D6.4
AT LEAST THREE SOPS ON SETTING UP AND CONDUCTING MEASUREMENTS WITH THE SMM, DIELECTRIC RESONATORS AND COAXIAL PROBES TO ENABLE A MAXIMUM OF REPRODUCIBILITY (T6.4)

Contractual delivery date:	M18
Actual delivery date:	M18

DOCUMENT INFORMATION

Version	V1	**Dissemination level**	X
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PROJECT INFORMATION

Grant Agreement n°	761036
Dates	1st November 2017 – 31 October 2020

DOCUMENT APPROVAL

Name	Position in project	Organisation	Date	Visa
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DOCUMENT HISTORY

Version	Date	Modifications	Authors
1	29.4.2018	Initial	Johannes Hoffmann

TABLE DES MATIERES

1 EXECUTIVE SUMMARY

1.1 Description of the deliverable content and purpose

This deliverable will gather at least three SOPs on setting up and conducting measurements with the SMM, dielectric resonators and coaxial probes to enable a maximum of reproducibility (T6.4).

The SOPs have been prepared based on the experience of the project partners. Additionally, the SOPs have been reviewed by stakeholders of the MMAMA project and by other interested parties.

1.2 Brief description of the state of the art and the innovation breakthroughs

N.A.

1.3 Corrective action (if relevant)

N.A.

1.4 IPR issues (if relevant)

N.A.

2 SOP ON SMM

Version 1

TITLE: Data acquisition with a Scanning Microwave Microscope

AUTHOR: Arne Buchter, Toai Le Quang, Denis Vasyukov, Johannes Hoffmann

SHORT DESCRIPTION: This document's intention is to provide a generic and instrument-unspecific guideline to help in the process of acquiring reliable and reproducible data with the Scanning Microwave Microscope. Areas covered are sample and microscope handling, data storage and a few quick reminders on data analysis. Extensive advice on data analysis is beyond the document's scope.

ABBREVIATIONS / TERMINOLOGY:
SMM – Scanning Microwave Microscope
VNA – Vector Network Analyzer
S11 – Scattering parameter

VERSION / DATE: v0.6 / 13.03.2019

In general a SMM consists of a Scanning Probe Microscope (SPM) which is interfaced with devices to measure vector microwave reflection and/or transmission. Therefore it is inevitable to respect requirements of the mostly mechanically sensitive scanning probe part of the SMM and the sensitive high frequency components. The following is a collection of advice for good practice to ensure stable and reliable operation of a SMM to obtain high quality data. The content is kept generic and avoids too much detail, which is instrument or lab specific. The document instead focusses on general good practice, which can be followed in any lab operating a SMM. There are three sections in the following. The first one deals with the lab and setting up a SMM in general and sample and data storage. This serves as the foundation and should in the best case only needed to be followed once, but reviewed on a regular basis. The second part provides advice on the actual measurement process which is to be followed in day-to-day activities. The last part provides some general advice on the analysis of SMM data.

2.1 GENERAL

Performing experiments with a SMM might expose the operator to laser light, high voltage, sharp objects, chemicals and other hazards thus it is important to ensure proper training of the SMM operator according to local safety regulations and device specificities. This includes familiarity with available handbooks, lab regulations or other available documentation specific to local premises and instruments.

Ensure stable lab climate (temperature, humidity) to enable stable operation of the SMM with minimum mechanical, thermal, and electrical drift. Avoid opening windows, direct sunlight on the experiment, sources of electromagnetic radiation, etc. In the best case humidity, temperature and pressure are logged to allow tracing back implausible SMM measurements and check possible influences of one of these parameters.

Ensure calm, vibration free environment for the SPM and VNA including the interfacing cables - especially while experiments are conducted. The SMM should be placed in a calm position in the lab, away from noise sources, ventilation and passersby. In the best scenario, the SMM should be shielded from external changes in electromagnetic field e.g. by putting it in a Faraday cage. It should be placed in an acoustic case and on a vibration isolation mount like an optical table or other damping platform. To reach maximum control on environmental variables the SMM can be placed in a glovebox which hosts a controlled nitrogen-environment and thus also avoids any condensation or water layers on sample and probe.

