A Fast Modelling-Based Technique for the Characterization of Graphene-Based Polymer Composites
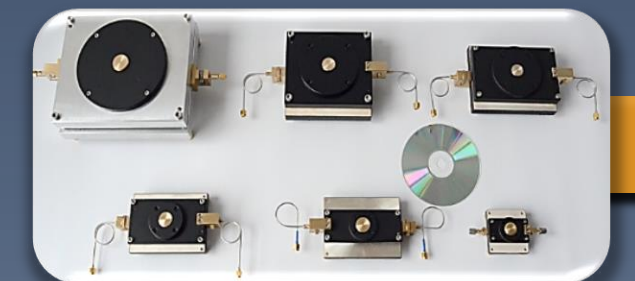
MEMO 2023

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QuickWave™
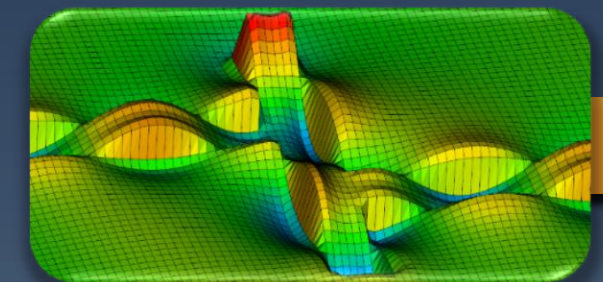
Materials Measurement

R&D Projects
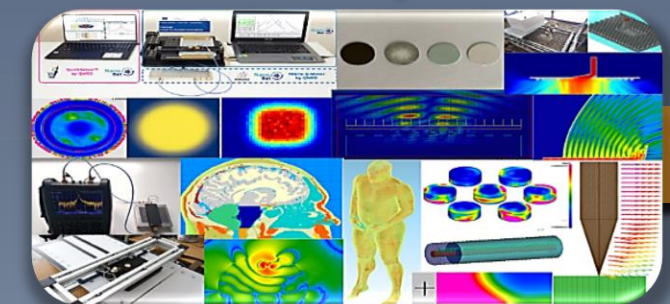
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Outline

1. Two Dimensional inverted Single-Post Dielectric Resonator – iSiPDR

2. Sample under Test – Thermoplastic polimer composite (ABS/GNP)

3. Characterization and Retro-modelling

4. Results and discussion

5. Conclusions

2D iSiPDR

Non-destructive and contactless materials characterization

Fig. 1 Measurement setup for 2D imaging of Graphene-Based Polymer Composites: 2D iSiPDR 10GHz scanner and dedicated Q-Meter

Design: Inverted configuration (iSiPDR) with a sample holder and incorporation into a movement mechanism.

Sample placement: Sample under test (SUT) is placed under the dielectric resonator post at the bottom of the cavity.

Frequency: **10 GHz.**

Working slot: Allows insertion of SUTs of up to **1 mm thickness.**

Scanner components: Standa 2D motorized translation stage with an incorporated metal reference plate as a sample holder.

Scanning procedure: Metal plate with a sample placed upon is moved over a grid of points with a **spatial resolution** varying in practice in the range of **0.5 – 5 mm**, depending on the SUT lateral size and requested accuracy of the surface imaging.

Scanning area: Spans over a **100 x 100 mm** surface, covering typical sizes of available material wafers.
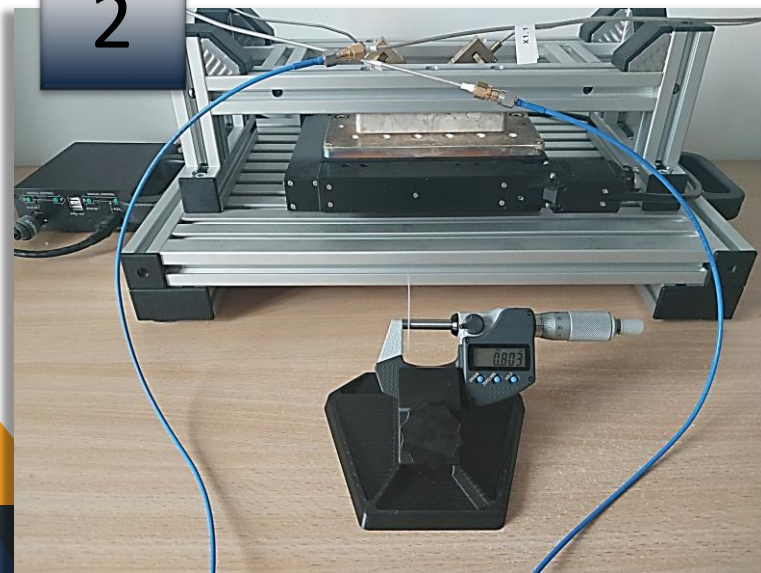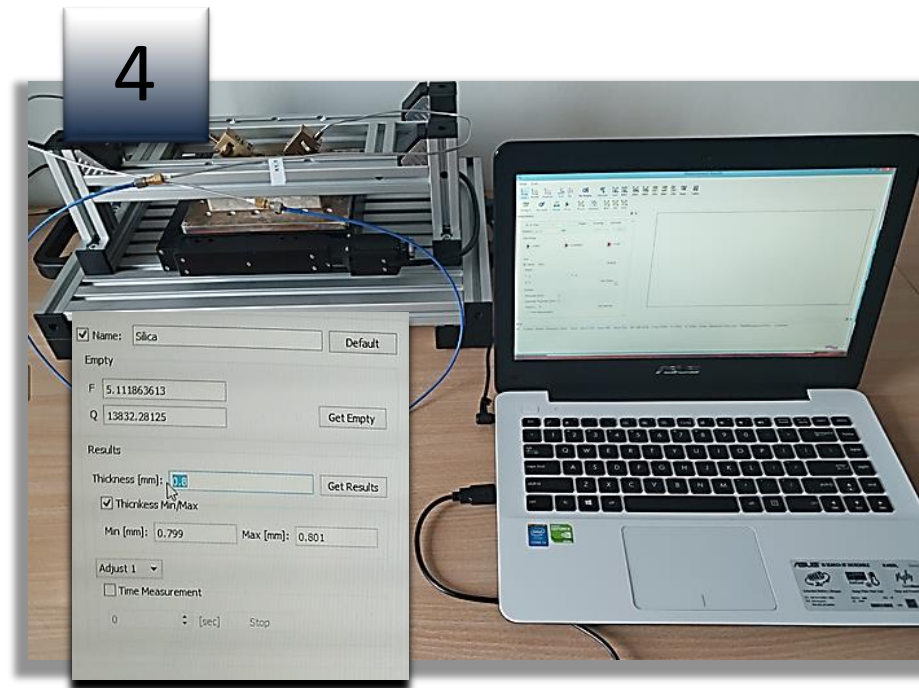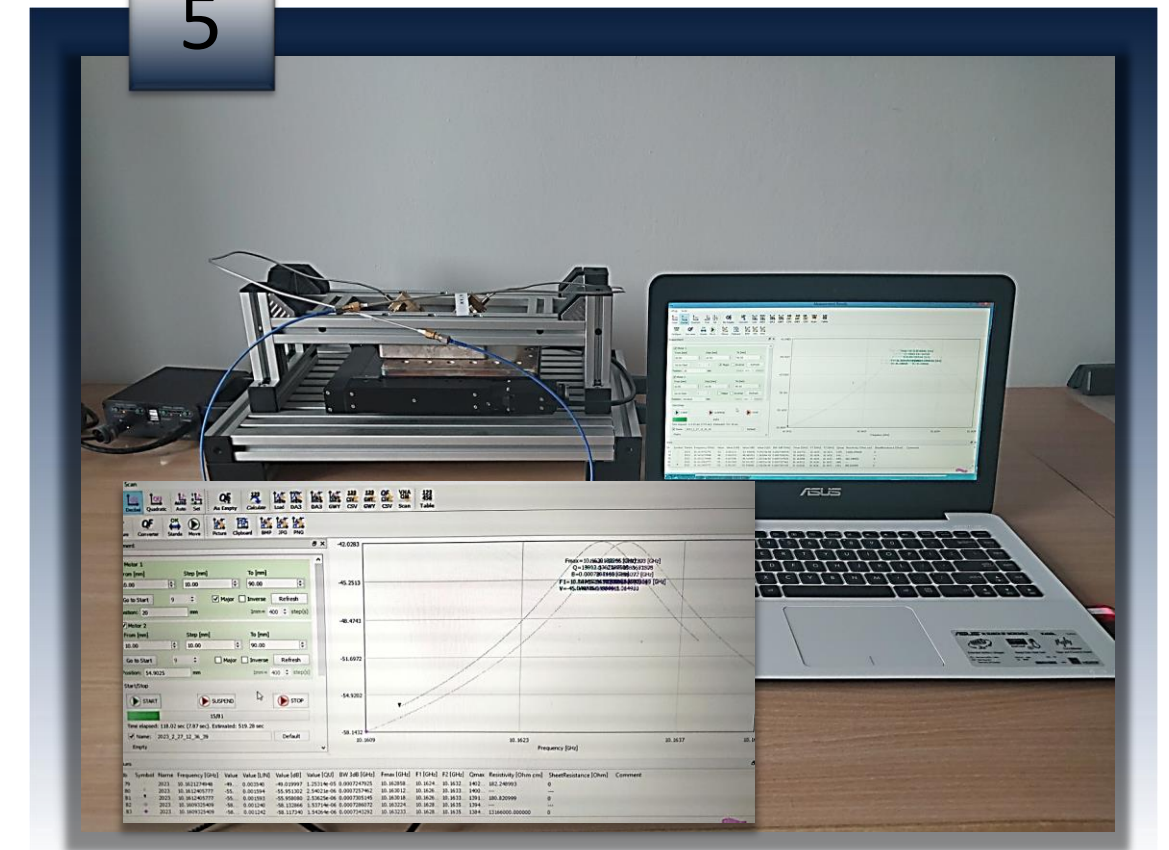
2D iSiPDR

