THz waves and meta-materials: Imaging and sensing

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Leuven Inc. 15.06.'10

ETRO

LAMI-ETRO contributions from

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dr. Irina Jaeger
dr. Saiful Islam
dr. Amna Elhawil
ir. Vladimir Matvejev
ir. Bin Zhu
NN

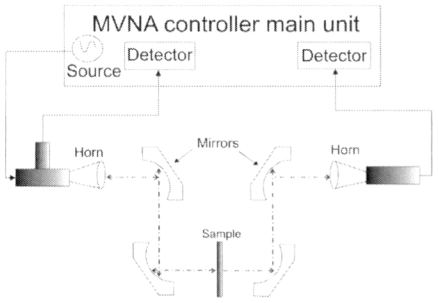
Prof. dr. Hichem Sahli (signal processing)

Measurement Infrastructure

Quasi-optical measurement setup

AB millimetre 8-350-2 (VNA):

- Frequency range: 40 to 660 GHz → 2000 GHz
- Quasi-Optical → on-wafer extension



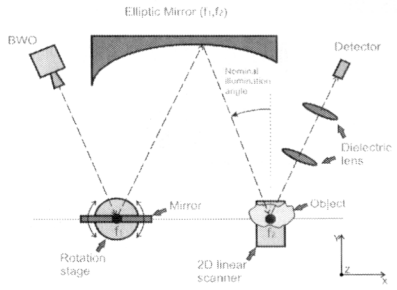
Characteristics: See datasheet ABMM

Antenna radiation & Imaging setups

Today in W-band (70-110 GHz) → extension to M-VNA

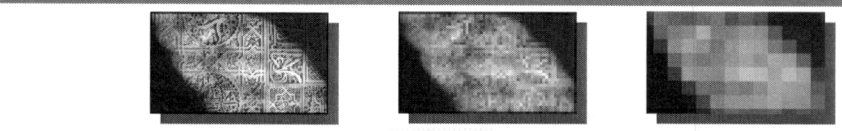
Antenna radiation patterns)

IMAGING



OUTLINE

THz-wave band





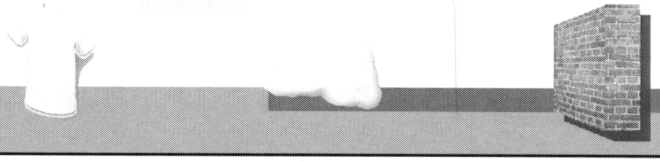
Higher Resolution



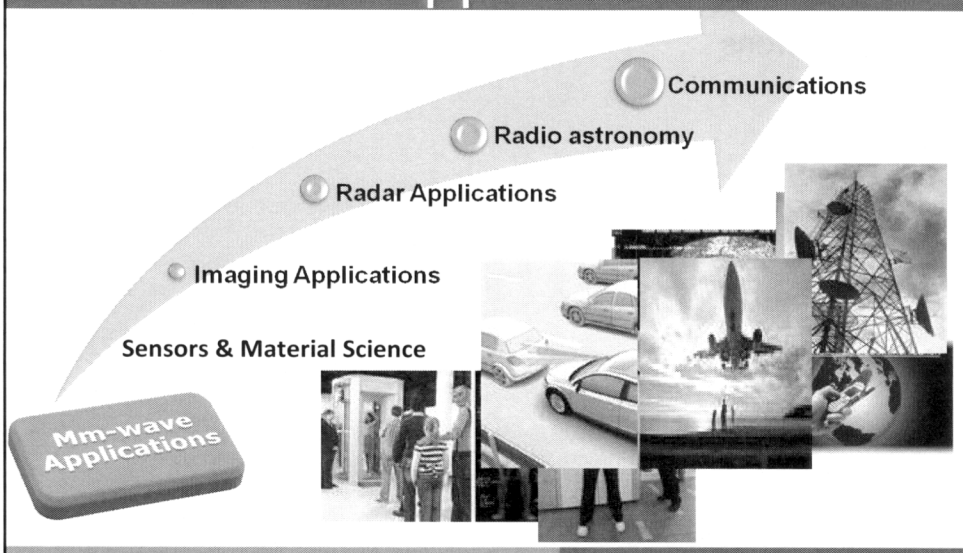
UV Visible Light IR THz Waves Microwaves FM Radio AM Radio

1 nm 500 nm 1 μm 1 mm 1 cm 10 cm 1 m 10 m 100 m

Larger Penetration



THz wave Applications



Active mm-wave system concept

74 – 110GHz.

10mW Power.

Material types

REFLECTORS TRANSPARENT ABSORBERS

Collaboration with department of physical chemistry and polymer science (FYSC-VUB)
***Prof.** Guy Van Assche & **ir.** Golzen Nicolaas-Alexander*