Ensure proper connection of coaxial cables from VNA to the SMM probe. Clean connectors, use torque wrench and ensure a stable position of all the cables and respecting their specific minimal bending radius. Moving cables can lead to significant change in S11 (especially the phase). Keep cables as short as possible to reduce losses. Make sure cables fit to the frequency range used in experiments

In the best case, samples and SMM tips should be stored in a clean, dry, dust free desiccator at stable temperature. There are other and better options for storage such as: Nitrogen dry box or vacuum chamber.

Ensure proper and unambiguous sample labelling to avoid mixing up during storage and experiment.

When handling samples and sensitive parts of the SMM it is advisable to wear nitrile gloves and use tweezers where appropriate. Plastic tweezers are appropriated for samples manipulations in “dice” configuration. Where necessary avoid electrostatic discharge by proper grounding of the operator. Moreover, while handling SMM tips, it is recommended to avoid any mechanical contact to the end of the tip, which can cause both mechanical and electrical damages.

Work according to local IT policies and ensure safe data storage with regular backups. Best to avoid local hard drive and use a server storage.

Keep raw data and post-processed data separate and make sure to not overwrite raw data during analysis treatment.

2.2 MEASUREMENT

Make sure all relevant devices are switched on. To ensure stable operation the VNA should be switched on, with source power off, for at least a day before measurements start.

Confirm tip status and make sure it is still in proper shape for measurements. Perform visual inspection of the sample before measurement and check for dust, scratches, etc. Depending on availability this can be done using an electron or optical microscope.

Before starting measurement an instrument specific warm up period should be respected.

Make sure to log all relevant metadata (sample, experimental settings, environmental conditions, etc.). An example of a comprehensive set of metadata for SMM and other microwave techniques can be found on DOI 10.5281/zenodo.2591367.

Choose suitable microwave frequency for the experiments. Setting parameters as input RF power, operation frequency, intermediate frequency bandwidth (IFBW), scanning area, scan speed, scan direction, number of points/pixels are chosen depending on the material physical and geometrical properties as well as the characteristic response of the microwave electronics.

Before doing SMM measurements on a sample of interest, test the electrical sensitivity of the SMM probe by scanning on well-defined samples, calibration kits for SMM. Currently, there are commercial kits, i.e. capacitive kits provided by Prime Nano consisting of different dielectric (typ. SiO_2) terraces or resistive/inductive and capacitive gold structures on silicon nitride membranes provided by METAS.

It is highly recommended to calibrate the SMM to ensure the traceability of measurements to the SI, *i.e.* to get reliable and comparable quantitative results.
Save data according to local naming convention using unique names. Useful schemes could include sample ID, microwave frequency, scan parameters (range, pixels), timestamp.
After experiments the SMM should be brought back to standard conditions agreed on in the lab and sample is to be unmounted and stored safely.

2.3 ANALYSIS

One or more of the following types of data are usually generated during SMM measurements:

- Uncalibrated S11 in real/imaginary or abs/angle
- Topography, phase, dissipation, excitation amplitude

These quantities can be either stored for points, line scans or complete 2D images or in rare cases even 3D images.

Cross-check topography and S11 to distinguish topography-induced S11 signals (unwanted) and those of purely material-related origin (wanted).
Even though Phase (S11) and Magnitude (S11) reproduce very often, it is a must to carefully check both for unique features.

Further analysis according to common practice.

3 SOP ON COAXIAL PROBE

TITLE: Data acquisition with Coaxial dielectric probes

AUTHORS: Georg Gramse, Ivan Alic, Manuel Kasper, Mykolas Ragulskis, Manuel Moertelmaier, Ferry Kienberger

SHORT DESCRIPTION: This document's intention is to provide a generic and instrument-unspecific guideline to help in the process of acquiring reliable and reproducible data with a coaxial dielectric probe. Areas covered are sample and coaxial probe handling, data storage and a few quick reminders on data analysis. Extensive advice on data analysis is beyond the document's scope.

