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

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INTRODUCTION

The aim of this paper is to demonstrate and fill the traceability gaps in the characterisation and assessment of materials used in the research and industry over the 1 GHz - 110 GHz range. Our choice of the frequency range is dictated by the need to extend and validate the relatively well established microwave techniques of material measurements (e.g. IEC 61189-2-721:2015, IPC-TM-650 2.5.5.13) to millimetre-wave (mmWave) technologies, including 5G and space applications. A recent industrial project led by the International Electronics Manufacturing Initiative [1] has demonstrated the challenges of characterising ultra-low-loss dielectric materials (and ultra-thin samples) relevant to 5G with the existing industrial best-practice approaches. It has highlighted the lack of Standard Reference Materials (SRMs) that would allow rigorous validation of the existing and forthcoming measurements methods. Last but not least, via an extensive round-robin campaign it has confirmed what an engineer would intuitively fear: that many substrate materials successfully used in microwave circuits may become unsuitable at mmWaves, due to increasing dielectric losses [2]. Similar observations are now being made by a sister project [3] with reference to metallic traces, specifically, to copper foils.

QWED team actively contributes to the abovementioned industrial projects. In parallel, we continue research and innovation activities in European framework projects concerned with materials modelling and characterisation (e.g. Horizon 2020 NanoBat [4], M-ERA.NET I4BAGS and ULTCC6G_Epac [5]) and contribute to the relevant European initiatives [6][7][8]. Herein, we combine our research and industrial experiences aiming to provide guidelines for the consistent electric and dielectric characterisation of materials from microwave to mmWave ranges. We shall also seek to identify specific needs of the space sector.

RESONATOR METHOD THEORY

The resonance phenomenon is relevant to many branches of physics and can be understood as observing a maximum magnitude of the output signal (Signal(out) in Fig.1a) in response to the input signal (Signal(in) of a fixed magnitude. A frequency at which such a maximum output signal is detected is called "resonant frequency". In classical electric circuit theory, a series RLC connection as in Fig.1b is a clear example. At the resonant frequency there is a net zero voltage drop over the series LC components and therefore a maximum voltage is detected over the receiver R. In fact, if the circuit is driven by a perfect voltage source (with zero output resistance, r=0), then the full driving voltage will be detected over the load R. The lumped resonance circuit of Fig.1b has only one resonant frequency f_r.

$$f_r = \frac{1}{(2 \cdot I \cdot \sqrt{LC}},\qquad (1)$$

where f_r – resonance frequency [Hz], I – current [A] , L – inductance [H], C – capacitance [F]

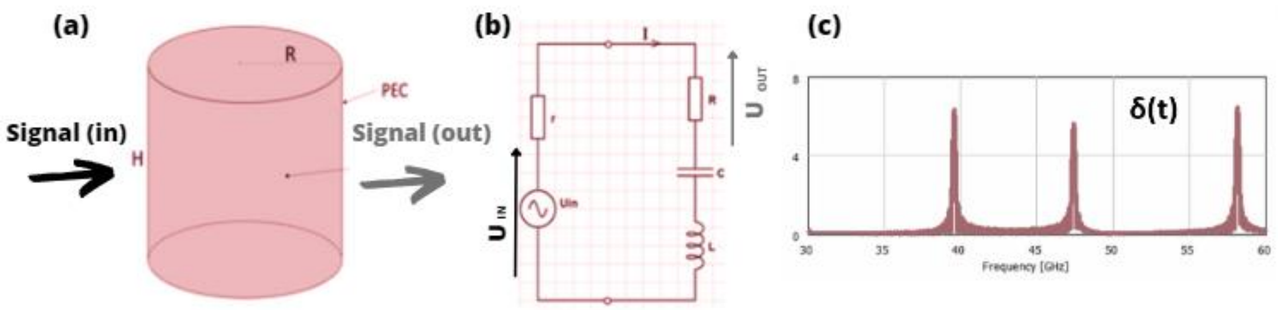

Fig. 1. Theoretical model of (a) cylindrical resonator, its (b) equivalent circuit for one selected resonance, and an (c) example of three consecutive resonances occurring in it.

