measured Q-Factor)

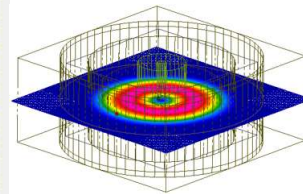
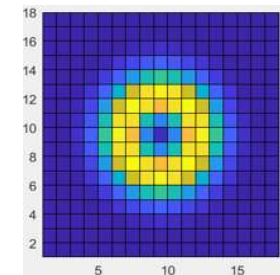
image further deconvolved  
using SPDR field pattern  
pre-simulated in QuickWave



Patterned PEDOT:PSS sample  
courtesy MateriaNova, Belgium

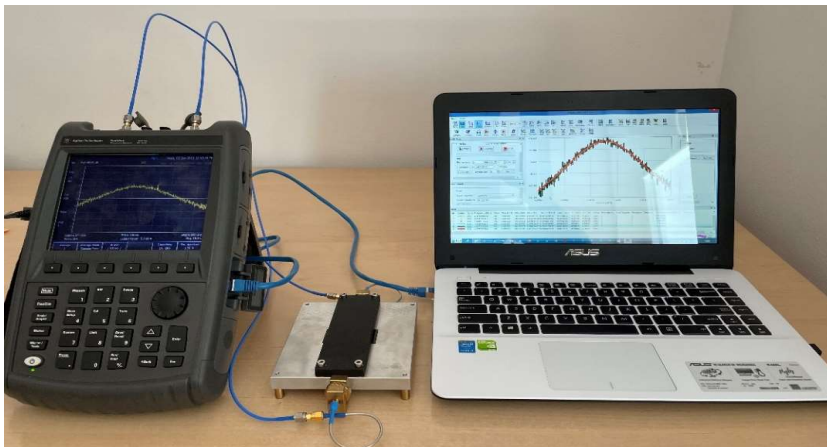
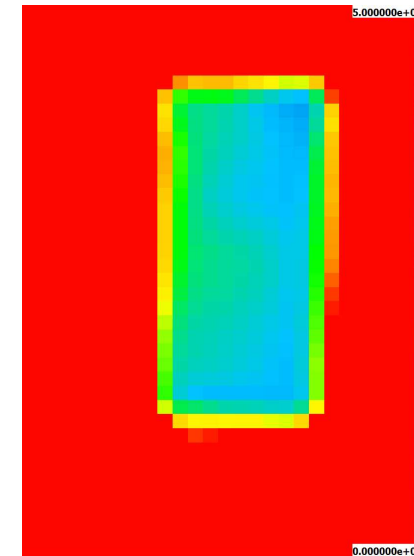
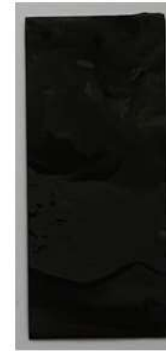


2D SPDR scanner





## 2D Imaging of Conductive Films – Application to Graphene Anodes



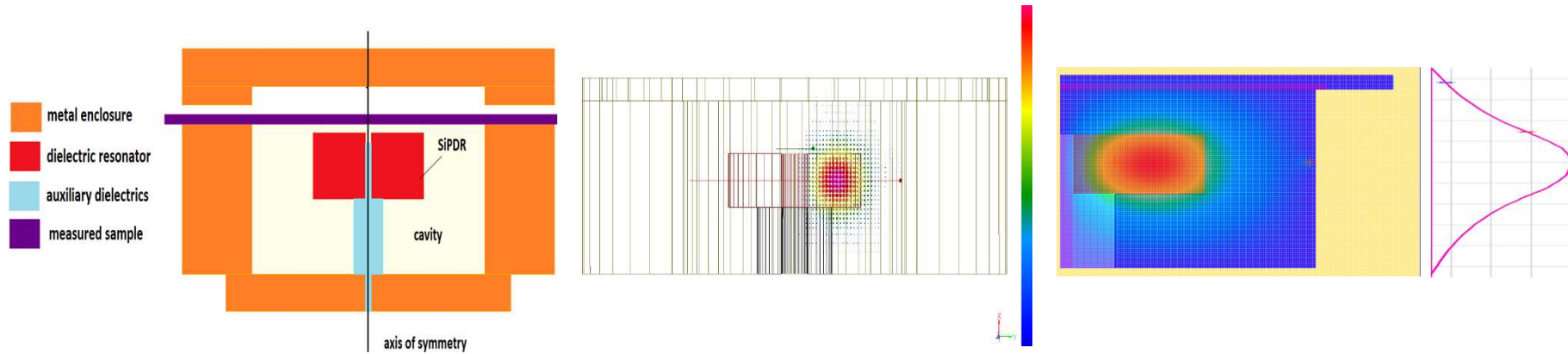
- ❑ Scanning area: 50 mm x 75 mm (25 mm margin around SUT)
- ❑ Uniform scanning step: 2 mm
- ❑ 1014 measurement points
- ❑ Avr thickness of the deposited graphene anode layer: 0.130 mm ± 0.02 mm
- ❑ Non-uniformities in  $R_s$  map due to sample thickness variation
- ❑  $R_s$  extracted for average thickness value
- ❑ An absolute value of  $R_s$  can vary within uncertainty of ± 15%
- ❑ Avr  $R_s$  of 19.3  $\Omega$ /sq. in exact agreement with point-wise 5GHz SiPDR device.