SBO-project: Active mmw imaging for concealed weapon detection

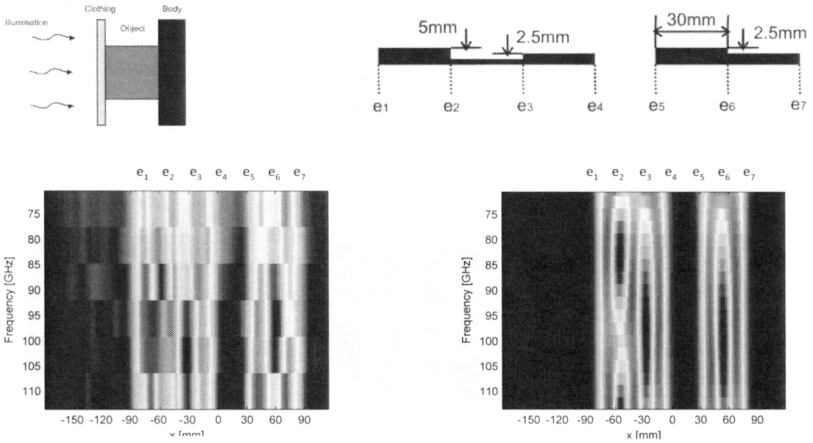
Vrije Universiteit Brussel

imec KATHOLIEKE UNIVERSITEIT LEUVEN UCL UNIVERSITEIT GENT

Most important results

SBO-IWT project activity April 2005-March 2010

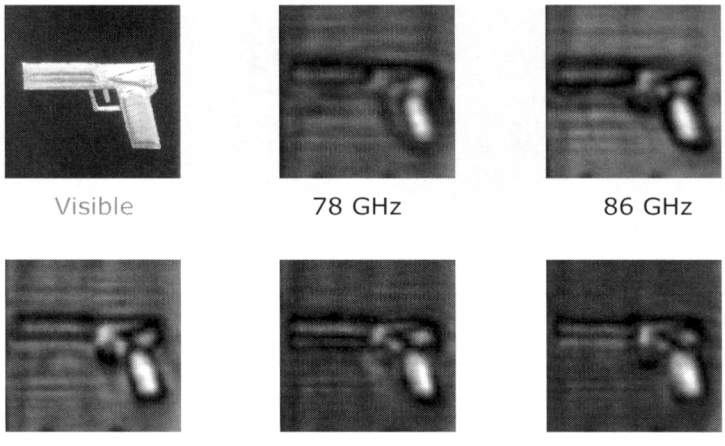
Multi-frequency illumination
--- Interference Patterns vs. Frequency



measurement

Scalar diffraction simulation

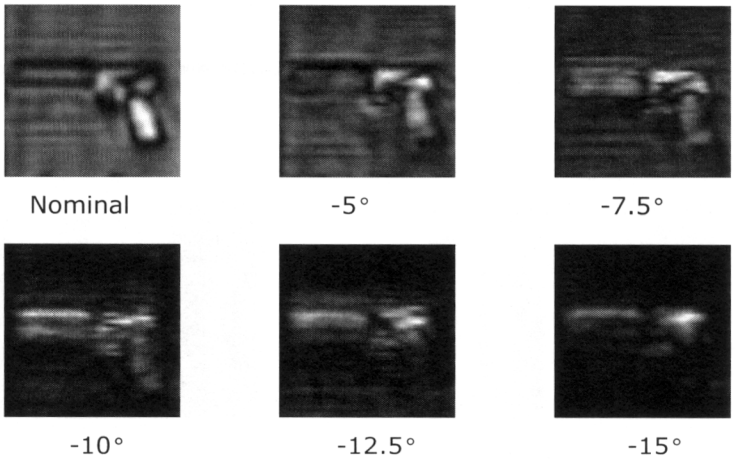
Multi-frequency illumination
--- Interference Patterns vs. Frequency



Visible | 78 GHz | 86 GHz

94 GHz | 102 GHz | 110 GHz

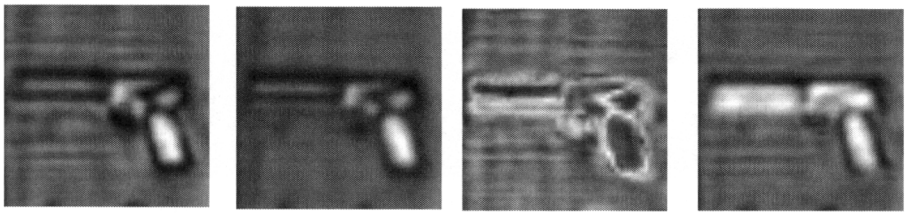
Multi-angle illumination
--- Interference Patterns vs. Angle



Nominal -5° -7.5°

-10° -12.5° -15°

Multi-frequency illumination
--- Interference Suppression

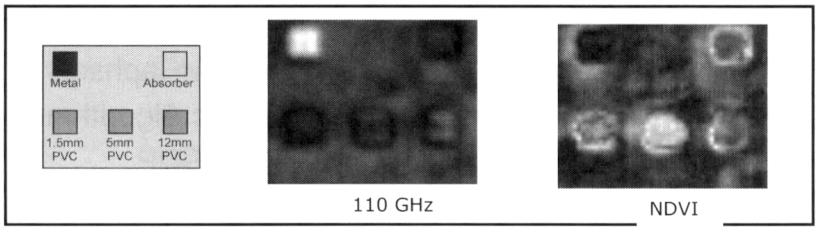


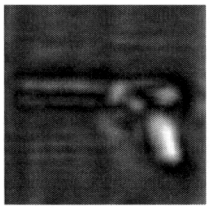
f =94 GHz Average(f) dfI Average(angle)

Averaging (f-images): interference noise partly suppressed
Averaging (angle-images) : interference noise substantially suppressed

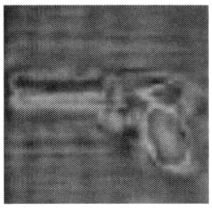
Differential frequency imaging ➔ small object feature extraction