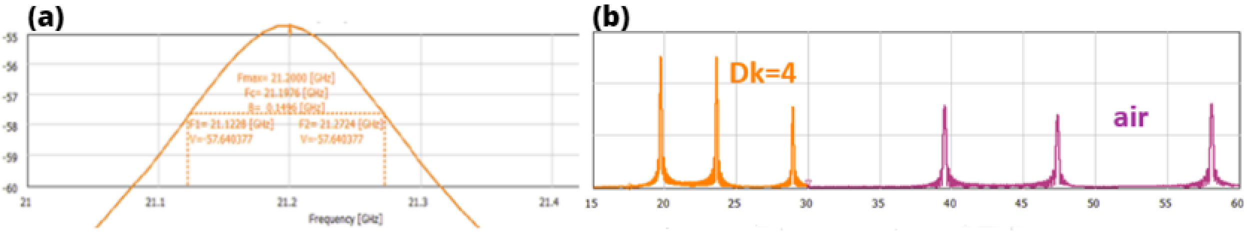

Fig. 2. Resonances seen on the transmission characteristics of a cavity resonator: (a) zoom around one of the resonances, illustrating the meaning of resonant frequency and 3-dB bandwidth, (b) transmission through the resonator filled with air and filled with a non-magnetic dielectric of Dk=4, showing the lowering of all resonant frequencies by a factor of 2 in the latter case.

Distributed resonant circuits, such as a cylindrical cavity resonator of Fig.1a, have infinitely many resonant frequencies, corresponding to resonant modes of different electromagnetic field distributions. Therefore, if the cylinder of Fig.1a is excited by a pulse (e.g. a delta pulse of flat frequency-domain spectrum), the output signal will contain infinitely many resonant peaks, at the resonant frequencies of the cylinder. Let us note that:

1. The amplitudes of the resonant peaks depend on the coupling of the input and output probes to the resonator.
2. For low and moderate coupling, the resonant frequencies are approximately equal to eigenfrequencies of the resonator, which are solutions of the Maxwellian eigenvalue problem. For canonical geometries the eigenvalue problem can be solved analytically; for example, for a cylinder of radius R, height H, and homogeneously filled with a lossless non-magnetic dielectric of dielectric constant Dk we obtain eq.(1):

$$f_{r,mnp} = \frac{c}{\sqrt{Dk}}\sqrt{(\frac{k_{mn}^{(,)}}{\pi R})^2 + (\frac{p}{H})^2}, \tag{2}$$

where m,n,p modal indices in angular, radial, and vertical directions; $f_{r,mnp}$ – frequency of mnp mode; c – speed of light; kmn – nth root of mth Bessel function (or its derivative, depending on the mode).

As stated before, infinitely many modes denoted by m,n,p indices are possible. Figures 3 and 4 show example distributions of the electric and magnetic fields.

3. The width of the resonance curve around each resonance is related to the losses in the resonator. In non- magnetic low-loss dielectrics we obtain eq.(2):

$$Q = 2\pi \frac{\iiint_V \varepsilon \vec{E}\cdot\vec{E}^* dv}{T\iiint_V \sigma \vec{E}\cdot\vec{E}^* dv} = \frac{\omega\varepsilon}{\sigma} = \frac{1}{D_f} \approx \frac{f_{res}}{\Delta f}, \tag{3}$$

where Q – quality factor , ε – real part of permittivity [F/m] equal Dk* ε_0, where ε_0-permittivity of vacuum, $\vec{E}$ – Electric field [V/m], σ – conductivity [S/m], T – period [s] , D_f – loss factor, f_{res} – resonance frequency [Hz], Δf – 3dB bandwidth [Hz]

It is therefore apparently straightforward to apply resonators as sensors of material properties. If a cavity resonator is homogeneously filled with a dielectric, then all its resonant frequencies will be tuned down by a square root of the material's Dk; while the material's loss factor Df will be calculated from the width of the resonance curve via eq. (2).