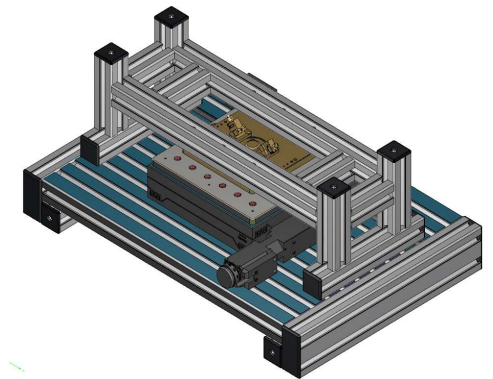
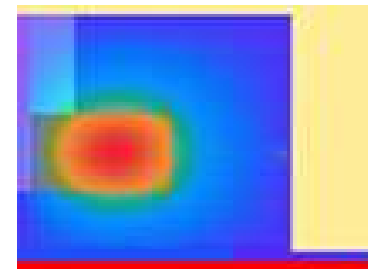
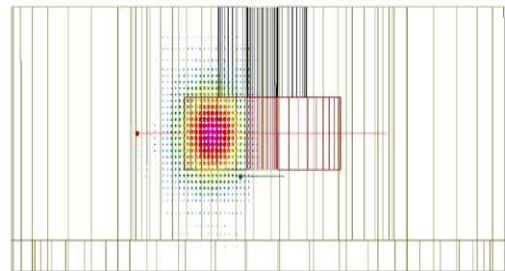
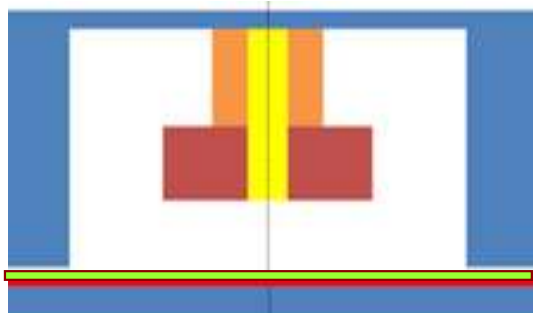


# 2D Imaging of Conductive Films – iSiPDR Scanner Design

Commercial 5GHz Single-Post Dielectric Resonator (SiPDR): schematics and E-field distribution



New 10GHz inverted SiPDR (iSiPDR) incorporated into 2D scanner



more sophisticated design & calibration:

