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1 Executive Summary (not exceeding 1 page)

Introduction The central aim of EMINDA was to develop traceable RF and Microwave dielectric materials metrology both to improve the electromagnetic (EM) properties of existing materials and to enable the uptake of new EM materials by European industries, especially electronics and ICT-related industries. Prior to EMINDA, traceable metrology for the EM properties of many key materials at required scales and frequencies had not been well developed in Europe. After EMINDA new National Metrology Institute (NMI) facilities are available to fill in gaps in traceable dielectric metrology provision. Support to industry can now be given for the development of electroceramics and for the characterisation of electronics substrates, and traceability is newly available for dielectric measurements at both nanoscale and microscale resolutions, which is of value for the development of multiphase composites and measurements on electronic circuits.

The Problem In order to develop faster electronics and effective microwave communication systems, industrial developers need to understand the EM performance of their materials at RF and microwave frequencies. This enables them to implement energy-efficient component designs and effective process-control during manufacturing. Measurements at lower frequencies provide no information on the high frequency performance of materials, so RF and microwave measurement systems must be used. Prior to EMINDA, the European metrological infrastructure for such EM materials characterisation was not as well developed as that in many other industrially important fields of metrology, European NMIs were thus unable to support these European industrial activities as effectively as was required. RF and microwave EM materials measurements are in general difficult to perform and it was also perceived that there was a need for more-general metrological guidance for industry from the NMIs on effective measurements in this field.

The Solution The project sought to develop new capabilities at European NMIs and to transfer the know-how gained in this activity to support European industries in order to solve their EM material measurement problems. The metrology tackled was chosen on the basis of two criteria. Firstly, it concentrated on the frequencies and dimensional scales that were of most practical importance for the European ICT and electronic industries, and, secondly, it concentrated on areas where the expertise of the EMINDA NMI partners could most effectively support industry. It covered microwave metrology for frequencies up to 80 GHz and, in some techniques, also at micro- and nanoscale resolutions on composites and 'on-wafer' for electronic components. An overarching objective was to develop a suite of traceable metrological tools for characterising passive (dielectric and magnetic) and active 'functional' EM materials across the spectrum. Broader support and guidance to industry was provided via measurement workshops, Good Practice advice and a measurement Club – the EMMA Club – which provided a forum for exchange of information.

Impact The project succeeded in developing its targeted measurement techniques: it introduced traceability into measurement areas where it was previously unavailable, it improved knowledge of sources of measurement uncertainty for other techniques where a reduction in the magnitude of uncertainties has proved possible and it has added to the range of calibration artefacts and calibration methods that can be used with these techniques to provide traceability. A number of the techniques studied are now available as NMI measurement services, for example the Near-Field Scanning Microwave Microscopes (NSMM) at NPL (UK), and others are either already in use by industrial and academic collaborators or else are available for their longer term use. Thus Keysight Technologies (Austria) have utilised research outputs from this project to provide traceability with nanoscale spatial resolution for EM microwave measurements using NSMMs while new RF measurement cells are in use by the Josef Stefan Institute (Slovenia) for characterising electroceramics traceably. Longer term impact from the project will be guaranteed by the existence of the new measurement facilities and know-how in the EMINDA NMIs.

On the broader industrial support front, significant effort has been put into transfer of know-how from EMINDA's NMI Partners to industry and research institutions. Thus, good practice advice is available on the EMINDA web-site and the establishment of the measurements club ('EMMA') has ensured that the project's outputs have reached a wide industrial community. . During EMINDA there were four international meetings of the Club and an international workshop on microwave dielectric metrology in Ljubljana (2013). Overall, well over 100 scientists and engineers (in addition to EMINDA partners) have attended these meetings which succeeded in transferring measurement knowledge to them, as demonstrated by the positive feedback received from them.

2 Project Context, Rationale and Objectives

To develop faster electronics the electronics industry needs to understand the performance of materials at higher frequencies in order to design the most effective and energy efficient components and systems and to enable effective process control during manufacturing.

Measurements at lower frequencies do not provide information on the microwave performance of materials. Consequently, the measurements tackled in this project were designed to provide information on the capacitive and conductive properties of materials, on how RF and microwave signals propagate through pure materials and on how signal power is absorbed or lost in them as these are critical to functionality and performance. The most important intrinsic material parameter that is used to quantify all of these properties is called the ‘complex permittivity’ of the material, and it is primarily for this quantity that the project set out to provide new and better industrially-relevant measurement tools for. The work was undertaken both to characterise and to help to improve the EM properties of existing materials and to support the development of new materials.

The availability of such measurements will support industrial innovation in new products with higher performance and improved energy efficiency and efficient production of these products. The project sought to both develop new capabilities in European NMIs in EM materials metrology at high frequencies and transfer the know-how to help industry to solve its EM material measurement problems.

The central aim of EMINDA was to develop traceable RF and Microwave Electromagnetic (EM) materials metrology to enable the uptake of new EM materials by European industries, especially electronics and ICT-related industries. It was recognised prior to the inception of EMINDA that the European metrological infrastructure for EM materials characterisation had not previously been as well developed as that in many other fields of metrology that are of importance for industry. In fact, prior to the inception of EMINDA, traceable metrology for the dielectric and magnetic properties of many key materials at required scales had never been fully developed, and in some cases measurement uncertainties had been only partially assessed - if they were assessed at all. Support for European Industries from NMIs in this area was highlighted as a means of remedying this situation. It was seen to be necessary that this support should be offered on **two fronts**:

- First, the development of key advanced measurement techniques
- Second, the provision of a broader infrastructure for EM materials metrology

On the second, broader, front the project aimed to widen the scope of traceable National Measurement Institute (NMI) measurement services for EM materials and to promote collaborative metrological ventures. This was partly approached through the establishment of a European measurement club which would run meetings for European scientists and engineers, workshops with training sessions and would promote good measurement practice. The industrial relevance of the work undertaken in EMINDA was much enhanced through consultation with our industrial collaborators and attendees at these club meetings and workshops, whose inputs were invaluable.

2.1 Advanced Measurement Techniques

On the first front the project targeted the development of advanced metrology for characterising passive (dielectric and magnetic) and active ‘functional’ EM materials across the spectrum from low frequencies (LF) 100 kHz through RF and up to microwave frequencies (80 GHz).

The metrological tools aimed to cover:

- A range of parameters that determine the dielectric, capacitive and conductive properties of materials: complex relative permittivity and permeability; magnetoelectric coefficients; coupling efficiencies in multiferroic and magnetoelectric materials and systems (charge, strain, stress, fields).
- A range of dimensional scales: at the micro- and nano-scale of individual materials and at the macro scale of substrates and thin-films.

- An understanding of the uncertainties inherent for each measurement technique in order to help industries to select the most effective and informative measurement methods appropriate to their measurement needs. For some of these techniques no uncertainty analysis had previously existed.
- Validated EM-field modelling as this is an essential factor in providing traceable measurements and uncertainties.

Specifically the project targeted advanced metrology at the frequencies and dimensional scales that were of most practical importance for European industries. It focussed on the measurement of the complex permittivity, $\varepsilon^* = \varepsilon' - j\varepsilon''$, of materials, where ε' is the real part of the permittivity, which quantifies the capacitive and transmissive properties of materials, ε" is the imaginary part of the permittivity, which quantifies power absorption ('loss') in the material and its electrical conductivity, and where $j = \sqrt{(-1)}$. Some of the research work in EMINDA (see Section 3.4) also addressed the measurement of magnetic complex permeability, μ*. (Ref. [2.1.1] provides a more detailed background of RF and Microwave electromagnetic materials metrology.) This commitment required:

- Metrology up to microwave frequencies, 80 GHz (the practical upper frequency limit set for EMINDA), to cover the ever increasing clock rates of electronic components
- Thin-film and micro- and nano-scale dielectric metrology, as these are the scales at which many emerging applications are being developed
- On-wafer materials metrology, both for substrates and thin films mounted on substrates, as this is the medium in which most RF & Microwave ICT is implemented
- Challenging metrology at the macroscale (centimetre scales) for high permittivity ceramics
- A focus on both passive dielectrics, which are used in substrate wave-front conditioning and support structures, and on active functional materials, such as ferroelectrics and magneto-electrics which are being targeted, for example, for tuning and switching applications

It should be noted that ICT and other industries require dielectric measurements to be performed at all scales down from the macroscopic (cm-scales) through micron scale down to the nanoscale. EMINDA addressed this wide range of scales to the extent that funding would allow.

The advanced measurement techniques chosen for development were those in which NMI-based metrology, and the existing expertise of European NMIs, could most effectively be pulled through to support European industries in this field.

In order to achieve the project's objectives three broad areas of metrology research were tackled:

Near-field Scanning Microwave Microscopes (NSMMs) at microwave frequencies are an emerging metrological method, which can potentially enable step changes in materials and device technology through the following measurements:

- Fully calibrated surface scanning of dopant levels in semiconductor circuits to assess their *in-situ* properties
- Charge-carrier dynamics and transport properties in general, including tunnelling
- Characterisation of domain dynamics in ferro- and piezoelectrics, multiferroics: relaxation and response times
- Probing high permittivity dielectrics used in on-chip passives for RF/wireless applications
- Assessment of thin-film uniformity and detection of defects and impurities in thin-film coatings
- Large area assessment of the integrity of thin films of new materials such as graphene
- Rapid combinatorial sampling of new electroceramic materials
- Characterisation of multiphase materials for optimising microwave processing
- Time-resolved conductivity and permittivity measurements

However, at the commencement of EMINDA, traceable, repeatable and reliable metrology for these techniques was in its infancy and it was perceived maximum benefit from NSMMs would not be obtainable without focused metrological research to ensure that measurements could be more quantitatively meaningful. EMINDA tackled this problem in two ways, respectively using Atomic Force Microscope (AFM) cantilevers for nano-scale probing and cavity-based sensors for micron-scale probing. The technical details of the work on NSMMs in this project are described below in Sections 3.1, which covers the AFM-based nano-scale probing, and 3.2 which covers micron-scale probing (cavity-based NSMM metrological research). Further details of NSMM measurements on active functional materials are given in Section 3.5

Co-Planar Waveguide (CPW) Based Measurements These on-wafer measurements cover broad-band methods that can characterise EM materials properties of both wafer substrates and of thin functional films mounted on those substrates. They have the advantage that they assess the RF & Microwave properties of materials in a medium (on-wafer) in which many of them will find end-use applications, and they are therefore particularly appropriate for assessing materials intended for ICT electronics applications. While other on-wafer transmission-line media are in common use (e.g. microstrip, stripline), CPW measurements have been shown to be the most traceable for metrological purposes and were therefore the chosen transmission line for EMINDA. Comprehensive traceability for these CPW methods was addressed for the first time ever in this project. All of these techniques rely on inverse electromagnetic field modelling to provide traceable links to materials properties. It is only possible to improve traceability by developing new validated modelling approaches, and these were therefore a key theme in this project, which tackled the modelling of both dielectric and magnetic thin films as well as modelling of CPW/substrate interactions. The technical details of co-planar dielectric measurements on substrate and laminas are described below in Section 3.3. CPW methods for measuring thin films were also investigated, in which the thin film is interposed between the metal CPW tracks and the substrate – this work is described in Section 3.4. Work on measuring such films with applied electrical and stress bias is discussed in Section 3.5

Macroscopic Measurements of bulk dielectrics are highly important to industry, for instance for dielectric resonators, for support components and for the conditioning of wavefronts, and they were also addressed in this project. Knowledge of the properties of substrate materials are important for optimising on-wafer electronics performance and to improve the metrology of EM substrates Split Cylinder (SC) measurements on laminar substrate materials have been developed significantly during EMINDA allowing measurements of permittivity to be undertaken at a much larger number of microwave frequencies – this work is described in detail in Section 3.3. Work was also undertaken to scan dielectric properties of thin-films and dielectric surfaces at macroscopic (mm – cm) scales using existing measurement techniques, cavities and Split-Post Dielectric Resonators (SPDRs) – this work is described in detail in Section 3.6. On another front, the challenging task of providing RF traceability for high permittivity ceramics was addressed through the development of new admittance cells (see Ref. [2.1.1]), allowing their operating range to be extended – this work is described in detail in Section 3.7. With these techniques, the aim was not only to improve the metrology and provide quantitative uncertainty information, but to extend the use of the techniques to new applications, for example the measurement and scanning of the surface conductivity of semi-conductors.

The very variety of methods that are needed to support industry in the measurement of complex permittivity on a broad range of materials at RF & Microwave frequencies must be emphasised. Some of these techniques operate at spot frequencies and others are broadband, some are targeted at laminar materials, some at thin films and some at bulk materials or at the monitoring of the EM properties of the surface of specimens. What they all have in common is their value to electronic and other industries in Europe. It should be pointed out that measurement uncertainties vary enormously from one technique to another and from one material to another, so it is not possible to state here an overall figure for uncertainties obtained in EMINDA. What can be stated about EMINDA is that in all areas of metrological research undertaken, better understanding of uncertainties has been achieved and that in many areas reduced uncertainties can now be obtained for the methods studied as a result.

Comparison of Techniques. A further key objective of this metrological work was to strengthen traceability and confidence in measurement through measurement comparisons between the different techniques studied in the EMINDA Project. Steps were put in place to ensure that Partners could benefit from this process. Wherever possible such comparisons were planned ahead of time to ensure, for example, that the same material specimens were of the correct shape and size to ensure that they could be measured by the different techniques and by the different Partners. Further details of Partner collaboration in EMINDA are provided in section 3.8.

Reference for Section 2.1.

[2.1.1] *'A Guide to the Characterisation of Dielectric Materials at RF and Microwave Frequencies'*, edited by R N Clarke, NPL Good Practice Guide, published by the Institute of Measurement and Control and NPL, 2003.

2.2 A Broader Infrastructure for EM Materials Metrology – Creating Impact

In addition to the future-oriented advanced metrological research just described, it was recognised that to create real impact in the field of RF and Microwave EM materials measurements, EMINDA needed to support European industries with their existing needs for metrology by establishing a better European infra-structure for this metrology. This aimed to create impact on three fronts:

- Via the EMINDA website, http://projects.npl.co.uk/eminda/project.html, scientists and engineers can easily access details of what the European NMIs can offer by way of measurement services and research support in this metrological area. Completion of EMINDA has enabled this offering to be expanded

- Via the EMINDA website, http://projects.npl.co.uk/eminda/good-practice/, they can access Good-Practice Advice on RF and Microwave EM materials measurements

- A European Electromagnetic Materials Measurements Club (the European EMMA Club) was established which enabled club members to exchange ideas with, and understand the needs of, European scientists and engineers. The EMMA-Club held two major international meetings and ran a metrology workshop during the course of the EMINDA project

More specifically, the aims of the EMMA club are:

- To provide a discussion forum for scientists and engineers who have an interest in measuring the electromagnetic properties of materials:
  - dielectric, magnetic, passive, functional and multifunctional materials
  - solids, liquids, natural, manufactured and metamaterials
  - at scales from macro to nano
- To present the latest scientific and technical developments relating to the measurement and applications of electromagnetic materials

To provide a consultation forum through which future European programmes of dielectric and electromagnetic materials research can be informed

The benefits and impact that have accrued from these support activities in the EMINDA project are described below in Section 4.2.