Measurement 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 - app invoked measurement.

2. Measure thickness of the sample.

3. Insert the sample into iSiPDR.

4. Insert the sample thickness into the PC app.

5. Material parameters are extracted automatically with each step.

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Sample under Test

Thermoplastic polimer composite

Acrylonitrile–butadiene–styrene (ABS) with graphene nanoplatelet (GNP)
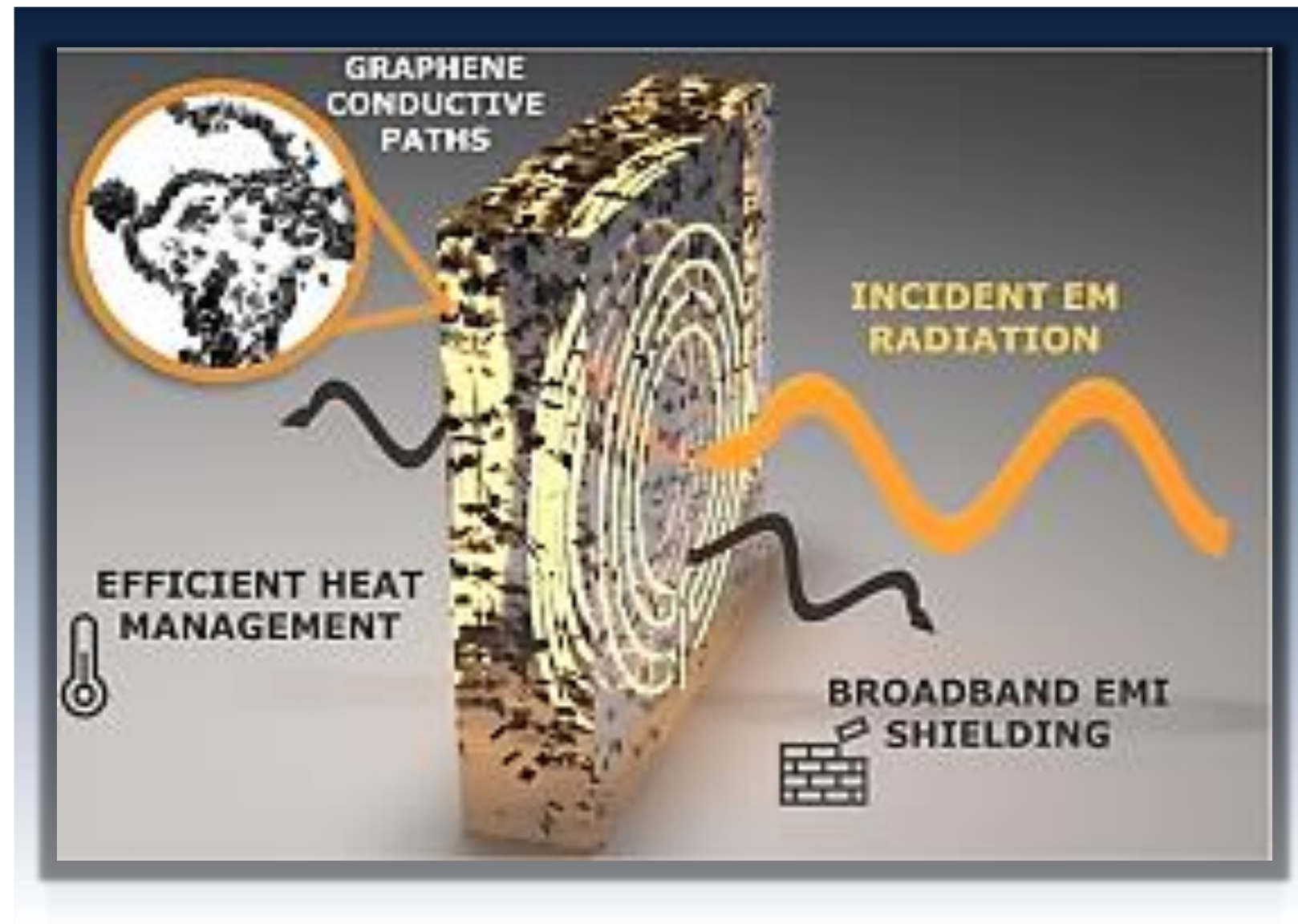
Resinex Poland supplied ABS in a powder form that had a melt flow rate of 43 g/10 min (220 °C/10 kg) and a density of 1.04 g/cm³ according to the provided technical datasheet. Sigma-Aldrich provided GNPs in the form of a powder with an average lateral dimension of 25 μm and a surface area of 120-150 m²/g. To fabricate ABS/GNP composites, the graphene powder was first mixed with ABS.

Hot press method was then used to fabricate nanocomposite samples. The prepared dried mixture was placed in a hydraulic press, where the mold temperature was set at 290 °C, and a pressure of 20 MPa was applied. As a result, composite samples were obtained.
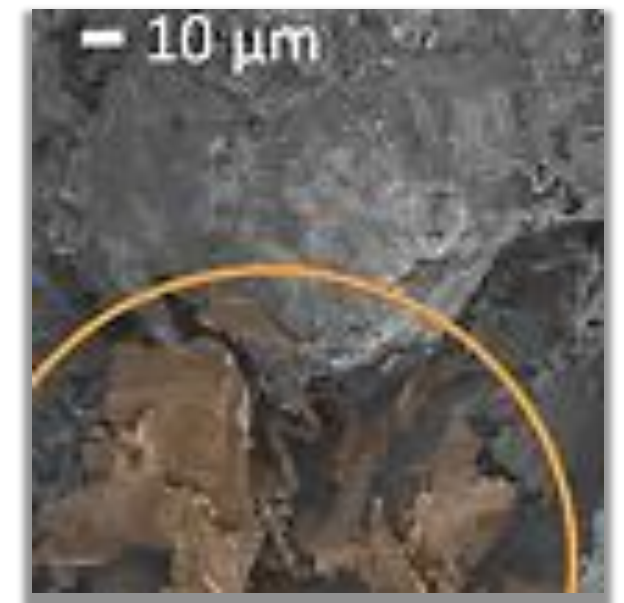
„Graphene-Based Thermoplastic Composites as Extremely Broadband and Frequency-Dependent EMI Absorbers for Multifunctional Applications"

Klaudia Żerańska-Chudek, Karolina Filak, Konrad Wilczyński, Agnieszka Siemion, Norbert Pałka, Konrad Godziszewski, Yevhen Yashchyshyn, Mariusz Zdrojek