**active sheet** facing the **DR head** → distance depends on the thickness of sample **substrate**



# 2D Imaging of Conductive Films – 10 GHz iSiPDR Scanning Setup

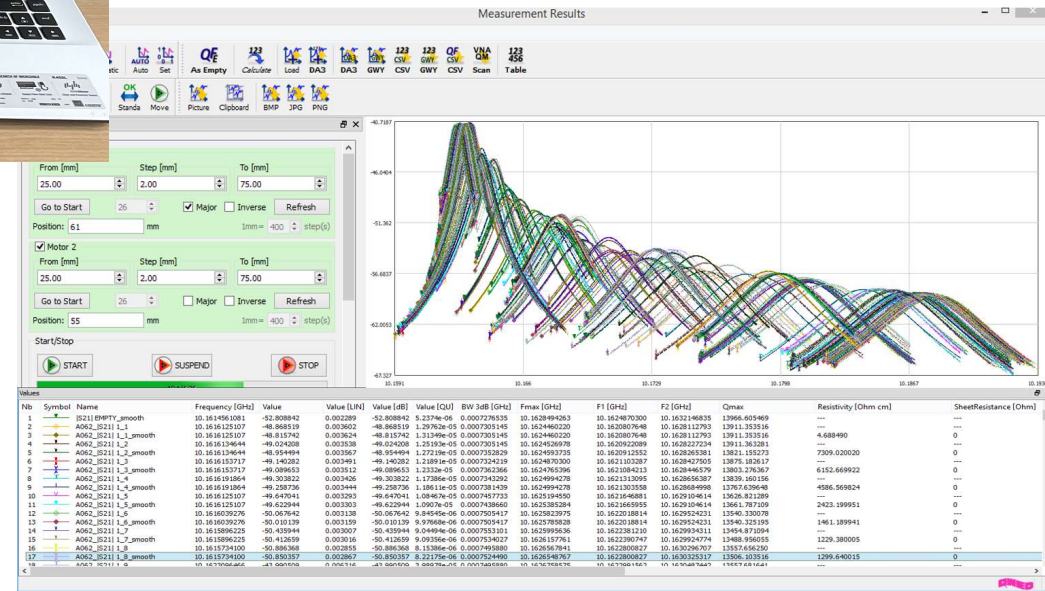


a family of  $|S_{21}|$  curves obtained in one scan

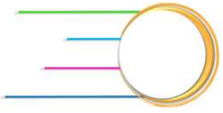
2D SiPDR scanner

Keysight FieldFox

Control App



# Concluding remarks



In the on-going iNEMI project, the four resonator methods (SCR, SPDR, BCDR, FPOR) are studied in terms of **accuracy**, **repeatability**, and **reproducibility**.

Each method has specific features, which can make it **preferable for a particular application** (e.g., different sample  $D_k / D_f$ , thickness, expected anisotropy; frequency & temperature range of use).

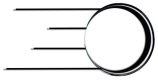
## Attention:

**Each resonator is just a passive test-fixture.**

All resonators must be used with VNA (*in some cases, scalar analyser is sufficient*).

Using the same resonator used with different network analysers (and signal processing software) may lead different results.

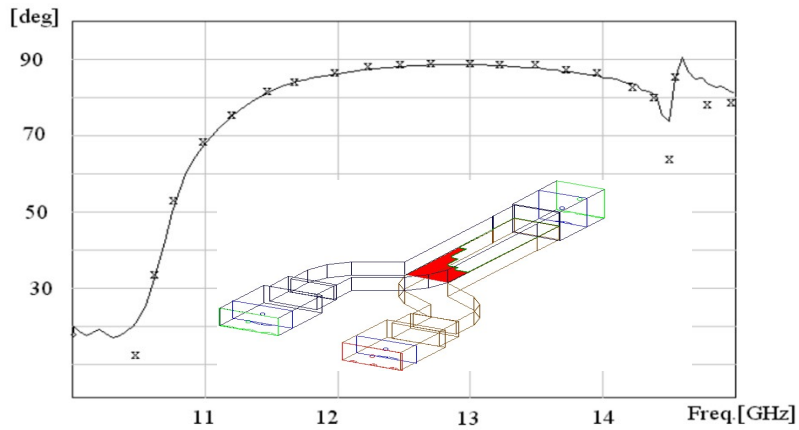
# QuickWave original applications in cosmic research & satellite telecommunication



