TITLE: Data acquisition with Single - Post Dielectric Resonators

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SHORT DESCRIPTION: This document's intention is to provide a guideline to help in the process of acquiring reliable and reproducible data with single - post dielectric resonators (SiPDR) [1], with a focus on the inverted (iSiPDR) configuration [2] developed and studied in the H2020 NanoBat project. Areas covered include preparation and handling of measurement setups and material samples. Field distributions in iSiPDR are shown, in order to facilitate the overall understanding of its operation and the nature of the measured data, but extensive advice on data analysis is beyond the document's scope. Appropriate literature is referenced and the complete measurement process, including the modelling needed for the data analysis, is documented in the Twinned MODA+CHADA format [3] currently promoted by EMMC and EMCC [4].

INSTRUMENT SPECIFICITY: This guideline is targeting to be instrument unspecific, subject however to two constraints:

1. At the time of writing, the only SiPDR commercially available on the open market is that from QWED, operating at 5 GHz [5]. While it well serves the purposes of a single quasi-pointwise material measurement (averaged over the SiPDR head), it is not suitable for the implementation in XY-motorised stages needed for 2D imaging of the material's surface, as previously successfully demonstrated in the case of the more classical SPDR resonators [6][7]. This is why in the NanoBat project [8], an inverted SiPDR (iSiPDR) design has been proposed, allowing the resonator to move over the imaged material sample, within the range of the XY-stage. In this SOP, the term "SiPDR" is used in a generic sense, denoting the commercial 5 GHz SiPDR and the new 10 GHz iSiPDR, and the SOP is relevant to both, unless otherwise explicitly stated. While interested users may also construct their own SiPDRs based on the open literature referenced hereinafter, and follow this SOP, it is not guaranteed that such devices provide the same simplicity of operation and accuracy of the results.
2. SiPDR needs a microwave signal generator with a function of measuring transmission (S21) characteristics between the SiPDR's two ports and extracting the resonant frequency and 3 dB bandwidth. In principle, those functionalities can be provided by any VNA or a simpler customised device. However, the operation of QWED SiPDRs is most straightforward when used jointly with QWED's Microwave Frequency Q-Meter, or any VNA compatible with QWED's Material Measurement Suite (MMS) software, or KEYSIGHT VNAs including Option 003 in their N1500A Material Measurement Suite.

ABBREVIATIONS / TERMINOLOGY:

SiPDR - Single - Post Dielectric Resonator

iSiPDR – Inverted Single - Post Dielectric Resonator

SPDR - Split - Post Dielectric Resonator

VNA - Vector Network Analyser

Q-Meter - Microwave Frequency Q-Meter from QWED

MMS – Materials Measurement Suite from QWED

S21 - Scattering parameter - transmission

SUT –sample under test

MW / RF - Microwave / Radio-frequency

ESD - Electrostatic discharge

FDTD – Finite-Difference Time-Domain method (of electromagnetic modelling)

BoR – Bodies-of-Revolution

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Introduction: principles of the SiPDR method

This document attempts to provide a generic guideline for use of single - post dielectric resonators (SiPDR), including the new inverted design (iSiPDR). The intention is to provide basic information needed to perform reliable, repeatable, and safe measurements. The text gives notes on resonator handling and maintenance, performing measurements, and common sources of errors. Special attention is paid to the different ways of operating SiPDRs within the three types of measurements setups, which use different types of microwave signal generators / meters and different software:

1. a non-specific VNA,
2. a VNA compatible with QWED's Material Measurement Suite (MMS) - applicable with QWED's SiPDRs,
3. QWED's Microwave Frequency Q-Meter - applicable with QWED's SiPDRs.