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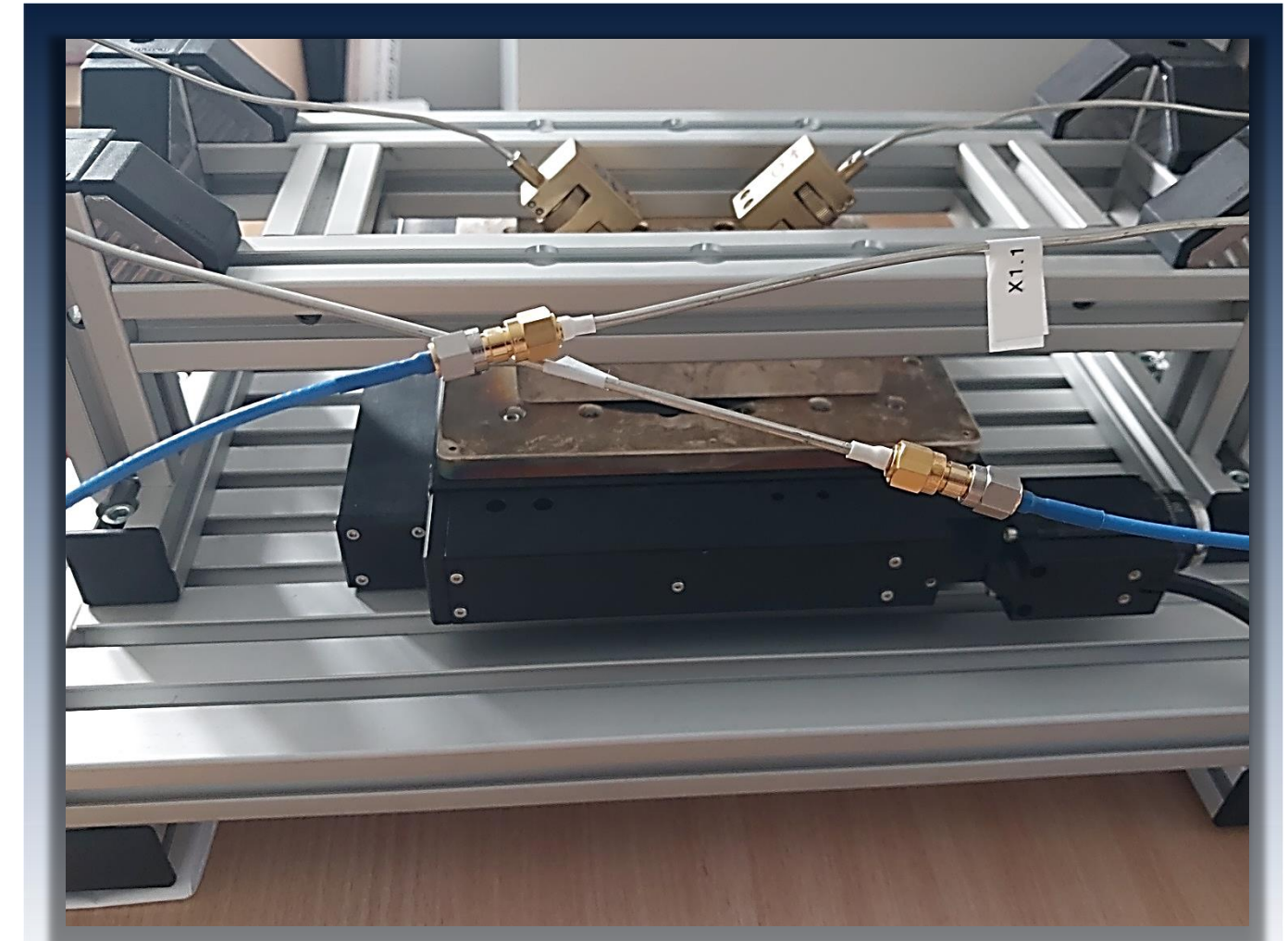WARSAW UNIVERSITY OF TECHNOLOGY

Sample under Test

Setting up the measuring system

ABS/GNP

Average thickness: 429.9 [μm]
Maximum: 480 [μm]
Minimum: 362 [μm]

Insert the sample
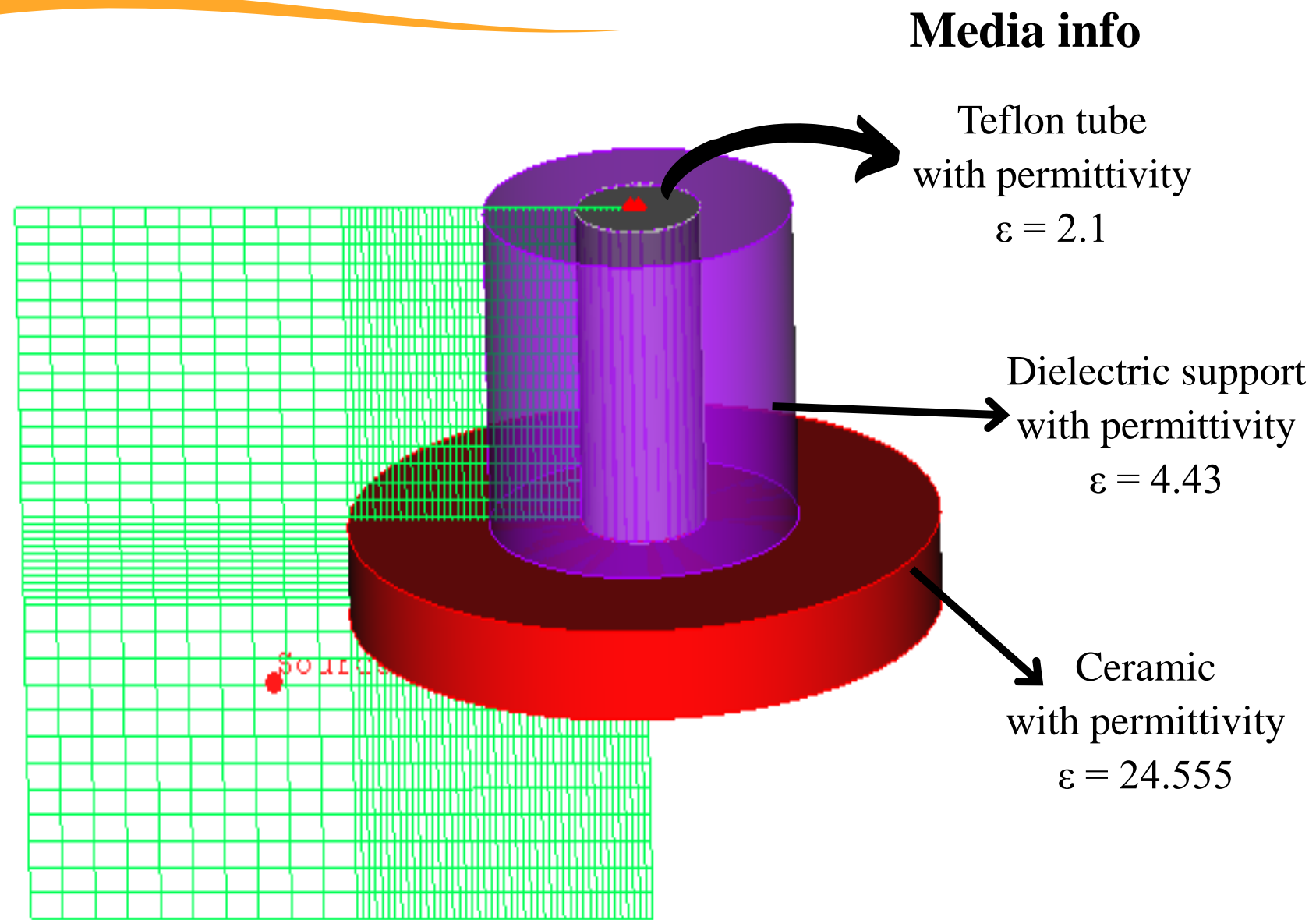into iSiPDR

Measurement setup for 2D imaging of graphene-based
polimer composites

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Retro-modelling

iSiPDR modelling

Media info

Teflon tube
with permittivity
$\varepsilon = 2.1$

Dielectric support
with permittivity
$\varepsilon = 4.43$

Ceramic
with permittivity
$\varepsilon = 24.555$

3D view of the iSiPDR in FreeCAD QW Modeller,
which automatically generates 2D BoR mesh.