## Septum polariser by SES

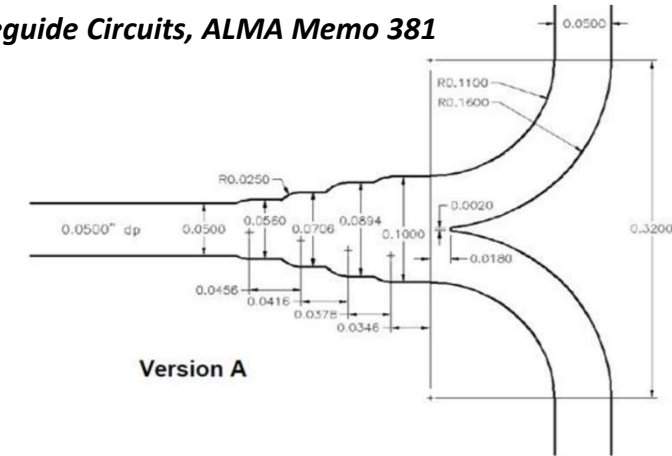
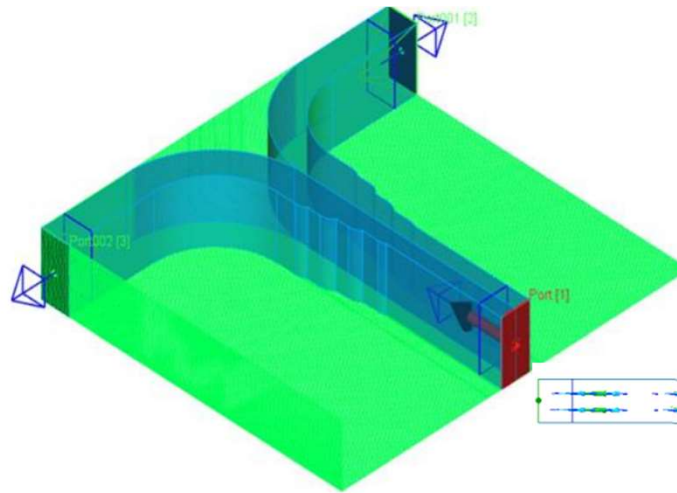
design & measurements: Saab Ericsson Space  
modelling: QWED, 1997

below: differential phase-shift

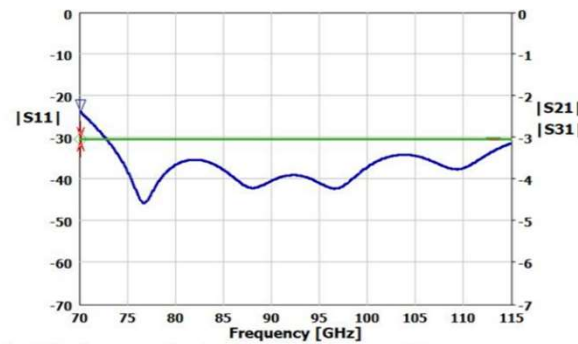
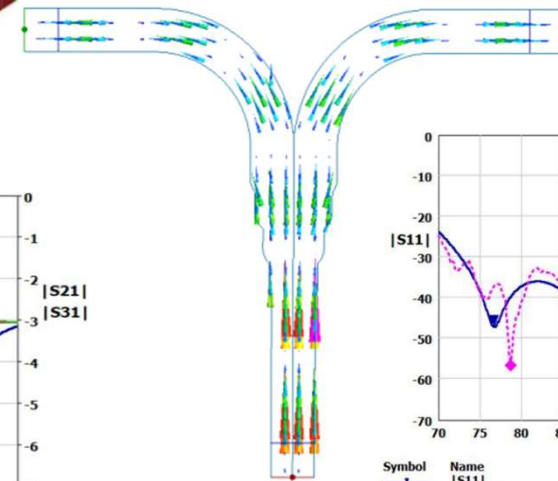


## E-plane Y-junction by NRAO

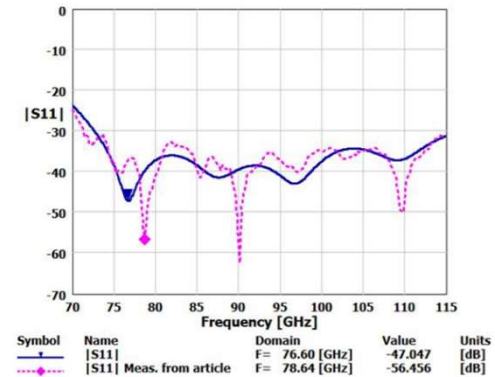
after A. R. Kerr, Elements for E-Plane Split-Block Waveguide Circuits, ALMA Memo 381



