*Project title*: Traceability for electrical measurements at millimetre-wave and terahertz frequencies for communications and electronics technologies (TEMMT)

Start date and duration: 1 May 2019, for 3 years and 3 months

Website: http://projects.lne.eu/jrp-temmt/

European Metrology Programme for Innovation and Research (EMPIR) project (part of Horizon 2020)



Traceability for electrical measurements at millimetre-wave and terahertz frequencies for communications and electronics technologies



#### Introduction

- Millimetre-wave and THz spectrum actively exploited by applications, e.g. 5G (wireless backhaul links), connected autonomous vehicles (radar sensors), space-born radiometers for Earth remote sensing, security imaging...
- Lack of traceability for electrical measurements >100GHz, although measuring instruments commercially available
- This project aims to establish traceability to the SI for 3 electrical measurement quantities: S-parameters, Power, Material properties (complex permittivity), at millimetre-wave and THz frequencies



#### **Objectives and workpackages overview**

#### WP1: Traceable connectorised S-parameter measurements

- Develop new metrology for waveguide to 1.5 THz
- Establish traceability for E-connector for 5G and automotive radars
- Exploit outputs from previous EMRP project SIB62 (HFCircuits)



#### WP2: Traceable planar S-parameter measurements

- Improve on-wafer measurements to 1.1 THz
- Develop new planar calibration/ verification standards
- Exploit outputs from previous EMPIR project 14IND02 (PlanarCal)

#### WP3: Traceable power measurements

- Extend calorimeters and transfer standards above 100 GHz
- Investigate traceability for commercial power meters at THz frequencies

#### WP4: Traceable material measurements

- Investigate material properties over a wide frequency range (140-750 GHz)
- Establish traceability for commercial material characterisation kits at THz frequencies





#### Knowledge transfer:

- Standards: IEEE P287, IEEE P1785, IECTC46, IEEE On-wafer Group
- ≥ 6 journal papers, ≥8 conference papers, ≥1 trade journal article
- Public reports on EURAMET comparisons for measurements of power and materials
- Technical Advisory Group

#### WP5: Creating impact

#### Training:

- 2 workshops delivered at annual European Microwave Week
- 2 training courses delivered at Project Partner locations
- Practical lab demonstrations of new measurement techniques using commercial equipment
- Researcher visits at key Project Partner organisations

#### Uptake and exploitation:

- Exploitation plan for disseminating developed standards and customer calibration services
- Comprehensive measurement capability based on outputs from this project and earlier European projects (SIB62, 14IND02)
- Establish link with European Microwave Association to increase
  project uptake
- Proposal to EURAMET for a coordinated network of EU NMIs

#### **19 Participants: 9 NMIs + 5 research institutes + 5 companies**

- NPL, CMI, GUM, LNE, METAS, PTB, TUBITAK, VSL, INTI
- Birmingham University, Chalmers University of Technology, Ferdinand-Braun-Institut (FBH), University of Lille, Military University of Technology in Warsaw
- Anritsu, FormFactor, Keysight, Rohde & Schwarz, VDI





Traceability for electrical measurements at millimetre-wave and terahertz frequencies for communications and electronics technologies



# WP1: Establishing Traceability to 90 GHz for the 1.35 mm Precision Coaxial Connector



#### Nick Ridler, James Skinner and Dan Stokes National Physical Laboratory, UK

1<sup>st</sup> TEMMT Training Course (online), 21<sup>st</sup> July 2021

## Overview



1. General Introduction – Nick Ridler (5 minutes)

2. Dimensional Traceability – Dan Stokes (15 minutes)

3. Electrical Traceability – James Skinner (15 minutes)

# 1. General Introduction



• Work Package 1 (WP1) – Description

• The 1.35 mm Precision Coaxial Connector

• The Research Team

# WP 1 – Description



# Purpose: To develop traceability and verification techniques for S-parameters

In 2 parts:

- (i) Coaxial line: 1.35 mm connector to 90 GHz
- (ii) Waveguide: in 3 bands covering 330 GHz to 1.5 THz



# WP 1 – Description



# Purpose: To develop traceability and verification techniques for S-parameters

In 2 parts:

(i) Coaxial line: 1.35 mm connector to 90 GHz

(ii) Waveguide: in 3 bands covering 330 GHz to 1.5 THz



# 1.35 mm Coaxial Connector

- A precision connector (with air interface)
- A "new" connector introduced in the past few years
- Designed for communications and radar related applications
- Standardised in IEC 61169 (Part 65)
- Standardisation in IEEE Std 287 is on-going







# 1.35 mm Coaxial Connector

- Recommended frequency range: DC to 90 GHz
- The E-band connector, or, "E connector"
- Internal diameter of outer conductor = 1.35 mm





# The Research Team



# Consortium of 4 world-leading National Metrology Institutes (NMIs):

INTI, Argentina



## LNE, France



NPL, UK



PTB, Germany



## 2. Dimensional Traceability



- What is our goal?
- Why Dimensional?
- Line diameters and measurements
- Challenge of connectors
- Comparison

## WHAT ARE WE TRYING TO ACHIEVE?















A "less" simple 1 port network.



What is  $S_{xxc}$  and  $S_{xxL}$ ?

 $\Gamma_x = S_c \oplus S_L \oplus S_x$ 

## WHY START WITH DIMENSIONAL METROLOGY?









### **MEASUREMENT SETUPS**

Outer conductor inner diameter

Air Plug Gauge





Inner conductor outer diameter

#### Air Ring Gauge



Microscopy









## UNCERTAINTY

#### Outer conductor using air gauging system

			Sensitivity			Inverse degrees of	Overall,
Description	Contribution	1	coeff	Distribution	Divisor	freedom	um
Lab temperature	2	°C	1.49E-02	Rectangular	1.73	0	0.017
Resolution	0.5	um	0.5	Rectangular	1.73	0	0.14
Parallax error	0.5	um	1	Rectangular	1.73	0	0.29
Probe linearity	0.2	%	0.15	Rectangular	1.73	0	0.017
Meter accuracy	1	%	0.15	Rectangular	1.73	0	0.087
Probe repeatability	0.1	um	1	Gaussian (k = 1)	1	0.0010	0.10
Flow adjuster hysteresis	0.519	um	1	Rectangular	1.73	0	0.30
Std dev of measurements	0.79	um	0.26	Gaussian (k = 1)	1	0.071	0.20
Calibration standard history	0	um	1	Gaussian (k = 1)	1	0	0
Calibration standard uncertainty	0.8	um	1	Gaussian (k = 2)	2	0	0.40
					Total	0.00071	0.6427
						1410	
					Coverage fa	ctor	2
					Expanded u	ncertainty	1.29
					(c.f. k = 2)		1.29



Measured deviations from nominalStd IOuter diameter average0.500um0

Std Dev m 0.79um

=> 1.3505mm ± 1.29um @ k = 2



0.82

#### UNCERTAINTY

#### Inner conductor using laser gauge

		Sensitivity			Inverse degrees	
Description	Contribution	coeff	Distribution	Divisor	of freedom	Overall, um
Resolution	0.1 um	0.5	Rectangular	1.73	0	0.029
Lab temperature	2°C	0.0064	Rectangular	1.73	0	0.0074
Cal standard misalignment	0.05 mm	0.033	Rectangular	1.73	0	0.00094
Gauge repeatability	0.1 um	1	Gaussian (k = 1)	1	0.0010	0.10
Height error	0.4 um	1	Rectangular	1.73	0	0.23
Laser noise	0.2 um	1	Gaussian (k = 1)	1	0	0.20
Inner conductor position	0.05 mm	0.059	Rectangular	1.73	0	0.0017
Calibration standard history	0 um	1	Gaussian (k = 1)	1	0	0
Calibration standard uncertainty	0.5 um	1	Gaussian (k = 2)	2	0	0.25
Std dev of measurements	0.21 um	0.26	Gaussian (k = 1)	1	0.071	0.055
				Total	2.67693E-05	0.4121
					37356	
				Coverage	factor	2
				Expande	d uncertainty	0.82

Measured deviations from nominalSInner diameter average0.180um

Std Dev m 0.21um

=> 0.5862mm ± 0.82um @ k = 2

(c.f. k = 2)

#### UNCERTAINTY



#### **Characteristic Impedance**

$$Z_0 = \frac{1}{2\pi} \sqrt{\frac{\mu}{\varepsilon}} \ln\left(\frac{b}{a}\right) \cong 59.94 \times \ln\left(\frac{b}{a}\right)$$

					Inverse degrees of	
Description	Contribution	Sensitivity coeff	Distribution	Divisor	freedom	Overall, Ohm
Outer conductor radius	0.6427um	0.044	Gaussian (k = 1)	1	0.00071	0.029
Inner conductor radius	0.4121um	0.10	Gaussian (k = 1)	1	2.68E-05	0.042
Permittivity	0.000078	25	Rectangular	1.73	0	0.001
				Total	8.26E-05	0.051
					12112	
				Coverage	e factor	2
Characteristic impedance	ce <b>50.025</b> Ω	± 0.102 Ω @	k = 2	Expande	d uncertainty	0.10



## **MEASUREMENT SETUPS**

Micro co-ordinate measurement machines (µCMM)





Microscopy





A "less" simple 1 port network.