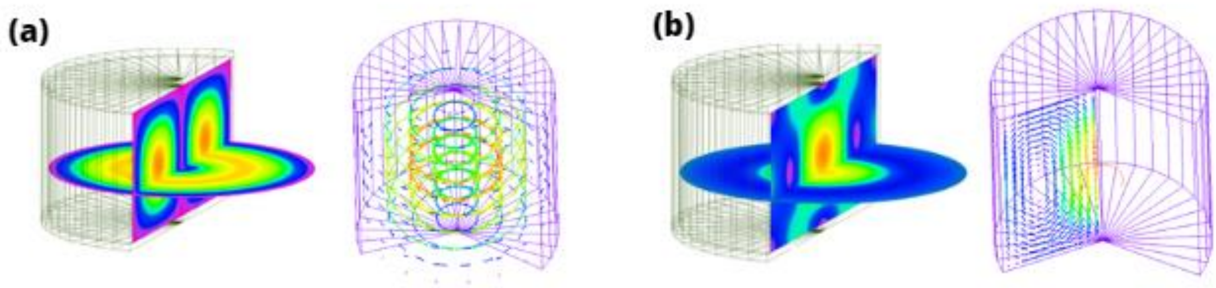

Fig. 3. Electric field (a) and magnetic field (b) of the basic mod TE_{011} occurring in the occurring in a cylindrical resonator at its operating frequency.

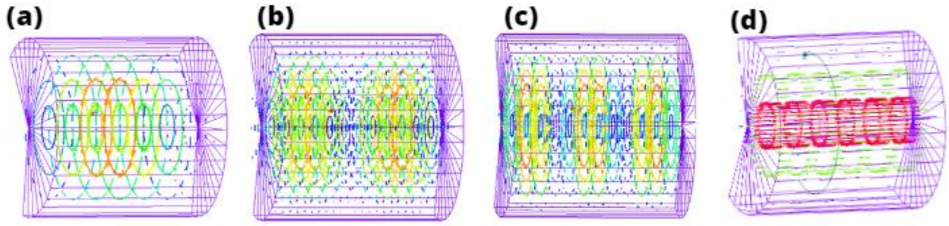

Fig. 4. Basic modes occurring in a cylindrical resonant cavity (a) TE_{011} (b) TE_{012} (c) TE_{013} (d) TM_{010}

Electromagnetic 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 characterisation. Some of them are difficult to excite. Software provided with the resonator is usually compatible only with modes pre-selected by the vendor. From the resonators benchmarked in [1][2], in practice only a Fabry-Perot Open Resonator (FPOR) is multimodal and provides a multi-frequency characterisation.

A final remark is concerned with anisotropic materials. Consider a cylindrical resonator as in Fig.4 and a flat sample placed in parallel to the circular bases. In the case of TE modes (as in Fig.4a,b,c) the sample will interact with in=plane electric field and therefore, the in-plane material parameters will be measured. In the case of TM modes (as in Fig.4d), the our-of-plane material parameters will be measured. From the resonators considered in [1], only a Balanced Circular Disk Resonator (BCDR) is dedicated to the out-of-plane measurements.

PARCTICAL MICROWAVE AND MILLIMETRE-WAVE RESONANCE METHODS

There are different types of resonators corresponding to different requirements for sample dimensions, frequency range or measured electromagnetic (EM) properties of the material. All are known for their accuracy and most for non-destructive measurements of materials at microwave frequencies. For each of these methods, the electromagnetic properties of the sample are obtained by analysing the shift in resonant frequencies and the change in Q-factors. Thus, two measurements are necessary to obtain results. A measurement of an empty resonator is always required as a reference point and then the sample under test is placed in it. The difference between these values and the measurement of the thickness of the sample allows one to determine the EM parameters from the calibration curves. Thus, it is also inherent and very important to accurately estimate the thickness of the material under test.

In most cases, the measurement procedures are not complicated. In particular, some resonators allow inserting the sample without any disassembly of the measurement system. For example, the Split-Post Dielectric Resonator (SPDR) method is based on [11] and illustrated in Fig.5 is straightforward. While in SPDR the sample is placed in strong electric field and therefore, high-loss materials cannot be measured, a similar Single-Post Dielectric Resonator (SiPDR) is dedicated to higher-conductivity (lower resistivity) materials. Figure 6 explains the use of SiPDR in a more advanced scanning setup, which produces a map of material parameters. The procedure for scanning the entire sample surface is only slightly more difficult for the user. It requires additional operation of the head positioning motors. It comes down only to entering the values in the master unit control application (MUCA) software. The procedure itself does not change from scanner to scanner. It is therefore demonstrated on an inverted Single-Post Dielectric Resonator scanner shown on Fig. 6, based on [12].