3 Research Results

The following sub-sections present the research results of EMINDA under headings that describe eight metrological areas upon which EMINDA focused. The ways in which this research contributes to metrology solutions in these areas is described. While these technical areas are identified separately here, it should be recognised that project partners working in each area benefitted enormously from the interchange of ideas, models, samples and cross-project collaboration generally in EMINDA, which occurred at partner's meetings, in workshops via conference calls and the Internet.

3.1 AFM Based NSMM Metrological Research – Nanoscale Scanning Metrology

Conventional EM material measurement techniques can measure the dielectric properties of samples which have a minimum size of millimetres or centimetres. As described above, because of the continuing miniaturisation of integrated circuits it is highly desirable to characterise sub-micrometre components. Other applications of this sub-micrometre metrology include the evaluation of fuel cell and battery membranes in which micro-cracks can lead to catastrophic failure.

This work concentrated on the measurement of permittivity and losses in sub-micrometre sized samples of dielectric material. The bulk of this work was undertaken in a collaboration between METAS, ETHZ and Agilent Technologies (now Keysight Technologies), but there were significant inputs from other members of the EMINDA consortium. These measurements were made with a cantilever based NSMM - an instrument similar to an atomic force microscope (AFM) but with a conducting tip, see Figure 3.1.1. The tip is connected via an impedance matching network to a vector network analyser (VNA), which measures the reflection coefficient from the tip, the reflection coefficient being sensitive to the sample's complex permittivity.

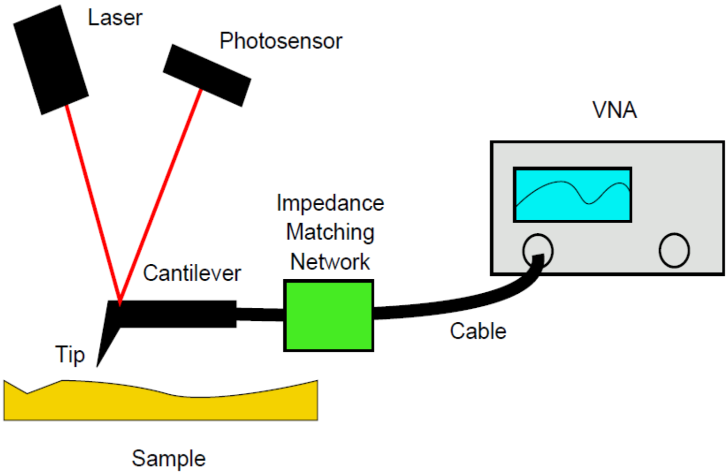


Figure 3.1.1 Principle of the cantilever-based NSMM Probe

At the start of the project, the first NSMM to be used for this work was based at Agilent Technologies in Linz, Austria and the project proceeded through a close collaboration between METAS and Agilent. The instrument was, prior to the commencement of the EMINDA project, effective for producing images but it was difficult to attribute a permittivity and loss tangent to each pixel in scanned dielectric images. Thus the research here concentrated on building models of how the material properties are translated to a measured value of the reflection coefficient on the VNA. Work included modelling of the tip-sample-interaction and the transfer function of the impedance matching network. In a second step the main uncertainty sources were identified and attributed to the relevant points of the measurement model. The uncertainties in the model are propagated through the model to the measurement result, see Ref [3.1.1]

A key research output was the application of a typical VNA 'one-port' measurement algorithm, for measuring reflection coefficient, to the NSMM. This works well under the assumption that the tip-sample interaction stays the same throughout the measurement, see Ref. [3.1.2]. The novelty in this approach is that it allows one to measure permittivity and loss tangent simultaneously. Previous approaches were not able to separate the two parameters.

A second key output is a boundary element algorithm, written by the researcher from ETHZ, which allows the computation of the tip-sample capacitance for rotationally symmetric tips within milliseconds, it is the variation of this capacitance that is detected by the VNA. Here co-operation between METAS, ETHZ and NPL helped project partners to understand just how models that provide one-port algorithms for NSMMs can improve their metrology. This more complete model of the AFM-based NSMM makes it possible to calibrate the VNA with varying tip-sample interaction. The speed of the simulator is crucial because there are often hundreds of different tip-sample distances which have to be evaluated for one calibration.

The third key output, also a result of a close collaboration between ETHZ and METAS, is a range of easy-to–manufacture calibration substrates for NSMMs which contain resistive and capacitive elements, see Figure 3.1.2.

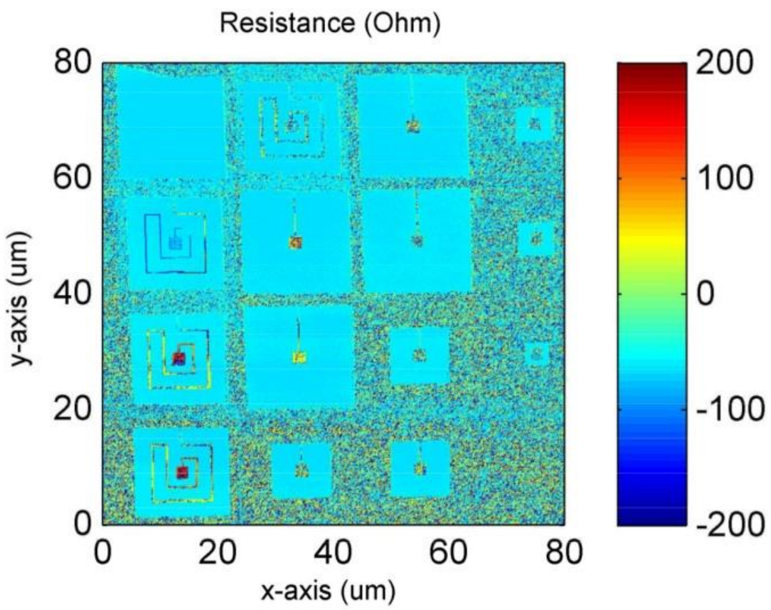


Figure 3.1.2 An NSMM scan of a calibration wafer built on a 30-nm thick silicon nitride membrane with a patterned 30-nm gold layer on top. The contact pads in the centre of the squares are connected to the surrounding Earth plane by thin resistive metal tracks of different lengths and thicknesses, thereby providing resistive elements that can be used to calibrate NSMMs for loss measurement

Experience gained in the earlier part of this project, working with Agilent Technologies enabled METAS to design and build their own NSMM during the EMINDA project, see Figures 3.1.3 to 3.1.6, which is a significant addition to the microwave materials measurement capability at METAS.

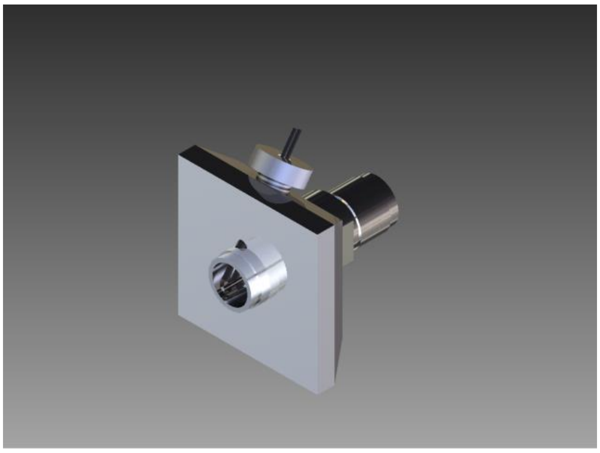


Figure 3.1.3 The head of the METAS NSMM. A tuning fork, seen at the top slightly inclined, is pressed against the tip, which in turn is inserted into a female 2.4-mm adapter (horizontal round section). The diameter of the NSMM tip wire is 500 micrometres

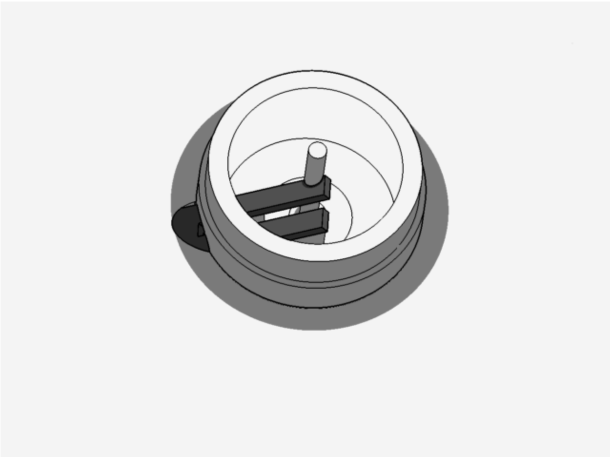


Figure 3.1.4. Detail view of the probe head of Figure 3.1.3. The wire for the tip is in the centre and the tuning fork is pressed against this wire. The ring around the wire and tuning fork is the casing of the 2.4-mm adapter. Note that an access hole is drilled through the casing for the tuning fork

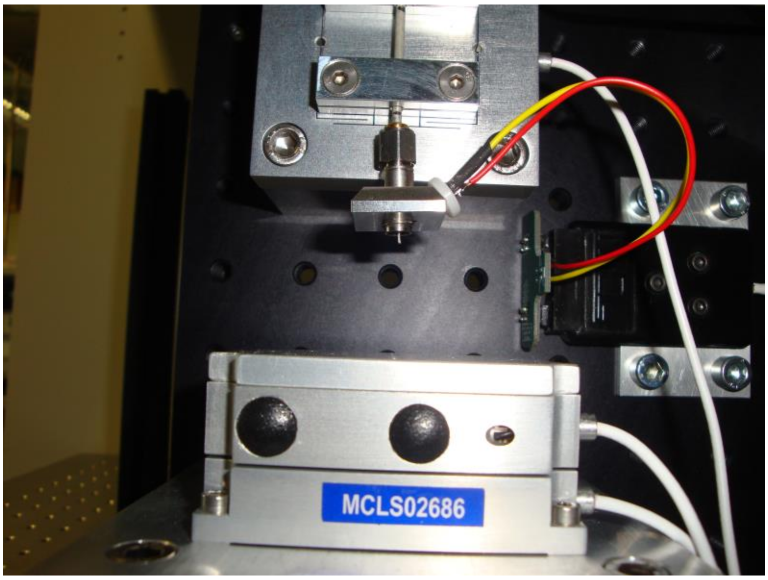


Figure 3.1.5 The assembled METAS NSMM. The lower block is a 200 micrometre x 200 micrometre piezo scanning stage. Above one can see the head depicted in Figures 3.1.3 and 3.1.4. The tuning fork is connected to the red and yellow cable. The semi-rigid coaxial line connection to the VNA and the head itself are held by a z-axis piezo stage with 30 micrometres range

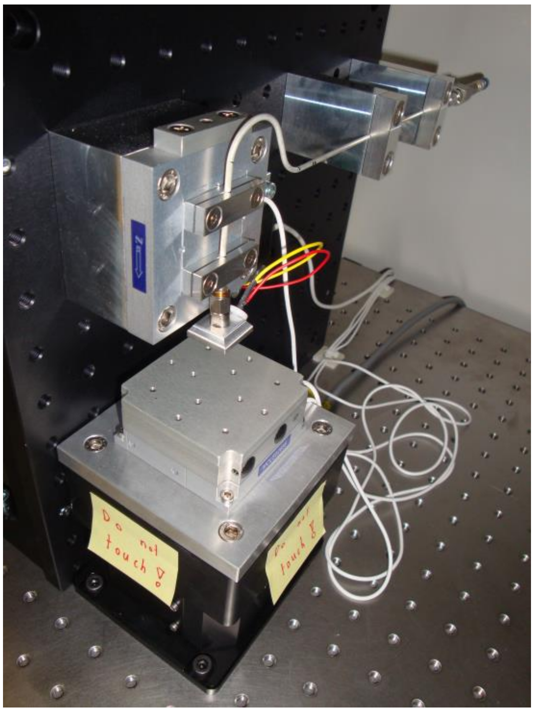


Figure 3.1.6 View of the whole METAS NSMM including the semi rigid access line between the VNA and the tip (top of diagram) and a coarse z-stage below the x-y-stage. The coarse z-stage permits travel of up to 20 mm vertically for the samples

In conclusion, the theoretical achievements include a comprehensive model of the NSMM, identification of error sources and propagation of these errors to the final result. The practical achievements are two different calibration algorithms with and without varying tip-sample interaction and an easy-to-manufacture calibration substrate which contains resistive and capacitive elements, see ref. [3.1.3].

References for Section 3.1

[3.1.1] Zeier, M., J. Hoffmann, and M. Wollensack. "*Metas. UncLib—a measurement uncertainty calculator for advanced problems*." *Metrologia,* **49.6** (2012): 809-815.

[3.1.2] Hoffmann, J., et al. *"A calibration algorithm for nearfield scanning microwave microscopes."* 12th IEEE Conference on Nanotechnology (IEEE-NANO), IEEE, 2012.

[3.1.3] Hoffmann, Johannes, et al. *"Measuring low loss dielectric substrates with scanning probe microscopes." Applied Physics Letters,* **105.1** (2014): 013102.

3.2 Cavity-based NSMM Metrological Research – Microscale Scanning Metrology

This research, which was largely based at NPL and Imperial College (IC), but with significant inputs from other EMINDA Partners, focussed on resonator-based NSMMs with spatial resolutions in the micron range. As stated above, this type of probe can study the surface properties of dielectrics and the integrity of thin films, potentially over a wide (cm-scale) area. Such instruments are used for measuring the complex permittivity of materials by means of a probe which is coupled to a resonant cavity, see Figure 3.2.1. A small fraction of the EM energy within the resonant system resides externally to the cavity, close to the probe tip. This is modified by the presence of a dielectric material placed at the probe tip. Measurements of the Q-factor and resonant frequency of the cavity/probe combination can therefore allow the complex permittivity of dielectric specimens to be determined. The EM response of the NSMM is analysed by means of a perturbation technique: the Q-factor and the resonant frequency of the resonator are determined by fitting to S-parameter measurements made on a Vector Network Analyser (VNA) and the small changes to these parameters that occur when the tip is close to a dielectric specimen are used to derive its complex permittivity.

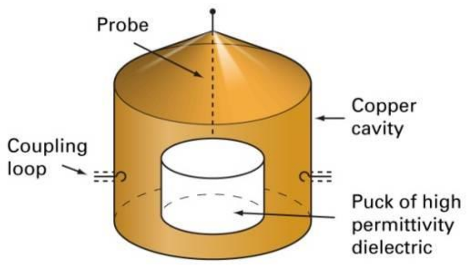
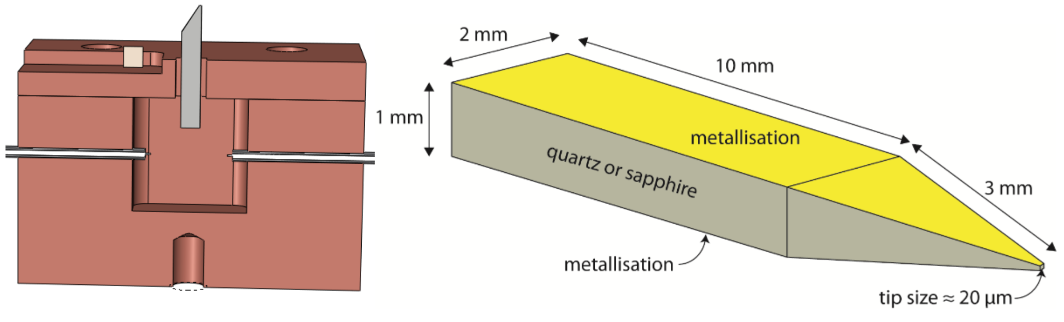


Figure 3.2.1 An NPL 2.4 GHz microwave cavity/probe combination that was used in the micron-scale NSMM research

Two fundamentally different designs of NSMM were studied within this project. Both of them were designed for measurements on the micron scale. The two designs are referred to as the RNSMM (Resonant Near-field Scanning Microwave Microscope), as shown in Figure 3.2.1 and the TNSMM (Transverse Near-Field Microwave Microscope), developed at IC, an example of which is shown in Figure 3.2.2. The RNSMM uses a length of tungsten wire with a spherical tip (50 µm or 100 µm diameter) as the probe. The TNSMM uses two parallel conductors formed on a strip of low loss dielectric (quartz or sapphire) sharpened to a point approximately 0.02 mm across. The advantage of this novel approach is that the E-field is confined to the locality of the parallel conductors. With this configuration the electric (E)-field is predominantly in the plane of the specimen surface. For the RNSMM, the electric field is less well confined and is largely perpendicular to the specimen surface. During EMINDA, both types of NSMM were integrated with a scanning system at NPL to provide a facility for the measurement of complex material on the micron scale, see Figure 3.2.3.