 $\Gamma_x = S_c \oplus S_L \oplus S_x$ 

#### COMPARISON

- Comparison of conductor diameters and characteristic impedances.
- 2 traveling standards: Beadless airlines.
- 5 participants:





NPLO

**National Physical Laboratory** 



## Electrical Metrology: Key Challenges

#### **Outline**



- Challenges in electrical metrology
  - Sourcing Components
  - Choice of calibration scheme
  - Measurement setup Interfacing
  - Uncertainty Analysis
- S-parameter Results
- Next Steps for 1.35 mm capability
- Opportunities for 1.35 mm coaxial





## Establishing Measurement Capability in 1.35 mm Coaxial





## **Challenge: Sourcing Components**



- New 1.35 mm connector recently introduced to market
- Gradual uptake, still only a limited number of suppliers selling 1.35 mm components
- Currently available off the shelf:
  - Various waveguide to 1.35 mm adaptors
  - 1.85 mm to 1.35 mm coaxial adaptors
  - 1.00 mm to 1.35 mm coaxial adaptors
  - 1.35 mm to 1.35 mm coaxial adaptors (through device)
  - Calibration Standards: Short Circuits, Open Circuits, 50 Ohm Loads, Offset Short Circuits
- Some custom devices available
  - o Airlines, mismatch adaptors





- Calibration required to take accurate measurement through correction of errors present in the system
- Quality of calibration proportional to knowledge of calibration standards
- Choice narrowed by ability to characterise calibration standards





- TRL Through Reflect Line
  - Through: Cabled connection between ports 1 & 2
  - Reflect: Short-circuit standards (male & female) connected to ports
  - Line: Section of coaxial line of known length, known as an 'airline'
  - Consistent full-band calibration achieved through choice of airline lengths
  - Multiple airlines typically used. Effectiveness of line assessed at each frequency to ensure suitable standard is available for across whole of desired frequency band
  - Traceability based on calculation of characteristic impedance of airlines

     derived from diameters of the inner and outer conductors of the coaxial line







- SOLT Short Open Load Through
  - Short: 1-port Short-circuit standard (reflect)
  - Open: 1-port open-circuit standard (reflect)
  - Load: 1-port 50 Ohm termination standard (absorb)
  - Through: Cabled connection between ports 1 & 2
  - Traceability based measurement of characteristics of standards, or database of reflection coefficients







- SOOT Multiple Offset Short Through
  - Short: 1-port Short-circuit standard (reflect)
  - Offset short: 1-port Short-circuit standard (reflect) with offset of known length
  - Through: Cabled connection between ports 1 & 2
  - Might have upwards of 5 offset short-circuits
  - o Can also utilise open, load standards
  - Consistent full-band calibration achieved through choice of short-circuit offset lengths
  - Traceability based on dimensional characterisation of offset short-circuits





- Main factors in decision:
  - Availability of components
  - Ability to characterise standards dimensional capability
  - Compatibility with measurement setup



### **Challenge: Measurement Setup**



- Equipment required to achieve setup capable of measuring in 1.35 mm coaxial to the full extent of its capability i.e. up to 90 GHz
- Main limiting factor is access to VNA

   E-Band not accessible to VNAs with 2.4 mm connector or larger / only 2 ports
- Single setup for full band / multiple banded setups





#### Challenge: Measurement Setup Case 1: 1.0 mm VNA



- VNA: Keysight N5290A PNA Millimeter Wave System
- Port connectors: 1.0 mm 110 GHz upper limit
- Connections: 1.0 mm cables connected to ports 1 & 2
- Adaptors: Rosenberger 1.0 mm (f) to 1.35 mm coaxial adaptors (m & f)
- Frequency range: Full-Band up to 90 GHz




### Challenge: Measurement Setup Case 2: 1.85 mm VNA



- VNA: Keysight N5274B PNA-X
- Port connectors: 1.85 mm 67 GHz upper limit
- Frequency Extension: VDI Waveguide E-Band Extender heads - connected to PNA-X ports 1,2,3 & 4
- Adaptors: Rosenberger E-Band Waveguide to 1.35 mm coaxial adaptors (m & f)
- Frequency range: 60 to 90 GHz (band-limited)
- Additional setup required for measurement below 60 GHz
  - using 1.85 mm to 1.35 mm adaptors





## 1.35 mm Setup via E-Band Extender Heads





## 1.35 mm Setup via 1.85 mm Coaxial





#### 1.85 mm Keysight PNA-X



### **Other Possible Cases**



- 1.0 mm coaxial extension e.g. Keysight V3050A Signal Analyzer Frequency Extender
- Waveguide tapers e.g. W-Band Waveguide Extender Heads (75 110 GHz) + W-Band to E-Band waveguide tapers attached to ports
- And more...



## **Challenge: Uncertainty Analysis**



- Chief contribution: characterisation of calibration standards
- Understanding of connector effects – resonances at high frequencies caused by connector pin gaps
- Uncertainty Budget contributions:



Connector pair interactive region





### Results – Coaxial Setup via 1.85 mm Up to 67 GHz





### **Results – Setup via E-Band Extender Heads** 60 to 90 GHz







### **Combined Results**







## **Next Steps**



- Continue to develop measurement capability
- Dimensional intercomparison between project participants
   In progress
  - Measurement of diameters of 1.35 mm airline inner conductor and outer conductor
- Electrical intercomparison between project participants
  - In progress
  - Calibration techniques
  - Methods of standard characterisation
  - Uncertainty analysis







- Provide s-parameter measurement services for 1.35 mm coaxial
- Establish power measurement capability for 1.35 mm power sensors
- Other measurement parameters





### **Thank You**





### **TEMMT-Workshop**:

WP 2- Guidelines for the design of calibration substrates, including the suppression of parasitic modes, influence of microwave probes and crosstalk effects up to W-band

### Gia Ngoc Phung, Uwe Arz, AG 2.23

Physikalisch-Technische Bundesanstalt, Braunschweig, Germany





- Motivation:
  - On-Wafer Measurements
  - Method of Investigation
- Statement of Problem
- Influence of Microwave Probes
- Propagation and Suppression of Surface Waves
- Summary and Conclusion



### **Motivation**

# PB On-Wafer Measurements (I)

- Main advantage: Characterization of any Device Under Test (DUT) without the influences of packaging
- Measurement performed with Ground Signal Ground (GSG) probes
- Measured raw data contains parasitic effects of
  - the neighborhood
  - the probes
  - measurement instrumentation itself
- How can we obtain the "true" performance of any device ?
  - Applying a calibration process to deduct the unwanted effects from the raw data
  - Here, multiline Thru Reflect Line (mTRL) calibration applied





# PB Method of Investigation: EM Simulation



Bridge model Probe model emulating measuring probes with least parasitics



### **Statement of Problem**

# PB Statement of Problem

- Several problems in on-wafer measurements detected but not clarified
- Focus of the talk:
  - Influence of microwave probes
  - Influence of surface waves





F.J. Schmückle, T. Probst, U. Arz, G.N. Phung, R. Doerner, and W. Heinrich, "Mutual Interference in Calibration Line Configurations," in 89<sup>th</sup> Automatic RF Techniques Group Microwave Measurement Conference (ARFTG) Digest, Honololu, HI, USA, Jun. 2017.



### **Influence of Microwave Probes**

# PB Starting Point of Investigation

- Different results of the same DUT measured with
- different probes from different manufacturers
  - GGB
  - Allstron
  - Infinity

# Which measurement shows the true performance of the DUT, if any ?



## PTB Probes from different manufacturers



(a) Picoprobe (top view, 100-μm pitch), (b) ACP (125-μm pitch), (c) Allstron (100-μm pitch), (d) Infinity Probe (125-μm pitch; all bottom view), and (e) |Z| Probe (125-μm pitch).