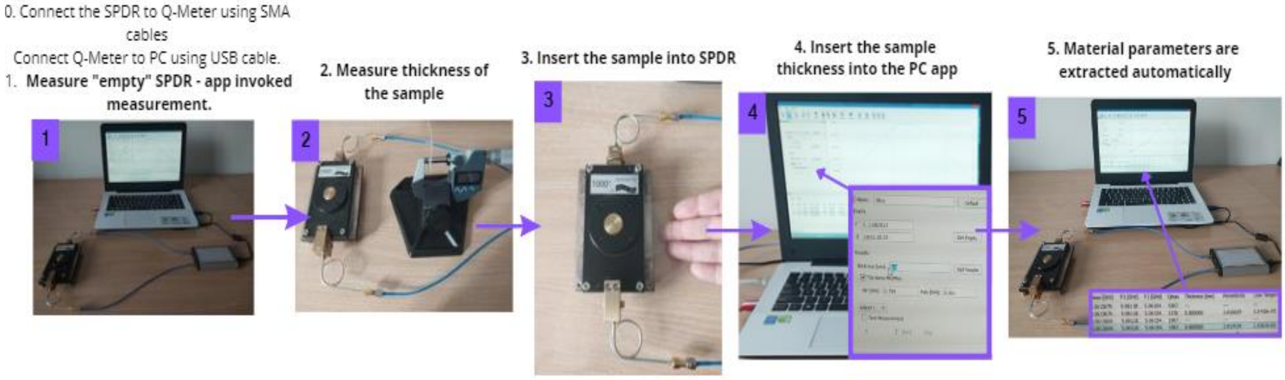

Fig. 5. 5 GHz Split-Post Dielectric Resonator (SPDR) measurement procedure.

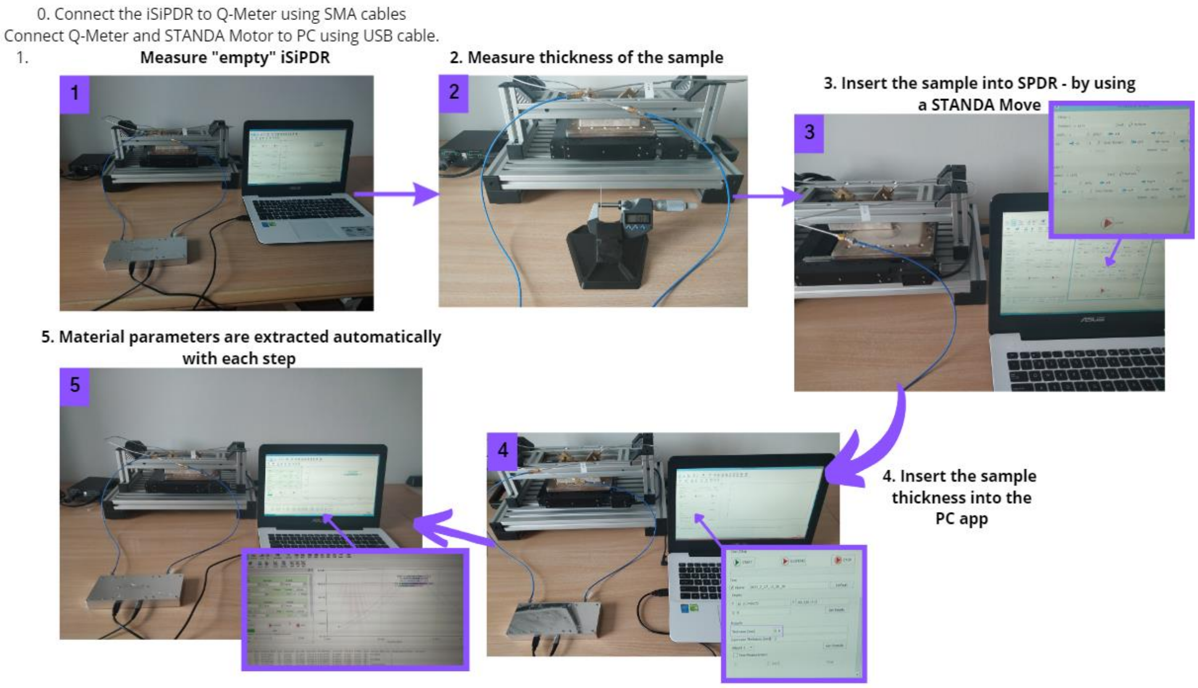

Fig. 6 10 GHz inverted Single-Post Dielectric Resonator (iSiPDR) measurement procedure.