Version A

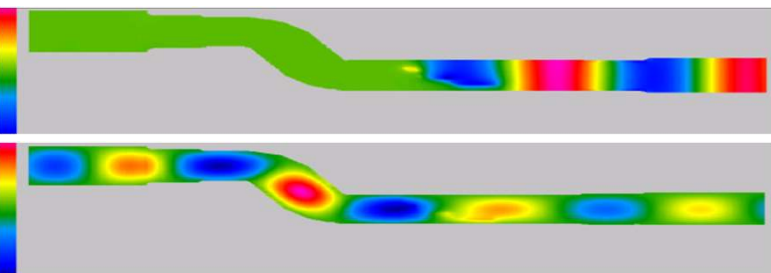


Symbol	Name	Domain	Value	Units
—	S11	F= 70.00 [GHz]	-23.587	[dB]
—	S21	F= 70.00 [GHz]	-3.011	[dB]
—	S31	F= 70.00 [GHz]	-3.012	[dB]

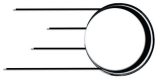


Symbol	Name	Domain	Value	Units
—	S11	F= 76.60 [GHz]	-47.047	[dB]
—	S11  Meas. from article	F= 78.64 [GHz]	-56.456	[dB]

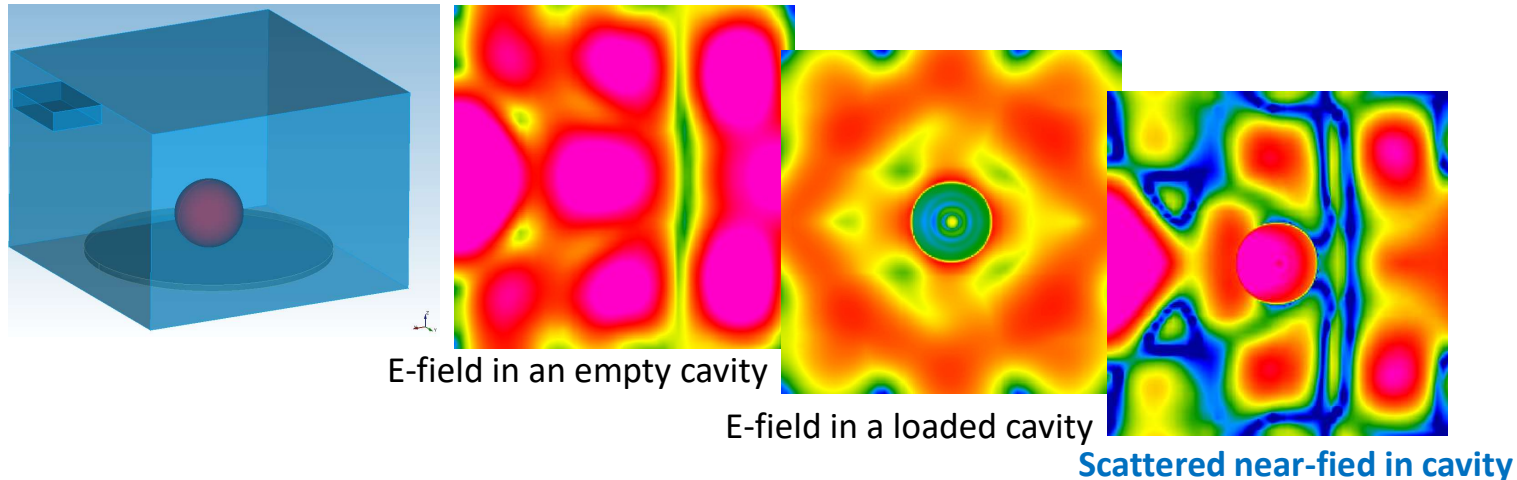
propagation of two polarisations  
at centre frequency