[3] Rumiantsev, Andrej and R. Doerner. "RF Probe Technology: History and Selected Topics." IEEE Microwave Magazine 14 (2013): 46-58.

## PTB Parasitic Effects : Systematic Study of Influence of Microwave Probes

- Simulation environment remains the same for all the calibration elements and DUTs
- Construction of a probe prototype
  - Exaggeration of the probe geometries in order to observe pronounced effects
  - Parametrization of the investigated probe geometries (length, slant and angle of probe)
  - Scaling of the cross-section of the probe



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# PB Structure under Probe Region



## Structure underneath the probe shadow

# PB Parasitic Effects : Neighbor in Probe Region (I)

- On metal chuck
  - For distance d<sub>in</sub> = 200 μm
  - Strength of resonance varies with probes







# PTB Parasitic Effects : Neighbor in Probe Region (II)

- On metal chuck
  - For distance d<sub>in</sub> = 400 μm
  - Strength of resonance varies with probes





Probe 1.2 Probe 2.2

# PTB Parasitic Effects : Neighbor in Probe Region (III)

- On metal chuck
  - For distance d<sub>in</sub> = 600 μm
  - Strength of resonance varies with probes





# PB Parasitic Effects : Neighbor in Probe Region (IV)

- On metal chuck
  - For distance d<sub>in</sub> = 800 μm
  - Strength of resonance varies with probes





# PTB Parasitic Effects : Neighbor in Probe Region (V)

- On metal chuck
  - For distance d<sub>in</sub> = 1000 μm
  - Strength of resonance varies with probes





# PIB Impact of Chuck Material

- Neighbor in probe region: DUT on different chuck material
  - Distance d<sub>in</sub> varies from 200 till 1400 μm
  - The larger the distance, the weaker the resonance.
  - Chuck material influences the strength of the resonance







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# **PB** Verification through Measurements

	pramme gramme gramme g
kmml     kmml     kmml     kmml     kmml     kmml       kmml     kmml     kmml     kmml     kmml       kmml     kmml     kmml     kmml     kmml	

# PB Simulation of Layout Design

Simulation of complete wafer with different excitations

- Smooth curve behavior with simplified bridge model
- Bridge model is too ideal to describe reality
- Resonant effects with probe simulation







# PB Measurement Results

 Probes from different manufacturers show different shape and deviations at the peak, due to the different probe geometries as predicted in simulations.





### Keep the sensitive regions of the probe shadow free of structures to avoid probe coupling to neighboring structure!!!





### Influence of Surface Waves

## PIB Parasitic Effects : Influence of Surface Waves (I)



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# PB Influence of Chuck Permittivity



Guideline 1: Use a thin substrate or one with low dielectric constant → Shifts the cut-off frequency of the surface waves upwards

### Or

Guideline 2: Use a chuck with the same dielectric constant as the wafer or above

$$\mathcal{E}_{r,chuck} \sim \mathcal{E}_{r,substrate} \text{ Or } \mathcal{E}_{r,chuck} > \mathcal{E}_{r,substrate}$$



### Validation of the Design Guideline for Surface Waves by Measurements

# PB Design Guidelines for Chuck Boundary Conditions

$$\mathcal{E}_{r,chuck} \sim \mathcal{E}_{r,substrate} Or \mathcal{E}_{r,chuck} > \mathcal{E}_{r,substrate}$$

 Substrate $\epsilon_{r,substrate}$	
Ceramic <b>E</b> <sub>r,chuck</sub>	

M. Spirito, U. Arz, G. N. Phung, F. J. Schmückle, W. Heinrich, and R. Lozar, "Guidelines for the Design of Calibration Substrates, including the Suppression of Parasitic Modes for Frequencies up to and including 325 GHz," EMPIR 14IND02 – PlanarCal, 2018, Physikalisch-Technische Bundesanstalt (PTB), 2018.

Fused silica substrate  $\varepsilon_r = 3.78$ 

Ceramic chuck  $\varepsilon_r = 6 \dots 7$ 

### No surface wave expected

Alumina substrate ε<sub>r</sub> = 9.7

Ceramic chuck  $\varepsilon_r = 6 \dots 7$ 

#### Surface wave expected

# PB Impact of Surface Waves

- Selected DUT I = 11400 µm to avoid probe coupling btw. the needles
- Fused silica: smoother transmission behavior
- Alumina substrate: more peculiarities and a strong resonance near 160 GHz.



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# PB Impact of Surface Waves

Fused silica substrate  $\varepsilon_r = 3.78$ 

Alumina substrate ε<sub>r</sub> = 9.7



**Less parasitics** 

- Excitation of surface waves
- Coupling to neighboring lines
- and excitation of the slotline modes there

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# PB Impact of Surface Waves

→ Smooth curve behavior for fused silica case



Verification of the guideline for surface waves



### **Summary and Conclusion**

# **PB** Summary of Guidelines

- Guideline: Influence of Microwave Probes
  - Keep the sensitive regions under the probe free of structures



# PB Summary of Guidelines

#### Guideline: Influence of Surface Waves

- Use a thin wafer or one with low dielectric constant to shift the cut-off frequency of the surface waves upwards
- Use a chuck with the same dielectric constant as the wafer or above

 $\varepsilon_{r,chuck} \sim \varepsilon_{r,substrate} \text{ Or } \varepsilon_{r,chuck} > \varepsilon_{r,substrate}$ 



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### Nano-robotic on-wafer probe station under scanning electron microscope

Kamel HADDADI



#### Microwave measurements for planar circuits and components

Short Name: PlanarCal, Project Number: 14IND02



Circuit Board - Space

http://planarcal.ptb.de/

#### PARTICIPATING EURAMET NMIs

METAS (Switzerland) **C** METAS

NPL (United Kingdom)

PTB (Germany)



VSL (Netherlands)



#### JRP SRT- EMPIR PlanarCal 2015-2018



The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

This project will **enable the traceable measurement and characterisation** of integrated circuits and components from radio-frequency to sub-mm frequencies with known measurement uncertainties.

- On-wafer up to 110 GHz
- University Lille IEMN focused on extension to nanoscale dimensions up to 30 GHz (tapered CPW TL vs IEMN set-up)
- Forschungsverbund Berlin e.V. (Germany)
- Fraunhofer-Gesellschaft zur Foerderung der angewandten Forschung e.V. (Germany)
- Friedrich-Alexander-Universität Erlangen Nürnberg (Germany)
- RF360 Europe GmbH (Germany)
- ROHDE & SCHWARZ GmbH & Co.
   Kommanditgesellschaft (Germany)
- o Technische Universiteit Delft (Netherlands)

### **On-wafer measurement : TEM guided approach**



$$a_{i} = \frac{\sqrt{\text{Re}(Z_{\text{ref}}^{i})}}{2|Z_{\text{ref}}|} (V_{i} + Z_{\text{ref}}^{i}I_{i})$$

$$\mathbf{b}_{i} = \frac{\sqrt{\text{Re}(\mathbf{Z}_{\text{ref}}^{i})}}{2|\mathbf{Z}_{\text{ref}}|} (\mathbf{V}_{i} - \mathbf{Z}_{\text{ref}}^{i}\mathbf{I}_{i})$$

$$\begin{bmatrix} b_1 \\ b_2 \end{bmatrix} = \begin{bmatrix} S_{11} & S_{12} \\ S_{21} & S_{22} \end{bmatrix} \cdot \begin{bmatrix} a_1 \\ a_2 \end{bmatrix}$$

- o 1.1 THz
- $\circ$  Contact pads = 30 x 30  $\mu$ m<sup>2</sup>
- $\circ$  Pitch = 25  $\mu$ m
- $\circ~$  Micro-Positioning 1µm /rot. 0.5°
- $\circ$  Optical vizualization

Marks, Roger B., and Dylan F. Williams. "A general waveguide circuit theory." *J. Research of the NIST* 97.5 (1992)

# **Objective/Challenge**

Determine the calibrated (and guided) S-parameters up to 30 GHz of CPW micro- and nanodevices with pads contacts smaller than 5 µm<sup>2</sup>