(a) (b)

Figure 3.2.2 One configuration for a TNSMM, as studied in this research at IC. (a) A probe/cavity assembly, with the probe (grey) emerging from the top, (b) A quartz probe in which the electric field at the tip lies in the plane of the tip face, at the extreme right, i.e. vertically in the diagram

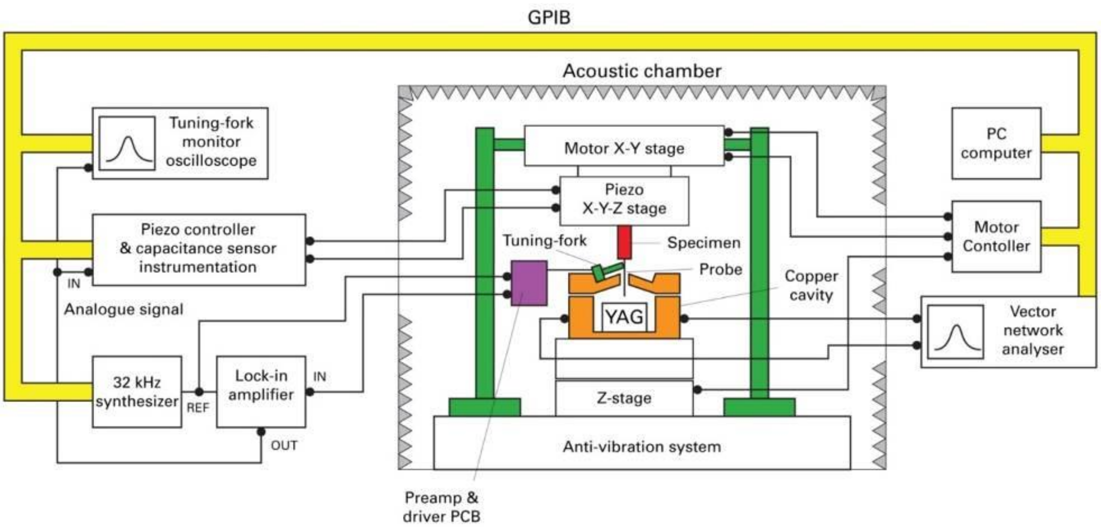


Figure 3.2.3 The NSMM scanning system at NPL

All NSMM systems require instrumentation to enable ‘contact mode’ to be established between the probe tip and specimen. In contact mode, the probe tip is actually about ~10 nm away from the surface. This is close enough to give reliable electrical measurement, yet there must be minimal pressure so that the probe is not deformed. In AFM-based instruments, see Section 3.1 Figure 3.1.1, contact mode is obtained simply by using a cantilever under gravitational force. For the RNSMM and TNSMM active systems for obtaining contact mode must be employed. The most common of these uses a quartz crystal tuning fork. For the NPL RNSMM it proved possible to use a miniature watch crystal 32-kHz tuning fork coupled to a stiff tungsten wire (diameter 0.1 mm) to allow shear-mode detection of contact mode by measuring the impedance of the tuning fork at its resonant frequency in a bridge circuit. Figure 3.2.4 shows one such configuration.

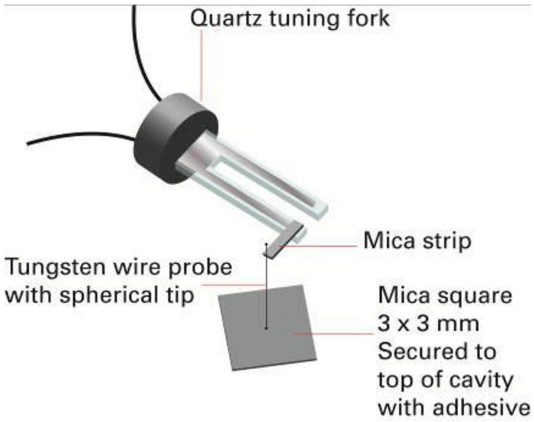


Figure 3.2.4 Tuning Fork proximity detection for the NPL RNSMM

During the course of this work it was shown that the wire probe must be considered to be a mechanical monopole, and that to obtain a good signal from it, it must also resonate mechanically at a frequency close to 32 kHz. The degree of acoustic coupling between the tuning fork and wire probe is a critical parameter. This presents formidable practical problems, especially as the cavity must function also as a microwave resonator. It is not possible to use miniature 32 kHz tuning forks to make a viable contact-mode detection system for the TNSMM, as the probes are larger and stiffer, so an optical system based on the deflection of a laser beam was used. This uses a Position Sensitive Photo-Diode (PSPD). This is an array of four photodiodes that enables deflection of the laser beam to be detected by subtraction of signals measured by individual elements. A piezo-actuator is used to excite vibrations in the probe which cause small deflections in the laser beam. The great advantage of this approach compared to using a tuning fork is that the excitation frequency can be varied to suit whatever mechanical resonances are available. Problems with making the tuning fork method work with the RNSMM proved to be a major obstacle to progress during EMINDA, and so the beam deflection method was also, towards the end of the project, implemented on the RNSMM. This should ensure that as part of the legacy of this project, a more robust RNSMM system will be available for industrial measurements.

Calibration of the RNSMM was performed using an electrostatic model proposed by Gao and Xiang (GX) Ref. [3.2.1] in 1998, using measurements on reference materials. This assumes that the tip itself is an isolated sphere. The frequency shift caused by the presence of a dielectric specimen can be calculated as a function of the gap between the sphere and specimen and the real part of dielectric permittivity of the sample. All of the available evidence would suggest that GX's approach remains valid at high frequencies; however their method of calibration for loss measurement is *ad hoc* and not well suited to the measurement of high loss materials, which was one of the goals of this work. In this project an alternative method for obtaining loss was used. This used the idea that the Q-factor and resonant frequency can be mathematically combined into a 'complex frequency'. This approach also readily treats the permittivity term in the GX formulation as a complex permittivity which is what is required for measuring lossy specimens. It was found that measurements using 'complex frequency' and standard GX formulations agreed well for low loss specimens, but diverged for high loss specimens.

In order to check upon this GX electrostatic model, a finite element modelling approach was also employed at NPL to model tip/specimen interactions. This used the COMSOL modelling package. Microwave EM fields in the region of the probe and specimen were modelled in the geometry shown in Figure 3.2.5, see Ref. [3.2.2]. This approach had two advantages. First, it allowed analysis of cavity perturbations in terms of the 'complex capacitance' between the probe and sample, complex capacitance being an alternative means of describing the capacitance/conductance combination seen by the probe. Secondly, a range of sample geometries were modelled to allow the effect of specimen imperfections (e.g. valleys and hills on the surface) to be quantified, and a number of inhomogeneous sample geometries were also modelled to allow the study the effects of permittivity variation across the face of specimens. This analysis contributed to the metrological understanding of NSMM measurement uncertainties, which the EMINDA project set out to quantify.

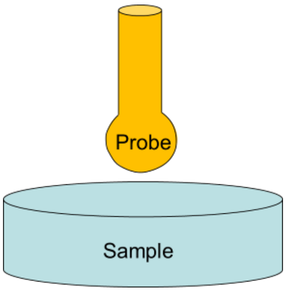


Figure 3.2.5 The RNSMM Probe/Sample Geometry modelled by COMSOL finite element analysis

One example of the complex capacitance analysis derived from this COMSOL Modelling is shown in Figure 3.2.6 which maps complex permittivity, ε' - jε'', onto the complex capacitance (*C*' - *jC*'') plane for one particular probe specimen geometry, where the 'imaginary capacitance', *C*'', is an alternative expressive for the conductivity or loss of the sample. The figure demonstrates how useful this form of modelling and mapping can be. It is clearly shown that the real capacitance, *C*', is affected by dielectric loss, ε''. This is a feature which previous simple capacitive models of probe/sample interaction could not capture, this finding again demonstrates how the EMINDA project has contributed to our understanding of NSMMs and it contributes significantly to making them fully traceable.

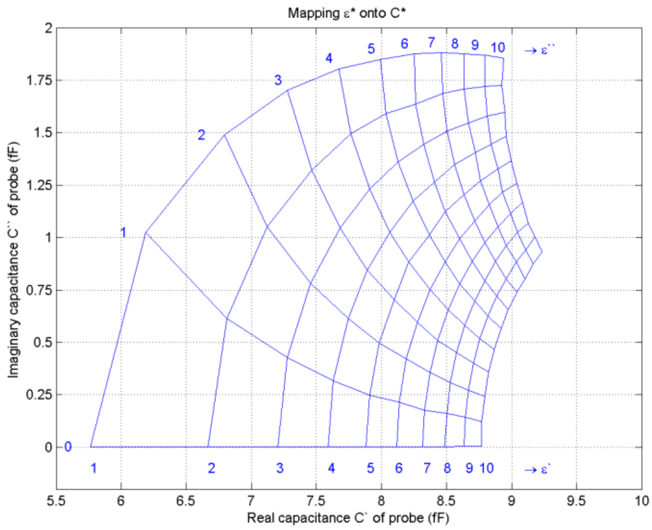


Figure 3.2.6 COMSOL Complex Capacitance analysis of NSMM probe measurements on lossy dielectric specimens displayed in terms of a mapping of the complex permittivity plane onto the complex capacitance plane, the latter being a complex parameter that the NSMM can measure. Full details are given in Ref 3.2.2

Gao and Xiang also published a two-layer theory for NSMMs, Ref. [3.2.3]. This was also implemented using the complex frequency to allow another important issue to be tackled: how to obtain a medium or high loss dielectric reference material for which traceable reference data is available so as to calibrate the NSMM for measurements on lossy dielectrics. Such Reference materials must be reproducible, and must be uniform in permittivity at the dimensional scales that the probe is sensitive to. This requirement excludes virtually all composite lossy materials such as carbon-loaded polymers because they are not uniform at the micron scale. A comprehensive search demonstrated that the only *solid* (as opposed to liquid) materials that make satisfactory reference materials are by nature low loss, e.g. fused silica and single crystals. However, by using the two-layer geometry it was possible to use a polar liquid held by a thin window as a uniform reference material, see Figure 3.2.7. Polar liquids generally have high loss in the microwave region of the spectrum and published traceable complex permittivity data is already available for them from NPL, see Ref. [3.2.4]. Using this two-layer regime in the EMINDA project it was demonstrated that polar liquids can be used to calibrate the RNSMM. A two-layer theory is also necessary to allow measurements on thin films – an important industry requirement. A paper is being prepared on this work.

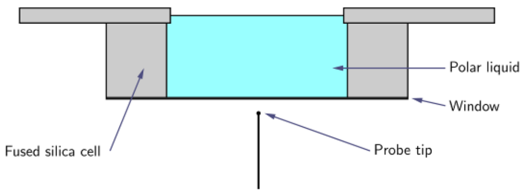
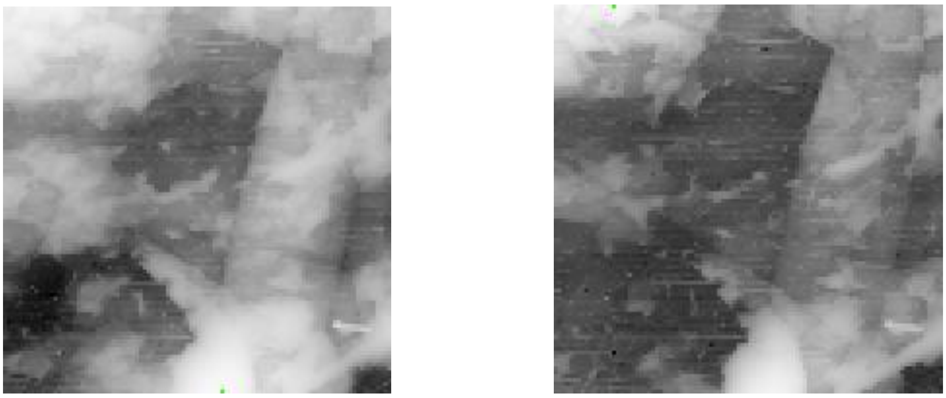


Figure 3.2.7 Calibration cell for the NSMM. A lossy reference liquid (blue) in the cell is detected by the probe mounted vertically below through a thin solid window

The uncertainty analysis of this type of probe is discussed further in Section 3.2.8.

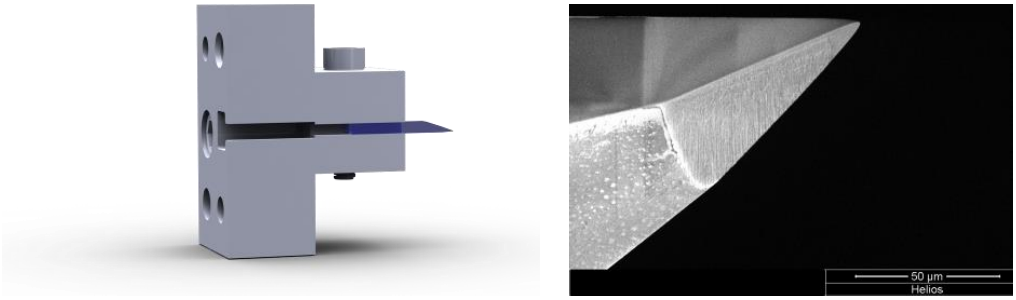
The RNSMM has already been used for assessing the properties of graphene and graphene oxide films, see Ref. [3.3.5], for which the EMINDA development work proved invaluable, see Figure 3.2.8. The industrial value of using the NSMM probe for characterising the internal structure of a ceramic dielectric resonator material has also been published in a Case Study on the EMINDA web-site, for full details see http://projects.npl.co.uk/eminda/case-studies/



(a) (b)

Figure 3.2.8 RNSMM scan of a graphene oxide film at a nominal frequency of 240 MHz. Each image covers a 0.1 x 0.1 mm area. (a) Cavity resonant frequency, 239.25 MHz (dark) to 239.3 MHz (light) (b) Cavity Q-factor: this varies from 1088 (dark) to 2796 (light)

Development of the TNSMM was largely carried out at Imperial College (IC). Initially the probe was attached to a resonant cavity, as shown in Figure 3.2.2, however the interplay of resonant-cavity modes and resonant modes in the TEM probe itself caused practical difficulties, so, in an alternative design, the cavity was replaced by a non-resonant feed based on a ridged waveguide so that only resonances in the probe were present, see Figure 3.2.9 (a). This produced strong probe/sample coupling, permitting effective measurement of complex permittivity. TNSMM probe tips were sharpened by Focussed-Ion-Beam (FIB) milling, as shown in Figure 3.2.9 (b) and metal films were deposited on opposite sides. Electrostatic models of probe tips were obtained using COMSOL for thin layer specimens. From these models it was possible to relate changes in resonant frequency to specimen permittivity. A Monte-Carlo process was developed to evaluate uncertainties for this technique. It was found that the largest uncertainty contribution is due to chipping or incompleteness of the metal film at the probe tip.