Multi-frequency illumination
--- Contrast Enhancement

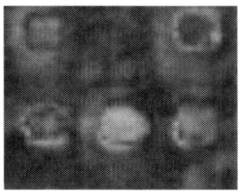




Single band



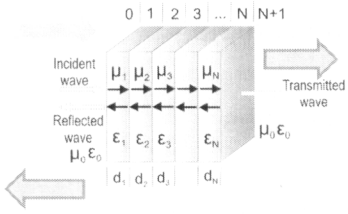
Interference suppression
Feature extraction

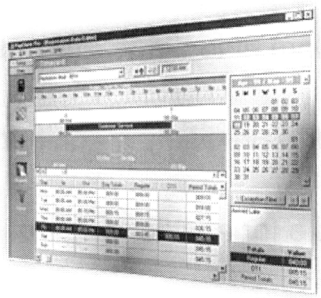


Interference suppression
Contrast enhancement

"Material characterization" = Dielectric Spectroscopy

(1) R, T vs freq → (2) Data Processing → (3) Results





Response of the material to
Electric field (ε)
and
Magnetic field (μ)

Extraction procedures for

- Single & Multi-layers
- Thick & Thin Films
- Meta-materials

- Nicholson-Ross-Weir
- Newton-Raphson
- Genetic Algorithms
- BFSG-SQP

Complex permeability (μ_r) & permittivity (ε_r)

Refractive index (n)	Conductivity (σ)	Resistivity (ρ)	Loss factor (tanδ)
$n = \sqrt{\mu_r \varepsilon_r}$	$\varepsilon_r = \varepsilon' + i\frac{\sigma}{\omega}$	$\rho = \frac{1}{\sigma}$	$\tan\delta = \frac{\varepsilon''}{\varepsilon'}$

Benefits: Quasi-Optical characterization

- Contact-less and non-destructive
- Less transmission losses
- No problem of leakage around the sample
- No need to precisely machined samples
- Applicable for a wide range of materials

Material types

Material Properties & THz range

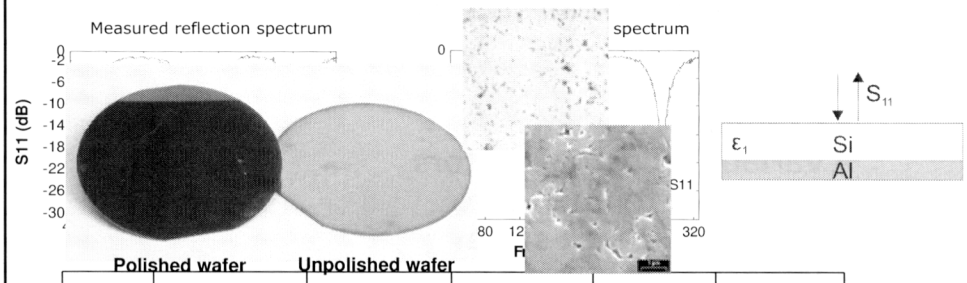
- Non-polar materials
  - Various industrial sectors
- Polar materials
  - Examples
  - Various industrial sectors
  - Impact of temperature
  - Impact of Phase state
  - Impact of under- or super-cooling
- Mixtures

Non-polar materials

- Solids from very diverse sectors
  - ...
- Liquids from very divers sectors
  - ...

Collaboration with department of physical chemistry and polymer science (FYSC-VUB)
Prof. Guy Van Assche & ir. Golzen Nicolaas-Alexander

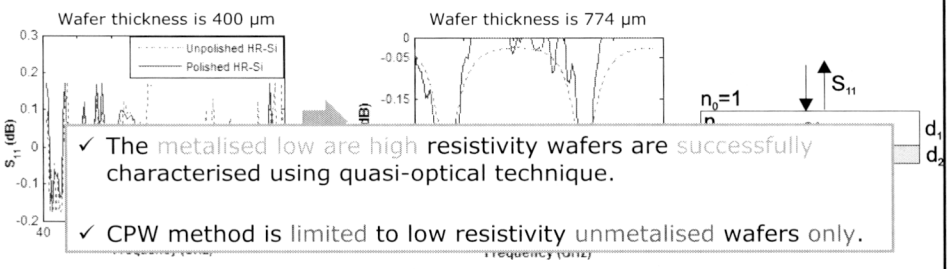
Non-polar: Silicon (LoRe)



Method	Sample	ρ (Ω.cm)	σ (S/m)	Re(ε_1)	rms error (10^{-3})
Quasi-optical	Polished LR-Si	9.5-11.03	9.96-10.5	11.70	1.38
	Unpolished LR-Si	10.8-12.9	7.76-9.27	11.70	0.53
CPW	Polished LR-Si	9-10	10-11.1	11.5	-

Collaboration with laboratoire d'Hypefrequences (EMIC-UCL)

Non-polar: Silicon (HiRe)

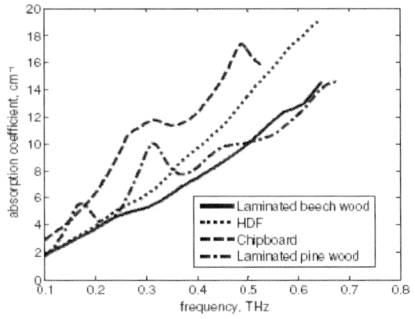


- ✓ The metalised low are high resistivity wafers are successfully characterised using quasi-optical technique.
- ✓ CPW method is limited to low resistivity unmetalised wafers only.