#### CONVENTIONAL



GSG probe Contact pad area  $= 30 \times 30 \mu m^2$ Pitch > 25  $\mu$ m



Robotic nanopositionner

Pitch =  $2.5 \,\mu m$ 

**GSG** Micro-probe

10nm /rot. ~  $1\mu^{\circ}$ 



Micropositionner 1µm /rot. 0,5°

**Optical visualisation** 



Scanning electron microscope 10nm









### Robotic Microwave On-Wafer probe Station in a Scanning Electron Microscope



### **MEMS probe:** Design and Fabrication

- CPW technology
- Die ~1 x 1 mm²
- $\circ$  400  $\mu m$  SOI process
- o 500 nm of gold metallization
- $\circ$  Thickness of the cantilever = 20  $\mu$ m
- $\circ$  Central line width = 2  $\mu$ m
- $\circ$  Gap width = 2.5  $\mu$ m
- $\circ$  Characteristic impedance = 50  $\Omega$

#### 1<sup>st</sup> generation of MEMS probes









J. Micromech. Microeng. 25 , 7 (2015) 075024

### **GSG probes & Calibration kit**

#### **Miniaturized Probe**

- GSG (Ground-Signal-Ground) topology
- MEMS (Microelectromechanical system)
- o Substrate (SOI HR, 400µm)
- o Different contacting material / size / shape





#### **Cal Kit characteristics**

- Substrate: GaAs (380µm)
- Metallization: Gold (500 nm)
- Resistive layer: NiCr(20nm)



### Integration up to 20 GHz



LEAF EquipEX





Magnitude and phase-shift measurement uncertainties of the reflection-coefficient  $\Gamma$  as a function of the magnitude of  $\Gamma$ 

### **Set-up vector calibration**

#### **Calibration Procedure**

- VNA Keysight<sup>TM</sup> N5245A PNA-X
- P<sub>RF</sub> = -10 dBm
- IFBW = 100 Hz

#### μ-Prober-in-SEM



н

(2) On-wafer probe tips ref. plans: One-Port SOL calibration

### **Calibrated Measurements**



#### Accurate on-wafer characterization of complex impedances

### **Application to 1D InAs nanodevice measurement**



Daffe, K., Marzouk, J., El Fellahi, A., Xu, T., Boyaval, C., Eliet, S., Grandidier, B., Arscott, S., Dambrine, G. and Haddadi, K., 2017, October. Nano-probing station incorporating MEMS probes for 1D device RF onwafer characterization. In *2017 47th European Microwave Conference (EuMC)* (pp. 831-834). IEEE.

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# 1 – Calibration and test tapered CPW structures design, fab & RF test





Fig. 1. Open-circuit calibration standard included in the fabricated wafer. The dimensions shown were selected to achieve a  $50-\Omega$  characteristic impedance across the entire structure.

Parameters	4 µm gap Meas. [nom]
Wi(μm) W(μm) D(μm) H(μm) Ws(μm) Wr(μm) Lr(nm)	102 [100] 299 [300] 65 [66] 1.7 [1.8] 2.4 [2.3] 3.9 [4] 110 [100]
	_

Fig. 2. Layout of the fabricated 3 inch wafer including all the access structures and calibration standards.















#### 2 – Silicon nanowires fabrication

Silicon nanowires (SiNWs) used in this work were prepared using the vapor-liquid-solid (VLS) mechanism.

The fundamental process is based on metal-catalyst-directed chemical vapor deposition of silicon. First, a thin film of gold (4 nm thick) was evaporated on the clean Si substrate. Gold nanoparticles with a wide size distribution were obtained as a result of metal dewetting on the surface.

Exposure of the gold-coated surface to silane gas at a pressure of 0.4 T with 40 sccm at 500  $^\circ$ C during 20 min in a dedicated furnace, led to SiNWs growth. The nanowires are terminated by

gold nanoparticles according to the VLS mechanism.



#### 3 – Handling, nanomanipulation and soldering under scanning electron microscopy





















#### 3 – Handling, nanomanipulation and soldering under scanning electron microscopy



View field: 15.00 µm Det: SE 2 µm AMIR / ANH SEM MAG: 14.44 kx Date(m/d/y): 03/05/18











View field: 6.53 mm SEM MAG: 33 x





B







SEM HV: 5.00 kV WD: 8.986 mm View field: 22.75 µm Det: SE

SEM MAG: 9.53 kx Date(m/d/y): 03/06/18



5 µm



AMIR / ANH









#### 3 – DC measurements (empty & loaded CPW structures)



3 – DC measurements (empty & loaded CPW structures)



**Process reproducibility : 2 test structures loaded with NWs** 



#### 3 – RF measurements (empty & loaded CPW structures)



**O**METAS

### **THANK YOU**











European Union European Regional Development Fund





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### **TEMMT-Workshop:** WP 3 - Calibration of RF power at D-Band

Gia Ngoc Phung, Karsten Kuhlmann, Jürgen Rühaak, AG 2.22

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- Motivation: RF power
- Measurement principles
  - Direct comparison
  - Microcalorimeter
- Measurement Results at D-Band
- Summary and Conclusion


### **Motivation**

### PTB Motivation

#### **RF** derived quantities

- Scattering parameters
- Calibration of power sensors
- Antenna gain and factor
- Feld strength
- Power density
- Specific absorption rate
- Channel characterization for Broadband communication up to the THz-range
- On-wafer-measurements
- Dielectric materials properties



# **PBRF Power Measurement**

### • Usually two requirements:

- Power sensor (detector)
- Power meter (base unit)





### **Calibration Method**

### **PTB** Microcalorimeter (I)





Example R900

- Symmetrical set-up
- Rect. waveguide or coaxial
- High thermal stability
- Large thermal time constant
- Nominal power: 1 to 10 mW

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18.07.2021

# **PB** Microcalorimeter (II)





### **PB** Measurement Quantities

Effective efficiency

$$\eta_{eff} = \frac{P_{ind}}{P_{RF,abs}} = \frac{P_{DC,sub}}{P_{RF,abs}}$$

Thermistor

Calibration factor

$$\eta_{cal} = \frac{P_{ind}}{P_{inc}} = (1 - |\Gamma|^2) \eta_{eff}$$

### PB Direct Comparison Method

#### Sensor calibration by direct comparison method



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# PB Uncertainty in the Frequency Bands

Frequency ranges	Microcalorimeter	Direct comparison
10 MHz - 8 GHz	0.002 (coaxial)	0.003 - 0.004 (coaxial)
8 GHz - 18 GHz	0.003 (coaxial)	0.005 - 0.007 (coaxial)
18 GHz - 26.5 GHz	0.002 (waveguide)	0.013 (coaxial) 0.007 (waveguide)
26.5 GHz - 40 GHz	0.0032 (waveguide)	0.018 – 0.020 (coaxial) 0.009 (waveguide)
33 GHz - 50 GHz	0.008 - 0.016 (waveguide)	0.015 - 0.018 (coaxial) 0.012 (waveguide)
50 GHz - 75 GHz	0.012 - 0.019 (waveguide)	0.016 (waveguide)
75 GHz - 110 GHz	0.016 - 0.026 (waveguide)	0.020 (waveguide)
Measurement time:		· · · · · · · · · · · · · · · · · · ·

Microcalorimeter: appr. 18 h per frequency point Direct comparison: appr. 2 h per frequency point Longer measurement time but better uncertainty



#### **Measurement Results at D-Band**

### PB Current Status of Power Sensors

- Current Status :
  - Use of thermistors for calibration in general
  - Commercial thermistor sensors (Hewlett Packard, Hughes, Millitech) from the 1980s:
    - poor input match
    - Iimited bandwidth
    - leakage
    - scarce
  - Development of novel power transfer started at Rohde & Schwarz (R&S) supported by PTB around 2010



# PB Generalized Efficiency

- Due to lack of thermistor sensors at D-Band
- $\rightarrow$  Development of a sensor is required
- →1st prototype of thermoelectric D-band power transfer standard designed, manufactured and characterized

- Characterization of thermoelectric power sensors
- →Introduction of generalized efficiency

# PIB R1400 Waveguide Calorimeter

R1400 waveguide calorimeter



Version: 1.02I\_DVM vom 6.12.2012 Messprogramm 102I\_NV3.exe Anfang: 2018-11-25, 20:24:20 Einsatz: Kal2\_R1400