ABBREVIATIONS / TERMINOLOGY:

VNA - Vector Network Analyzer:
S11 - Scattering parameter
ESD - Electrostatic discharge
CDP - Coaxial dielectric probes
RFI - radio-frequency interference
MUT – material under test

VERSION / DATE: v0.1 / 08.04.2019

3.1 Introduction

This document attempts to provide a generic and instrument unspecific guideline for use of dielectric probes. The intention is to provide basic information needed to perform reliable and repeatable and safe measurements. Text gives notes on probe handling and maintenance, performing measurements, and common sources of errors.

Coaxial dielectric probe is a rigid coaxial structure of well-defined, known dimensions and electrical characteristic. The fields at the probe end “fringe” into the material and change as they come into contact with the MUT. The material is measured by immersing the probe into a liquid or touching it to the flat face of a solid (or powder) material. The reflected signal (S11) can be measured and related to dielectric permittivity. Probe is typically connected to VNA that performs S11 sweep over desired frequency range.

3.2 Equipment and operator safety

Performing CDP measurements may expose operator to material under test. Necessary persuasions should be taken to avoid injury or exposure to harmful substances.

VNA is sensitive to static electricity, 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.

3.3 Calibration and measurement

A typical measurement system using a coaxial probe method consists of a network analyzer or impedance analyzer, software to calculate permittivity, and a coaxial probe, probe stand and cable.

Ensure proper connection of coaxial cables from VNA to the CDP. Therefore all connectors should be checked in case there is any damage on the inner or outer connector or any dirt. To tighten cables the appropriate torque wrench should be used which ensures good connection and prevents damage to the connectors. Ensure a stable position of all the cables and respecting their specific minimal bending radius. Moving cables can lead to significant change in S11 phase thus voiding the probe calibration. Keep cables as short as possible to reduce signal losses. If application allows use rigid cables and avoid movement. Make sure cables are specified to work in the frequency range used in experiments.

Before measuring, calibration of the probe must be performed. A three-term calibration corrects for the directivity, tracking, and source match errors that can be present in a reflection measurement. In order to solve for these three error terms, three well-known standards are measured. The difference between the predicted and actual values is used to remove the systematic (repeatable) errors from the measurement. The three commonly used known standards are air, a short circuit, and distillate and de-ionized water. Even after calibrating the probe, there are additional sources of error that can affect the accuracy of a measurement.

Stable environment is important for experiment repeatability, environment temperature should be stable and known, sample temperature should be known, controlled and recorded. Sample contamination should be avoided by use of clean containers, tweezers and other instruments that contact the sample including the probe itself. Clean the probe between measuring calibration materials (especially in case of liquids) and MUTs. If using cleaning agent such as alcohol, make sure it evaporated from the probe surface.

The MUT volume must be sufficient for correct results, sensing volume depends on probe aperture. As a rule of thumb the MUT depth should be several times greater than the probe aperture, container diameter, (lateral dimensions of the sample) should be double of the aperture diameter. These are general rules and exact minimal dimensions should be experimentally determined. Minimum depth of the MUT can be determined experimentally by applying conductive metal plate behind MUT, if no change in measured S11 or dielectric parameters is observed, sample depth is sufficient.

If measuring liquids, ensure there are no air bubbles at the probe end after immersion of the probe. Measuring rigid solids is difficult because it is not possible to ensure good contact between the probe end and the material; any tilt and the air gap will introduce a large error.

Soft solids can be measured if it is possible to ensure contact without air gaps. It should be noted that force applied to the sample in contact with the probe can cause changes in material properties.

After a measurement session it is good to measure a known standard to check if calibration is still valid and if system parameters have changed. Ideally known standard is measured after calibration and once more after measurement session.