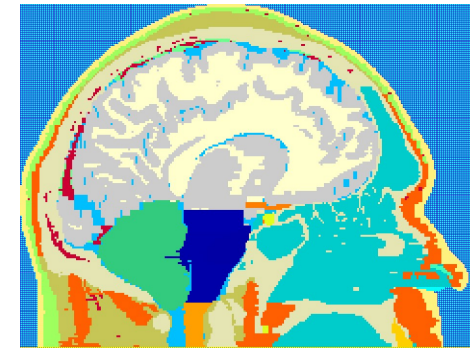
# QuickWave modelling EM field interaction with tissues (for food processing & medical applicators)



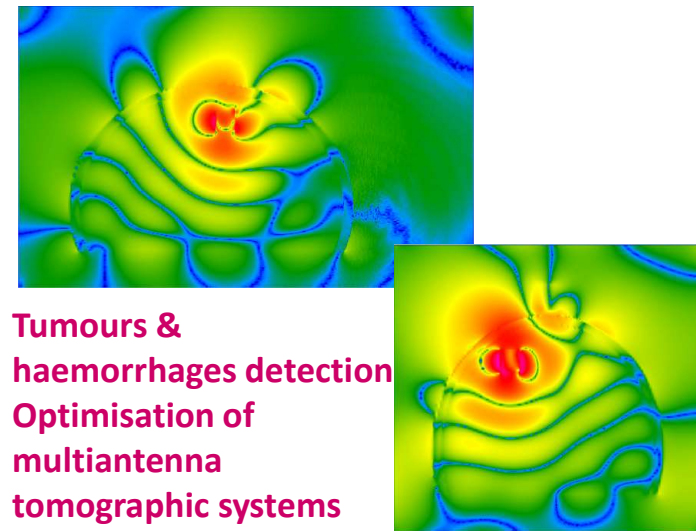
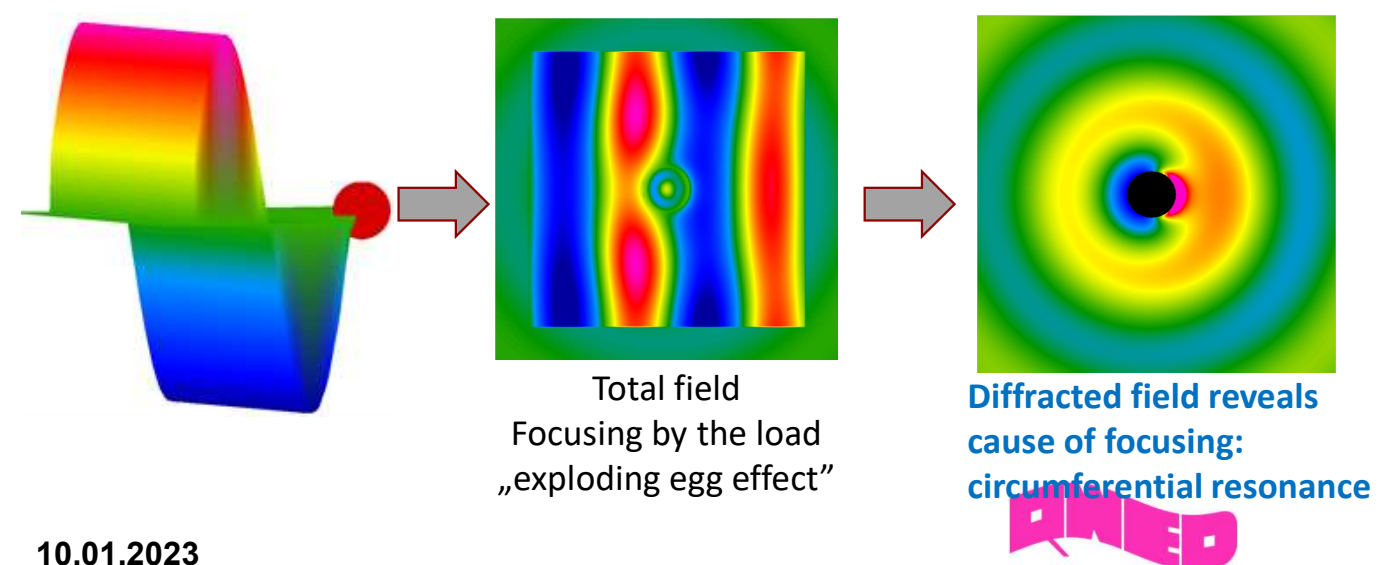
Separation of incident and diffracted fields (*option implemented per request of P.O.Risman, Malardalen University*)