(a) (b)

Figure 3.2.9 (a) TNSSM Probe mounted in a ridged waveguide for broadband measurements.
(b) TNSMM probe tip sharped by Focussed-Ion-Beam (FIB) milling

As part of this project, modelling of the interaction between the tip and samples was performed using COMSOL. This will be the subject of a paper in due course. By scanning across a junction between dielectric materials (using an especially manufactured specimen), the resolution of measurements was investigated.

Measurements on functional materials (applying a bias) were obtained using both RNSMM and TNSMM, these are described further in Section 3.5. It is intended to publish all of the EMINDA work on both NSMM configurations in peer reviewed journals as soon as possible.

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[3.2.2] N. Smith, K. Lees, A Gregory and R. Clarke, *'Modelling a Resonant Near Field Scanning Microwave Microscope (RNSMM) Probe'*, Proceedings of the 2014 COMSOL Conference, Sept. 2014.

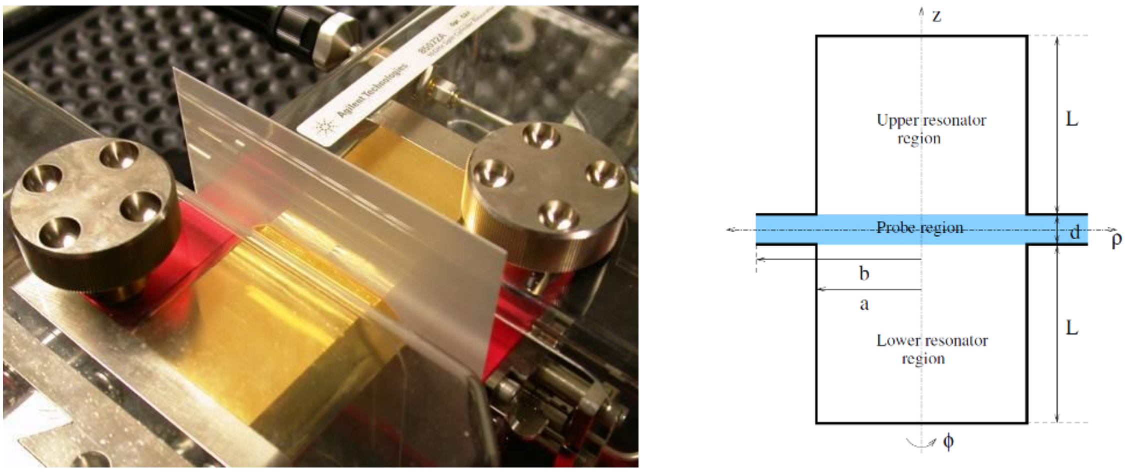
[3.2.3] C. Gao, B. Hu, P. Zhang, M. Huang, W. Liu, I. Takeuchi, *'Quantitative microwave evanescent microscopy of dielectric thin films using a recursive image charge approach', Appl. Phys. Lett.*, vol. **84**, no. 23, pp. 4647-4649, 2004.

[3.2.4] A P Gregory and R N Clarke, 2009, NPL Report MAT 23, *Tables of the Complex Permittivity of Dielectric Reference Liquids at Frequencies up to 5 GHz*, NPL, March 2009.

[3.2.5] Andrew Gregory, Ling Hao, Norbert Klein, John Gallop, Cecilia Mattevi, Olena Shaforost, Kevin Lees and Bob Clarke, '*Spatially resolved electrical characterisation of graphene layers by an evanescent field microwave microscope'*, *Physica E* (2012), http://dx.doi.org/10.1016/j.physe.2012.10.006.

3.3 Traceable Broadband Dielectric Measurements of Substrate & Laminar Materials

This research covered two techniques for measuring the complex permittivity of electronic substrates and of laminar dielectric specimens in general: the **Split Cylinder (SC) Resonator** method and the **Co-Planar Waveguide (CPW)** broadband transmission line method. In both cases, frequency coverage and uncertainties were significantly improved as a result of research in the EMINDA project, with comprehensive uncertainty budgets being developed for both methods. The materials and structures measured by these techniques are ubiquitous in all contemporary ICT electronic circuitry where on-wafer construction of circuits is now the norm. Improvements in the quantification of their dielectric properties contributes significantly to the efficacy of circuits that are deposited onto them and allows performance to be optimised more effectively.



(a) (b)

Figure 3.3.1 The Split Cylinder (SC) Measurements system at PTB, (a) measurement on a substrate material, (b) plan of the SC cavity

The SC method, see Figure 3.3.1, is ideally suited for measuring laminar materials at multiple resonant frequencies. The research on this technique was largely carried out at PTB, but with significant inputs from the other EMINDA Partners. The SC has the low-uncertainty (BF33, ε_r = 4.5) advantage of resonant methods, with the benefit of wide frequency coverage (10 – 30 GHz). An SC mode-matching model utilising TE_{0np} modes, which is known from the literature, has been implemented at PTB, and the complex permittivity of several samples of different materials (GaAs, Al_2O_3, SiO_2, AF45, BF33) have been measured in the frequency range 10-30 GHz, see Refs. [3.3.1], [3.3.2]. The results were in good agreement with reference values given in the literature. However, for the first time, a comprehensive uncertainty budget recognising the influences of geometrical and material parameters as well as measurement quantities has been developed utilising the Monte-Carlo method and the classical GUM method (linear propagation of uncertainties), see Ref. [3.3.1]. Both methods led to the same values of uncertainty, which lay in the range of 5 % for relative permittivity and 10 % for loss tangent.

In addition to TE_{0np} modes, the SC resonator exhibits other TE and TM modes in the frequency range covered. In some cases, these modes will resonate close to the frequency of the TE_{0np} modes distorting the resonances of interest, see Figure 3.3.2. An extended mode-matching model predicting all the excited TE and TM modes in the considered frequency range has been developed, see Refs. [3.3.2] and [3.3.3]. The new model enables a better identification of the modes of interest and predicts a possible distortion, i.e. asymmetry in these resonances due to coupling or overlapping with other modes, see Ref. [3.3.2].

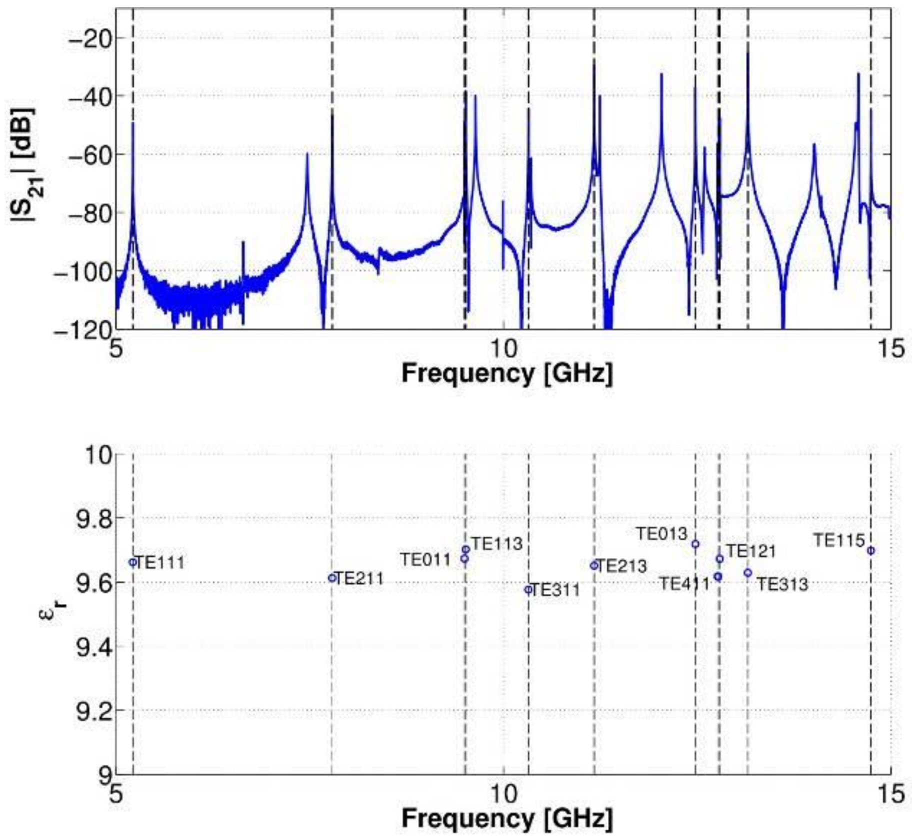


Figure 3.3.2 Extended SC mode-matching with the PTB model, including (odd p) TE_{mnp} modes, showing resonances as measured on the VNA and their modal interpretation underneath

Several methods for the evaluation of asymmetric resonances have been examined. A simple approach to account for asymmetry of the measured resonance curve in the uncertainty budget was been proposed during the EMINDA project, see Figure 3.3.3.

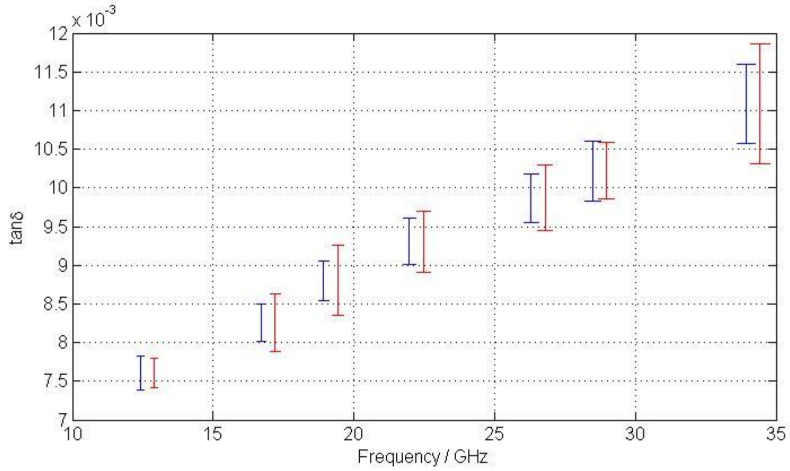
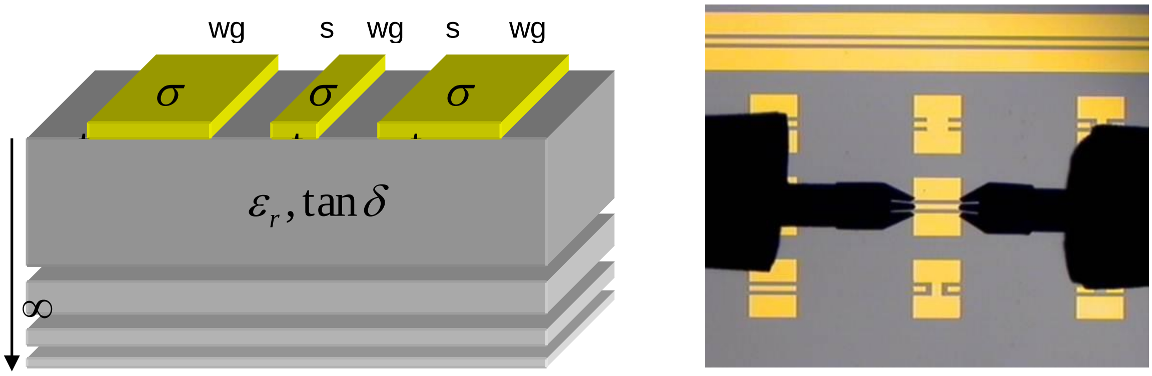


Figure 3.3.3 Loss factor uncertainties due to measurement (blue) and asymmetric (red) quality factor uncertainties (BF33, $\varepsilon_r = 4.5$)

Split cylinder measurements at PTB were compared with Split-Post Dielectric Resonator (SPDR) measurements at NPL and at GUM for five samples of different materials (Al_2O_3, SiO_2, AF_{45}). The results showed good agreement within the uncertainty range for both relative permittivity (5%) and loss tangent (10 %). This validation exercise provides confidence in the newly developed methods, which can now be used in support of European industry.

The research at PTB and LNE into the broadband Co-Planar Waveguide (CPW) method, see Figure 3.3.4, focussed on the identification of major uncertainty contributions but it also proved to have the added benefit of improving the uncertainties for loss measurement of very low loss substrates.

The main advantage of using transmission line CPW methods in permittivity measurements is its capability for broadband and frequency-continuous characterisation of the substrate dielectrics under test, the CPW is metalised onto these substrates. At frequencies below 10 GHz, substrate materials are measured routinely using these CPW methods. However, these measurements have typically been performed without any assessment of uncertainty. As the frequency increases, additional sources of uncertainty become relevant, and one of the targets of this research was to characterise uncertainties thoroughly so that higher frequency measurements would be possible. Based on closed-form expressions for CPW waveguide properties and on CST Microwave Studio simulations, several CPWs were designed at PTB and LNE, utilising different CPW lengths and cross-sections. Four CPW test structures on two different substrate materials (SiO_2, Al_2O_3) and with two different metallisation thicknesses (0.5 µm, 1.1 µm) were fabricated at the CNRS-LAAS laboratory and measured at PTB and LNE.



(a) (b)

Figure 3.3.4 (a) CPW - co-planar waveguide: 'wg' is metalised waveguide, 's' is substrate dielectric. (b) Measurements on a CPW calibration substrate. The probe station (black) is attached via transmission lines to a Vector Network Analyser VNA which performs the CPW measurements

A preliminary uncertainty budget recognising the influences of geometrical and material parameters was developed utilising the Monte-Carlo method and the classical GUM method, Ref. 3.3.3. The results showed good agreement with SC measurements of the same substrate materials (SiO_2, Al_2O_3). Based on the experience gained from these measurements, further research was performed to extend the CPW method to higher frequencies, nominally 80 GHz, according to the original EMINDA plan. Two optimised CPW test structures were designed and fabricated on two different substrate materials: quartz (6 CPW lines and 1 'reflect' standards, of 3 different cross-sections) and alumina (5 lines and 1 'reflect', with 3 different cross-sections) with metal thickness 0.5 µm, see Figure 3.3.5. Measurements and permittivity extractions up to 110 GHz were performed for the optimised CPWs. A comprehensive uncertainty budget was developed recognising input uncertainties of geometrical and physical parameters as well as measurement uncertainties.