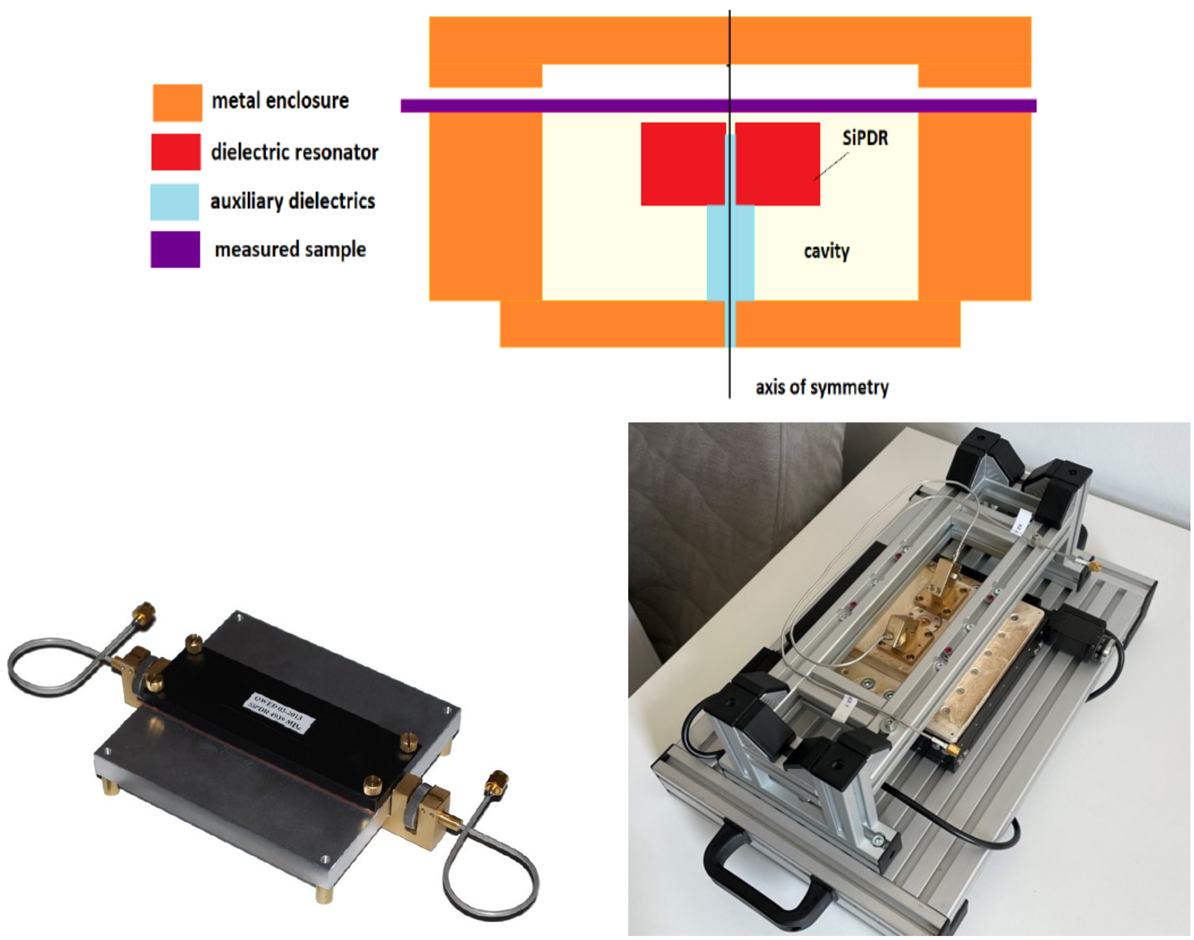

Fig. 1: Schematics (upper) and photos of example SiPDR units from QWED: commercial stand-alone 5 GHz SiPDR (lower left) and new iSiPDR incorportated into 2D scanner (lower right).