Computer software calculates dielectric properties from S11 data, the final results always depend on the SW algorithm and probe model used, its limitations should be known and taken into account.

4 SOP ON DIELECTRIC RESONATOR

TITLE: Data acquisition with Split - Post Dielectric Resonators

AUTHORS: Malgorzata Celuch, Janusz Rudnicki

SHORT DESCRIPTION: This document's intention is to provide a guideline to help in the process of acquiring reliable and reproducible data with a split - post dielectric resonator (SPDR). Areas covered include preparation and handling of measurement setups and material samples. Field distributions in SPDR 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 but also errors are pointed out in the existing IEC norm.
INSTRUMENT SPECIFICITY: This guideline is targeting to be instrument unspecific, subject however to two constraints:

1. At the time of writing, the only SPDRs commercially available on the open market are those from QWED. While interested users may construct their own SPDRs based on the open literature referenced hereinfurther, it is not guaranteed that such devices provide the same simplicity of operation, in particular, that they come equipped with their device-tailored calibration software and require no further calibration by the user.
2. SPDR needs a microwave signal generator with a function of measuring transmission (S21) characteristics between the SPDRs two ports and extracting the resonant frequency and 3dB bandwidth. In principle, those functionalities can be provided by any VNA or a simpler customised device. However, the operation of QWED SPDRs is most straightforward when used jointly with KEYSIGHT VNAs including Option 003 in their N1500A Material Measurement Suite or QWED Microwave Frequency Q-Meter.

ABBREVIATIONS / TERMINOLOGY:
SPDR - Split - Post Dielectric Resonator
VNA - Vector Network Analyser:
Q-Meter - Microwave Frequency Q-Meter from QWED
S21 - Scattering parameter - transmission
MUT / SUT – material under test / sample under test
MW / RF - Microwave / Radio-frequency
ESD - Electrostatic discharge

VERSION / DATE: v0.1 / 08.04.2019

4.1 Introduction: principles of the SPDR method

This document attempts to provide a generic guideline for use of split - post dielectric resonators (SPDR). 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 atttention is paid to the different ways of operating the only commercially available SPDRs from QWED within the three types of measurements setups: a nonspecific VNA, KEYSIGHT VNAs including Option 003 in their N1500A Material Measurement Suite or QWED Microwave Frequency Q-Meter.

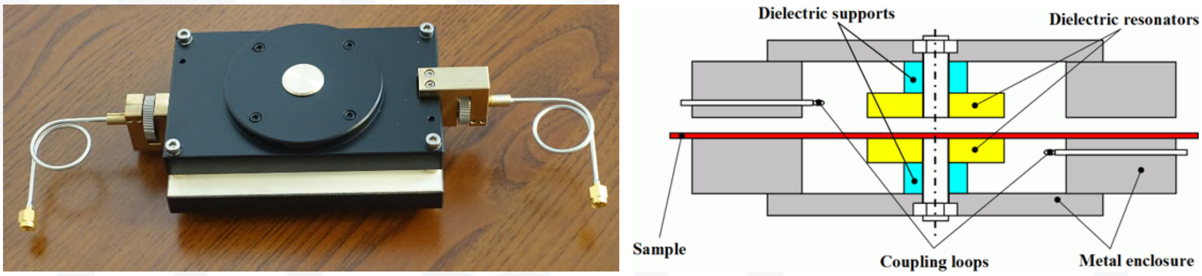


Fig. 1: Example SPDR and its internal construction after [1][2][3].

