Final Publishable JRP Summary for IND02 EMINDA Electromagnetic Characterisation of Materials for Industrial Applications up to Microwave Frequencies

Overview

Advances in electronics technology lie behind all developments in Information and Communications Technology (ICT). There is a world-wide demand for ever-faster electronics. Modern electronics is very much materials–based, i.e. ‘solid-state’, and in today’s ubiquitous on-wafer (‘chip’) electronics a key role is played by both *active*, ‘functional’ materials, such as semiconductors, piezoelectrics and ferroelectrics, and *passive* ‘dielectric’ materials that provide substrate (wafer) and support-structures and which play an essential role in the transmission of signals from place to place. With clock-rates in computers already in the microwave (i.e. GHz) region of the spectrum and rising ever higher in frequency, it has become important to characterise the electromagnetic (EM) properties of all these materials at RF (Radio Frequency) and Microwave frequencies (their operational frequency range) to enable industry to design and manufacture ultrafast electronics applications.

The project developed a range of metrological tools to provide accurate measurement of key EM parameters to support these emerging industrial needs.

Need for the project

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.

Scientific and technical objectives

There was a need to target metrology at frequencies and dimensional scales that were seen to be of most practical importance for developing European ICT and electronic industries. This required metrology up to microwave frequencies of 80 GHz, to cover the ever increasing clock rates of electronic components, and metrology at the micro- and nano-scale on the surface of individual materials and at the macro scale of the silicon wafers (substrates) onto which electronic circuits are printed and for thin-films of functional materials that may, in the future, replace silicon.

A number of advanced measurement techniques have recently become available that offer the ability to provide high level metrological capabilities for ultrafast electronics. These tools need to be better understood

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and their performance characterised to ensure that NMIs can meet the developing measurement needs of the European electronics industry. Therefore the overarching project objective was:

To develop **a suite of traceable metrological tools** 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.

To meet these objectives the project focused on the development and characterisation of a range of new measurement techniques at relevant high frequencies:

- **Nanoscale and Microscale Near-Field Scanning Microscopes** to determine the dielectric, capacitive and conductive properties of materials at small spatial scales across a material surface. Understanding these properties at this scale enables the selection of materials with specific features for electronics and other applications.
- **Co-planar Waveguide (CPW) and Split Cylinder (SC)** techniques to provide traceable measurements on **substrate** materials and on functional (e.g. ferroelectric) **thin films** mounted on substrates. These are needed to assess the quality and performance of traditional silicon substrates at high frequencies during the manufacturing process and to assess the performance of the thin-films.
- The project also sought to extend the existing measurement techniques to assess the performance of **bulk dielectric materials** (typically, high permittivity ceramics) at the radio frequencies at which they will ultimately be used.

On the broader front, in supporting European Industry, the project addressed the following objectives:

- Set up new measurement services for EM materials and to inform industry of the European NMI measurement capabilities via the project website.
- Support European industry through a Measurement Club – the EMMA Club.
- Ensuring that project activities are relevant to industrial needs by working closely with industrial collaborators and by carrying out practical Case Studies with industry.

Results

The project developed a range of new metrological tools and capabilities as described below.

Nanoscale Near-Field Scanning Microscope Metrology (NSMMs)

NSMMs are used to scan the dielectric, capacitive and conductive properties of materials at 'small' spatial scales across a material surface. In the project two types of NSMM were developed, one with a spatial resolution of tens of nanometres and the second with a spatial resolution of tens of micrometres (see next section). Applications of NSMMs include measurement of dopant profiles in semiconductors, checking of uniformity of ceramic materials, evaluating the dielectric loss of mineral specimens for microwave processing and checking the uniformity of thin films, such as graphene.

METAS, working with ETHZ and Keysight Technologies (formerly Agilent), successfully developed new algorithms, calibration artefacts and skills for measuring microwave-frequency permittivity and other electromagnetic material parameters at nanometre scales using cantilever-based NSMMs. Their work made major steps forward when compared with previous approaches. Progress in this field was sufficient to allow METAS to develop their own NSMM during the project. A number of scientific papers have been published on this work, and calibration artefacts developed by METAS and ETHZ are now in use by Keysight Technologies.

Microscale Near-Field Scanning Microscope Metrology (NSMMs)

Work at NPL and Imperial College (IC) has advanced our understanding of NSMMs used in the micrometre range. Probe/sample proximity is an important parameter to monitor in these measurements and two independent systems have been employed for this purpose: acoustic damping of tuning-fork-induced vibrations of the probe and laser based optical detection - the latter implemented for the first time for this type of NSMM. The project has advanced the measurement of dielectric loss by these NSMM systems, both in terms of numerical modelling, where a new complex algorithm has been applied, and through the development of a new artefact calibration standard. At NPL, it is now possible to make traceable measurement of complex permittivity at microwave frequencies and these facilities are available to support European industries.