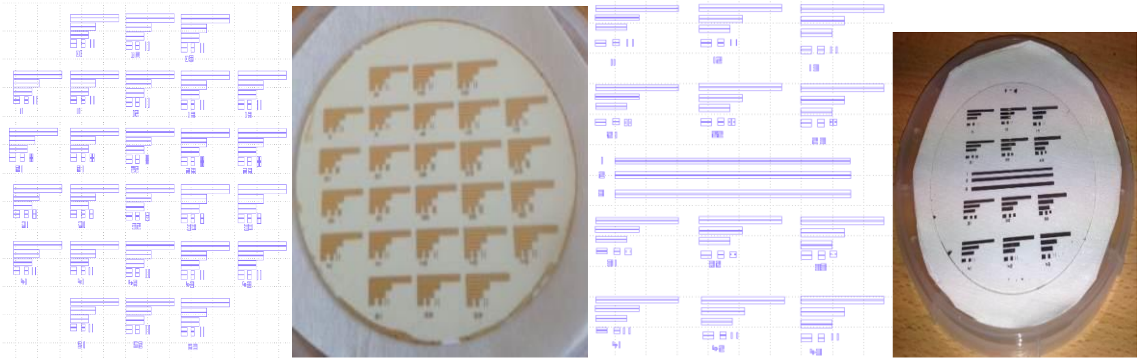


Figure 3.3.5 CPW test structures manufactured for the EMINDA research on CPW metrology (see text)

Uncertainty analyses of the extracted relative permittivity and loss tangent up to 70 GHz showed that among the geometrical and material parameters metal thickness and conductivity constitute the main contributions to the total uncertainties. In the case of relative permittivity the metal thickness influence is dominant; whereas the metal conductivity influence becomes dominant above 18 GHz in the case of loss tangent. The conductivity of the CPWs was measured at GUM utilising the split-post dielectric resonator (SPDR) technique.

A new broadband, semi-numerical method to extract the loss tangent of low-loss dielectric substrates from on-wafer S-parameter measurements of coplanar waveguides of different lengths was introduced. The method, which works up to at least 80 GHz, makes use of two closed-form coplanar waveguide models, one of them assuming quasi-TEM behaviour, the second one accounting for modal dispersion. The method was applied successfully to alumina, quartz and GaAs substrates, thereby demonstrating an improvement of the loss factor measurement range by a factor of at least 10 in comparison to established transmission-line techniques, see Figure 3.3.5 and Ref. [3.3.4]. The new method uses starting values for the loss tangent at low frequencies given by the manufacturer or measured by other techniques. The determination of the metal conductivity is greatly simplified, because only a plausible range with upper and lower bounds needs to be known to the algorithm. Therefore a precise measurement of the conductivity is no longer necessary, see Ref. [3.3.5].

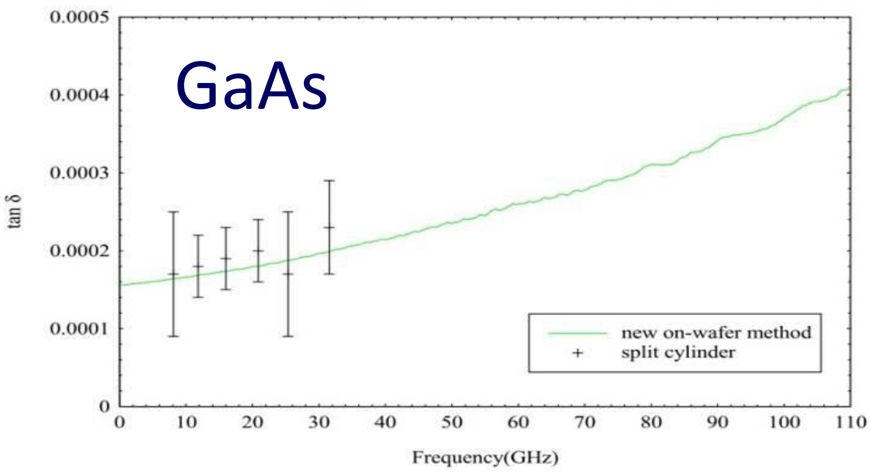


Figure 3.3.6 Measurements on the loss tangent of a gallium arsenide substrate by the enhanced CPW method at PTB, compared with SC measurements, also by PTB

References for Section 3.3.

[3.3.1] K. Kuhlmann, U. Arz, *'Uncertainties in Split-Cylinder Resonator Measurements'*, 79th ARFTG Conference Digest, 2012, pp. 121-124.

[3.3.2] S. Zinal, U. Arz, *'An Extended Mode-Matching Model for Improved Relative Permittivity Measurements Using a Split-Cylinder Resonator'*, *Advances in Radio Science*, Vol. **12**, 2014 ('accepted for publication').

[3.3.3] U. Arz, D. F. Williams, *'Uncertainties in Complex Permittivity Extraction from Coplanar Waveguide Scattering-Parameter Measurements'*, 81th ARFTG Conference Digest, 2013.

[3.3.4] U. Arz, *'Loss Tangent Extraction Based on Equivalent Conductivity Derived from CPW Measurements'*, 18th IEEE Workshop on Signal and Power Integrity, Ghent, 2014.

[3.3.5] U. Arz, *'Microwave Substrate Loss Tangent Extraction from Coplanar Waveguide Measurements up to 125 GHz'*, 83rd ARFTG Conference Digest, 2014.

3.4 On-Wafer Traceability for Advanced & Functional Thin Films

As in Section 3.3, the research here made use of Co-Planar-Waveguide (CPW) transmission line, and Vector Network Analyser (VNA) probe stations for measurements on them, see Figure 3.3.4. Here however, the intention was to characterise thin functional films printed onto the substrate material between the substrate and the CPW metallisation, see Figure 3.4.1. This is a very important measurement geometry to address metrologically, because it is one in which many newly developed EM functional materials, such as ferroelectrics, piezoelectrics and magneto-electrics will be integrated into ICT circuits.

Metrological research on thin films in this type of geometry was seen from the very outset as being very challenging and as presenting higher risk factors than most of the activities in EMINDA. For example, as this particular thin-film modality is new to end-user applications, the very availability of suitable functional specimens for the project constituted one of its major risk factors. In the event, the intention to perform measurements at NPL on multiferroic thin films did not come to fruition due to the unavailability of suitable specimens. Effort was therefore switched to the measurement of other functional materials. It should also be emphasized that uncertainties for complex permittivity measurement by these techniques were expected to be significantly higher than for the other techniques covered in EMINDA. Nevertheless, the importance to industry of pursuing these measurements is clear, as a far better understanding of the method is needed if significant progress is to be made.

This CPW approach to measuring the dielectric properties of thin films may be compared to that described below in Section 3.6 which employs Split-Post Dielectric Resonator (SPDR) measurements. SPDR measurements already provide very high accuracy for some types of thin film, but only at spot frequencies (one frequency per SPDR). If the CPW measurements can be made metrologically sound and if uncertainties can be quantified and reduced the CPW method should offer the potential advantages of large frequency coverage and the ability to apply bias to actuate changes in the RF and Microwave properties of the thin films, thereby widening their functionality. In the long term, viable bias modalities would include the application of static electric or magnetic fields or mechanical stress. The EMINDA work here may be seen as a contributory step towards achieving this goal.

Work in this area was largely carried out at LNE and NPL, but with significant inputs from other partners and from project collaborators who supplied EM functional specimens. The central aim of the work was to develop traceable measurement techniques for EM functional thin films over the frequency range 5 GHz – 50 GHz. LNE were actually able to extend this to 2 GHz – 110 GHz.

LNE characterised ferroelectric thin films mounted under CPW metalisation, as shown in Figure 3.4.1, Barium Strontium Titanate (BST) and Lead Titanate Zirconate (PZT). BST and PZT are the most common ferroelectric materials studied, and are considered to be good candidates for many electronics applications such as sensors, capacitors and actuators.

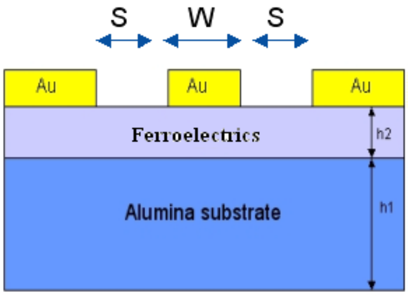


Figure 3.4.1 A multi-layered CPW structure: the thin-film to be measured (for clarity, shown much thicker than in practice), in this case a ferroelectric, is deposited between the CPW (W) metallisation and the substrate (S)

For the extraction of the relative permittivity of a ferroelectric thin film, LNE carried out a multiline 'Thru-Reflect-Line' (TRL) calibration with 'Multical', a software package developed at the National Institute of Standards and Technology (NIST) in the USA. The VNA measurement requires the S-parameters of the CPW line to be measured and the propagation constant of the CPW line must be measured to in order to extract the permittivity.

The permittivity, ε_2, of the thin film in this structure is obtained from the conformal mapping method and may be expressed as follows, Ref. [3.4.1]:

$$\varepsilon_2 = \frac{1}{q_2}\left(\varepsilon_{eff} - 1 - q_1(\varepsilon_1 - 1)\right) + \varepsilon_1 = \varepsilon'_2 - j\varepsilon''_2 \qquad (1)$$

$$q_i = \frac{1}{2}\frac{K(ki)}{K(k'i)}\frac{K(k'_0)}{K(k_0)}, i = 1,2 \qquad (2)$$

$$\tan\delta_2 = \frac{\varepsilon''_2}{\varepsilon'_2} \qquad (3)$$

Where ε_1 is the relative permittivity of the substrate, ε_{eff} is the 'effective' measured permittivity; q_1 and q_2 are the filling factors and tan δ_2 is the loss tangent of the BST thin film. As the height of the ferroelectric thin film h_2 (400 nm) is small compared to W (60 µm), the CPW inner conductor width, and S (28 µm), k_2 is small and this leads to numerical error in calculating elliptic functions. To overcome this difficulty LNE employed the asymptotic formula for the ratio of elliptic function Ref. [3.4.2]:

$$\frac{K(ki)}{K(k'i)} = \frac{\pi}{\ln\left[\dfrac{2\left(1+\sqrt{\sqrt{1-k_2^2}}\right)}{1-\sqrt{\sqrt{1-k_2^2}}}\right]} \qquad (4)$$

For $0 \leq k \leq 0.707$, $k_2 \rightarrow 0$: (5)

$$\frac{K(k2)}{K(k'2)} = \frac{\pi}{\ln\left[\dfrac{16}{k_2^2}\right]}$$

Then, $$q_2 = \frac{1}{2}\frac{\pi}{\ln\left[\frac{16}{k_2^2}\right]}\frac{K(k'0)}{K(k0)} \quad (6)$$

Figures 3.4.2 (a) and (b) show measurements on a BST film. They exhibit an increase of the relative permittivity and the loss tangent at low frequencies (below 10 GHz), which can be attributed to the calibration errors of CPW lines at low frequencies. The BST thin film exhibits a high permittivity value which stabilises at the value of 350 at around 30 GHz.

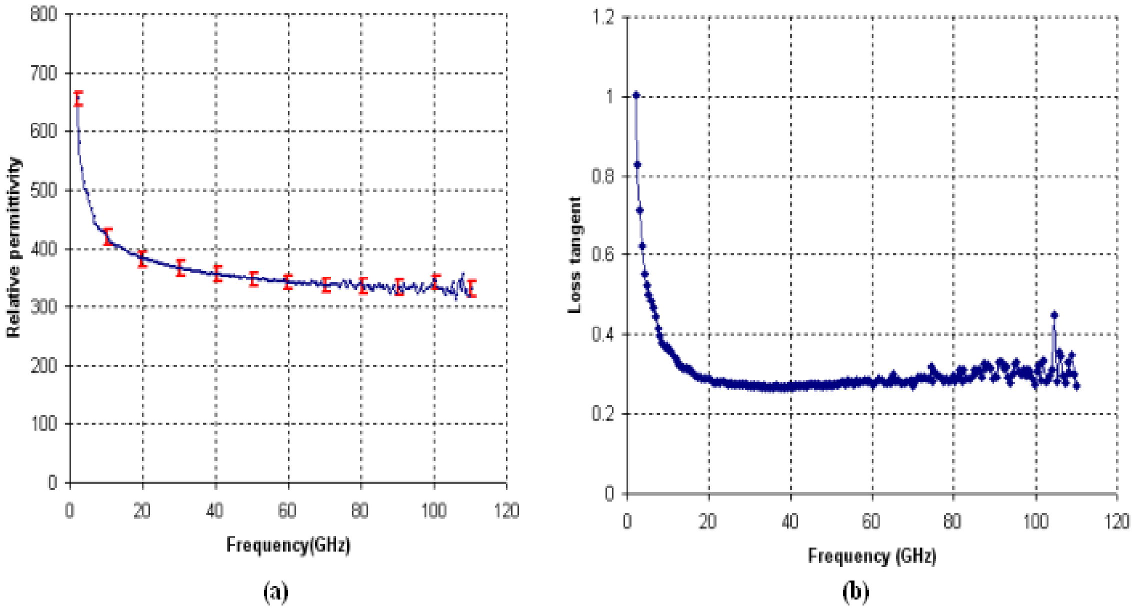


Figure 3.4.2 Permittivity (a), and Loss Tangent (b), of BST as measured at LNE

Figure 3.4.3 (a) and (b), below, shows LNE measurements on PZT. This material also exhibits an increase of the relative permittivity and the loss tangent at low frequencies (below 10 GHz) like the BST, which can be attributed to the calibration errors of coplanar lines at low frequencies. However, in this case, it can be seen that the loss tangent increases significantly as the frequency increases. At 2 GHz the PZT thin film exhibits a very high value (at around 1350), but this value decreases very quickly before reaching 250 at 110 GHz.

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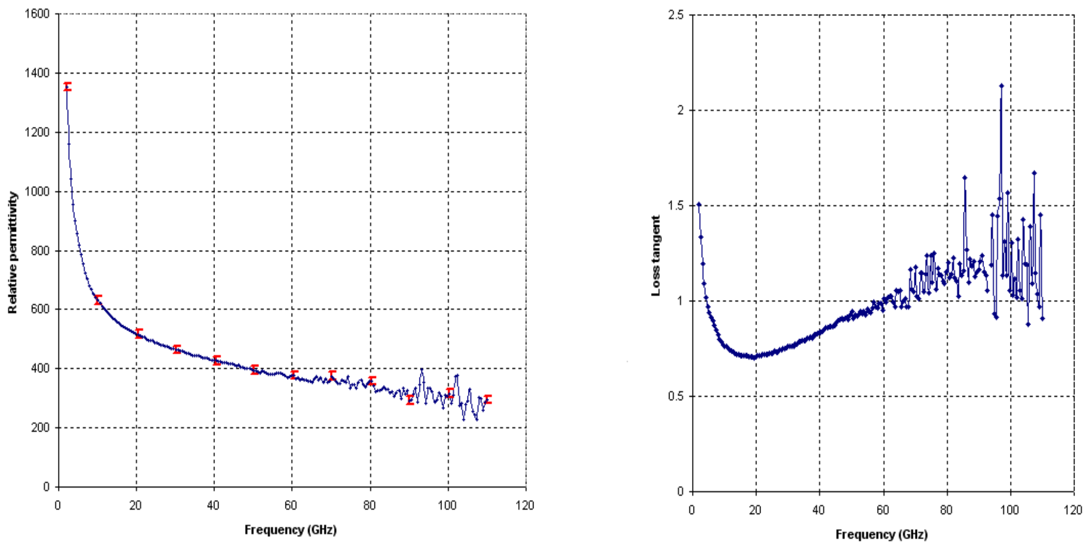


(a) (b)

Figure 3.4.3 Permittivity (a) and Loss tangent (b) of PZT as measured at LNE

At NPL similar CPW thin-film specimens were employed, but the aim was to develop traceable measurement techniques for functional thin films on substrates over the frequency range 5 GHz - 50 GHz, both with and without biasing being applied. The work concentrated on two technical approaches namely CPW measurements and Split-Post Dielectric Resonator (SPDR) measurements. CPW measurements have the advantage of large frequency coverage and the ability to bias the functional dielectric, whereas SPDR measurements provide very high accuracy at a spot frequency.