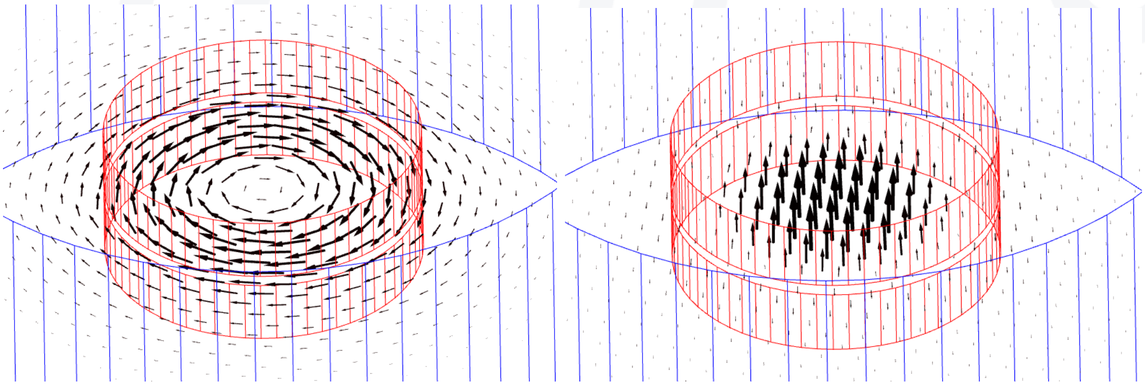


Fig. 2: Electric (left) and magnetic (right) field distribution in SPDR at the resonant frequency; the ceramic posts are here marked in red, the field lines in black, and the metal enclosure in blue.

SPDR is a rigid structure of well-defined, known dimensions and materials. Looking from the outside (Fig. 1 left) SPDR 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 a pair of dielectric cylindrical posts made of high-permittivity low-loss ceramics (yellow in the diagram of Fig. 1 right, red in Fig. 2). At the operating frequency, a resonant mode if formed, with electromagnetic fields mostly confined between those ceramic posts, as shown in Fig. 2. Losses in the metal enclosure are thereby minimal, moreover, since the magnetic field has only a vertical component, the currents in the side wall of the enclosure are only circumferential, and there is no radiation through the slot. All electromagnetic energy injected through the coupling loops in contained within in the SPDR “head” (inside the enclosure); an estimated 90% of energy is confined between the ceramic posts. *Note that the mode used in SPDR measurements is axisymmetrical, contrary to Figures B.2 and B.3 of norm [2], which are incorrect.*

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 (Fig. 3). The IEC norm [2] refers to frequencies between The IEC norm [2] refers to frequencies between 1.1 GHz and 20 GHz, the commercially available resonators from QWED cover several discrete nominal frequencies between 1.1 GHz and 15 GHz. SPDRs for other frequencies can be designed following the theory of ref. [1], however, at lower frequencies SPDRs become big and bulky, while it higher frequencies manufacturing tolerances and losses in the applied materials deteriorate the measurement accuracy.

The SPDR method requires making two consecutive measurements: one of the empty SPDR and one with the SPDR loaded with the sample. The principle of the method resides in extracting the real part of the SUT permittivity from the change of the resonant frequency, and the imaginary part - from the change of the Q-factor. The details of the SPDR method are presented in publication [1] and summarised in the IEC norm [2].

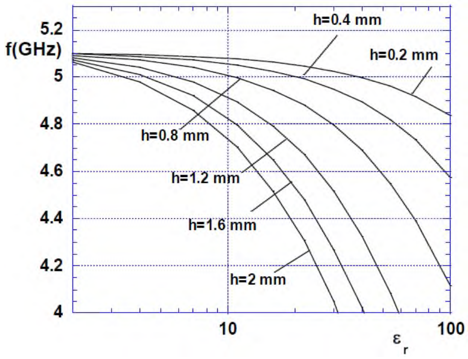


Fig. 3: Resonant frequency (f) of an example (nominally) 5 GHz SPDR loaded with a SUT of height h and permittivity relative permittivity ε_r.

4.2 Handling and safety of resonators

SPDRs are passive structures, and the measurement mode is well shielded from the environment (Fig. 3), 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 and leakage though the slot may occur.

SPDRs 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 SPDR should be handled with care and kept clean. The coupling loops within the resonator (Fig. 1) are specifically delicate. QWED SPDRs 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 individual calibration, each SPDR comes with its own (unit-specific) version of the software. A backup copy of the software should be kept in a safe place and clearly linked to a particular SPDR unit.