Fig. 2 Dimensions of the iSiPDR
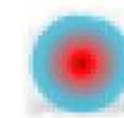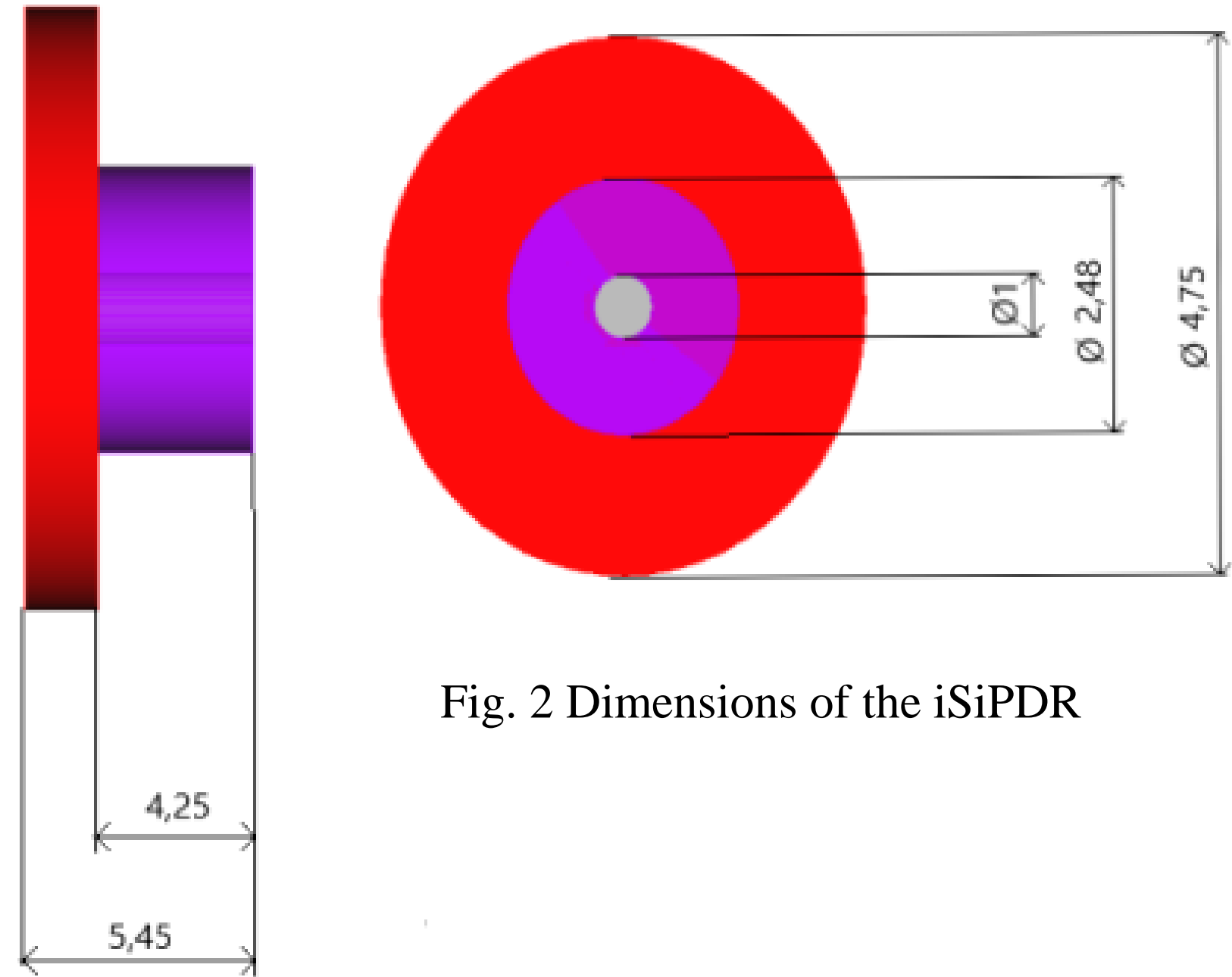
Source parameters

Point source excited by Eφ field with frequency
from **5** to **11** GHz.

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Retro-modelling

iSiPDR modelling

E Total electric field **H** Total magnetic field

V2D

Fig. 3 Vector display of electric field
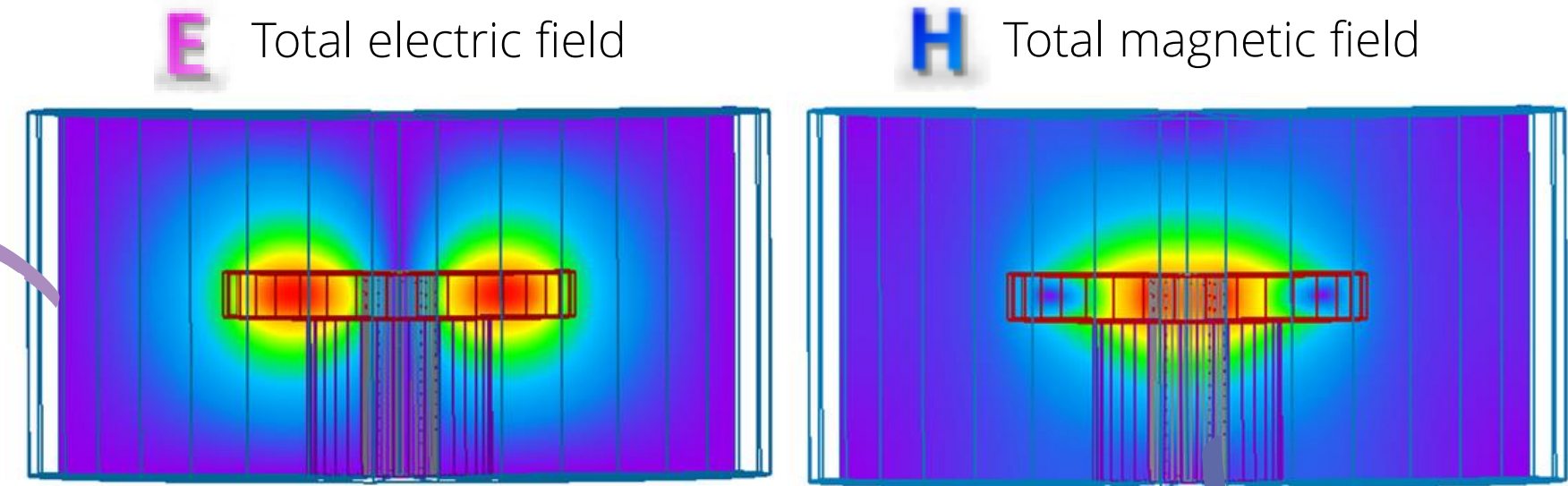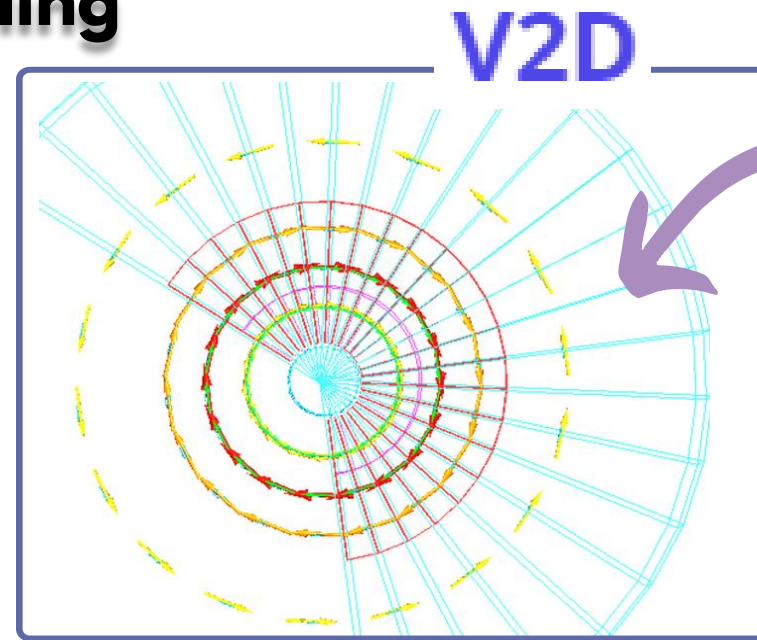
Tab. 1 SiPDR simulation parameters for different types and meshing