The SPDR work was successful with measurements of thin films down to 50 nm being demonstrated, but the CPW studies were only partially successful. Application of biasing, electrical and mechanical, was demonstrated (see Section 3.5) and traceable measurements on purely dielectric films were demonstrated between 2 GHz and 110 GHz. However, modelling of magnetic and multi-ferroic films could not go forward because no suitable samples could be sourced and delivered during the period of the EMINDA project. However, the measurement techniques required have been developed successfully and can be used for future work. The aim of the CPW modelling was both to extract results from magnetic materials and to show how biasing on magnetic and multiferroic films would affect the S-parameters of CPW devices. These aims could not be met during the currency of the EMINDA project.

The original plan for magnetic measurements was to modify the standard conformal mapping technique employed by Gevorgian to allow for magnetic properties. The approach was to use the duality principle between permittivity and permeability, Ref. [3.4.2]. Although a theoretically valid approach, the solution showed behaviour that is not consistent with the known physics of the materials measured. Consequently, an alternative spectral approach was then developed as a complete 'forward' model of the waveguide, which calculates the propagation constant and the impedance for a given set of geometric and material parameters from known material parameters. The intention was then to develop an iterated 'reverse' algorithm, which would allow the electromagnetic material parameters, complex permittivity, ε*, and complex permeability, μ*, from VNA-measured parameters. Again, during EMINDA, it was not possible to create a stable algorithm that successfully converges. An alternative approach which uses the commercial package Microwave Studio (MWS) to calculate the permittivity and permeability is currently in use, however this takes significant time as MWS can only perform the 'forward' computation which takes half-an-hour per run.

The NPL work on the application of bias in the CPW technique is discussed in Section 3.5.

At GUM thin film measurements based on SPDRs were modelled using mode-matching and Rayleigh-Ritz techniques for a specific SPDR. The model allows for the de-embedding of the thin film permittivity from: (a) SPDR measurements of the bare substrate, (b) SPDR measurements and substrate and film and (c) the film thickness. The method has been evaluated at 20 GHz, but the modelling has not been validated yet against other methods, but in principle this could be achieved by comparisions with the Split Cylinder measurement technique. A preliminary uncertainty analysis demonstrates that the uncertainty of the thin-film thickness is the most important contributing factor in the uncertainty budget in these measurements, so thickness measurement should be performed with the highest possible accuracy.

References for Section 3.4

[3.4.1] S.Gevorgian, *'Ferroelectrics in Microwave Devices, Circuits and* Systems' ,pp247-260.

[3.4.2] K.Gupta, R.Garg, I.Bahl, P.Bhartia, *'Microstrip lines and slotlines'*, 2nd ed. Boston, MA: Artech House, 1998, ch.7.

3.5 Measurements on Functional Materials with Applied Bias

The application of bias to functional thin film specimens was studied in EMINDA at both NPL and IC. Significant support for this work was offered by other project partners and by EMINDA collaborators who supplied specimens. Measurements were undertaken on the NSMM systems described in Section 3.2 and by using the CPW thin-film method described in Section 3.4. EMINDA addressed these measurements because the future utility and value of functional films in ICT electronics will depend significantly upon applications in which such biases are applied, e.g. for tuning or switching. The work described here may be seen as a first step towards putting the traceable RF and Microwave metrology of thin-films under bias onto a firm footing for an industrial (as opposed to an academic) environment.

Measurements in both the RNSMM and TNSMM on functional materials whilst applying a bias were undertaken, and bias-related modification of ε' was detected (details to be published).

In the CPW measurements two methods of biasing were successfully developed**:** voltage and mechanical. CPW voltage biasing is based on the inclusion of 'Bias T' networks into the microwave measurement system. These are placed into the microwave measurement system before the VNA calibration plane and so do not affect the uncertainty of the measurements. This approach safely allows up to 100 V to be applied to the CPW system whilst safely avoiding the possibility that it might be coupled to the rest of the network by error. In practice it was found that the voltage that can be applied is limited to 30 V. There are two reasons for this, in both cases the applied voltage has to be limited to avoid breakdown of the thin film:

- The assumption that the voltage is evenly distributed across the film is not valid. FDTD modelling using CST Microwave Studio shows that there is a higher voltage at the edge of the CPW tracks, see Figure 3.5.1

- The CPW tracks are not perfect and these imperfections lead to areas of higher field

Mechanical biasing has been achieved by using the device shown in Figure 3.5.2. It is based on designs used for previous piezoelectric studies at NPL. The device allows the centre of the substrate to be bent whilst it is mounted on a VNA probe station. The level of bending has to be constrained because excess bending will cause the substrate to crack. A silicon substrate of 500 µm thickness, permits the application of up to 18 ppm of strain. No additional uncertainties in the microwave measurements have been found to be introduced by this biasing technique. By the end of the EMINDA project, with the level of strain and the thin films available it was not possible to measure any strain effects at RF and microwave frequencies. The work does successfully show, however that bending of silicon substrates within working devices need not affect the performance of microwave circuits.

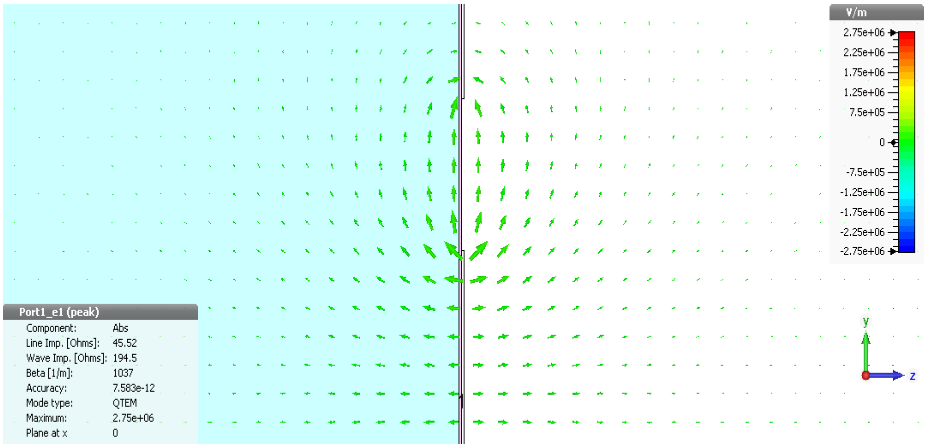


Figure 3.5.1 A CST Microwave Studio electric field model of the field close to the surface of a CPW transmission line. The Figure shows a cross-section through the line with the substrate (blue on the left) and air on the right. The thin film on the substrate surface is shown in grey, and runs from top to bottom. The metallised CPW transmission line is shown as slightly wider grey, compare with Figure 3.4.1. The length of the green arrows shows the electric field strength in the region close to and between the CPW inner and outer conductors. Note that the field strength is greatest close to the sharp edge of the metal conductors

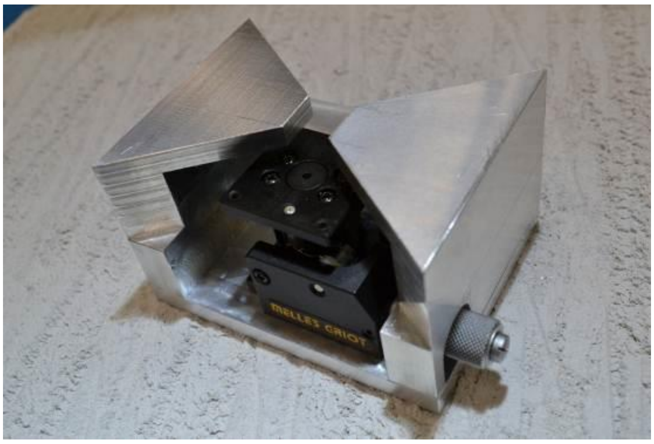


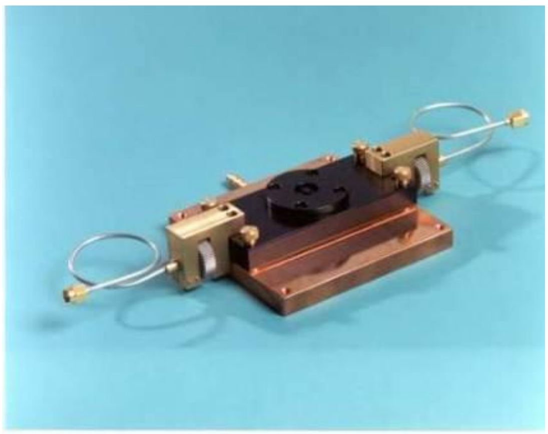
Figure 3.5.2 Wafer mount constructed at NPL for testing the effect of stress on permittivity for piezoelectric films

Arising out of the CPW studies in EMINDA University of Birmingham approached NPL with the intention of setting up a dialogue on methods for data extraction for biased thin films. They became a collaborator in the project and NPL and Birmingham have started sharing both experimental and theoretical data and plan to continue beyond the end of EMINDA to ensure the expertise gained in this project is passed on to this leading University.

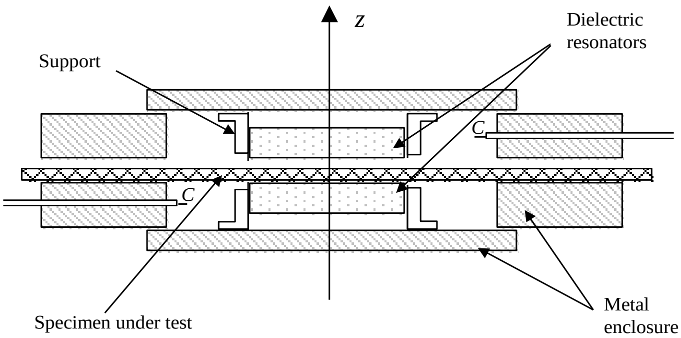
3.6 Resonator Based Measurements on Thin Films and Surfaces

The work described here was undertaken using existing resonator measurement cells but it was aimed at extending the range of measurements they are capable of and at quantifying the uncertainty contributions that arise in this type of measurement. The work was largely undertaken at GUM, but with significant support from other partners and with materials supplied by them and EMINDA collaborators. Note that all of this work is described in detail in Refs. [3.6.1], [3.6.2] and [3.6.3].

One of the targets here was to measure sheet resistance and resistivity of conducting and semiconducting materials, this is a parameter which is of major interest to designers of electronic devices. Split and single-post dielectric resonators (SPDRs) are suitable instruments to measure sheet resistance on wafers and on thin films deposited on dielectric substrates, see Figure 3.6.1. They allow the determination of the conductivity of CPWs with more accuracy than with other methods (e.g. the Van-der-Pauw method). Additionally, important materials including graphene, conducting polymers, semiconductors, wafers, and epitaxial films (SiC, GaN) deposited on low loss dielectric substrates such as sapphire can be investigated using these techniques.



(a)



(b)

Figure 3.6.1 A Split-Post Dielectric Resonator (SPDR): (a) 10 GHz SPDR, (b) side view of SPDR

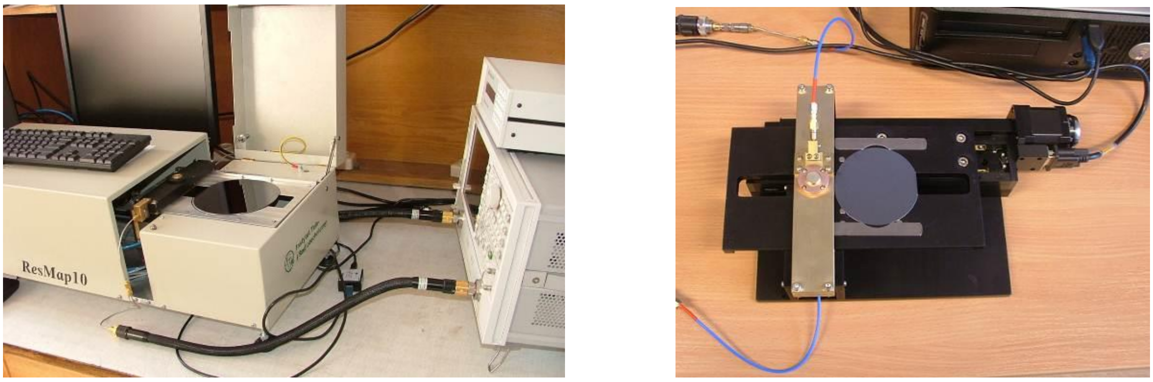
Two kinds of surface conductivity were targeted in the EMINDA modelling and measurements. The first was the conductivity (and or resistivity) of bulk materials having thickness that is a few times larger than the skin depth at the test frequency. The second was conductivity of thin conducting films having thickness smaller than the skin depth (down to 50 nm). All measurements at GUM were performed employing quasi-$TE_{0\delta}$ modes excited in measurement cells that employ very low loss dielectric resonators, as shown in Figure 3.6.1. For the $TE_{0\delta}$ modes the electric field has only the azimuthally field component so the electric currents in the sample is circumferential and the method is essentially, therefore, contactless.

Single post dielectric resonator/surface conductivity interactions were modelled by the mode-matching and Rayleigh Ritz method and the modelling was validated against a four-point probe method and the Eddy Current method.

Nine samples of bulk materials were measured. Seven of them were copper-clad epoxy glass laminates, one was gold plated alumina and another was gold plated silicon. The effective conductivity and sheet resistance on both sides of metal layers was measured for all specimens. The conclusion is that for copper-clad epoxy glass laminates, effective conductivity on the substrate-metal interface is up to three times smaller than the conductivity of bulk copper, due to the surface roughness. This difference is relatively small for gold plated ceramic and silicon.

The sheet resistance of several material samples was mapped using scanning systems, see Figure 3.6.2, including semiconductor wafers (bulk), metal films on single crystal quartz, graphene films on PET and polyaniline samples on fused silica. The measurements have been validated against the four-point probe method and the Eddy Current method. Measurements of graphene epitaxial films on SiC and GaN epitaxial films on sapphire were performed. Samples were obtained from our EMINDA collaborator, the Institute of

Electronic Materials Technology (Warsaw). The measurements were validated against the DC method. Figure 3.6.3 shows the result of one such mapping exercise performed on a 6” silicon wafer.