Kal2 R1400

# PBR&S Thermoelectric Power Sensors



# PB Connection of the R&S Sensor





### PB Generalized Sensor Efficiency (I)

- Generalized Sensor Efficiency :
  - Figure of merit used to characterize the RF or thermal performance of thermoelectric power sensors
  - Based on four steps
    - Linearity Measurement of Thermopile
    - Multiple Offset Short Measurements
    - Averaging P<sub>avg</sub> from the Short Measurements
    - Calculation of Generalized Sensor Efficiency

Linearity Measurement of Thermopile

Multiple Offset Short Measurements

Averaging Pavg from the Short Measurements

### PB Generalized Sensor Efficiency (II)

- Linearity Measurement of Thermopile
  - Measurement of DC Voltage
  - Measurement of DC thermal voltage
  - Measurement of DC resistance

$$V_{DC} = v_0 + v_2 - v_1$$

$$e_{DC} = e_0 + e_2 - e_1$$

 $P_{dc} = \frac{V_{dc}^2}{2}$ 

 $\underline{P_{dc}}$ 

Heating coefficient

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Linearity Measurement of Thermopile

Multiple Offset Short Measurements

Averaging Pavg from the Short Measurements

(3) Generalized Sensor Efficiency

(1)

(2)

(4)

### PB Multiple Offset Short Measurements (I)



Linearity Measurement of Thermopile

Multiple Offset Short Measurements

Averaging Pavg from the Short Measurements

### PB Principle of Generalized Sensor Efficiency (I)

The generalized efficiency

$$\eta_{gen}(f) := \left. \frac{P_{dc}}{P_{RF,abs}(f)} \right|_{V_{th,1}=V_{th,2}}$$

$$p_{cor} = \frac{Q \cdot (1 - \Gamma^2)}{1 - \frac{Q \cdot (1 + \Gamma^2) \cdot k_2 \cdot V_{th1}}{m \cdot e_1}};$$
$$Q = \frac{P_{AVWG}}{2 \cdot P_{dc}};$$
$$\eta_{gen}(f) = \left(1 + p_{cor} \frac{1 + |\Gamma|^2}{1 - |\Gamma|^2}\right) \cdot \frac{k_2}{m} \cdot \frac{V_{th,1}}{e_1}$$

Linearity Measurement of Thermopile

Multiple Offset Short Measurements

Averaging Pavg from the Short Measurements

### PTB Principle of Generalized Sensor Efficiency (II)



Linearity Measurement of Thermopile

Multiple Offset Short Measurements

Averaging Pavg from the Short Measurements

### PB Principle of Generalized Sensor Efficiency (II)





### **Direct Comparison Method**

# PB Measurement setup R&S sensors



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### PB Measurement of R&S Thermoelectric Sensors



### PIB Raw Measurement Data





# PB Direct Comparison Results





$$\eta_{cal,X} = \eta_{gen} \cdot \frac{P_{N,Ref}}{P_{x,Ref}} \cdot \frac{U_{TS,X}^2}{U_{TS,N}^2} \cdot \frac{R_{TS,N}}{R_{TS,X}} \cdot \frac{\left(1 - \left|\Gamma_{TS,N}\right|^2\right)}{\left(1 - \left|\Gamma_{TS,X}\right|^2\right)} \cdot \frac{\left|1 - \Gamma_G \cdot \Gamma_X\right|^2}{\left|1 - \Gamma_G \cdot \Gamma_N\right|^2}$$

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National Metrology Institute



### **Summary and Conclusion**

# PB Summary and Conclusion

- State-of-the-art of RF power traceability:
  - So far, direct comparison and microcalorimeter measurement for thermistors and thermoelectric power sensors established up to W-Band
- New:
  - Waveguide calorimeter developed, manufactured and characterized up to 170 GHz
  - Direct comparison method at D-Band established for RF power calibration
  - Novel thermoelectric transfer standard, 1st prototype available
  - First results demonstrated reasonable
  - On-going work:
    - Establishing RF power traceability at D-Band
    - Extension to higher frequency range up to G-Band (140 GHz 220 GHz) and J-Band (220 GHz - 325 GHz)



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### **Design of D-Band Thin-Film Power Sensor**

### Milan Salek, Yi Wang

Emerging Device Technology Research Group, Department of Electronic, Electrical and Systems Engineering The University of Birmingham

21 July 2021







### Part 1

- Introduction to our approach
- Matched load design
- Sensor prototype
- Measurements
- Summary
- Part 2
  - D-band power measurement using the BHAM sensor (Murat Celep, NPL)



### Power sensor structures suitable for sub-mm-Waves





#### Thin film bolometer

T. Inoue, I. Yokoshima, and M. Sasaki, "High-Performance Thin-Film Barretter Mount For Power Measurement In W-Band," *Electron. Lett.*, vol. 21, no. 5, pp. 170–172, 1985.



#### Thermoelectric sensor

R. H. Judaschke, K. Kuhlmann, T. M. Reichel, and W. Perndl, "Millimeter-Wave Thermoelectric Power Transfer Standard," *IEEE Trans. Instrum. Meas.*, vol. 64, no. 12, pp. 3444–3450, 2015.



#### Matched load

N. Erickson, "A fast and sensitive submillimeter waveguide power meter", 10<sup>th</sup> Int. Symp. Space Terahertz Technology, March 1999, pp. 501-507



### Our approach



#### Power sensor specification

- Frequency range of 110 170 GHz
- Operating resistance of thin film ~200 Ω

#### Approach

- Matched load to absorb RF power;
- Low-resistivity silicon substrate (RF absorber + ease of fabrication);
- Platinum line for sensing.

### Main challenge

- Impedance matching (bandwidth, sensitivity to materials and fabrication tolerance)
- Sensor chip fabrication
- Thermal insulation




## Matched load design

-10

-20

.30

-50

-60 110

S11 (dB)



160

170

S-Parameters Vs Frequency [Magnitude in dB]

140

150

#### **Conductivity of Si substrate**

- S11 is under -28 dB when the electrical conductivity of silicon is  $10 \sim 100$  S/m (1  $-10 \ \Omega \cdot cm$ ).
- Substrate thickness (100 600 um) has little effect on the matching.

#### Angle of the sensor chip

When the angle is below 20°,  $S_{11}$  is below -20 dB across the D-band.



120

130

Thin-film sensor chip



- Silicon substrate: ~ 200 μm
- Silicon dioxide (DC isolate silicon from platinum): ~ 1 μm
- Pt thin film: ~ 200 nm
- The sensor chip is  $10.4 \text{ mm} \times 5.65 \text{ mm}$ .
- The meander thin-film line has a length of 30.5 mm and width of 80 µm.
- Each square gold pad has a side length of 2 mm.









- Electrical interconnection with DC: through spring loaded pin connectors.
- Housing: CNC machined from C109 tellurium copper
- Chip: fabricated at PTB; diced by a femtosecond laser cutter. DC resistance = 208.4  $\Omega$  (A) and 201.3  $\Omega$  (B).











38 chips per 3 inch wafer









## Thermal simulation



#### Assumption

- The RF power of 2 mW peak was used for thermal simulations.
- The ambient temperature is set at 23 °C.





**RF** measurements





The measured return losses of both power sensors are consistent and over 15 dB across the band, lower than the 28 dB prediction from simulation.





- The response time is slow.
- The sensor response is significantly ۲ influenced by the environment.

Thermal insulation is not effective and reference temperature sensing is required!



0.6000

Switch on

20 min

and off every

Time

## **D-band power measurement using the BHAM microwave sensor**



#### **Murat Celep**

BHAM microwave sensor was attached to the D-band micro-calorimeter system.

The system has been used to measure absolute microwave power.





#### **Calorimeter characterization**



BHAM microwave sensor was connected to the micro-calorimeter and thermopile was characterized with BHAM sensor using DC known power (thermopile/BHAM sensor coefficient).





#### **Calorimeter characterization**



S-parameters of the calorimeter were measured for the 3-ports.

$$\begin{bmatrix} S \end{bmatrix} = \begin{bmatrix} S_{11} & S_{12} & S_{13} \\ S_{21} & S_{22} & S_{23} \\ S_{31} & S_{32} & S_{33} \end{bmatrix}$$



#### **Microwave power measurement**



DUT power meter (Erickson PM5) was attached to the reference port.

Microwave power was applied to the calorimeter port 1.

Heating effect of the dissipated microwave power on the BHAM sensor was measured through thermopile output voltage.