In NSMM metrology, it is essential to understand how EM fields interact with the probe and sample and several approaches were adopted in the project for modelling these field interactions. This work was pursued in four NMIs, both at the nanoscale and the microscale, and has significantly progressed understanding. Thus, measurement errors introduced by stray capacitances have been shown to be significant, requiring practical designs to reduce them. Likewise, simple capacitive models have been shown to be inadequate, especially for the measurement of dielectric loss. Microwave resonances in the body of specimens can be induced via the NSMM probe and a better understanding of the measurement limitations caused by them has now been achieved. The modelling has contributed to far better uncertainty estimates for this class of measurement. Traceable microwave NSMM measurements are now within the remit of two NMIs in Europe.

Traceable Broadband Dielectric Measurements of Substrate Materials

All solid state electronics at microwave frequencies are implemented 'on wafer' and the dielectric characterisation of the substrates on which electronic circuits are printed is an important contribution to quality and performance control. At PTB, the metrology of Split Cylinder (SC) measurements on laminar substrate materials has been developed significantly allowing measurements of permittivity to be undertaken at a much larger number of microwave frequencies. A thorough uncertainty analysis has been undertaken for this method. Measurement results have been compared with Split-Post (SPDR) measurements at other NMIs (NPL and GUM) for five different materials and this has shown good agreement within the uncertainty range for both relative permittivity and dielectric loss. This PTB work helped all three NMIs to gain a better understanding of measurement uncertainties and improved confidence in measurement.

Similar collaborative activity between PTB and LNE has improved optimisation of Co-Planar Waveguide (CPW) test structures fabricated on quartz and alumina substrates. Measurements and permittivity extraction up to 110 GHz have been performed at LNE and PTB on these structures, thereby surpassing one of the chief goals of the project: to evaluate dielectric measurement methods up to at least 80 GHz. In a major achievement, PTB implemented new modelling approaches for the CPW measurement of complex permittivity, improving sensitivity for very low loss measurements to levels normally only associated with resonant techniques. This approach has also delivered a comprehensive uncertainty budget for the method. As part of this activity, GUM has extended capabilities for measuring surface conductivities of laminar specimens using SPDR metrology which has a key role in evaluating semiconductor materials.

On-Wafer Traceability for Advanced & Functional Thin Films

It is internationally anticipated that silicon-based electronics will be nearing its limit of performance over the next decade or two. There is therefore much interest in evaluating other materials, which can replace some of the functions of silicon and also, very importantly, cut down on energy dissipation. The functional materials of interest (e.g. ferroelectrics, piezoelectrics, magnetoelectrics, multiferroics) will be utilised in circuits in the form of thin films. It is therefore important to develop and evaluate these materials in thin film form. Both NPL and LNE studied the measurement of thin dielectric films by CPW methods. NPL addressed the measurement of magnetic and other, e.g. ferroelectric, films, while LNE undertook measurements on barium strontium titanate and lead zirconia titanate films which both showed agreement with literature values for permittivity and led to a better understanding of the uncertainties in these measurements than has previously been published in the literature. LNE has developed an optimised structure for permittivity measurements and performed successful measurements to 110 GHz, again exceeding the project's 80 GHz target. These techniques are now available at both NMIs to support European industries in their measurements.

NPL has developed the methodology that would allow the decoupling of magnetic and electric field within thin film specimens deposited onto a substrate in a CPW geometry. This will allow the coupling coefficient of a suitable multiferroic material to be characterised. During the project such materials were not available so the technique could not be fully trialled, but the capability has been established and can be deployed when the materials become available.

Measurements on Bulk and High Permittivity Ceramics

While small-scale and thin film dielectric metrology is important for the further development of electronics, there remain a number of industrially important areas in which bulk dielectric components are used and for which accurate metrology to characterise the intrinsic performance of materials has been sorely lacking. One such area is the characterisation of very high permittivity (> 1000) electroceramics at RF. SIQ, with modelling support from NPL, tackled this problem. As a result, they have significantly improved capability and confidence in measurements on high permittivity dielectrics (permittivity typically > 500). This work is already proving of value to electroceramic researchers. The NPL/SIQ collaboration on the modelling of the measurement-cell/sample interactions has contributed greatly to the improvement and quantification of uncertainties in these measurements. Measurement cells developed at SIQ are now in use in the laboratory of a research collaborator institution (The Josef Stefan Institute) that is developing electroceramics.