(a) (b)

Figure 3.6.2 SPDR scanning systems: mapping of silicon wafer resistivity as a function of position, (a) at 5 GHz, (b) at 14.5 GHz. In both cases the SPDR has been retracted to the left and the 6” silicon wafer is to the right of it

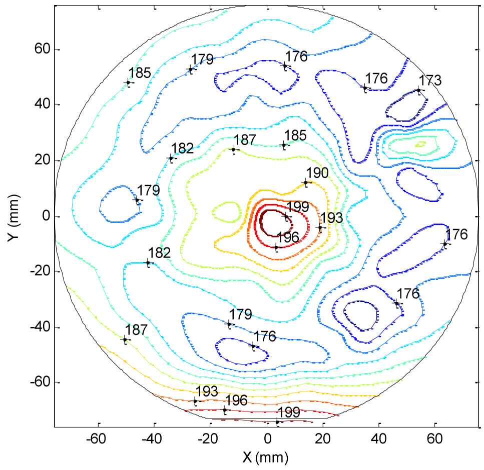


Figure 3.6.3 A mapping of the resistivity of a 6” silicon wafer, as measured by the SPDR method at 5 GHz. The resistivity units are Ω.m

Generic uncertainty budgets for this approach to conductivity mapping, employing the SPDR technique, were developed. The typical uncertainty value of conductivity measurements is a few percent. The main contributing factors to the uncertainty budget are Q-factor uncertainty and thickness uncertainty. The results of these measurements have been presented at international conferences, see Refs. [3.6.1], [3.6.2] and [3.6.3]. It was concluded that, following EMINDA, further investigation of the effective conductivity of rough metal surfaces would be of value to the European ICT industrial community and recommendations to this effect are being made in a separate EMINDA report, Ref. [3.6.4].

References for Section 3.6

[3.6.1] Jerzy Krupka, ‘Introduction to Advanced Dielectric Measurement Techniques: ‘Substrate Measurements with Split Post Dielectric Resonators’, IMS 2012 Workshop Electronic Notes, 18.06.2012

[3.6.2] Jerzy Krupka, Łukasz Usydus and Henryk Kołtuniak, *'Surface resistance and conductivity of rough surfaces of metals on printed circuit boards and metalized ceramic substrates'*, MIKON Conference Digest, 2012. 21.05.2012

[3.6.3] Jerzy Krupka, *'Contactless methods of conductivity and sheet resistance measurement for semiconductors, conductors and superconductors'*, *Measurement Science and Technology*, **24** (2013) 062001. doi:10.1088/0957-0233/24/6/062001

[3.6.4] *'Report on the further Advancement of EM Materials Metrology in Europe'*, EMINDA authors, forthcoming, in print.

3.7 RF Measurements on Bulk and High Permittivity Ceramics

This research focussed on challenging microwave measurements on bulk ceramic specimens, which, though often undertaken for industry in the past, have not been thoroughly studied before with a view to quantifying their uncertainties. Measurement difficulties arise from the fact that the permittivities, ε', of these materials are very high, typically with ε' well above 100, often above 1000 and even above 10,000. While these values can be measured capacitatively at lower frequencies, below RF, they present a problem at higher RF and microwave frequencies, as the high permittivity creates gross mismatches for travelling waves. Without due attention to measurement-cell design, measurement uncertainties can as a result exceed 100 %. The aim of this EMINDA research was to reduce and quantify such uncertainties.

This work was largely carried out at GUM and SIQ, but with significant input, especially on modelling, from other EMINDA Partners. GUM focused on microwave measurements while SIQ developed new measurement cells for RF and lower microwave frequencies, working closely with two EMINDA collaborators in Slovenia, the Josef Stefan Institute and the University of Nova Gorica. The importance of these materials for the European electronics industries cannot be underestimated, they are used in dielectric resonators (DRs, e.g. for tuning) and in microwave capacitors.

GUM developed Dielectric Resonator (DR) measurements of two types. The first using quasi-TE_{0mn} mode resonances and the second using whispering gallery mode (WGM) resonances. In both cases sample permittivity is derived from measured resonant frequency and loss tangent or conductivity is determined from the resonance Q-factor. These parameters are measured on a VNA. In the former case, eight samples of bulk ceramic were measured, the samples being provided by SIQ, Slovenia. The permittivities of these specimens ranged from 40 to around 300, while dielectric loss tangents ranged from 10^{-4} to 10^{-2}. This technique had the additional benefit of allowing the measurement of the thermal coefficient of permittivity. The modelling required in this method has been validated on the basis of measurements of standard reference material samples made of sapphire YAG and BZT ceramic. With the help of these measurements a generic uncertainty budget for the quasi-TE_{0mn} mode resonances method was developed. The uncertainty of the real part of permittivity was found to be predominantly dependant on the uncertainty in sample dimensions. For the quasi-TE_{0mn} mode method the uncertainties of both ther diameter and the height of the sample are important. For uncertainty of dielectric loss tangent the following conditions apply:

1. If the contribution of dielectric losses in the Q-factor of the resonator is dominant then the uncertainty of dielectric loss tangent is similar to the uncertainty of the Q-factor measurement

2. If the contribution of dielectric losses is much smaller than the contribution of parasitic losses then uncertainty of the dielectric loss tangent increases, in extreme cases to infinity. For this reason only samples having a dielectric loss tangent larger than 10^{-6} can be measured by employing TE_{0mn} modes

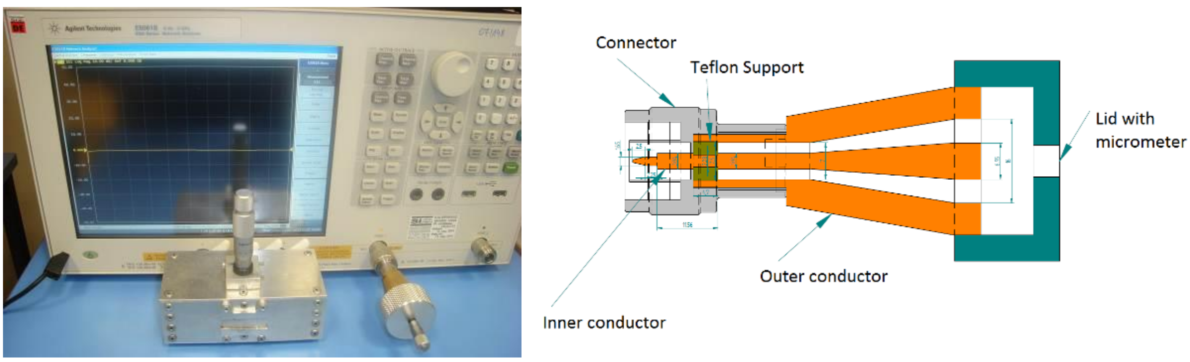
As a result of this EMINDA research, the software for the quasi-TE_{0mn} mode method is now implemented in computer programs – one for each cavity resonator. This software is available at GUM and the technique can therefore be disseminated for use by industrial laboratories.

The second DR approach studied by GUM made use of whispering gallery mode (WGM) resonances, which are better suited to measurements at millmetre-wave frequencies, e.g. above 60 GHz. The material specimens were: alumina ceramic, BMT ceramic, single crystal gallium arsenide and single crystal quartz. Each sample was measured employing several whispering gallery modes. It was thus possible to obtain dielectric loss tangent versus frequency characteristics of these samples. For the anisotropic quartz specimen both parallel and perpendicular components of permittivity were measured employing quasi-TE and quasi-TM modes WGM families.

These measurements helped GUM to develop a generic uncertainty budget for the WGM method. It was found that the uncertainty of the real part of permittivity was predominantly dependant on the uncertainty of the sample dimensions. This uncertainty for the WGM method depends mainly on sample diameter uncertainty and only to some extent on the uncertainty of the sample's height. For modes having sufficiently large azimuthal mode number, the Q-factor due to parasitic losses is much larger than the Q-factor due to dielectric losses. In such a case the contribution of parasitic losses can be neglected and the dielectric loss tangent uncertainty is similar to the uncertainty of the Q-factor measurement.

By comparing the quasi-TE_{0mn} to the WGM method it can be concluded that the losses of very low-loss materials should be measured only with the latter, WGM, technique, as the former does not have sufficient sensitivity to do so. In measurements of materials having dielectric loss tangent > 2×10^{-4}, TE_{0mn} modes are preferable because they are not degenerate and are therefore easier to identify.

At lower RF and microwave frequencies, SIQ carried out research into the design and construction of cells for measurements on high permittivity ceramics, specifically PTC (Positive Temperature Coefficient) ceramics which have major applications in heating elements. Test cells were produced by SIQ with modelling support from NPL. One design is a test cell for use on four-terminal pair (4-TP) impedance analysers or LCR meters (i.e. meters that measure inductance, capacitance, resistance and conductance) and the other is for use on coaxial impedance analysers or VNA's. See Figures 3.7.1 and 3.7.2.

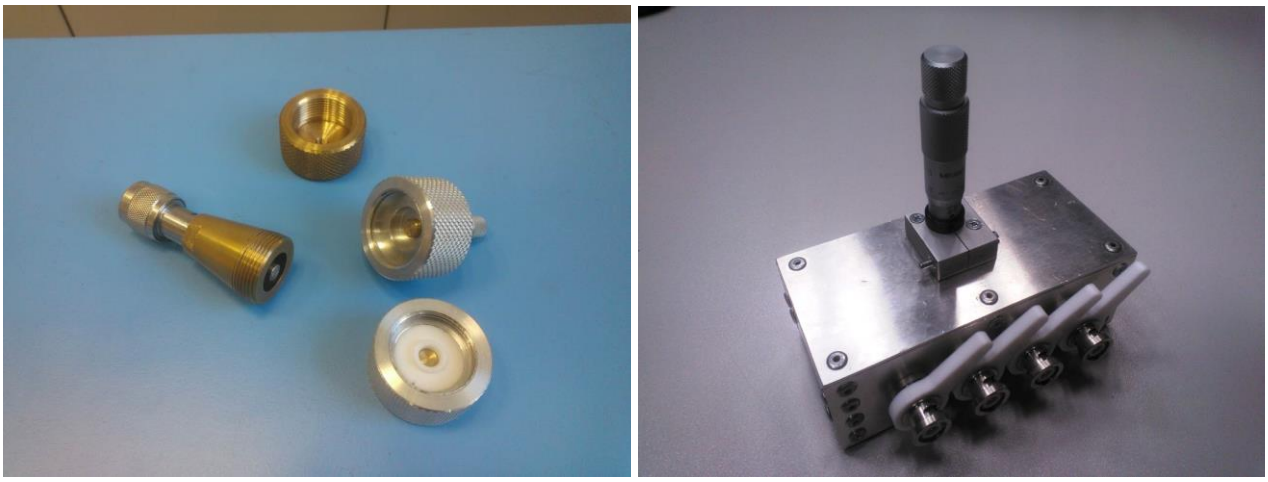


(a) (b)

Figure 3.7.1 Test cells constructed at SIQ for the measurement of high permittivity ceramic: (a) a cell for use on a Four-Terminal-Pair (4-TP) bridge, (b) a coaxial cell for reflectometric measurements on a VNA

In order to evaluate the measurement uncertainties of these cells, calibration and reference materials were obtained. Reference samples for permittivity were manufactured from a variety of materials with dimensions similar to the samples of PTC ceramics produced and developed by the EMINDA Industrial Collaborator JSI

(Josef Stefan Institute). These have permittivity values in the range from 10 to 366. A separate set of calibration materials with nominal permittivities of 1200 and 13000 were purchased. These materials were characterised electromagnetically by the laboratory that sourced them for SIQ, but this does not guarantee any metrological traceability and so a subset of these reference materials with permittivity ranging from 10 to 366 have been characterised traceably by GUM (MG) in Poland using a resonance technique. These samples provide the first reference materials for calibrating and checking such measurements.



(a) (b)

Figure 3.7.2 SIQ RF test cells for ceramics which were modelled at NPL: (a) coaxial cells, (b) a sealed cell showing coaxial connectors for connecting to a 4-TP bridge, see Figure 3.7.1 (a)

Calibration routines for both types of test cells were developed at SIQ. They stipulate methods and calculations which enable traceable measurement of permittivity using the test cells and appropriate analysers. The calibration procedure specifies how the analysers have to be calibrated before the measurements, using reference samples and other artefact standards. This provides a comprehensive approach for calibrating the measurement cells when they are used away from SIQ by other laboratories, including industrial laboratories.

As part of the uncertainty analyses undertaken, the influence of metallisation on specimens has been studied and taken into account in the uncertainty budget. Metallisation of the surfaces of specimens is essential in these measurements to prevent unwanted air gaps between the specimen and cell, which would otherwise generate very large measurement errors. But the metallisation itself can introduce errors, e.g. it could introduce loss mechanisms which are not intrinsic to the dielectric under study. Other influential parameters, e.g. reference sample temperature and dimensional uncertainties, have been included in the uncertainty budget. It has been shown that major contributions to uncertainty are the dimensions of the sample and the uncertainty of the permittivity of the reference samples. Temperature has a major influence on the uncertainty thus temperature should be controled/measured and quoted with the measurement results.

A major goal of this research at SIQ was to automate this measurement process so that it could be employed effectively away from SIQ, especially in an industrial production line. A basic remote-control and acquisition system for measurement results was developed at SIQ for our EMINDA Industrial Collaborator JSI (Josef Stefan Institute) to use with their impedance analyser. This software has been integrated so that all calculations of permittivity and loss are now done in the same software as instrument control and data acquisition. The software can operate on a range of LCR meters or 4-TP impedance analysers. Uncertainties are calculated for each of the parameters automatically. The software also allows 1-port (reflectometric) measurement on VNAs to be used to calculate all parameters from the S11 parameters that are measured by the VNA. This instrumentation and software has been set up at JSI for measurement of ceramic samples during production. The 4-TP cell is now used on an impedance analyser that covers the frequency range from 5 Hz to 13 MHz. Measurements using this analyser have been routinely used since January 2014. A second analyser has been employed for measurements using the coaxial cell and an analyser that measures the S11, reflection coefficient. The analyser covers a frequency range from 1 MHz to 1000 MHz. Complete calibration and measurement routines have been programed into the operating software by SIQ so that calculation of complex permittivity is done in real time after the measurement is performed. In this way measurement time has been significantly reduced and also confidence in measurements has significantly improved by calibrating the cells using reference samples, which provides traceability of the measurements together with the uncertainty. Similar work for our EMINDA partner the University of Nova Gorica has formed the basis of a Case Study which has been published on the EMINDA web-site, http://projects.npl.co.uk/eminda/case-studies/

3.8 Partner Collaboration – Modelling and Measurement Comparisons

Sections 3.1 – 3.7 describe how the various techniques enhanced or newly developed in EMINDA were championed by individual NMIs. But it is very important to emphasise that EMINDA was an integrated project, with each of the Partners supporting each of the research efforts to some extent – either through technical advice, production or loan of specimens or via collaboration in modelling. This co-operation in IND02 EMINDA continued very successfully throughout the project.

Thus Agilent, METAS, NPL, and the researchers in at ETHZ and IC effectively co-operated in the field of NSMMs on the problem of modelling specimen/sample interaction and the effect of stray EM fields - these are issues which affect the uncertainty of all NSMM measurements and they are now appreciated better as a result of EMINDA. Also, in the field of NSMM measurements, there were a number of measurement sessions in which METAS and ETHZ scientists worked at Agilent’s centre in Linz, Austria. METAS and LNE also co-operated to perform NSMM measurements. Similar collaborative activity between PTB and LNE led to optimisation of two Co-Planar Waveguide (CPW) test structures which have been fabricated on quartz and alumina substrates. NPL was able to support SIQ by modelling the measurement cells described in Section 3.7.