Output of the DUT power meter ( $P_{DUT}$ ) was measured at the same time.



#### **Microwave power measurement**





#### **DUT effective efficiency**



#### Effective efficiency of the DUT was calculated using below equation.

$$EE = \frac{P_{DUT}}{P_{mwd}} \frac{|S_{21}|^2}{|S_{31}|^2} \frac{1 - |\Gamma_{STD}|^2}{1 - |\Gamma_{DUT}|^2} \frac{|1 - \Gamma_3 \Gamma_{DUT}|^2}{|1 - \Gamma_2 \Gamma_{STD}|^2}$$





## THz broadband spectroscopy: instrumentation and performance

Mira Naftaly



### **Dielectric properties**, quantities and units

Technologies for broadband dielectric measurements at THz and sub-THz frequencies

Low-loss materials at THz and sub-THz frequencies



## **Dielectric properties, quantities** and units

"Dielectric" quantities

- Complex permittivity:
- Loss factor or tan-delta:
- "Spectroscopic" quantities
- Absorption coefficient:
- Extinction:
- Refractive index:

 $\varepsilon' + i\varepsilon''$  $\tan \delta = \frac{\varepsilon''}{\varepsilon'}$ 

## **Conversion between quantities**



$$k = \frac{c}{4\pi f} \alpha$$

$$\varepsilon' + i\varepsilon'' = (n + ik)^2 = (n^2 - k^2) + i(2nk)$$

$$n = \sqrt{\varepsilon' + k^2} = \sqrt{\frac{1}{2} \left[\varepsilon' + (\varepsilon'^2 + \varepsilon''^2)^{1/2}\right]}$$
$$k = \frac{\varepsilon''}{2n}$$

$$\tan \delta = \frac{\varepsilon''}{\varepsilon'} = \frac{2nk}{n^2 - k^2}$$



## Frequency and wavelength unit conversion

Frequency	Wavelength	Wavenumber	Energy
(THz)	(μ <b>m</b> )	(cm <sup>-1</sup> )	(meV)
ν	$\lambda = c/v$	$\sigma = v/c$	$eV = h_V c / 10^8$
1	299.8	33.35	4.136
299.8	1	10000	1240
0.02998	10000	1	0.1240
0.2418	1240	8.065	1



Dielectric properties, quantities and units

Technologies for broadband dielectric measurements at THz and sub-THz frequencies

Low-loss materials at THz and sub-THz frequencies





- Time-domain spectroscopy
- Frequency-domain spectroscopy
- VNA-based spectroscopy
- Fourier transform spectroscopy

## **THz spectrometer instruments**



### **Closed-loop**

- TDS Time-domain spectrometer (pulsed)
- FDS Frequency-domain spectrometer (CW)
- VNA Vector network analyser (CW)
- Coherent detection measures field amplitude and phase

### **Open-loop**

- FTS Fourier transform spectrometer (CW)
- Scanning spectrometer any combination of a tunable source and a broadband detector
- Incoherent detection measures field intensity

### **Coherent** systems

strongly dominate broadband terahertz measurements

## Open-loop and closed-loop systems



An open loop system consists of:

- an emitter and a detector which operate independently;
- optics to guide radiation from emitter to detector.



A closed loop system consists of:

- an emitter and a detector which are activated by the same source;
- optics to guide radiation from emitter to detector.



## **Time-domain spectrometer (TDS)**



TDS is the dominant device for broadband THz measurements – accounting for >90% of published results.



### **TDS performance**

- Broadband operation
- One-shot spectral acquisition
- Large bandwidth:
  - 5-6 THz as standard
  - up to 20 THz is possible
- Frequency resolution 1-10 GHz

#### TDS components:

- Pump laser femtosecond pulsed
- Differential variable delay
- THz emitter photoconductive antenna (most common)
- THz detector photoconductive antenna (most common)
- THz beam guiding optics

## Photoconductive THz emitters and detectors



**Emitted beam is polarized Detection is polarization sensitive** 



## **TDS operation**



Uses a <u>single-cycle THz pulse</u>

Data is acquired in time domain

by scanning the *probe pulse* over the *THz pulse* using *variable time-delay*.



## **Spectral data from TDS**



#### Amplitude and phase spectra obtained via Fourier Transform.



## **Parameter extraction in TDS**



#### Most TDS measurements are performed to obtain $n \& \alpha$ !

#### Calculating **<u>refractive index</u>** and **<u>absorption coefficient</u> of material from TDS data:**

Field amplitude: Phase: Refractive index: Absorption coefficient: Sample thickness:

$$E_{ref} \& E_{sample}$$

$$\phi_{ref} \& \phi_{sample}$$

$$n$$

$$\alpha [L^{-1}]$$

$$d [L]$$

$$n(\omega) = 1 + \frac{(\phi_{ref} - \phi_{sample})c}{2\pi f d} \qquad (1)$$

$$T(\omega) = 1 - \frac{(n-1)^2 + k^2}{(n+1)^2 + k^2} \qquad (2)$$

$$k(\omega) = \frac{\alpha c}{2\pi f} \qquad (3)$$

$$\alpha(\omega) = -\frac{2}{d} \ln \left(T \frac{E_{sample}}{E_{ref}}\right) \qquad (4)$$

Note: when k is non-negligible, Eqs. 2-4 must be calculated iteratively.

## **Example: lactose monohydrate**





## Frequency-domain spectrometer (FDS)



FDS has a narrower measurement bandwidth than TDS, but has the advantage of much higher frequency resolution.



#### **FDS performance**

- Broadband operation
- Frequency scanning
- Bandwidth: up to <u>3 THz</u>
- Frequency resolution <50 MHz

#### FDS components:

- Two stabilised CW lasers with offset wavelengths
  - THz is generated as the difference frequency
- THz emitter photoconductive mixer
- THz detector photoconductive mixer
- THz beam guiding optics

## Example: whispering-gallerymode resonance



Phase-sensitive (coherent) detection gives rise to **phase "fringes"** (these are not standing waves!) Therefore an **envelope** function must be applied to the data.



#### (Figure courtesy of Dominik Vogt, University of Auckland, New Zealand)

## **VNA-based FDS**



VNA-based spectrometers have a narrower measurement bandwidth than TDS or FDS, but higher frequency resolution.



extenders with horns

#### Components:

- VNA with frequency extenders
- Horn antennas or other optics
- All-electronic

#### More information in other talks!

#### **VNA performance**

- Frequency scanning
- Bandwidth: up to <u>1.7 THz</u>
- Frequency resolution <0.1 MHz



MCK by Swissto12

# Fourier Transform Spectrometer (FTS)





FTS components:

- Broadband source (e.g. Hg lamp)
- Broadband power detector
- Optics
- Precision scanning mechanism

#### **FTS performance**

- Broadband operation
- Single-scan full-spectrum
- Bandwidth:
  - 1-180 THz standard
  - 0.05-840 THz available

National Physical Laboratory

- Frequency resolution
  - 1 GHz standard
  - <0.1 GHz available</li>

## **FTS operation**



#### Data is acquired as an interferogram



- Oscillations are etalon fringes due to standing waves in the sample.
- Fringes disappear when the sample has strong absorption
- Fringes are necessary for extracting refractive index and absorption



## **Parameter extraction in FTS**



Step 1: *n* is extracted from the fringe spacing:

 $\Delta f = c/2nd$  (ideal case)

Step 2:  $\alpha$  is extracted from the etalon transmission function:

$$T(f) = I_T(f)/I_0(f) = \frac{1}{\mathcal{M} + \mathcal{F} \sin^2 \beta d}$$
$$\mathcal{F}(f) = \frac{4R}{(1-R)^2}$$
$$\mathcal{M}(f) = \frac{\left(1 - Re^{-2\alpha d}\right)^2}{(1-R)^2 e^{-2\alpha d}} > 1$$
$$R(f) = \frac{(n-1)^2}{(n+1)^2}$$
$$\beta = 2\pi f n/c$$

#### Note: extracting n from fringe spacing is <u>non-trivial</u>!



Parameter extraction in FTS is <u>not straightforward</u>, with many potential sources of error. Samples must be sufficiently thin and low-loss to produce fringes.