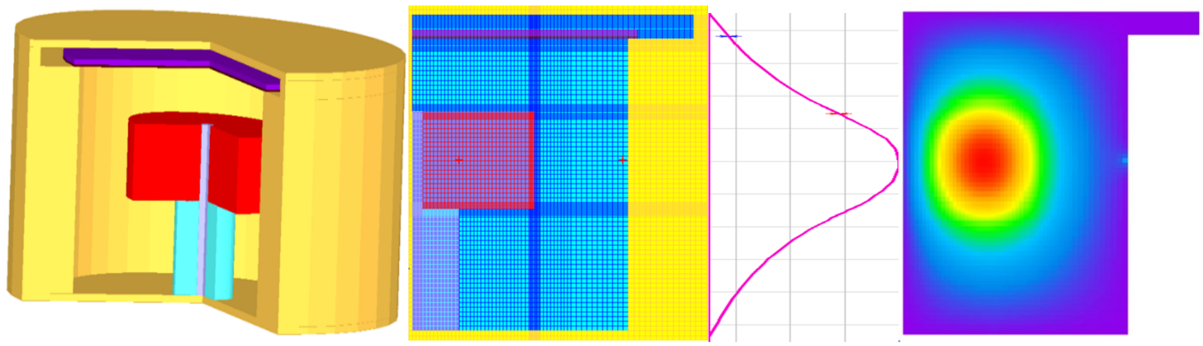

Fig. 2: Electromagnetic design model of an example SiPDR (from left to right): 3D CAD model (shown 270 deg section, colours as in Fig. 1), BoR FDTD meshing (half of the SiPDR long-section, colours as in Fig. 1 plus darker blue denoting air), and simulated electric field magnitude at the resonant frequency illustrated in 1D (along the height / axis of rotational symmetry) and 2D (in half of the long-section).

Fig. 3: Photo of an operating setup including 10GHz iSiPDR 2D scanner (left; position of the actual iSiPDR indicated) and Keysight FieldFox (centre) driven by a laptop (right) with QWED control application.

SiPDR is a rigid structure of well-defined, known dimensions and materials. Looking from the outside (Fig. 1 lower left) SiPDR appears as a metal enclosure with a slot for easy insertion and support of a laminar sample. However, it should be stressed that the actual “resonator” is the dielectric cylindrical post made of high-permittivity low-loss ceramics (red in Figs. 1, 2), which stores the electromagnetic (EM) energy. At the operating frequency, a resonant mode if formed, with EM fields being axially symmetrical and mostly confined in the ceramic post, as shown in Fig. 2. Losses in the metal enclosure are thereby minimal. Moreover, since the magnetic field has only a vertical component at the side wall of the enclosure, the currents in the side wall are only circumferential, and there is no radiation through the slot. All electromagnetic energy injected through the coupling loops in contained within in the SiPDR “head” (inside the enclosure); an estimated 90-95% of energy is confined in ceramic post. The E-field magnitude decreases nearly linearly from the post to the ground plane (upper in Figs. 1, 2), as clearly demonstrated by its 1D envelope in Fig. 2. What is crucial for the SiPDR measurement method, as opposed to the more classical SPDR [7], is that the sample (violet in Figs. 1, 2) is placed in weak E-field. It therefore weakly interacts with the resonator, but in a manner well-controlled by means of the electromagnetic modelling. This allows measuring losses in highly-lossy (low-resitivity) materials, which cannot be measured in SPDRs. **For such materials, the dielectric constant becomes irrelevant and the measured parameter is either resistivity (for bulk samples) or surface resistance (well-characterising thin conducting films on high-resistivity substrates)** [9].

The resonant mode has a particular resonant frequency depending on resonator's dimensions and materials but also, to some extent, on the electrical properties of the sample under test (SUT). Thus, each resonator is designed for a particular nominal frequency and the actual measurement is taken at a frequency close to the nominal one. SiPDRs for other frequencies can be designed following the theory of ref. [1], however, such designs become impractical at frequencies much lower than 5 GHz or higher than 10 GHz: at lower frequencies such SiPDRs become big and bulky, while at higher frequencies the manufacturing tolerances and losses in the applied materials deteriorate the measurement accuracy.