SPDR 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 SPDRs 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 SPDRs 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 SPDR's own software happened.

4.3 Sample types, preparation and safety

SPDRs are originally intended for the measurements of the **complex permittivity of laminar dielectric materials**. Other documented applications include thin ferroelectric films deposited on low loss dielectric substrates as well as sheet resistance and conductivity of various conducting materials such as commercial resistive layers, thin conductive polymer films or high resistivity semiconductors. As per Fig. 2, electric properties of SUT are measured in the tangential electric field (and hence are in-plane permittivity and conductivity); while magnetic properties are measured in the normal magnetic field (and hence are out-of-plane complex permeability).

The sample under test (SUT) should be flat and thin enough to fit into the SPDR's slot. SUT's lateral dimensions must be big enough to cover the SPDR "head" (somewhat smaller SUTs are measurable but with reduced accuracy) but also the width must be small enough to fit through the slot (which is a mechanical constrained that can be alleviated by a customised SPDR construction). There are no formal requirements on the minimum thickness of the SUT, also SUT rigidness and surface polishing are not absolutely necessary, thanks to the fact that *air slots between the SUT and the fixture do not affect the measurements* (which is quite a unique feature of the SPDR measurement method, resulting from the use of the SPDR tangential electric field, Fig. 2). However, for very thin SUTs the extraction of permittivity becomes ill-conditioned (see Fig. 3), and non-uniformity (Δh) of the SUT height (h) transfers into inaccuracy of its measurement. According to the IEC norm [2], to which QWED SPDRs comply, the errors of measuring SUT relative permittivity and loss tangent are within:

$$\Delta\varepsilon/\varepsilon=\pm(0.0015+\Delta h/h)$$

$$\Delta\tan\delta=\pm2*10^{-5}\text{ or }\pm0.03*\tan\delta\text{ whichever is higher}$$

Note that the IEC norm [2] includes Table 1 "Speciment dimensions" and Table 2 "SPDR test fixture's parameter" for several nominal frequencies. Those tables should be interpreted with caution, taking into account our earlier remarks. The size of SPDR "head" and gap (Table 2 of [2]) may be somewhat different, depending e.g. on the ceramics used for the posts (undefined in [2]). On the other hand, maximum SUT dimensions (Table 1 of [2]) are not absolute electromagnetic constraints, but can be increased with a particular mechanical construction. QWED standard SPDRs (as per table below) comply with the IEC norm [2] for "head" and gap sizes, generally allow for slightly wider samples, and pose no fundamental restictions of SUT length. A recent design within the MMAMA project which additionally allows for surface scanning with an SPDR will be subject to separate SOP, to be prepared after more tests and validation.

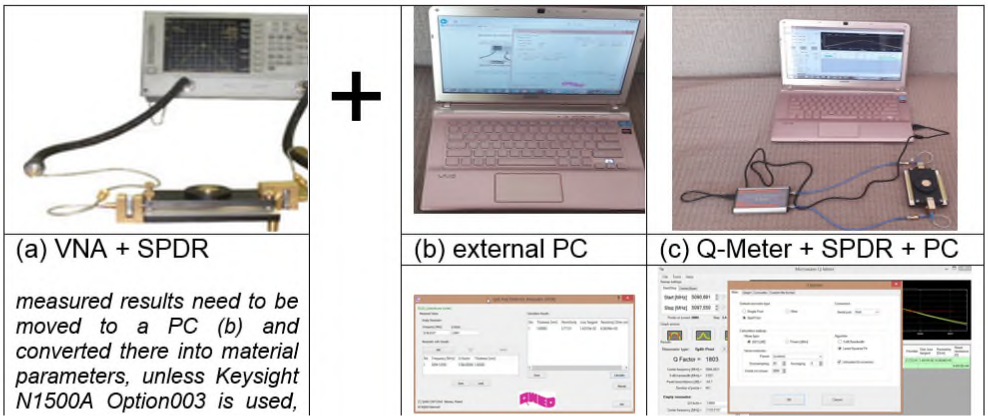
Nominal frequency [GHz]	**Min size of SUT [mm] (diameter D or square D x D)**	**Max thickness of SUT [mm]**	**Max width of SUT (standard version)**
1.1	120	6.00	150
1.9	70	4.00	100
2.5	55	3.10	100
5.1	30	1.95	90
10	22	0.95	90
15	14	0.60	40