Simulation Type	Simulation Time (min:sec)	Simulation iteration	Number of cells			Cells with boundaries	Cells per wavelength	Used RAM
			X	Y	Z			
3D	82:06	4665941	17	17	7	3528	5	344 kB
3D + QProny	8:01	100000	111	109	38	506805	80	48 MB
V2D Body of Revolution	0:41	123235	17	22		1092	15	106 kB
	2:19	223878	31	42		3220	30	314 kB
	2:53	425289	61	82	1	11180	60	1 MB
	7:42	853361	120	164		41664	120	4 MB
	19:45	1743020	238	325		159236	240	15 MB
V2D + QProny	2:01	100000	104	125		27864	120	3 MB

V2D
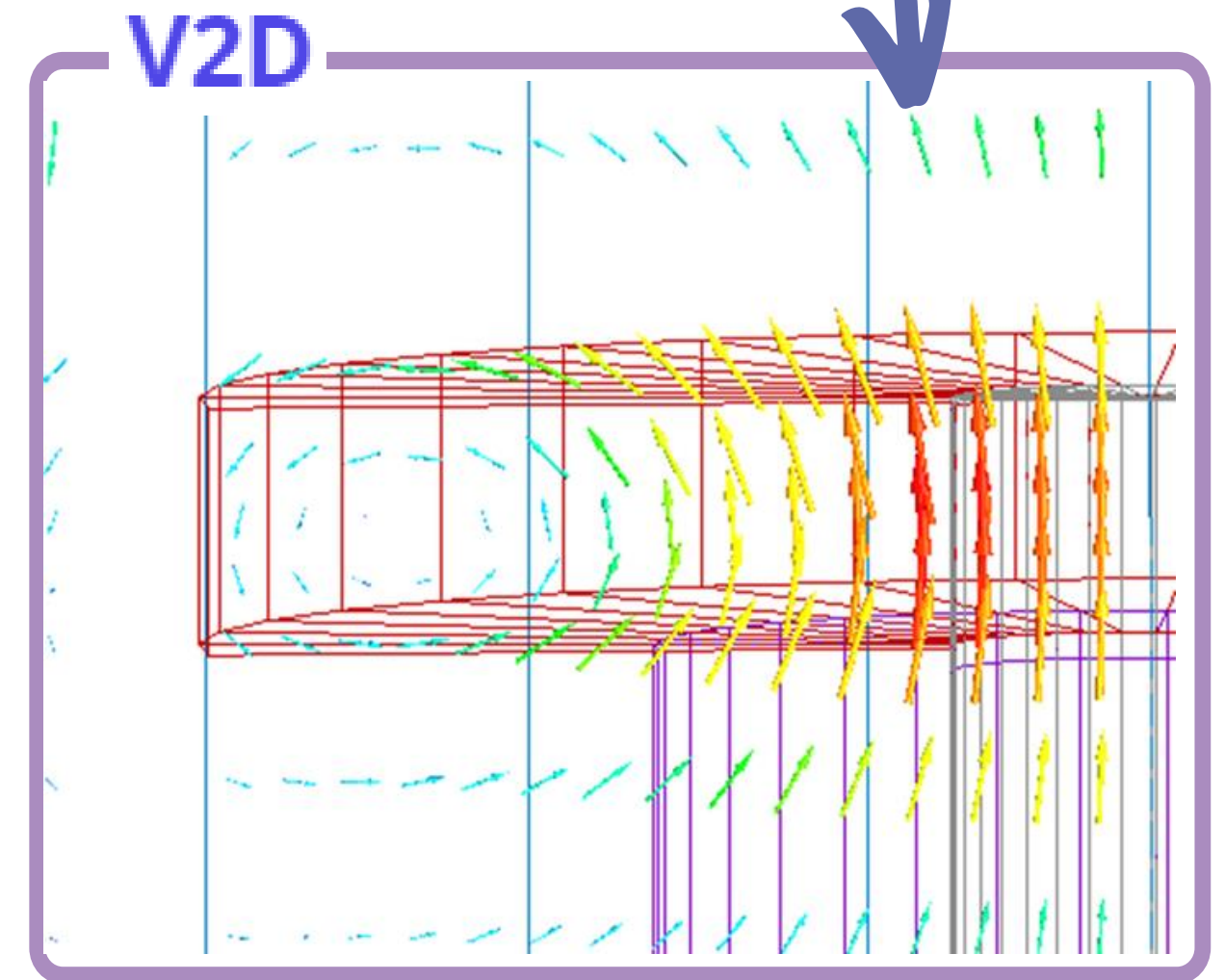
Fig. 4 Vector display of magnetic field

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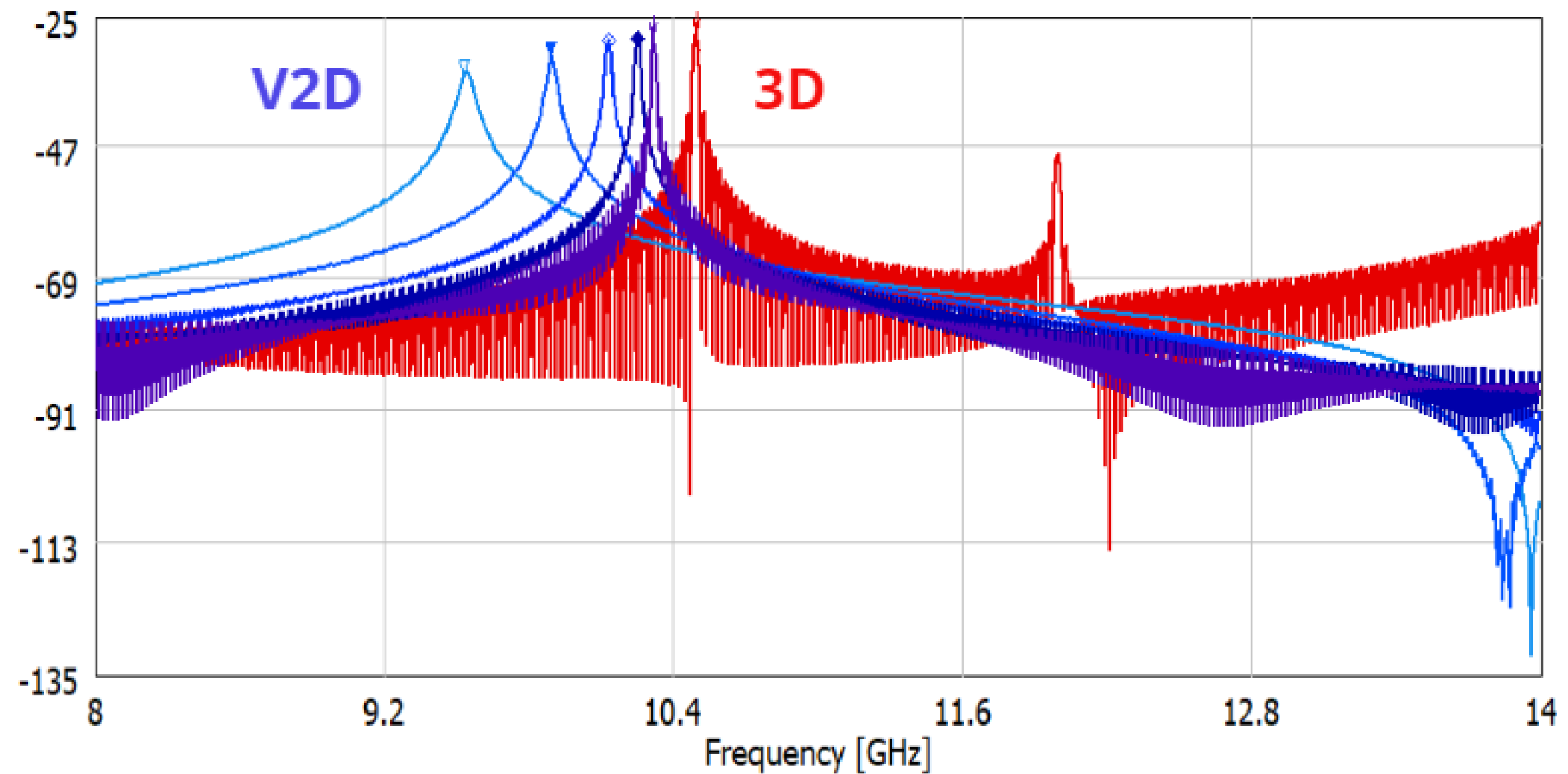WARSAW UNIVERSITY OF TECHNOLOGY