A stand-alone SiPDR, as shown in the lower left of Fig. 1, provides information about the part of the sample interacting with the resonant fields (see Fig. 2). This information is therefore averaged over a certain area of the sample, but for the sake of brevity, we often call it a “point-wise measurement”, meaning a measurement averaged around the point located centrally to the ceramic post. To image bigger samples and/or to obtain a map of sample parameters over a bigger area, an SiPDR has to be mounted into a 2D scanning device. In the scanner of Fig. 3 (or Fig.1 lower right) the resonator is located above the sample (which is opposite to the stand-alone unit, where the sample is over the resonator) and when we want to be specific, we call it an inverted SiPDR (iSiPDR).

The SiPDR method requires making two consecutive measurements: one of the empty SiPDR and one of the SiPDR loaded with the sample. The principle of the method resides in extracting the material resistivity (for bulk samples) or surface resistance (for thin films) [9] from the change of the resonant frequency and Q-factor. The mapping curves for doing so are obtained by full-wave electromagnetic modelling, as exemplified by Fig. 4.

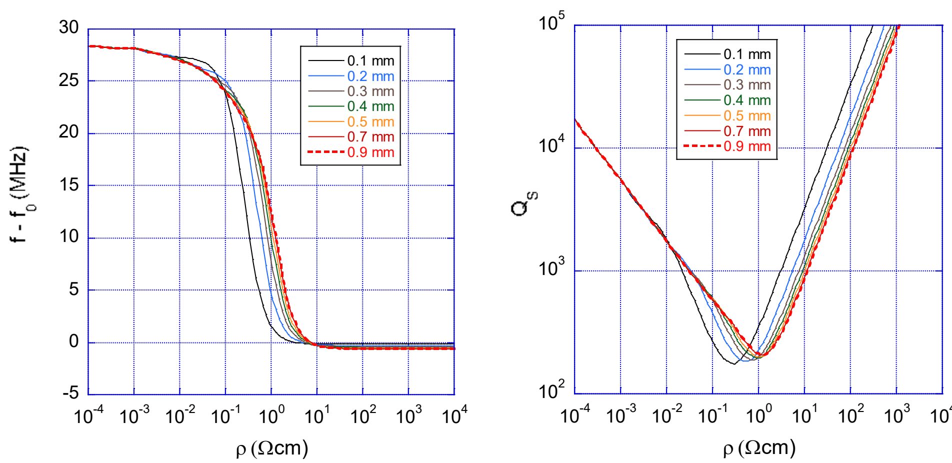

Fig. 4: Resonant frequency shifts (left) and Q-factors (right) versus resistivity ρ for bulk samples of different thickness h, measured in an example SiPDR [5] of nominal frequency 5 GHz.

Handling and safety of resonators

SiPDRs are passive structures, and the measurement mode is well shielded from the environment (Fig. 2), so in normal use no electromagnetic hazards arise. However, if a resonator is erroneously driven by a signal significantly outside of its nominal operating frequency band, undesired modes may be excited that may be subject to leakage. Therefore, when an SiPDR is connected to and driven by a VNA (or an alternative active device), output power of that active device should not be increased about its standard value and frequency band should not be wider than expected for 3 dB bandwidth measurement of the SiPDR.

SiPDRs provide accurate measurements because they are precisely designed and mounted, and each unit is individually calibrated upon manufacturing to adjust for the manufacturing tolerances. Therefore, each SiPDR should be handled with care and kept clean. The coupling loops within the resonator (Fig. 1) are specifically delicate. QWED SiPDRs are therefore terminated with semi-rigid cables and to prevent those cables from damage, additional flexible coaxial cables connecting them to the high-precision cables of the VNA are recommended.

Because of its individual calibration at origin, each SiPDR comes with its own (unit-specific) version of the software. A backup copy of the software should be kept by the user in a safe place and clearly linked to a particular SiPDR unit.