## Comparative advantages – a personal view



**FDS** 

**FTS** 

**VNA** 

TDS

#### Criteria

#### Science

- Bandwidth (FTS)
- Frequency resolution (VNA)
- SNR & dynamic range (TDS, VNA)
- Unambiguous parameter extraction (TDS)
- Accuracy & precision

#### Industrial

- Speed of measurement
- Ease of measurement
- Repeatability
- Size of instrument
- Suitability for in-line applications
- Cost



Dielectric properties, quantities and units

Technologies for broadband dielectric measurements at THz and sub-THz frequencies

Low-loss materials at THz and sub-THz frequencies





### **Few materials are THz-transparent!**

- Inorganic crystals
- Non-polar polymers
# **Inorganic crystals**



- Carbon group crystals
  - Diamond
  - High resistivity silicon
  - High resistivity germanium
  - Hexagonal silicon carbide
- Oxides
  - Quartz
  - Sapphire
- Nitrides
  - Aluminium nitride
  - Gallium nitride
  - Silicon nitride



Frequency (THz)







#### Silicon Si High resistivity (undoped)





Crystal properties	Chemical formula	Si	
	Crystal type	Isotropic	
	Crystal system	Cubic	
		Fd3m	
Optical properties	Transparency (visible)	NO	
	Colour	Metallic grey	
	Birefringence	NO	
	Refractive index @ 1.55 µm	3.4777	
	Band gap eV	1.12	
Physical properties	Density g/cm <sup>3</sup>	2.329	
	Moh's hardness	6.5	





# Germanium Ge

#### High resistivity (undoped)





Crystal	Chemical formula	Ge
properties	Crystal type	Isotropic
	Crystal system	Cubic
		Fd3m
Optical	Transparency (visible)	NO
properties	Colour	Metallic grey
	Birefringence	NO
	Refractive index @ 2.8 µm	4.052
	Band gap eV	0.66
Physical	Density g/cm <sup>3</sup>	5.323
properties	Moh's hardness	6.0





# Hexagonal silicon carbide SiC



Crystal properties	Chemical formula	SiC
	Crystal type	Uniaxial
	Crystal system	Hexagonal
		$C_{6v}^4$ -P6 <sub>3</sub> mc
	Polytypes	4H-SIC; 6H-SIC
Optical properties	Transparency (visible)	YES
	Colour	Colourless
	Birefringence	YES
	Refractive index @ 590 nm	o – 2.56
		e – 2.60
	Band gap eV	3.23 (4H); 3.05 (6H)
Physical properties	Density g/cm <sup>3</sup>	3.21
	Moh's hardness	9.5



Tarekegne et al. Optics express 27 (2019): 3618-3628.









Crystal	Chemical formula	SiO <sub>2</sub>
properties	Crystal type	Uniaxial
	Crystal system	Trigonal
	Polytypes	$P3_12; P3_22$
Optical	Transparency (visible)	YES
properties	Colour	Colourless
	Birefringence	YES
	Refractive index @ 590 nm	o – 1.544
		e – 1.553
	Band gap eV	8.4
Physical	Density g/cm <sup>3</sup>	2.649
properties	Moh's hardness	7





# Sapphire Al<sub>2</sub>O<sub>3</sub>





Crystal properties	Chemical formula	Al <sub>2</sub> O <sub>3</sub>
	Crystal type	Uniaxial
	Crystal system	Trigonal
		R3c
Optical properties	Transparency (visible)	YES
	Colour	Colourless
	Birefringence	YES
	Refractive index @ 590 nm	o - 1.7680
		e - 1.7600
	Band gap eV	9.9
Physical properties	Density g/cm <sup>3</sup>	3.97
	Moh's hardness	9





# **THz-transparent crystals**



THz refractive index	Absorption @ 1 THz (cm <sup>-1</sup> )	Absorption @ 3 THz (cm <sup>-1</sup> )	Absorption @ 10 THz (cm <sup>-1</sup> )	Transparency in the visible
2.38	0.1	0.12	0.27	Yes
3.42	0.1	0.1	0.3	No
4.01	0.2	1.3	20	No
3.13	0.1	0.4	6	Yes
2.11	0.2	1.2	45	Yes
3.1	1.0	9	68	Yes
	THz refractive index 2.38 3.42 4.01 3.13 2.11 3.1	THzAbsorptionrefractive index@ 1 THz (cm <sup>-1</sup> )2.380.13.420.14.010.23.130.12.110.23.11.0	THzAbsorptionAbsorptionrefractive index@ 1 THz (cm <sup>-1</sup> )@ 3 THz (cm <sup>-1</sup> )2.380.10.123.420.10.123.420.10.14.010.21.33.130.10.42.110.21.23.11.09	THzAbsorptionAbsorptionAbsorptionrefractive index0 1 THz (cm <sup>-1</sup> )0 3 THz (cm <sup>-1</sup> )0 10 THz (cm <sup>-1</sup> )2.380.10.120.273.420.10.120.273.420.10.10.34.010.21.3203.130.10.462.110.21.2453.11.0968





How to recognise non-polar polymers?

#### Polymers containing only C and H (or F) atoms

#### **Polyethylene** Appearance: milky-white





High density polyethylene (HDPE) Low density polyethylene (LDPE) Linear low density polyethylene (LLDPE) High molecular weight polyethylene (HMWPE) Ultra high molecular weight polyethylene (UHMWPE)









# **Poly-methyl-pentene PMP (TPX)**



# **Cyclo-olefin copolymer COC**



Appearance: colourless & transparent







#### **Polystyrene** Appearance: colourless & transparent









# Polytetrafluoroethylene PTFE (Teflon)



Appearance: bright white







# Paraffin wax, jelly and liquid





- Alkanes whose formula is C<sub>2</sub>H<sub>2n+2</sub>.
- Wax has chains of 20-40 atoms; liquid has chains of 6-16 atoms; jelly is a mixture of longer and shorter chains.
- Wax and jelly are both partially crystalline, and appear translucent.
- Liquid paraffin is colourless and transparent.

Paraffins can be used as mounting or suspension media for a wide variety of materials and powders, and as optical contact media.

### Paraffin





# **THz-transparent polymers**



Polymer	THz refractive	Absorption @	Absorption @	Absorption @	Transparency
	index (mean)	1 THz (cm <sup>-1</sup> )	3 THz (cm <sup>-1</sup> )	10 THz (cm <sup>-1</sup> )	in the visible
LDPE	1.51	0.2	1.6	~2	No
HDPE	1.53	0.2	1.6	~3	No
PTFE	1.43	0.5	2.8	>50	No
COC	1.52-1.53	0.2	0.8	~2	Yes
PMP (TPX)	1.46	0.3	0.8	~2.5	Yes
PP	1.52	0.3	~1.5	~3.5	Yes
PS	1.58	1.5	2.5	~5	Yes
Paraffin liq.	1.47	0.5	1.7	NA	Yes
Paraffin wax	1.49	0.8	4.2	NA	No

Note: Polymers that are <u>transparent in the visible and at THz</u> have similar refractive indices in both regions ( $n_{visible} \cong n_{THz}$ ). This aids THz beam path alignment using visible light.



# Thank you





# Millimetre-wave characterisation of dielectric materials using a guided free-space technique

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1<sup>st</sup> TEMMT Training Course, 21-July-2021

## **Outline**

- Introduction
- Material Characterisation Kit (MCK)
  - Assessment at WR-15 band
  - New calibration methods
  - Data filtering techniques
- Conclusions

#### **Introduction – Permittivity and Permeability**

#### Permittivity

#### Permeability

"

$$\frac{\varepsilon}{\varepsilon_{0}} = \varepsilon_{r} = \varepsilon_{r}' - j\varepsilon_{r}''$$

interaction of a material in the presence of an external electric field.

$$\frac{\mu}{\mu_0} = \mu_r = \mu_r' - j\mu_r''$$

interaction of a material in the presence of an external magnetic field.