Retro-modelling

iSiPDR modelling

Fig. 5 Transmission |21| of SiPDR for different types and meshing, As the number of cells increases, the difference between the frequencies in BoR (V2D) gets smaller and smaller until they overlap

Retro-modelling

Optimizing Simulation process

Grid Search

Results

V2D + QProny

Thickness++ Eps++

Simulation

1. Resonance Frequency – First insight.

2. Quality factor extraction, additional frequency check (more accurate).

3. Change permittivity of the sample by 1% .

4. Change permittivity of ceramics by 1%.

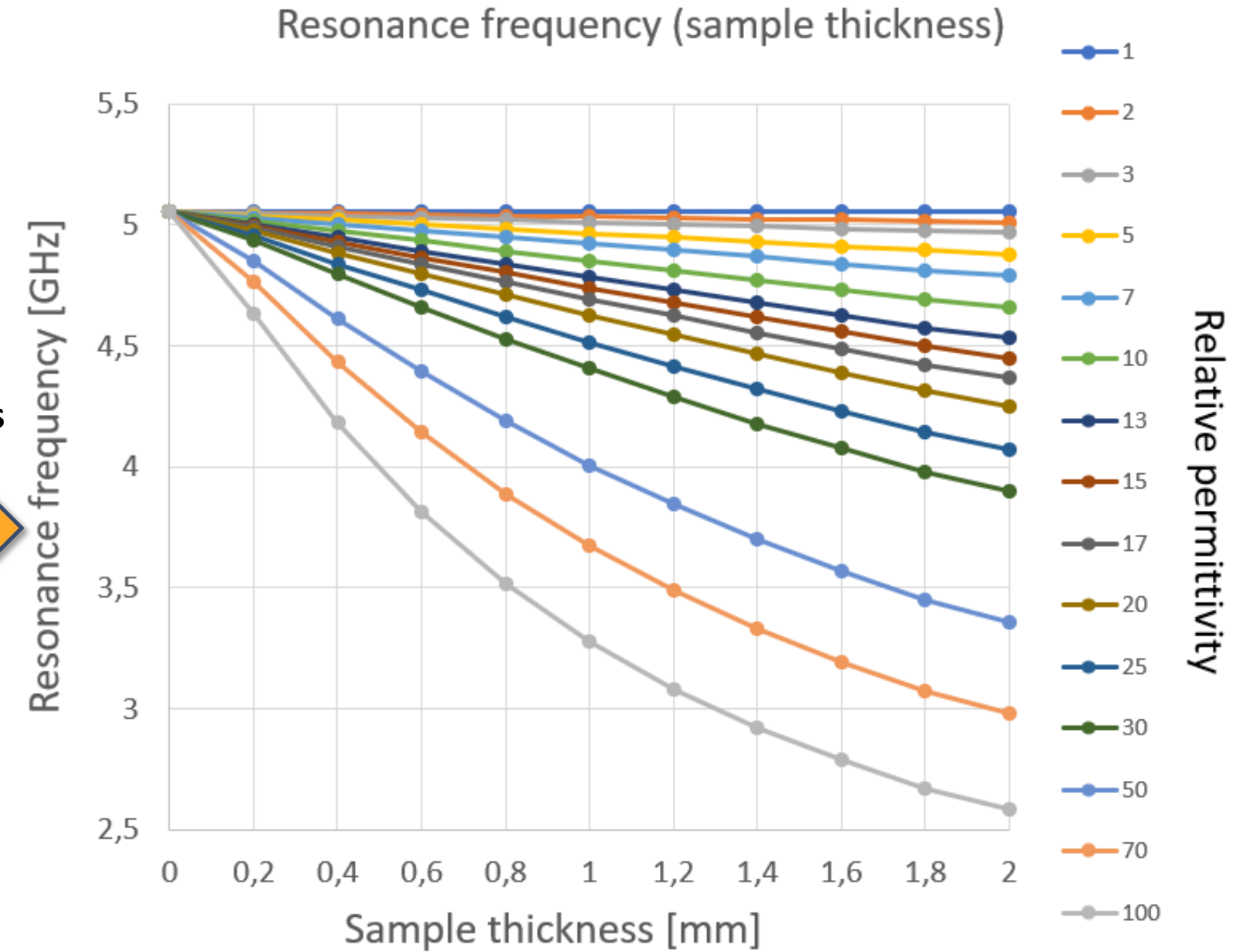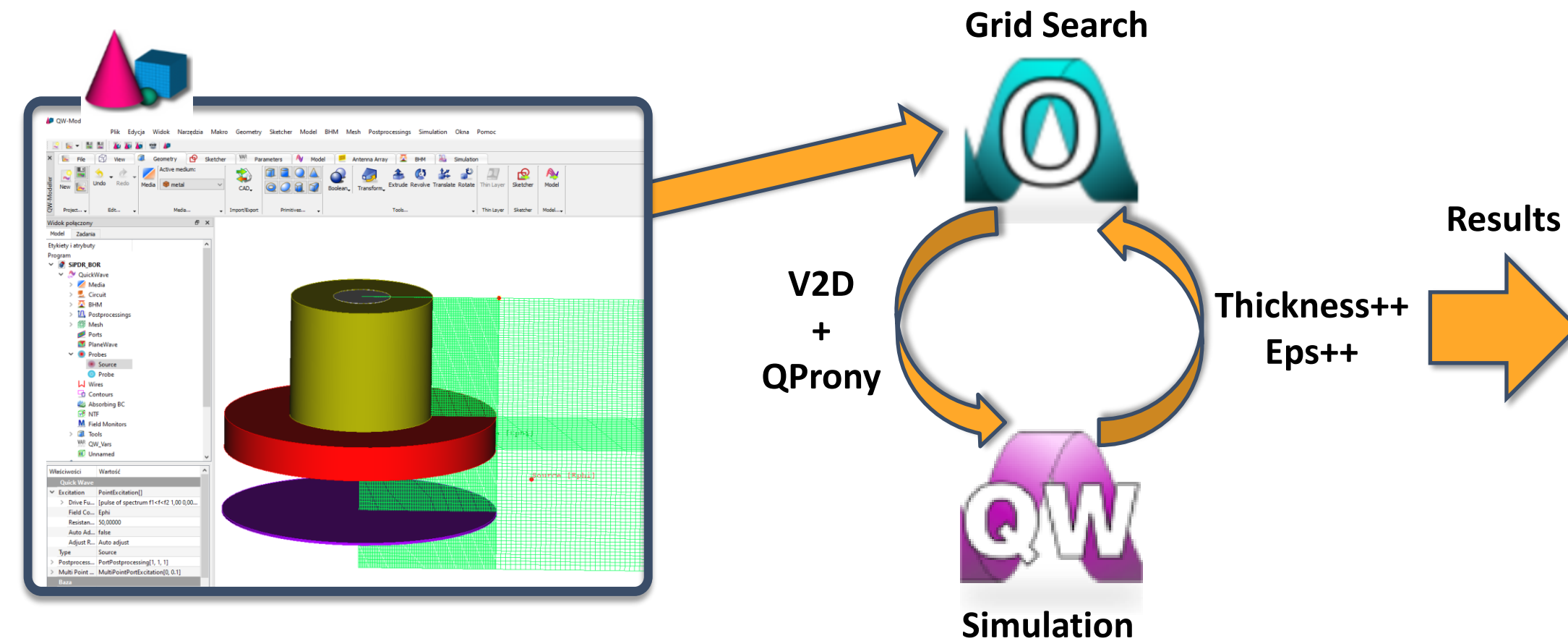Resonance frequency (sample thickness)