SiPDR must not be kept or used outside its operating temperature range dictated by the applied materials and cables. QWED declares the range from -270 deg C to 110 deg C for its standard SiPDRs but customised higher-temperature units have also been used.

A practical observation stemming from QWED's 20+ years of experience in producing and marketing its test-fixture is, that damages in normal use are rare; most reported damages concerned the semi-rigid cables; in only one case a resonator was damaged during shipment (or rather custom clearance) and a loss of the resonator's own software happened.

Sample types, preparation and safety

SiPDRs are originally intended for the measurements of resistivity of bulk materials or sheet resistance (surface resistance) of thin conductive material films on high-resistivity substrates. The range of material parameters measurable in SiPDRs is given in Table 1 (lower row). Together with SPDRs (for which a SOP can be found in [7]), they make the dielectric resonator method applicable to a wide range of materials, from ultra-low-loss dielectrics to ultra-high-conductivity (mobility) semiconductors. For conductors (metals), one more topology, based on a sapphire resonator [10] is used.

	Conductivity [1/(Ωm)]	Resistivity [Ω cm]	Surface resistivity [Ω/sq]
Range od SPDR applications	$2\ 10^{-3}$ to 0.5	from $2\ 10^{2}$ to $5\ 10^{4}$	from $2\ 10^{3}$ to 10^{7}
Range of SiPDR applications	0.1 to 10^{6}	from 10^{-4} (*) to 10^{3}	from 10^{-1} to $2\ 10^{4}$

Table 1: Range of material parameters measurable in SiPDRs and SPDRs.

The sample under test (SUT) should be flat and insertable into the SiPDR's slot. SUT's lateral dimensions must be big enough to cover the SiPDR "head" (somewhat smaller SUTs are measurable but with reduced accuracy) while the thickness must be small enough to fit through the slot (which is a mechanical constrain that can be alleviated by a customised SiPDR construction). There are no formal requirements on the minimum thickness of the SUT, also SUT rigidness is strongly recommended. For thin film characterisation, such films should be deposited on low-loss susbstrates.

The operator inserts a SUT into the SiPDR slot. This exposes the operator to the material under test as well as the material to the operator. Necesary care must be taken to avoid personal injury in case of harmful substances and to avoid damaging SUTs if they are soft or fragile. The use of gloves, masks, or tweezers is often recommended and in any case the instructions from the material's manufacturer must be adhered to. Note that it is also possible to manouver delicate SUTs placed on a low loss dielectic foil such as polyethhylene or PTFE - such thin supporting sheets do not noticeably affect on the measurement, moreover, their effect can be eliminated by taking the SiPDR loaded with the supporting sheet (instead of the empty SiPDR) as reference. If, despite all caution, a sticky SUT leaves traces on an SiPDR, the SiPDR must be carefully cleaned.

For each SiPDR unit, at least one verification SUT should be maintained, preferably marked by an engraving pen or other suitable method.

Measurement setups, equipment and operator safety

SiPDR measurements can be performed at room temperature or in environmental test chamber. In all tests the ambient test temperature must not exceed beyond the operating temperature range of a particular SiPDR declared by its manufacturer. Similarly as in the case of SPDRs for which the IEC norm [2] declares that the temperature variation should not exceed 1 deg C during test, a good practice in the SiPDR case is to make the measurement of the loaded unit immediately after the measurement of the empty unit. Relevant environmental conditions (temperature, humidity) should be recorded together with the test results.

SiPDR + VNA measurement setup

A classical measurement setup consists of an SiPDR and a VNA (such as FieldFox in Fig. 3). VNA is sensitive to static electricity, hence special care should be taken to avoid damage from ESD. Conducting wrist straps should be used to prevent high voltages from accumulating on workers bodies, also anti-static mats or conductive flooring materials are desirable. No highly-charging materials should be in the vicinity. The inner conductors of any RF cables, connectors or probes should never be touched, or come in contact with electrostatically charged surfaces.