Actual and potential impact

Regarding practical measurement, the NMI partners in the project can now offer much better support to European industries as a result of the work undertaken. A number of new measurement systems have been developed that can provide measurements for customers. In some cases customers are already making use of the new capabilities:

- At SIQ: Four terminal cells are available for measuring high permittivity ceramics up to 30 MHz, 10 mm diameter and 5 mm thickness. A coaxial cell has also been developed for measurements at higher frequencies. These cells and the techniques for using them developed in the project are now being used by the Josef Stefan Institut and the University of Nova Gorica for the development of new ceramics for industrial applications.
- At GUM: A new measurement capability for dielectric measurements on semiconductors and measurements on ferroelectric thin films is available.
- At GUM: New Split-Post resonators are in use for determining sheet resistance. These can now be deployed by NMIs or by industry.
- At METAS: A new NSMM with a measurement frequency range from 1 GHz to 50 GHz and a scan area of 200 µm x 200 µm has been built. Calibration artefacts have been designed and tested which can be used for calibrating NSMMs in industrial laboratories.
- At NPL: The NSMM can now perform traceable measurements of both permittivity and loss. It is available for, and has already been used for, research investigations for industry (see below) and in academic research - one example of this is the use of the NPL NSMM in a research study on the structure of graphene layers (see Publications, below).

Throughout the project, on the broader industrial support front, significant effort was put into transfer of know-how from the NMI partners to industry and research institutions.

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/

The project established the European Electromagnetic Materials Measurements and Applications Club (EMMA Club) to promote knowledge transfer, good practice and know-how for dielectric and EM materials measurement: 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 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 the project and options for this are being considered.

User uptake

Industrial users are already making use of the new metrological capabilities developed in the project:

Keysight Technologies (formerly Agilent), a manufacturer of instrumentation for the electronics sector, participated as a key partner in the project and is already benefitting from research on traceability for Atomic Force Microscope (AFM)-based NSMMs. They are making use of calibration artefacts and knowledge developed in the project to expand the range of electronic materials properties that can be measured and hence make their NSMMs more useful and attractive to Industrial buyers. The collaboration between the NMIs and Keysight is continuing beyond this project.

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, collaborated with project partners to improve their understanding of the performance of the dielectric resonators they manufacture for use as filters in communications base stations. The NPL NSMM was used to scan the permittivity variations in electroceramics used in dielectric resonators. 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 hhas proceeded and should allow studies of improved sintering to be undertaken.

The enhanced capability developed in the project also enables the NMIs to work more closely with academic institutions in ongoing research investigations relevant to longer-term developemtns in ultrafast electronics. For example, SIQ developed a remote-controlled measurement acquisition system which is now in use by the University of Nova Gorica for measurements at low cryogenic temperatures up to 2 MHz in frequency. University researchers can now make measurements more efficiently and reliably and with better repeatability. NPL worked with 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.

Discussions with industry and academia are ongoing to ensure that the project continues to have impact: the NSMM calibration work with Keysight, especially that relating to the calibration procedures, has continued; the cells developed at SIQ will be further developed with the Josef Stefan Institute and the University of Nova Gorica. NPL is discussing applications of the micron-scale NSMM with potential industrial customers. More generally, discussions with industry on their needs for further EM materials metrology developments will continue via the EMMA-Club.

The continued development and use of the metrological infrastructure developed in European NMIs to characterise EM materials at higher frequencies will enable the design and manufacture of components, systems and the corresponding manufacturing capabilities in fast electronics and so support the continued success of the European electronics industry.

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JRP-Coordinator: Mr Bob Clarke, NPL Tel: +44-20-8943-6156 E-mail: bob.clarke@npl.co.uk JRP website address: http://projects.npl.co.uk/eminda/	
JRP-Partners: JRP-Partner 1 NPL, UK JRP-Partner 2 EJPD, Switzerland JRP-Partner 3 MG, Poland JRP-Partner 4 LNE, France	 JRP-Partner 5 PTB, Germany JRP-Partner 6 SIQ, Slovenia JRP-Partner 7 Agilent, Austria
REG 1-Researcher (Associated Home Organisation):	Jens Niegemann, Switzerland ETHZ, Switzerland
REG 2-Researcher (Associated Home Organisation):	Stephen Hanham, UK IC, UK
REG 3-Researcher (Associated Home Organisation):	Christian Hafner, Switzerland ETHZ, Switzerland
REG 4-Researcher (Associated Home Organisation):	Stephen Hanham, UK IC, UK

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