Partners have continued to exchange dielectric specimens for measurement, both to help test the capability of measurement techniques and to improve confidence of measurement. Split cylinder (SC) measurements at PTB have been compared with SPDR measurements at NPL and GUM. This activity has included measurements on laminar substrates as well as conductivity measurements of metallisation and has helped all three NMIs to gain a better understanding of measurement issues. GUM has provided a number of specimens for PTB, LNE and NPL who would not have been able to access these materials without this input. SIQ has helped NPL to acquire specimens from the Josef Stefan Institute in Ljubljana, one of our IND02 EMINDA Collaborators.

4 Actual and Potential Impact

In Section 4.1 we present key project outputs from EMINDA detailing how the NMIs in EMINDA - METAS, LNE, PTB, GUM SIQ, NPL – are generating impact as a result of research undertaken in EMINDA. Section 4.2 looks at the wider impacts that have arisen from the EMINDA project, while Section 4.3 describes opportunities for longer term impact.

Key project outputs from EMINDA fall into two areas. First, new measurement facilities, and the expertise to run them, have been developed by the EMINDA NMIs. These facilities are now available for customer services and industrial research investigations. Secondly, on the broader industrial support front, significant effort throughout the project was put into the transfer of metrological know-how from the NMI partners to industry and research institutions. Section 4.1 describes key project outputs and impact generated by the new metrological tools established within the NMIs, while impact generated on the broader front is described in Section 4.2. As intended at the start of the project, the new measurement facilities reside in the laboratories of the individual EMINDA NMIs and are available, usually, if used as part of a measurement service for a fee charged by the individual NMI, but if employed in an agreed collaboration with industry or a research laboratory no direct fee is charged. It should be emphasised that, although these facilities now reside in individual NMIs and are now supported by the NMIs in which they reside, their creation in EMINDA was a joint collaborative effort between EMINDA partners and there is every intention that this close collaboration will continue in future, given suitable resources, in order to improve the offering from European NMIs in support of European RF and Microwave industries.

4.1 Intermediate Impacts from EMINDA Metrological Research

METAS and ETHZ collaborated on the generation of artefact standards for calibrating AFM-based NSMMs. They were designed by ETHZ and were tested at METAS. They are now available for use in industrial laboratories. These NSMMs are typically used for measuring dopant densities in semiconductor materials in electronics and such measurements are employed to improve the efficiency and reduce power loss in circuits – they can therefore have a wide impact. The instrument manufacturer Agilent Technologies – now Keysight Technologies - manufactures such NSMMs and is an unfunded partner in this project. Keysight are already benefitting from research undertaken by METAS and ETHZ. Keysight has taken up an NSMM calibration technique from this work and its algorithm is downloadable as an official add-on to the classic Agilent Technologies AFM-program 'PicoView'. Results from the METAS/ETHZ NSMM studies have been disseminated in scientific publications, and in presentations at conferences.

SIQ has developed new metrological tools for electroceramic material development. Thus, SIQ collaborated with the Jozef Stefan Institute (JSI) in Slovenia to establish new measurement capabilities for electroceramics. The software that is associated with these capabilities has been written so as to integrate measurements with computations to give a real-time capability that can be used in production-line environments. These capabilities are already in use by JSI:

- Measurement of dielectric properties of ceramic samples at temperatures from room temperature up to 500 °C and up to 1 MHz in frequency (sample size up to 10 mm in diameter and 5 mm thick)
- Measurement of dielectric properties of ceramic samples at room temperatures at frequencies up to 13 MHz (sample size up to 10 mm in diameter and 5 mm thick) using a 4TP test cell
- Measurement of dielectric properties of ceramic samples at room temperatures at frequencies up to 1000 MHz (sample size up to 7 mm in diameter and 3 mm thick)

SIQ also worked with EMINDA collaborator UNG (University Of Nova Gorica) on a Case Study which dealt with error corrections for their material characterisation system for low temperatures, see http://projects.npl.co.uk/eminda/case-studies/. This work has enabled UNG to acquire a better understanding of the materials they are developing. Results from these collaborative studies at SIQ have been disseminated in scientific publications and in presentations at conferences.

This work with JSI and UNG will enable manufacturers of high permittivity electroceramics to improve their performance and to provide better specifications to potential customers. Among the applications of such high performance materials, low loss ceramic capacitors are required ubiquitously in electronics. Another variety of electroceramic for which this work has impact is PTC (Positive Temperature Coefficient) ceramics. These materials are used in energy-efficient heating elements. At higher frequencies high permittivity ceramics are used in dielectric resonators, one application of which is for filters for radio channels, both in base stations and receivers. The potential impact for this EMINDA work is therefore very broad, contributing to electronics, telecoms and energy saving.

At NPL the micron-resolution NSMM (see Section 3.2) has already been used in a research investigation with Imperial College to characterise films of graphene. A paper describing this investigation has been published (Gregory *et al* (2014), see Section 3.2). For applications that require deposition of graphene and other functional thin films over large areas (e.g. on centimetre scales) there is clearly a requirement to check the uniformity and integrity of deposition. The NPL NSMM has this capability, as shown in Figure 3.2.8 above. Another application for the NSMM is the dielectric measurement of multiphase materials such as minerals. Microwave heating for mineral extraction relies upon high power differential dielectric heating in the various phase components of rocks to shatter the rock. NPL has been working with Nottingham University's National Centre for Industrial Microwave Processing (NCIMP) to apply such measurements to the optimisation of such processing techniques, which promise very large energy savings.

In another EMINDA research collaboration NPL worked with Powerwave Technologies Inc - a company working on the design and manufacture of RF and microwave filters, antennas and power amplifiers for the mobile telecommunications industry - to improve understanding of the performance of dielectric resonators for use as filters in communications base stations. The NPL NSMM was used to scan the permittivity variations in the electroceramics used in these resonators, providing quantitative information that can be used to improve the manufacture and performance of these materials. The materials used in the filters are sintered at elevated temperatures and performance depends upon good quality sintering. The micron-scale scans of permittivity possible with the NSMM now enables manufacturers to understand how well the sintering has proceeded and should allow studies of improved sintering to be undertaken. This work is described in a Case Study that has been published on the EMINDA website, see http://projects.npl.co.uk/eminda/case-studies/.

In a second Case Study NPL worked with EMINDA Partner the University of Nottingham on a Microwave Chemistry feasibility study to monitor chemical polymerisation reactions on-line. The purpose of the study was to investigate the next steps into the practical design of an on-line monitor for such reactions. NPL characterised the microwave dielectric properties of a number of the reagents as a preliminary step in this undertaking.

Key Outputs Leading to Future Impact

The following key metrological advances achieved in EMINDA have been targeted at industrial requirements that are anticipated to have industrial impact in the near future.

The research on NSMMs at METAS has enabled METAS to develop a new NSMM of its own, operating from 1 GHz to 50 GHz with a scan area of 200 µm x 200 µm. Longer-term impact may be expected from this facility in future as it is now available for research investigations.

LNE has developed a CPW technique for characterising functional thin films which can be used in research investigations for customers and collaborators. The results from LNE studies have been disseminated in scientific publications and in presentations at conferences

Using existing facilities, PTB have solved a number of problems that limited the application of Split Cylinder Resonators (SCRs), leading to two first-time achievements. First, a capability for traceable measurement with SCRs with a comprehensive uncertainty budget. Secondly, a resonator-model capable of dealing with asymmetric resonances - this greatly increases the number of frequencies at which the SCR can carry out reliable measurements. The SCR can now provide a traceable method for accurately characterising the dielectric properties of substrates and laminar materials. These are important parameters which must be known by on-wafer electronics designers if they are to optimise circuit design.

In work also targeted at dielectric measurement on electronics substrates PTB and LNE have performed measurements and permittivity extractions at frequencies up to 110 GHz on new coplanar waveguide test structures, thereby surpassing one of the chief goals for EMINDA: to evaluate dielectric measurement methods up to at least 80 GHz. In addition, a new on-wafer measurement method based on the combination of the two well-known Heinrich CPW models has been developed at PTB enabling the extraction of the loss tangent up to at least 80 GHz. Results from PTB studies have been disseminated in scientific publications and in presentations at conferences

GUM have developed a number of new techniques which are being used in research investigations. They now have a traceable measurement capability for mapping the surface conductivity of semiconductors and thin films which is available for customer measurements and research investigations – this is an important capability as it informs manufacturers about the quality of the semiconductor materials they are using.

GUM have also developed traceable measurement techniques for Quasi-TE_{0mn} mode complex permittivity measurements on ceramics, such as the dielectric resonator materials that are used in electronic channel filters. The software that enables this technique to be used is available from GUM and can be used in other laboratories. GUM have likewise developed traceable measurement techniques for whispering gallery mode (WGM) complex permittivity measurements on ceramics, this is important because it enables measurements to be performed at much higher millimetre-wave frequencies. This capability is available for customer measurements and research investigations. Results from GUM studies have been disseminated in scientific publications and in presentations at conferences

At NPL the micron-resolution NSMM (RNSMM) continues to be enhanced to expand the range of measurements on dielectric materials that can be offered to customers. Two enhancements were being implemented at the end of the EMINDA project. First, the facility to use polar reference liquids as reference materials to calibrate the NSMM will greatly expand the range of traceability for loss measurement which can be disseminated to research collaborators and industrial customers. A paper on this technique for the traceable measurement of loss using the NSMM is being written. Secondly the implementation of a beam deflection method by NPL and Imperial College will make the NPL NSMM more robust and therefore more cost-effective for customer measurements because of the reduction in down-time. Results from these NPL and IC studies have been disseminated via scientific publications and in presentations at conferences, negotiations for its first potential customer are in hand.

How EMINDA is supporting Industrial requirements and European Policies

All of these new facilities and impacts relate to the specific and high-level needs described at the start of the EMINDA project. Specifically the project has developed new traceable Electromagnetic (EM) materials metrology which enables the uptake of new EM materials by European industries, especially the electronics and ICT industries – as illustrated above. At a high level, in its targeted areas of metrology, it has developed European metrological infrastructure for EM materials characterisation so that it can match, in terms of industrial impact, the efficacy that other fields of metrology have already achieved. The intermediate impacts achieved so far will feed into wider and longer-term European socio-economic policy by improving the efficacy and quality control of the European industries that must carry through those policies. Most directly the industries that benefit lie in the electronics and ICT sectors, but there will also be impacts for the aerospace industries, for defence contractors and in health, medicine and safety – in short in all industrial and social areas where RF and microwave electromagnetic fields interact with materials.

4.2 Impact from Widening Support for European EM Materials Metrology

Throughout the EMINDA project, on the broader industrial support front, significant effort has been put into transfer of know-how from EMINDA's NMI Partners to industry and research institutions. The setting up of the European EM Materials Measurements Club (EMMA-Club) as a European Club has been particularly effective in this respect, see http://projects.npl.co.uk/eminda/emmaclub/. There have been two full international meetings of the Club: in Paris (2012) and London (2014), an international workshop on microwave dielectric metrology in Ljubljana (2013), and two local club meetings in Warsaw (2012) and Braunschweig (2013). Overall, well over 100 non-Partner scientists and engineers have attended these meetings at which they were able to discuss dielectric metrology in detail with Partners, besides receiving the benefits of access to the papers and posters presented at the meetings and direct contact with their authors. Feedback from EMMA-Club attendees has been extremely positive about the value of these events for themselves and for their companies and there has been much support for the continuation of the club beyond the end of EMINDA – we are therefore looking at options for this.

Two direct outputs from the last EMMA-Club meeting may be mentioned. The University of Birmingham approached NPL to discuss problems with data extraction in the CPW method (Section 3.4). Both laboratories are now sharing experimental and theoretical data and plan to continue beyond the end of the project to ensure the expertise gained in this project is passed on. Discussions with meeting attendees also tell us that they benefit from meetings between each other – the Club acts as a valuable forum to facilitate communication across the whole RF & Microwave materials measurements community in Europe.

The EMMA-Club and the IND02 EMINDA web-site are specifically designed to put Partners in touch with potential end-users and to inform them of the benefits that IND02 EMINDA and NMI capabilities can offer them – for example see the 'NMI Capabilities' page on the JRP web-site: http://projects.npl.co.uk/eminda/nmi_capabilities.html. All Partners ensure the take-up of results by end users through personal contacts. Measuring material samples of interest to end users helps them to evaluate whether the technology is interesting for them. In the longer term, publications are used to transmit knowledge to end users.

Good Practice in measurement is very important. It can make all the difference between delivering accurate measurements that can be trusted and inaccurate measurements in which we can have no confidence. Good Practice can also contribute significantly to the cost-effectiveness of measurements and can potentially save us a good deal of time. The project published three good practice guides, available at http://projects.npl.co.uk/eminda/good-practice/ - these were requested by industrial members of the EMMA-Club, and their use is demonstrated through the Case Studies, undertaken by NPL and SIQ which are posted on the EMINDA web-site, http://projects.npl.co.uk/eminda/case-studies/.

4.3 Impact beyond the end of the JRP

The benefits of the EMMA-Club and networking available from the IND02 EMINDA web-site are valuable outputs from the project. Feedback from EMMA-Club attendees confirms that it has impact for them. We are seeking ways to keep the club alive beyond the end of the project.

All of the new measurement facilities developed in the project (see Section 4.1) are targeted at support for European industries and research institutions in the long term: well beyond the lifetime of IND02 EMINDA. They will be used for measurement services and collaborative research investigations.

The EMINDA project could not itself address all of the metrological requirements of European industries for RF and Microwave dielectric metrology. Industrial contacts gained through EMINDA have helped the Partners to identify areas of work that are required to have impact for industry in the future. Recommendations for further work, some following up on EMINDA activities and some in new areas are made in a separate report which is a deliverable from the project.

In addition IND02 EMINDA Partners will continue to publish articles and to present papers on work that has been undertaken in this project.

5 Website Address and Contact Details

The EMINDA website is http://projects.npl.co.uk/eminda/

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6 List of Publications

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Karsten Kuhlmann and Uwe Arz, 'Uncertainties in Split-Cylinder Resonator Measurements', 79th ARFTG Conference Digest, pp. 121-124, 22.06.2012. ISBN: 978-1-4673-1230-1,

Uwe Arz, 'Introduction to Advanced Dielectric Measurement Techniques Millimeter-Wave Measurements: On-Wafer Techniques', IMS 2012 Workshop Electronic Notes, 18.06.2012

Jerzy Krupka, 'Introduction to Advanced Dielectric Measurement Techniques: Substrate Measurements with Split Post Dielectric Resonators', IMS 2012 Workshop Electronic Notes, 18.06.2012

Jerzy Krupka, Łukasz Usydus and Henryk Kołtuniak, 'Surface resistance and conductivity of rough surfaces of metals on printed circuit boards and metalized ceramic substrates', MIKON Conference Digest, 2012. 21.05.2012

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Andrew Gregory, Ling Hao, Norbert Klein, John Gallop, Cecilia Mattevi, Olena Shaforost, Kevin Lees, Bob Clarke, 'Spatially resolved electrical characterisation of graphene layers by an evanescent field microscope', *Physica E: Low-Dimensional Systems and Nano-structures*, **56**, February 2014, pp. 431–434, 2012. http://dx.doi.org/10.1016/j.physe.2012.10.006

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Hao, John Gallop, Mark Stewart, Kevin Lees and Jie Chen, 'Multi-functional MEMSINEMS for Nanometrology Applications', Proceedings of the 13th IEEE International Conference on Nanotechnology, Beijing, China, August 5-8, 2013, p1119-1124. Doi: 10.1109/NANO.2013.6721047