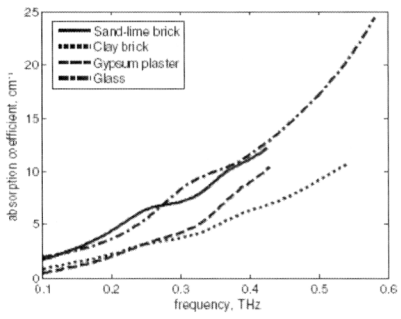
Method	Sample	Thickness (μm)	ρ (Ω.cm)	σ (S/m)	Re(ε_r)	rms error (10^{-3})
Quasi-optical	Bonded HR-Si	774	1330	0.075	11.72	0.67
CPW	Unpolished HR-Si	375	100-150	0.66-1	11.75	-

Collaboration with laboratoire d'Hypefrequences (EMIC-UCL)

Construction Materials



Various Wood Types



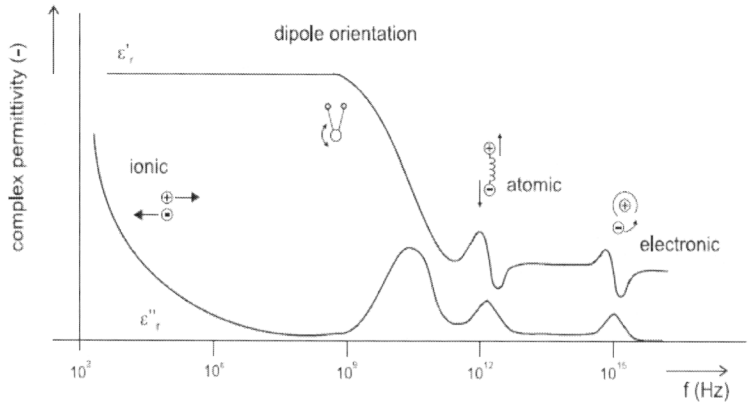
Bricks, Plasters, Glasses

Polar materials

- Solids from very diverse sectors
  - ...
- Liquids from very divers sectors
  - ...

Polar Materials : 7

Basic material science



Dielectric models: pure materials

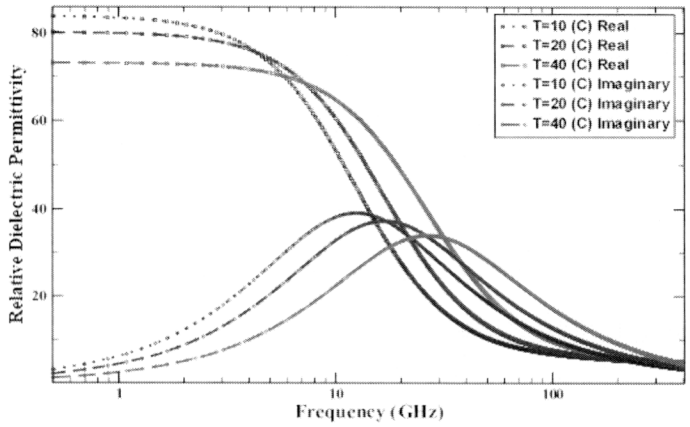
Multi-Debye

Cole-Cole

Havriliak-Negami

Water = polar material.

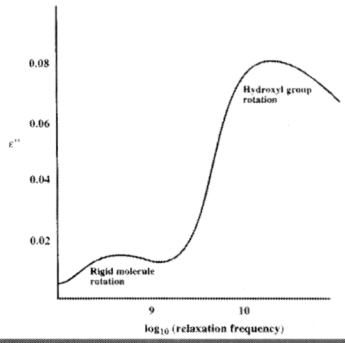
Dielectric permittivity Water



Polar Liquids

Rotation of the whole molecule
- relevant for THz if molecular weight limited

Rotation of parts of the molecule
- for larger molecules with pendant substituents
- functional groups attached to larger molecules
- more localized rotations



Collaboration with department of physical chemistry and polymer science (FYSC-VUB)
Prof. Guy Van Assche & ir. Golzen Nicolaas-Alexander

Other polar materials: relaxation times

Alcohols:
- aliphathic alcohols dipole moments close to H2O
- relaxation times from 50 to 1000 ps

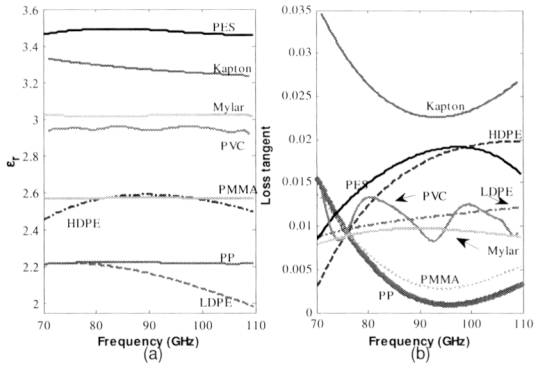
Nitriles, ketons, esters, chlorohydrocarbons:
- typically smaller relaxation times: 2 to 20 ps