This scanner is capable to measure the surface impedance of metamaterials and resistive films, and for non-contact conductivity measurements of semiconductor wafers. Also provides qualitative testing of anode materials for lithium-ion battery cells such as those composed of graphene based composites. The effectiveness of such measurements was confirmed in the [4]. As in SPDR, from the calibration curves, the conductivity of the sample is estimated and then the resistivity for bulk samples having thickness that is the same order or larger than the skin depth. For thin films, a surface resistivity value can be obtained. It should be noted that samples under test must be very flat and not bended. This can significantly affect the results of extracted parameters. Its operating frequency is 10 GHz. This value is a major limitation of resonators. It is possible to make a measurement, but at a single frequency. It happens that EM parameters change depending on the frequency. Wanting to verify that the potential benchmarking is consistent for each method. It would be necessary to exclude the influence of the method on the results. In this case, a very good solution is to use a broadband Fabry-Perot Open Resonator (FPOR) [14]. Fig. 7 shows the measurement procedure. FPOR operates on Gaussian odd modes spaced every 1.5 GHz giving a large set of measurements for frequencies from 20-110 GHz. When the dielectric material is low loss it will be necessary to use a sapphire resonator such as the one in Fig. 8

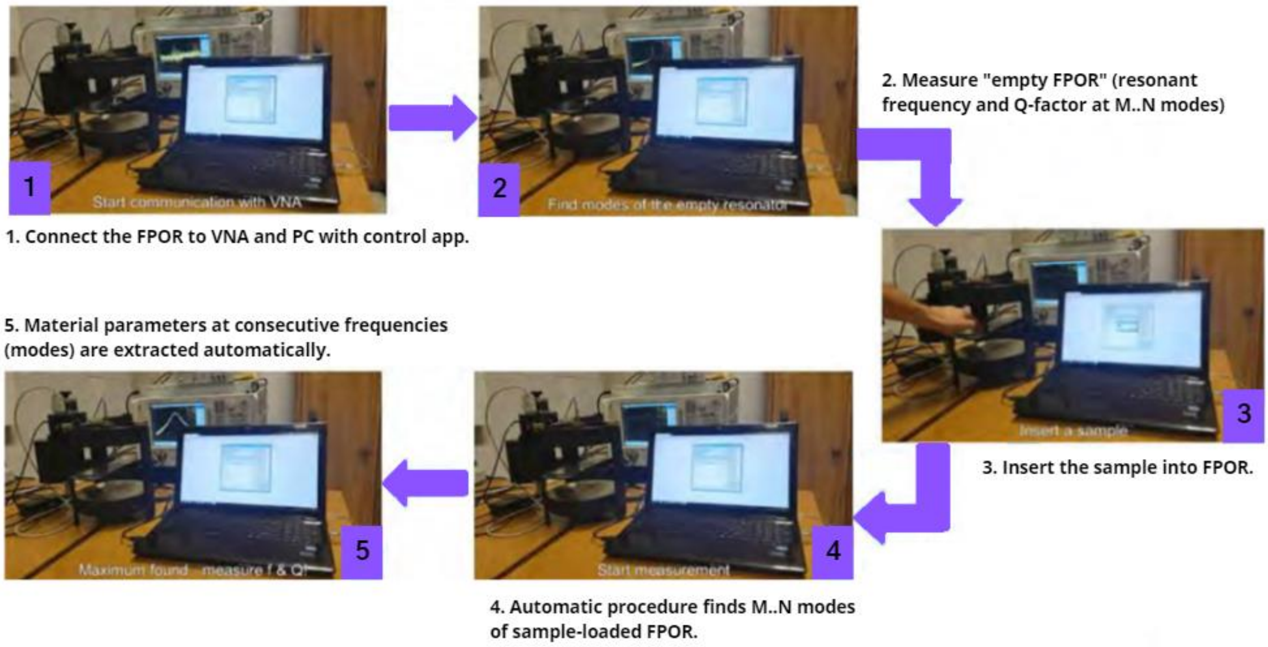

Fig. 7 20-110 GHz Fabry-Perot Open Resonator (FPOR) measurement procedure.