Fig. 6 Example of resonant frequency results obtained for 5 GHz SPDR after grid search of BoR FDTD simulation after varying sample thickness and relative permittivity
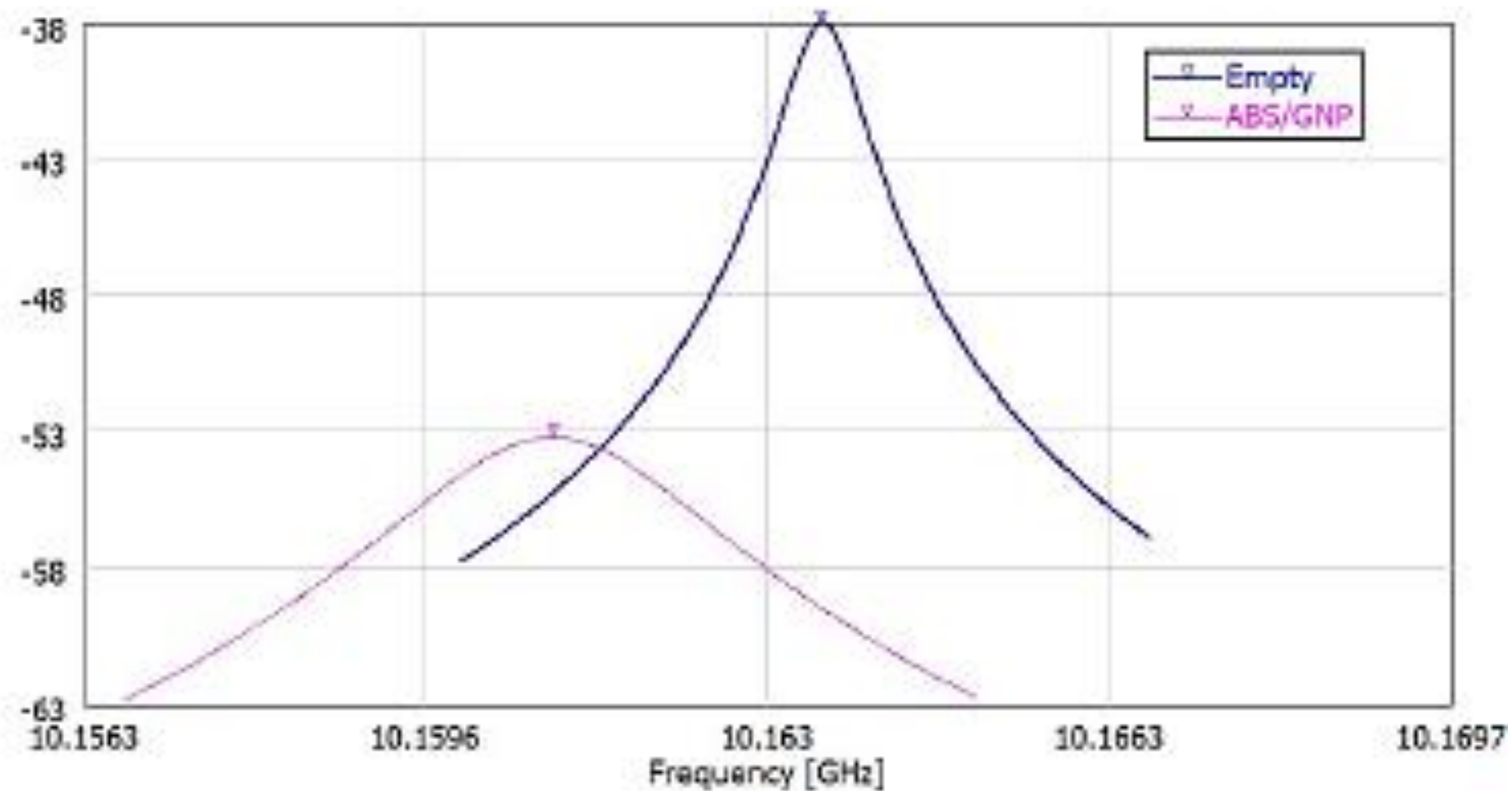
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Fig. 7 Transmission (abs (S21)) through the 10 GHz iSiPDR mounted
in the scanner and placed at two selected positions: over an empty region (blue) and at a selected
point over the ABS/GNP sample (pink).

For fast and accurate extraction of resonant frequencies and Q-factors at many scanning steps, the measured transmission curves are considered only in a limited frequency range close to the resonance, so that the extraction not burden the speed of data acquisition from the scanner and their post- processing.

Shielding properties of graphene-based polymer composites (GBPC) are enhanced by uniform dispersion of graphene flakes within the polymer matrix. Little attention has been given thus far to the rigorous studies of spatial inhomogenetities of the electrical and dielectric properties of GBPCs, due to nonuniform filler distribution in practical fabrication.