The frequency range of the VNA must naturally cover the operating frequencies of the SiPDRs to be applied; and the dynamic range should be at least 60dB as recommended by the IEC norm for SPDRs [2], but preferably 80-100 dB. Allow at least 30 min for the VNA to warm up and stabilise.

In case of KEYSIGHT VNAs including Option 003 in their N1500A Material Measurement Suite, if this is your first work with a particular SiPDR, make sure this SiPDRs specific software has been installed. In case of other VNAs, it is strongly recommened to use QWED control application (such as MMS); otherwise be prepared to copy the measured data and transfer it to a separate PC, with the SiPDR specific software installed on it.

Connect the SiPDR to the VNA and follow the VNA instructions. Start with the empty SiPDR. Enable the option of S21 magnitude measurement. Set the centre frequency at the nominal frequency of the SiPDR. Read the actual resonant frequency (peak of S21) and 3dB bandwidth. Check the minimum values of S11 and S22, if different by more than the second decimal place, use SiPDR nuts to change the positions of the coupling loops and repeat S21 measurements. Collect the resonant frequency and Q-factor (or 3dB bandwidth) of the empty resonator. Repeat the same steps for the SiPDR loaded with SUT.

In case of KEYSIGHT VNAs including Option 003 in their N1500A Material Measurement Suite, the material parameters will come up on the VNA screen. In case of other VNAs, use a dedicated application (such as QWED MMS) or move the raw measured results to the PC with the conversion software installed.

SiPDR + Q-Meter measurement setup

A fully-fledged VNA provides significantly more functionalities than needed for the SiPDR measurements. A microwave engineer having access to a VNA and knowing how to operate it will typically choose the VNA setup. However, SiPDRs have also found applications in various institutions, including food industries [9] and material science, where neither microwave equipment nor engineers are available. For those users, low-cost easy-to-use devices substituting the VNA in SiPDR measurements have been developed.

An example of such devices is Microwave Frequency Q-Meter from QWED [6], available for several frequency ranges compatible with the operating frequencies of QWED SPDRs and SiPDRs. Essentially, Q-Meter is a computer controlled microwave oscillator system for quick and automatic SiPDR measurements. A dedicated, user friendly and highly configurable application allows controlling the measurement process and enables easy management of the measurement results. A portable setup consisting of iSiPDR (built into the scanner), Q-Meter and a laptop is shown in Fig. 4. Its operating instructions will not be quoted here, as they are device- and application-specific. It is worth noting however, that the application itself guides the user through the measurement process.