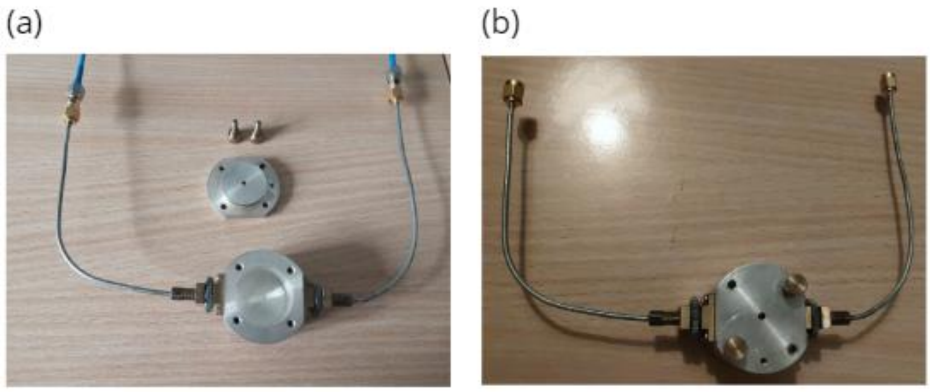

Fig. 8. TE01δ mode Dielectric Resonator with an open (a) and closed (b) cavity.

The Dielectric ResonatorTE01δ [15] is specially designed with closed cavity allows the examination of the resonant frequency shift and Q-Factor of self-resonating samples such as metals. All these methods have very similar measurement procedures.

RESULTS AND DISCUSSION

To create the benchmark, three types of resonators were selected due to the availability of test equipment in the resonator market and published standards and test data. The first of these are SPDR-type resonators. Standardized by the IEC. QWED and Keysight have them available from 1.1 to 15 GHz. The already commercially unavailable 20 GHz SPDR was also used. Another is a fully automated FPOR. Measurements were made on it from 20-110 GHz with 1.5 GHz steps. The last type is the Split Cylinder Resonator (SCR) [16] with a sample insertion slot. It is closest to the canonical cylinder cavity supporting TE011 mode. It is standardized by IPC and available at 10, 20, 28, 40, 60 and 80 GHz. Necessary for obtaining measurements and results are Vector Network Analysers (VNA). They were used to obtain transmissions. Based on them, resonant frequencies and Q-Factor are derived. EM parameters were extracted using proprietary software dedicated to each type of resonator. Samples with a low dielectric constant, a thickness of less than 0.2 mm and a very low dispersion coefficient were selected for comparison of all methods. The requirements of 5G technology were taken into account. Samples were transferred between laboratories that participated in the measurements [17]. Thus, strict procedures were established for transporting as well as storing samples. Due to the above, two types of materials were decided upon: Cyclo Olefin Polymer (CPO) and Precision Teflon. Fig. 9 shows the results of measurements of the dielectric constant of the COP and Teflon samples, obtained independently in each laboratory also marked on the legend. The results obtained range from 1% to 2.34. Figure 10 shows the loss tangent for COP and Teflon. For COP above 30 GHz, it remains constant as a function of frequency. Below this value I retain an interesting relationship. However, it is confirmed by other independent resonance methods. The scatter in the FPOR results obtained for Teflon above 50 GHz is due to the switching of frequency extenders to the VNA used.