Acids:
- again larger from 70 to 200 ps

Aromatic compounds
- (Benzenes): 10 to 50 ps

Polar & Non-Polar Polymers

- Thickness(es) from 0.04 – 5 mm.
- PP, PVC, Mylar, PES, Kapton and PMMA (1st time in W-band)



a) Real part of the permittivity and b) the loss tangent

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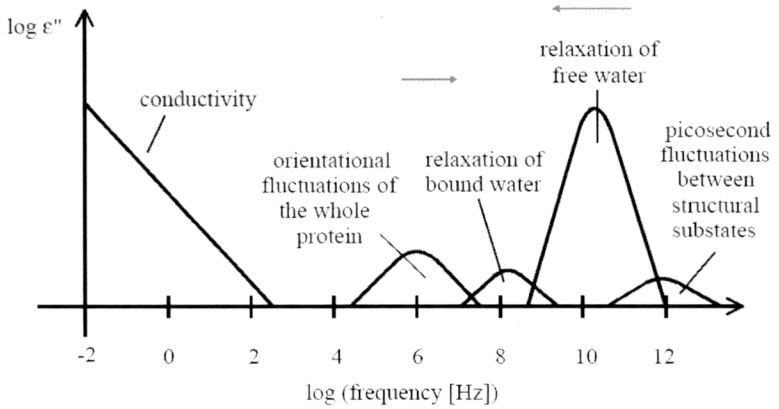
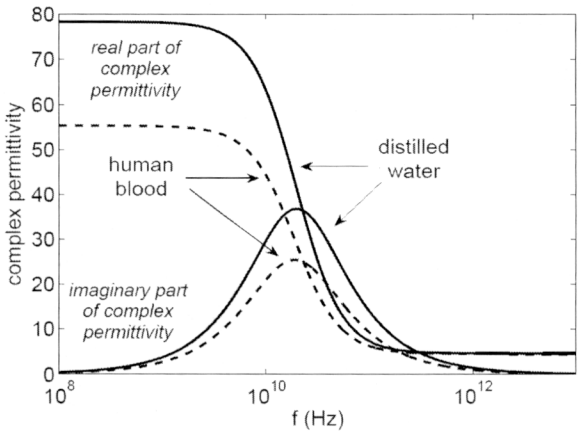
Proteins



Hemoglobine, Lysozime,...

Water versus Blood



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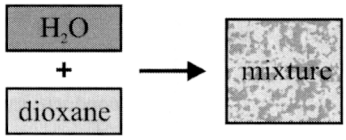
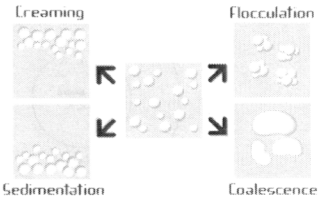
Prof. *Guy Van Assche* & ***ir.*** *Golzen Nicolaas-Alexander*

Dielectric mixing models



Classification: Colloidal systems

Medium / Phases		Dispersed phase		
		Gas	Liquid	Solid
Continuous medium	Gas	**NONE** (All gases are mutually miscible)	**Liquid** aerosol Examples: fog, mist, hair sprays	**Solid aerosol** Examples: smoke, cloud, air particulates
	Liquid	Foam Example: whipped cream, Shaving cream	Emulsion Examples: milk, mayonnaise, hand cream	Sol Examples: pigmented ink, blood
	Solid	**Solid foam** Examples: aerogel, styrofoam, pumice	Gel Examples: agar, gelatin, jelly, silicagel, opal	**Solid sol** Example: cranberry glass



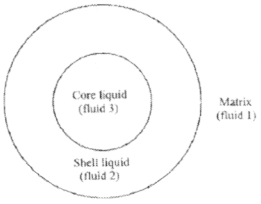
Examples of emulsions

Emulsions: w-in-o versus o-in-w
- coagulation; flocculation, sedimentation, coalescence...
- study of emulsifiers
- challenges: droplet size,...
- cosmetics:

- double emulsions with core-shell droplet morphology,...
- micro-emulsions
- micelles, reverse micelles...

Nano-Colloids

Suspensions



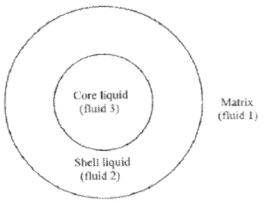
Dielectric mixing models

Molecular aspects of dielectric relaxation

Solvation Dynamics in H20

Interfacial science, surfactant molecules, ...

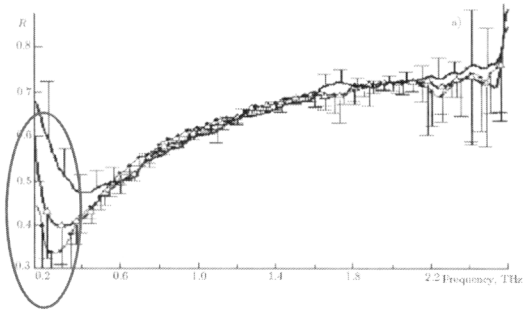
Hydration shells



Biotechnology: amino acids, proteins, DNA

O. P. Cherkasova et al. 52 (7), 518 -523 (2009)

Amino acid: tryptophan
Protein: trypsin (M = 21 kDa; 233 aa)
Ox seralbumin (M = 67 kDa; 607 aa)
Calf thymus DNA (M = 12 kDa)



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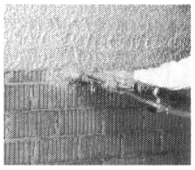
Sensitive to dielectric property variations

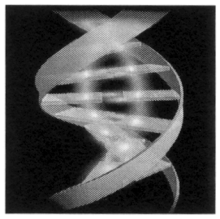
- Dielectric property variation might be
  - Abrupt → boundaries
  - Local → inclusions, defects, in-homogeneities, surface cracks
  - Gradient → gradient in material change