Experimental results for large (**10cm x 10cm**) GBPC panels, demonstrating a tremendous effect of the fabrication process parameters on inhomogeneities of the surface resistance, which degrade the shielding efficiency.
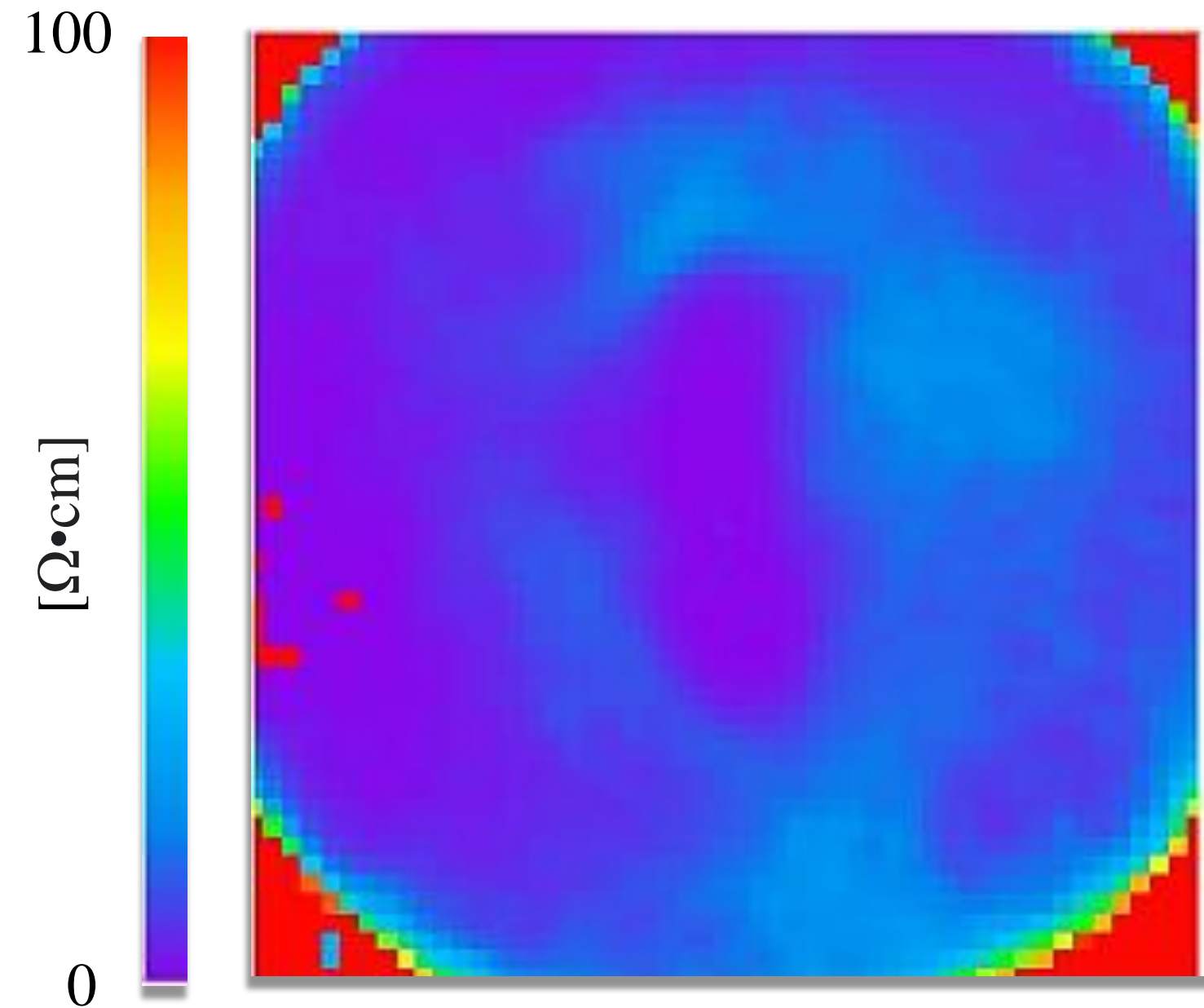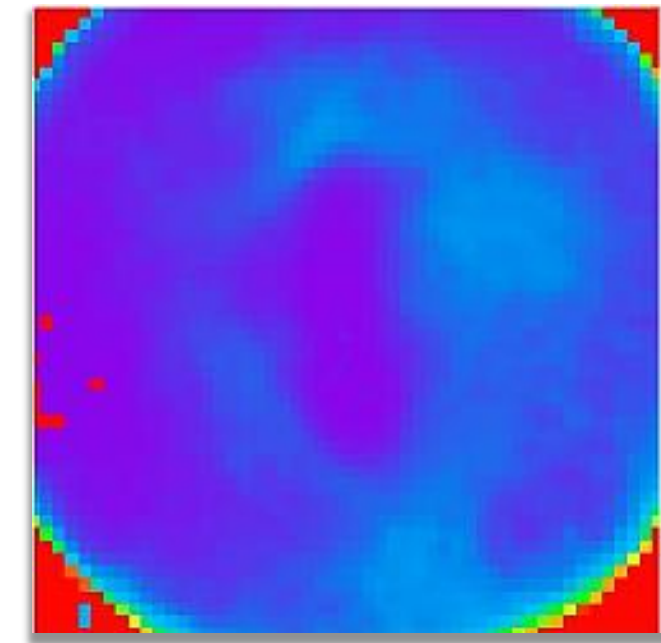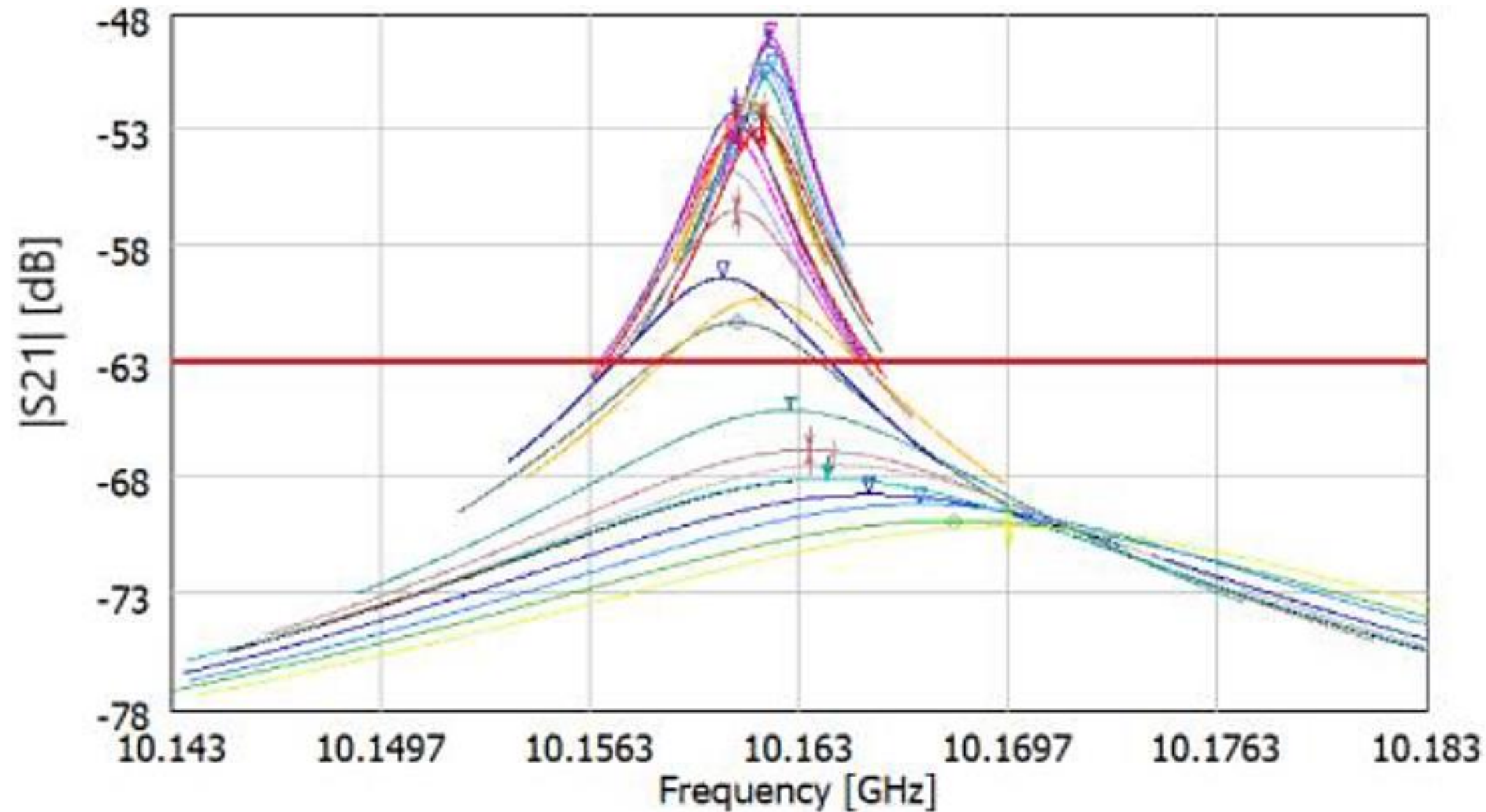

Fig. 9 2D maps of resistivity of ABS/GNP sample for iSiPDR scanning.

After analyzing all the results, it can be seen that the resonant frequency varies up to **10 MHz** up and down, for consecutive steps. At the very center of the sample we obtain the curves below the red straight line. These resonance curves are much wider thus indicating lower Q-factors calculated for them. Thus, the algorithm used returned very low resistivity values

Fig. 10 Transmission in a 10 GHz scanner depending on the position of the head along the middle of ABS/GNP sample.

Conclusions

• The microwave dielectric resonator technique was successfully applied to characterize graphene-based composites.

• A 10GHz Inverted Single-Post Dielectric-Resonator (iSiPDR) mounted into a 2D scanner enabled the construction of 2D **Q-factor maps** for different fragments of the scanned material.

• Q-factor maps were converted into **resistivity maps** using retro-modeling, which utilized ultra-fast BoR FDTD electromagnetic simulations and advanced Prony post-processing.

• In the initial samples of ABS/GNP, significant surface inhomogeneities were detected, indicating the need for improvement in the material fabrication process.

• The practical significance of the measurement was not limited because the overall losses of the sample are relevant for EMI applications.

• For monitoring graphene powder inhomogeneities, future work will focus on better controlling the deposited layer thickness.

• The modeling-based extraction procedure will be enhanced to incorporate pre-measured maps of sample thickness in future studies.

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Acknowledgement

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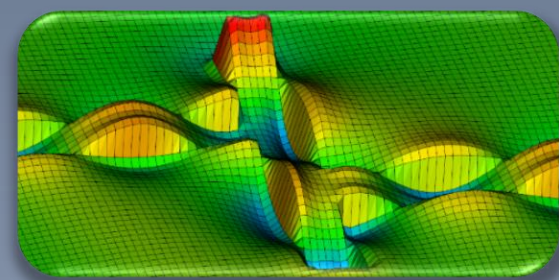
ULTCC6G_EPac

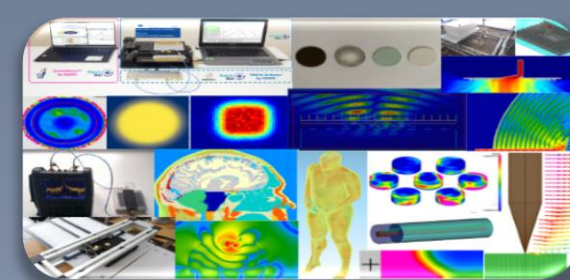*QuickWave™ Software*

Materials Measurement

R&D Projects

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