## Detection of inhomogeneities in tissues



AustinMan model\* converted to QuickWave EM software for Mälardalen University, Sweden



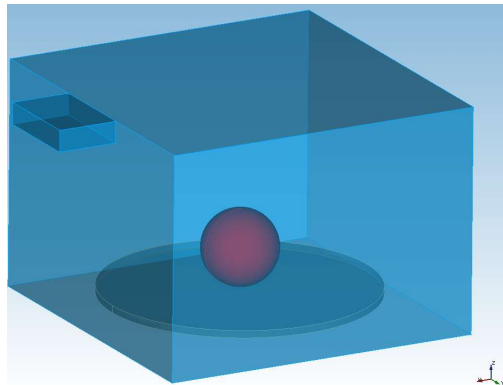
- ✓ Tumours & haemorrhages detection
- ✓ Optimisation of multi-antenna tomographic systems

# Illustration & cross-verification of QuickWave Multiphysics Regimes in Elsevier Book

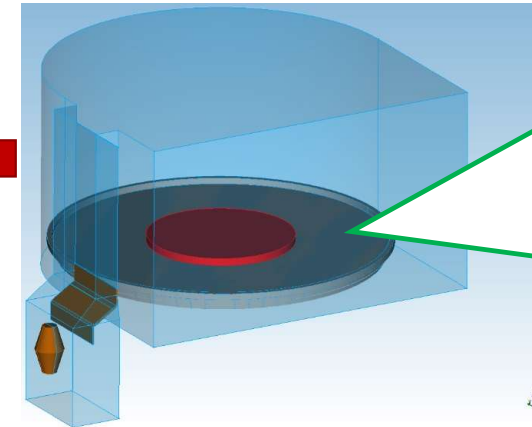


Simple microwave heating benchmarks  
& microwave heating phenomena studies\*

Design & analysis of real-life microwave oven cavities, incl.  
complicated cavity shapes and advanced feeding system\*



- heat transfer & load dynamics
- Load rotation & arbitrary movement during heating
- Source parameters tuning – regime for solid state sources
- Temperature dependence of material parameters



HFSS v11

Temperature(C)
1.0000e+002
5.9270e+001
8.0573e+001
6.1057e+001
7.7330e+001
7.1820e+001
6.0730e+001
6.0000e+001
6.9260e+001
6.0573e+001
4.2057e+001
6.0573e+001
3.7430e+001
3.8130e+001
2.0730e+001
7.0000e+001

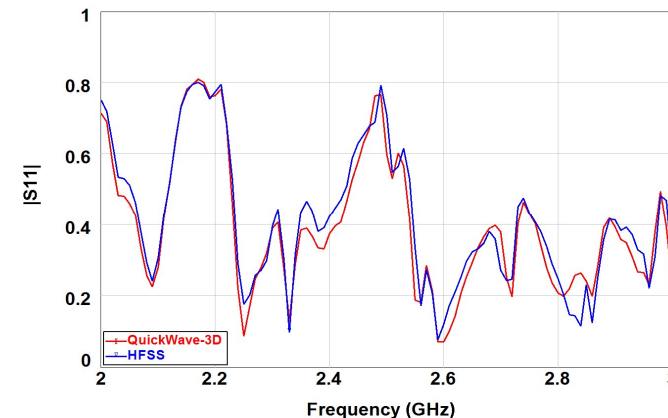
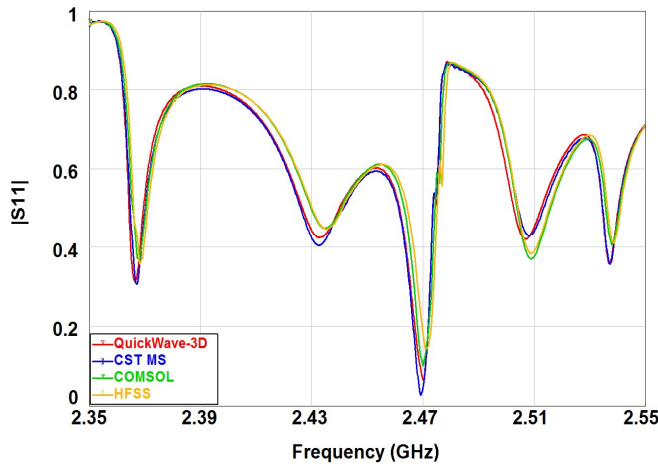
QuickWave 3D & BHM