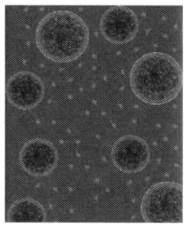
Industrial impact

- R & D level to develop
  - Innovative materials
  - Manufacturing processes
- Measurements in Process Systems
  - Reduce energy consumption
- Lab sensors
- Quality Control of Finished Products

Sectors





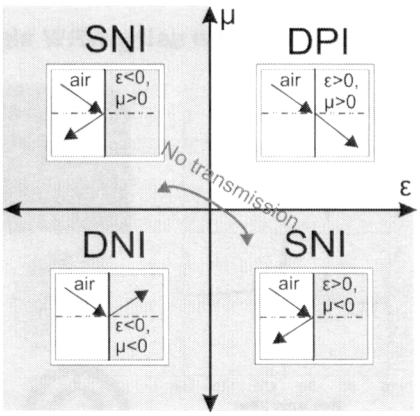
META MATERIALS

(Meta)materials

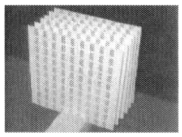
1. THz range:
    1. Parameter extraction methods: DNI and SNI
    2. Eauivalent Circuits Modelling
    3. Thin Film Sensing

2. Optical range
    1. Software development
    2. Fabrication Methods
    3. Parameter extraction

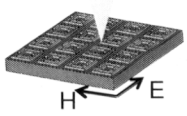
Metamaterials



Metamaterials characterization



Multilayer LHM (ε<0 and μ<0)



Single layer LHM (ε<0)

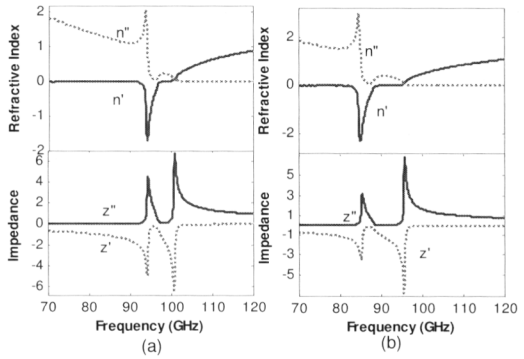
Characterization is performed using:

1. Direct method: to extract μ, ε and n using NRW algorithm .
2. Inverse procedure (GA): to find the optimum parameters of Drude and Lorentz models of μ and ε that give the best fitting of S-parameters.

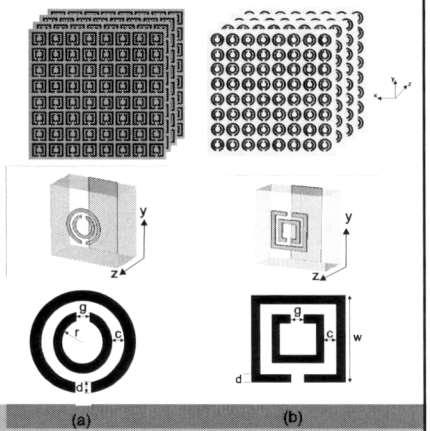
Metamaterials

Characterization methods:

1.Direct method : Extracting μ, ε and n using NRW algorithm .



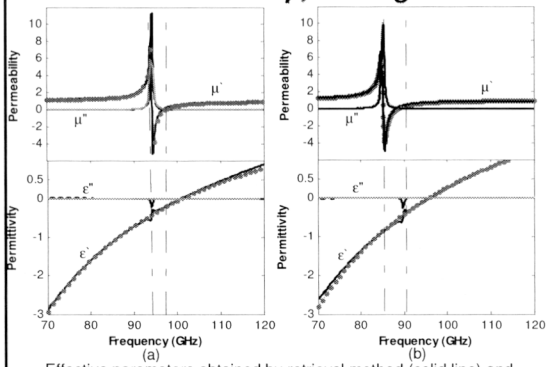
The extracted real part (solid line) and imaginary part (dotted line) of the refractive index and the Impedance of LHM. a) circular-shaped SRR. b) Square-shaped SRR.



Single layer materials

Characterization methods:

2.Inverse procedure: Finding the optimum parameters of Drude and Lorentz models of μ, ε that give the best fitting of S-parameters.



Effective parameters obtained by retrieval method (solid line) and fitting of dispersive models using GA (dotted line). a) Circular-shaped CMM. b) Square-shaped CMM.

$$\varepsilon_{eff} = \varepsilon_{\infty} - \frac{\omega_{ep}^2}{\omega(\omega + i\gamma_e)}$$

$$\mu_{eff} = \mu_{\infty} - \frac{\omega_{mp}^2 - \omega_{m0}^2}{\omega^2 - \omega_{m0}^2 + i\gamma_m \omega}$$

Parameter	Circular-shaped CMM	Square-shaped CMM
ε_{∞}	2.8221	3.0689
ω_{ep}	2.π. 168.39 GHz	2.π. 168.02 GHz
γ_e	1.95 MHz	3.26 MHz
μ_{∞}	1.033	1.0023
ω_{mp}	2.π. 97.26 GHz	2.π. 88.57 GHz
ω_{m0}	2.π. 94.29 GHz	2.π. 85.13 GHz
γ_m	3.23 GHz	4.40 GHz
RMS-error	3.6256e-3	3.5055e-3

where

ε_{∞} is the electric permittivity at the high frequency limit,
ω_{ep} is the electric plasma ("cutoff") frequency.
ω_{e0} is the electric resonant frequency