The operator inserts a SUT into the SPDR 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 manufacturer must be adhered to. Note that it is also possible to manouver delicate SUTs placed on a paper or teflon foil - such thin supporting sheets do not noticeably affect on the measurement, moreover, their effect can be eliminated by taking the SPDR loaded with the supporting sheet (instead of the empty SPDR) as reference.

In application to copper clad laminates, all cladding must be removed by etching, then a SUT must be cleaned and dried. Any SUT should be conditioned at the SPDR operating temperature prior to testing. SUT thickness must be measured with a micrometer of 0.001 mm resolution (or better). For each SPDR unit, at least one verification SUT should be maintained, preferably marked by an engraving pen or other suitable method.

4.4 Measurement setups, equipment and operator safety

SPDR measurements can be performed at room temperature or in environmental test chamber [2]. In all tests the ambient test temperature must not exceed beyond the operating temperature range of a particular SPDR declared by its manufacturer. The IEC norm [2] further strictly declares that the temperature variation should not exceed 1 deg C during test. In practice this means that the measurement of the SPDR loaded with SUT must quickly follow the measurement of the empty SPDR, and test environment (temperature, humidity) should be recorded together with the test results.


(a) VNA + SPDR

measured results need to be moved to a PC (b) and converted there into material parameters, unless Keysight N1500A Option003 is used,

(b) external PC

(c) Q-Meter + SPDR + PC

Fig. 4: Measurement setups bulit with SPDR: classical with SPDR connected to a VNA (a) which except for selected KEYSIGHt VNAs needs data tranfer to a PC (b); compact consisting of SPDR, Q-Meter and PC running a dedicated Q-Meter software.

VNA + SPDR measurement setup

A classical measurement setup consists of an SPDR and a VNA (Fig. 4a). VNA is sensitive to static electricity, 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 SPDRs to be applied; and the dynamic range recommended by the IEC norm [2] is more than 60dB. 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 SPDR, make sure this SPDRs specific software has been installed. In case of other VNAs, be prepared to copy the measured data and transfer it to a separate PC, with the SPDR specific software installed on it.

Connect the SPDR to the VNA and follow the VNA instructions. Start with the empty SPDR. Enable the option of S21 magnitude measurement. Set the centre frequency at the nominal frequency of the SPDR. 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 SPDR 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 SPDR 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, move the raw measured results to the PC. In the latter case, maintain special care to clearly annotate those results.

VNA + Q-Meter measurement setup

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

An example of such devices is Microwave Frequency Q-Meters from QWED, available for several frequency ranges compatible with the operating frequencies of QWED SPDRs. Essentially, Q-Meter is a computer controlled microwave oscillator system for quick and automatic SPDR 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 SPDR, Q-Meter and a laptop is shown in Fig. 4c. 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.

4.5 Forthcoming revisons and variants of this SOP

This SOP summarises the principles of SPDR measurements based on the original literature [1] and the pre-existing IEC norm [2], also incorporating critical observations from QWED engineers made during the 20 years of their own experience in manufacturing and using SPDRs as well as assisting other SPDR users in the market segments supported so far. It is hoped that the industrial implementation of QWED SPDRs at MMAMA partners will bring new observations that will be further added to this SOP. Also, another SOP will be prepared for the newly constructed large surface SPDR scanner.

While the split-post configuration was originally selected for dielectric resonator measurements in the MMAMA project, current activities indicate that the single-post configuration may be more relevant to some of the considered materials, e.g. highly conductive composites. When experience is gained, a separate SOP on single-post dielectric-resonator measurements will be prepared.