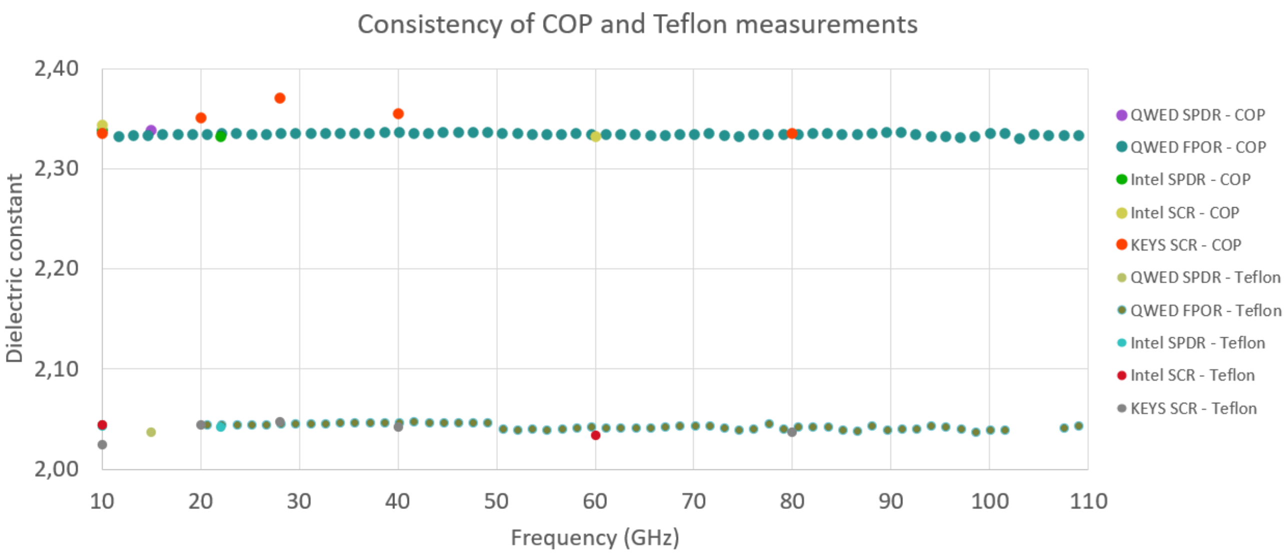

Fig. 9 Summary of results of dielectric constant measurements obtained by SPDR, SCR and FPOR methods for Cyclo Olefin Polymer (COP) and Teflon.

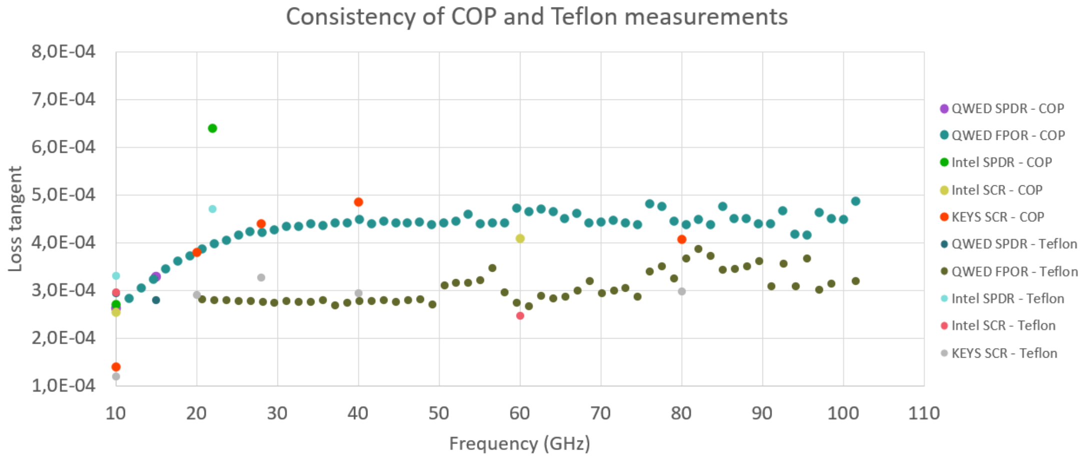

Fig. 10 Summary of results of loss tangent measurements obtained by SPDR, SCR and FPOR for COP and Teflon.

CONCLUSION

In this paper we have revisited the principles of resonator-based techniques for the electromagnetic characterisation of materials. We have also summarised the results of recent benchmarking studies of such techniques applied at mmWave frequencies. Our SMW presentation will further elaborate on the theory and practice, with a view to:

1. providing space engineers with **practical guidelines** concerning the accuracy, repeatability, and reproducibility of **popular techniques** for material characterisation in microwave and mmWave ranges (SPDR, SCR, FPOR, BCDR - acronyms as defined in [1][2]),
2. presenting **new instruments and techniques**, including those based on our own research (e.g. 2D scanners of dielectric surfaces and conductive films, recognised as **EU Innovation Radar** [9]) or coming from the collaborating laboratories (e.g. ultra-fast quasi-frequency-continuous Fabry-Perot measurements [10] extended to 110 GHz),
3. discussing recent trends [6][7] on exploring the **synergies between materials' modelling and characterisation**, or physics-based and data-driven models, towards the establishment of Digital Twins.

Besides sharing our experience, we aim to raise a discussion on the specific needs of the space research and industry, and to **formulate the challenges in terms of materials characterisation and assessment**. We seek **new collaborations and frameworks** to further develop our technologies, in order for such challenges to be effectively addressed by us and met.

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