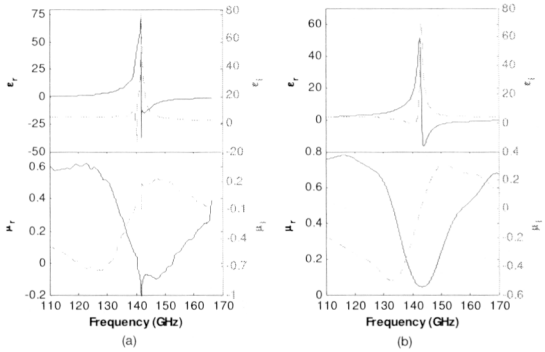
where

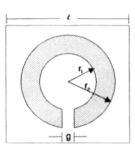
μ_{∞} is the magnetic permeability at high frequency.
ω_{mp} denotes the magnetic plasma frequency at which (in the lossless case) μ_{eff}=0.
ω_{m0} is the resonance frequency at which μ_{eff} diverges

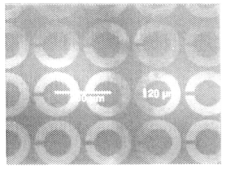
Metamaterials

Characterisation methods:

1.Direct method : Extracting μ, ε and n using NRW algorithm .





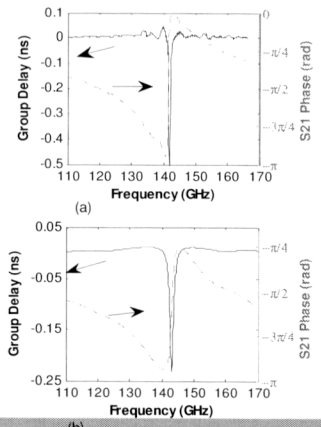


Retrieved permittivity and permeability from a) measured b) simulated data. (Solid) is real part and (dotted) is imaginary part.

Metamaterials

Characterization methods:

3.Calculating the phase velocity and group delay.



$$\lambda_g = \frac{z}{v_g} - \frac{z}{c}$$

where c is the light speed, v_g is given by

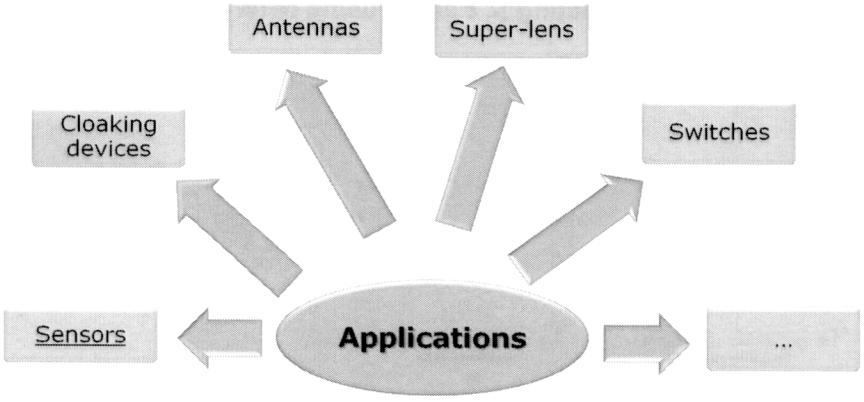
$$v_g = \frac{d\omega}{dk} = \frac{c}{\hat{n} + \omega\, d\hat{n}/d\omega}$$

For abnormal group velocity, the group delay is negative

$$\lambda_g = \frac{z}{v_g} - \frac{z}{c} = \frac{d}{d\omega}\left(\omega \frac{z}{c}[\hat{n}(\omega) - 1]\right) \equiv \frac{d\phi}{d\omega} < 0$$

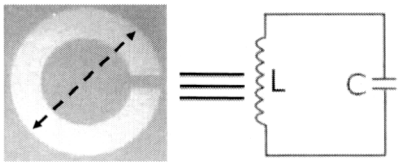
ϕ is the unwrapped phase of the transmission ***coefficient*** (S_{21}).

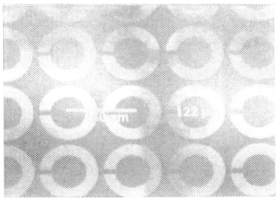
Meta-material Applications



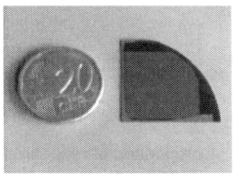
Thin-film sensing

Equivalent Circuit Models: SRR





Re < λ/10



$$f_0 = \frac{1}{2\pi\sqrt{LC}}$$

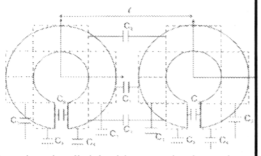
Equivalent circuit model of SRR

→Calculating F_0 from the geometric parameters :

Capacitances:

Coupling capacitance: $C_c = 1/((2/C_{r1})+(2/C_{r2})), C_{ri} = \frac{\varepsilon_0 L}{2}\left[\frac{K(k_0')}{K(k_0)}+\varepsilon_r\frac{K(k_1')}{K(k_1)}\right]$