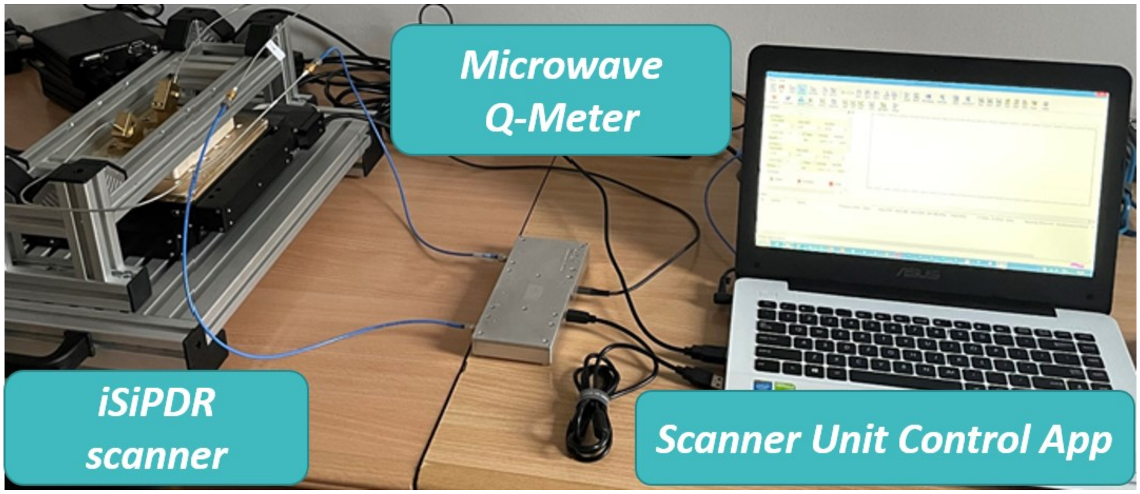

Fig. 4: Photo of the imaging setup comprising 10 GHz iSiPDR 2D scanner (left), QWED Q-Meter (centre), and a laptop running Q-Meter and scanner control software (right).

2D SiPDR scanning setup

In the NanoBat project, SiPDR measurements have been extended to 2D surface imaging, following the concept introduced in [6] for SPDRs. To this end, an inverted SiPDR (iSiPDR) at 10 GHz has been developed and built into a 2D scanner. The scanner has been designed with the use of 2D XY motorised stage from Standa. Several modifications of the resonator have been made to adjust to the mechanical construction, e.g., its inversion (which requires more sophisticated calibration, since the distance between the SUT's active surface and the ceramic head changes with the changing SUT substrate's thickness) and angular connection of the feeds. As shown in Figs. 3 and 4, connections have implemented and tested to two portable network analysers: KEYSIGHT FieldFox and a 10 GHz Q-Meter (developed by QWED in MMAMA project and enhanced in NanoBat). The whole setup is driven by an application running on laptop. The main specifications for the 2D SiPDR scanner and example results will be subject to a dedicated NanoBat deliverable (D1.3).

It is concluded that by incorporating the resonator into the scanner the following advantages extensions of the SiPDR technology are achieved:

- Samples of size larger than permitted by the standard SiPDR construction can be characterised.
- Automatic scanning images surface inhomogeneities, which may be programmed in the material fabrication process or caused by artifacts.
- The 2D scans obtained in a controlled manner can be further post-processed through image processing techniques to obtain higher resolution maps of resistivity or sheet resistance. As a result, the effective spatial resolution of SiPDR measurements is enhanced below the size of the resonator head and samples smaller-than-head can be measured.

Operation of the 2D scanner requires more caution than that of stand-alone resonators and three main issues will be pointed out:

- The flatness and stiffness of SUTs are now important not only for the accuracy of the extracted material parameters, but also from the viewpoint of smooth SUT movement within the iSiPDR head, to avoid mechanical blocking.
- When a scan of thousands of spatial point is made, which takes several hours, thermal variations of the iSiPDR parameters begins to influence the extracted material parameters. It is therefore recommended to minimise the measurement time by collecting only the raw transmission curves; curve smoothing, extraction of the resonant frequencies and Q-factors, and then their conversion to the material parameters, is performed at the post-processing stage.
- It is also recommended to measure the empty resonator both at the start and at the end of the scanning process. A shift in the iSiPDR resonant frequency and Q-factor will be a measure of the additional material imaging error (absent in the classical use of SiPDRs, where the reference and sample measurements are performed instantaneously one after the other). Studies on temporal and thermal stability of the SiPDRs are currently under way in order to formulate further recommendation for temperature monitoring and multiple temperature-depended scanner calibrations.

Forthcoming revisions and variants of this SOP

This SOP summarises the principles of SiPDR measurements based on the original literature [1] and QWED's further modelling activities, e.g. [2]. It also incorporates critical observations from QWED engineers made during the 20 years of their own experience in manufacturing and using material test-fixtures as well as assisting their users in the market segments supported so far. In particular, the industrial implementation of QWED SiPDRs to NanoBat battery materials has brought new observations and led to the development of the large surface SiPDR scanner, presented in the final section. On-going studies are concerned with long-term stability of SiPDR measurements that must be considered when imaging large material surfaces in the course of many hours; the results will be discussed in forthcoming revisions of this SOP.

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