4.6 References

[1] J. Krupka, A. P. Gregory, O. C. Rochard, R. N. Clarke, B. Riddle, and J. Baker-Jarvis, “Uncertainty of complex permittivity measurements by split-post dielectric resonator technique”, *J. Eur. Ceramic Soc.*, vol. 21, pp. 2673-2676, 2001.
[2] European Standard: IEC 61189-2-721:2015
[3] http://www.qwed.eu/resonators_spdr.html

Introduction

This document attempts to provide a generic and instrument unspecific guideline for use of dielectric probes. The intention is to provide basic information needed to perform reliable and repeatable and safe measurements. Text gives notes on probe handling and maintenance, performing measurements, and common sources of errors.

Coaxial dielectric probe is a rigid coaxial structure of well-defined, known dimensions and electrical characteristic. The fields at the probe end “fringe” into the material and change as they come into contact with the MUT. The material is measured by immersing the probe into a liquid or touching it to the flat face of a solid (or powder) material. The reflected signal (S11) can be measured and related to dielectric permittivity. Probe is typically connected to VNA that performs S11 sweep over desired frequency range.

Equipment and operator safety

Performing CDP measurements may expose operator to material under test. Necessary persuasions should be taken to avoid injury or exposure to harmful substances.

VNA is sensitive to static electricity, 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.

Calibration and measurement

A typical measurement system using a coaxial probe method consists of a network analyzer or impedance analyzer, software to calculate permittivity, and a coaxial probe, probe stand and cable.

Ensure proper connection of coaxial cables from VNA to the CDP. Therefore all connectors should be checked in case there is any damage on the inner or outer connector or any dirt. To tighten cables the appropriate torque wrench should be used which ensures good connection and prevents damage to the connectors. Ensure a stable position of all the cables and respecting their specific minimal bending radius. Moving cables can lead to significant change in S11 phase thus voiding the probe calibration. Keep cables as short as possible to reduce signal losses. If application allows use rigid cables and avoid movement. Make sure cables are specified to work in the frequency range used in experiments.

Before measuring, calibration of the probe must be performed. A three-term calibration corrects for the directivity, tracking, and source match errors that can be present in a reflection measurement. In order to solve for these three error terms, three well-known standards are measured. The difference between the predicted and actual values is used to remove the systematic (repeatable) errors from the measurement. The three commonly used known standards are air, a short circuit, and distillate and de-ionized water. Even after calibrating the probe, there are additional sources of error that can affect the accuracy of a measurement.

Stable environment is important for experiment repeatability, environment temperature should be stable and known, sample temperature should be known, controlled and recorded. Sample contamination should be avoided by use of clean containers, tweezers and other instruments that contact the sample including the probe itself. Clean the probe between measuring calibration materials (especially in case of liquids) and MUTs. If using cleaning agent such as alcohol, make sure it evaporated from the probe surface.

The MUT volume must be sufficient for correct results, sensing volume depends on probe aperture. As a rule of thumb the MUT depth should be several times greater than the probe aperture, container diameter, (lateral dimensions of the sample) should be double of the aperture diameter. These are general rules and exact minimal dimensions should be experimentally determined. Minimum depth of the MUT can be determined experimentally by applying conductive metal plate behind MUT, if no change in measured S11 or dielectric parameters is observed, sample depth is sufficient.

If measuring liquids, ensure there are no air bubbles at the probe end after immersion of the probe. Measuring rigid solids is difficult because it is not possible to ensure good contact between the probe end and the material; any tilt and the air gap will introduce a large error.

Soft solids can be measured if it is possible to ensure contact without air gaps. It should be noted that force applied to the sample in contact with the probe can cause changes in material properties.

After a measurement session it is good to measure a known standard to check if calibration is still valid and if system parameters have changed. Ideally known standard is measured after calibration and once more after measurement session.

Computer software calculates dielectric properties from S11 data, the final results always depend on the SW algorithm and probe model used, its limitations should be known and taken into account.