Loss tangent 
$$\tan \delta = \frac{\varepsilon_r}{\varepsilon_r}$$

[Shelley Begley: "Electromagnetic properties of materials"]

#### **Introduction – Typical Measurement Methods**



 TDS (Time-Domain Spectrometry) is another popular technique utilised for characterising material properties at THz frequencies

[Shelley Begley: "Electromagnetic properties of materials" & R N Clarke *et al*: "A guide to the characterisation of dielectric materials at RF and microwave frequencies" ]

#### **Material Characterisation Kit (MCK)**



[A Dimitriadis et al: "Dielectric measurements at mm-wave frequencies with the material characterization kit (MCK)"]

### **Material Characterisation Kit (MCK)**



- Effectively a *guided free-space* technique: MCKs are more compact and have less stringent requirement on alignment, compared to conventional free-space systems
- HE<sub>11</sub> hybrid mode (linear polarization): mode purity >98%

[A Dimitriadis et al: "Dielectric measurements at mm-wave frequencies with the material characterization kit (MCK)"]

#### **Material Characterisation Kit (MCK)**



[A Dimitriadis et al: "Dielectric measurements at mm-wave frequencies with the material characterization kit (MCK)"]

#### **Research into MCKs**

- NPL currently has 4 sets of MCKs, covering 50-75GHz, 75-110GHz, 140-220GHz, 500-750GHz
- Research focus:
  - Measurement uncertainties
  - Enhanced calibration techniques, e.g. TRL
  - Algorithms for extraction of complex permittivity from S-parameters



Calibration techniques recommended by manufacturer

Next, we will review 3 pieces of work undertaken by NPL

### MCK 1: Investigation at WR-15 (50-75GHz)



- S-parameters measured on PNA-X with VDI frequency extender heads
- Permittivity extracted from S-parameters using NIST precision model (only  $S_{21}$  used)
- Free-space calibration based on Short/Thru, together with time-gating. No need for prior waveguide calibration

Y. Wang, X. Shang, N. M. Ridler, T. Huang and W. Wu, "Characterization of Dielectric Materials at WR-15 Band (50–75 GHz) Using VNA-Based Technique," in IEEE Transactions on Instrumentation and Measurement, vol. 69, no. 7, pp. 4930-4939, July 2020

### MCK 1: Investigation at WR-15 (50-75GHz)



• Similar measurements performed for materials: Astra MT77, Rogers 3003, TPX, HDPE, Alumina, Silicon

Y. Wang, X. Shang, N. M. Ridler, T. Huang and W. Wu, "Characterization of Dielectric Materials at WR-15 Band (50–75 GHz) Using VNA-Based Technique," in IEEE Transactions on Instrumentation and Measurement, vol. 69, no. 7, pp. 4930-4939, July 2020

### MCK 1: Investigation at WR-15 (50-75GHz)

- Good agreement between measured results using MCK & published values in literature
- Preliminary estimates of the uncertainty obtained see the paper for details. Generally, systematic errors dominate the overall uncertainty, i.e. accuracy of S-parameters and sample thickness important

Samm1a	Thickness	ε'	٤'	Samula	Thickness	tan δ	tan δ
Sample	(mm)	(extracted)	(literature)	Sample	Sample (mm)		(literature)
PTFE	5 99	2 008	2.1 @ 12 GHz, [25]	PTFF	5.00	0.002	0.000 3 @ 12 GHz, [25]
1112	5.55	2.000	2.06 @ 92.5 GHz, [26]	11112	5.99		0.000 389 @ 92.5 GHz, [26]
Astra	1.60	2 988	3.0 @ 79 GHz, [19]	Astra	1.60	0.004	0.001 7 @ 79 GHz, [19]
MT77	1.00	2.900	3.0 @ 10 GHz, [27]	MT77	1.00	0.004	0.001 7 @ 10 GHz, [27]
Rogers	1 50	3 1 1 0	3.0 @ 60 GHz, [20]	Rogers	1.50	0.001	0.001 3 @ 60 GHz, [20]
3003	1.50	5.110	3 @ 10 GHz, [28]	3003	1.50	0.001	0.001 3 @ 10 GHz, [28]
TPX 2.81	2.099	2.13 @ 38 GHz, [29]	TDV	2 01	0.003	0.004 3 @ 38 GHz, [29]	
		2.136 @ 700 GHz, [30]	IFA	2.01		0.001 74 @ 700 GHz, [30]	
HDPE 5.97		2 301	2.3 @ 700 GHz, [30]	LIDDE	5.07	0.002	0.000 224 @ 700 GHz, [30]
11DTE 5.97	5.97	2.501	2.2505 @ 85 GHz, [31]	HDPE	5.97	0.002	0.000 225 217 @85 GHz, [31]
Alerecting 10.18		0.619	9.4 @ 2.5 THz, [33]	Alexanian	Alexandrea 10.10	0.001	0.021 @ 2.5 THz, [33]
Aluinina	10.18	9.018	9.424 @ 17 GHz, [32]	Alumina	10.18	0.001	0.000 31 @ 17 GHz, [32]
Silicon	3.06	3.06 11.678	11.7 @ 700 GHz, [30]	Giliaan	3.06	0.001	0.000 26 @ 700 GHz, [30]
			11.67 @ 1 THz, [33]	Silicon			0.000 4 @ 1 THz, [33]

Y. Wang, X. Shang, N. M. Ridler, T. Huang and W. Wu, "Characterization of Dielectric Materials at WR-15 Band (50–75 GHz) Using VNA-Based Technique," in IEEE Transactions on Instrumentation and Measurement, vol. 69, no. 7, pp. 4930-4939, July 2020



- Thru-Reflect-Line (TRL) calibration applied to MCK, to obtain robust S-parameters for calculation of permittivity and loss tangent
- Investigation undertaken at WR-5 (140-220GHz) and WM-380 (500-750GHz)
- TDS was used for benchmarking
- TRL calibration offers improvement in results

		WR-5	WM-380
	Mid-band frequency (GHz)	180	625
	Ridge diameter (mm)	8.200	5.000
Ridge Slot	Slot diameter (mm)	9.052	5.250
	$\lambda/4$ corrugated waveguide (mm)	0.422	0.120
nnàn/	$\lambda/4$ free-space (mm)	0.416	0.120
	Air gap length used (mm)	0.420	0.120

- Line standard can be either a metal waveguide or air gap, both work effectively
- Air gap is desirable as no additional standard required
- Time-gating was still applied, and the optimum gate width varies with samples and can be calculated



 TRL shows improvement in results for some difficult samples with relatively large dielectric constants. Good agreement between MCK (with TRL) and TDS results. See next 2 slides







- Data filtering technique (i.e. Savitzky–Golay filter) was applied to measured Sparameters, eliminating the need for time-gating
- Generally Savitzky-Golary filter results are consist with time-gating results, and the former works better for specimens with non-ideal quality
- MCK results were compared with open-resonator data, showing good agreement

D. Ma, X. Shang, N. M. Ridler and W. Wu, "Assessing the Impact of Data Filtering Techniques on Material Characterization at Millimeter-Wave Frequencies," in IEEE Transactions on Instrumentation and Measurement, vol. 70, pp. 1-4, 2021, Art no. 6005904


- Acetal co-polymer sample 2mm thickness
- Responses after MCK TRL calibration
- Time-gating was not applied

- Acetal co-polymer sample 2mm thickness
- Time-gating was applied

• Time-gating can effectively clean up the data



• For the same acetal co-polymer sample, Savitzky-Golay filter works as effectively as time-gating



Comparison carried out on glass sample (thickness 1.6mm), LDPE sample (thickness 6mm), quartz sample (thickness 4.1mm), and PTFE sample (thickness 12mm) too

#### TABLE I

#### Comparison of $\varepsilon'$ Values

Sample	MCK (Time-gating)	MCK (Savitzky-Golay filter)	Open-resonator
LDPE	$2.253\pm0.040$	$2.250\pm0.038$	$2.286 \pm 0.005$
Glass	$\boldsymbol{6.727 \pm 0.059}$	$6.718 \pm 0.044$	$\boldsymbol{6.70\pm0.04}$
Quartz	$4.378\pm0.037$	$4.374\pm0.041$	$4.433 \pm 0.013$

### TABLE II

#### COMPARISON OF tan $\delta$ VALUES

Sample	MCK (Time-gating)	MCK (Savitzky-Golay filter)	Open-resonator
LDPE	$0.002\pm0.013$	$0.002\pm0.013$	$0.00023 \pm 0.00001$
Glass	$0.023\pm0.014$	$0.024\pm0.015$	$0.0221 \pm 0.0012$
Quartz	$0.001\pm0.012$	$0.0010 \pm 0.013$	$0.000032 \pm 0.000012$

### Conclusions

- New guided free-space technique using Swissto12 MCKs was briefly described
- 3 pieces of recent work on assessing/improving MCK measurement method were summarised
- Other research work related to MCK, e.g. on extraction algorithms and new calibration methods, was also undertaken. See <u>TEMMT</u> <u>project website</u> – publication section
- Overall, MCK is a promising new technique enabling fast and easy material characterisations.

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