(deg C)  
100  
-20

Freezing to file  
the state of the  
simulation

De-freezing on  
arbitrary computer  
& at convenient  
time

Courtesy of Whirlpool Inc. – Whirlpool MAX oven



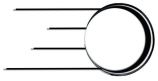
With QuickWave EM  
computation as fast as  
**1 min 18s** on a **low-cost  
video card** – supporting  
**all graphic cards with  
OpenCL**

\* M.Celuch, P.Kopyt & M. Olszewska-Placha in eds. M. Lorence, P. S. Pesheck, U. Erle, *Development of packaging and products for use in microwave ovens*, 2nd Ed. Elsevier 2020.

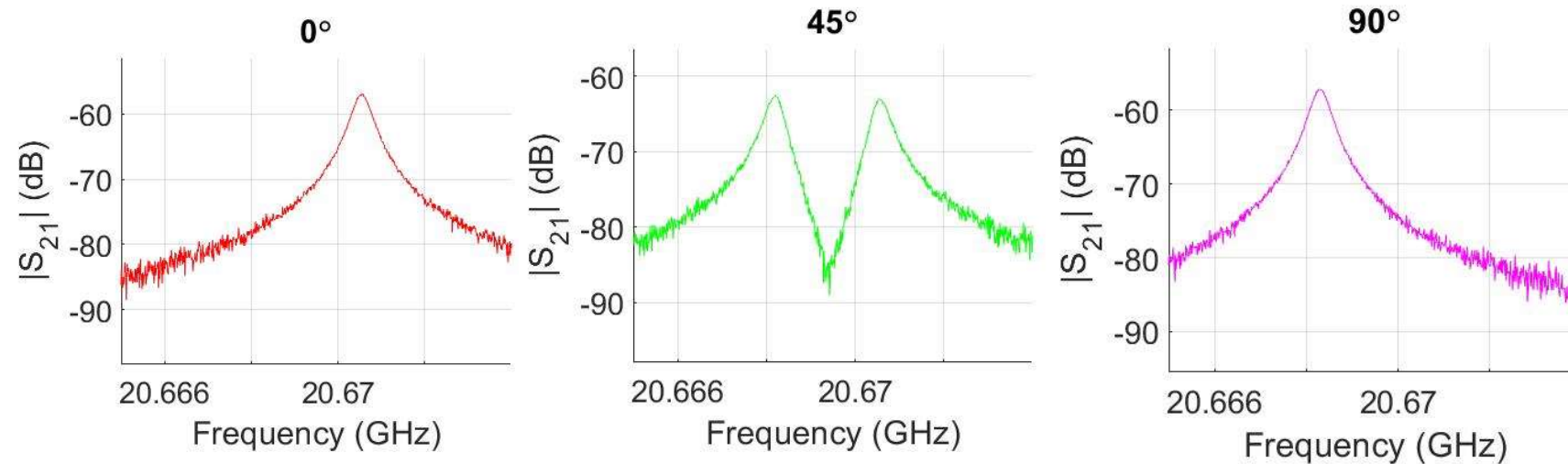




# Fabry-Perot Open Resonator (FPOR) - operation



With appropriately designed feeding loops, FPOR is capable of **linear E-field polarization** and hence **detecting in-plane anisotropy**:



Resonances detected for **BoPET** sample ( $t = 0.100$  mm), turned in xy plane.

**BoPET** (biaxially-oriented PET) involves thermal drawing in two in-plane directions with substantially different draw ratios, followed by **crystallization**. Hence, it is **in-plane anisotropic**.

For **PETG** (non-crystalline copolyesters, **isotropic**), resonant frequency does not depend on angular position of the sample.

T.Karpisz et al, " Measurement of in-plane anisotropy of dielectric materials with a Fabry-Perot open resonator", Proc. MIKON 2020