Surface capacitance: $C_s = \frac{2\varepsilon_0(t+w)}{\pi}\log(4r_i/g)$



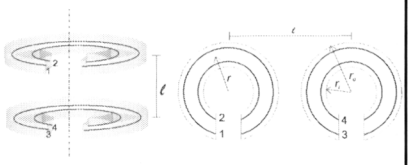
The ring is divided into sub-domains

Inductances:

Self-inductance: $L_s = \mu_0 r\left(\log\left(\frac{2r}{w}\right)+0.9+0.2\left(\frac{w}{2r}\right)^2\right)$

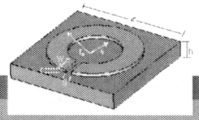
Mutual inductance: $M_0 = \mu_0\sqrt{a_1 a_2}\left\{\left(\frac{2}{k}-k\right)E_1-\frac{2}{k}E_2\right\}$

$L_m = M_0 F$

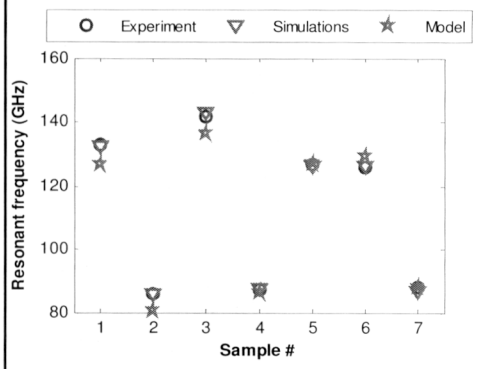


Filaments in coaxial and parallel positions

$$f_0 = \frac{1}{2\pi\sqrt{(L_s+4L_m)(C_g+C_c+C_s)}}$$

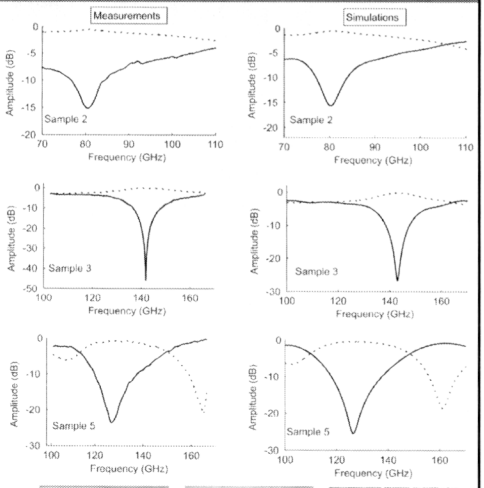


Equivalent circuit model of SRR



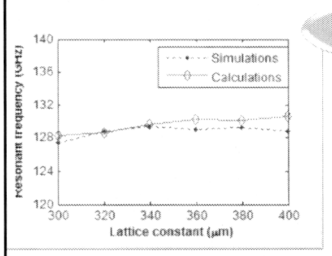
Comparison of resonant frequency of the samples from experimental, simulations and our model

✓ Maximum error is 4.7 %





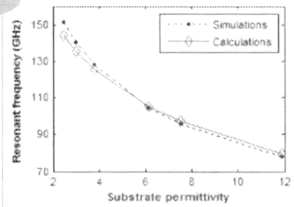
Equivalent circuit model of SRR



Lattice constant

Substrate

Model validity

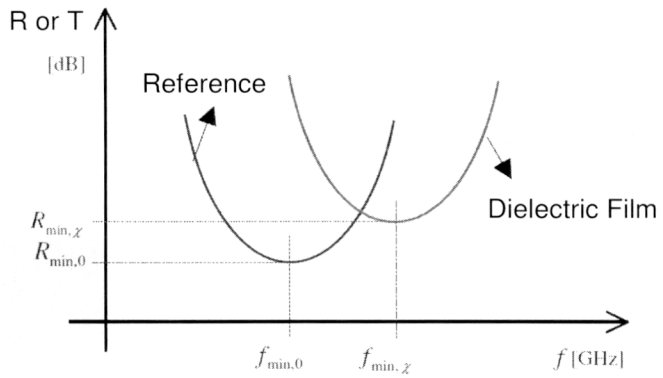


✓ Sub-domain modeling works effectively. (max. error is 1.37 %)

✓ Conformal mapping approximation works well. (max. error is 4%)

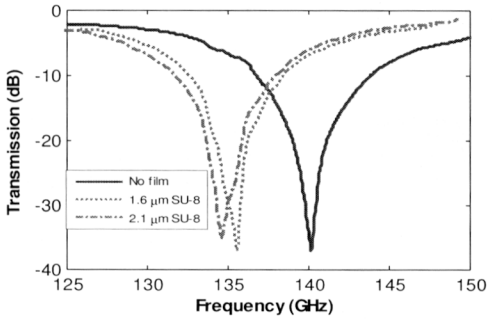
Thin Film Sensing

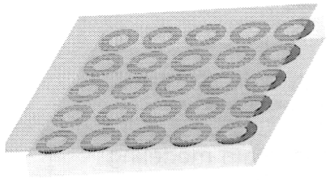
Resonance Shift due to dielectric perturbation of the deposited film



Thin-film sensing: Experimental Example

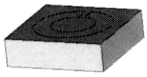
Micrometer thin SU-8 photoresist overlayer
on Quartz substrate



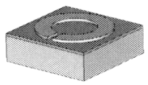


D_{layer} = lambda/1000 !!!

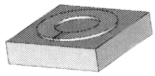
Thin-film location: sensitivity study



1) Film deposited on rings : 2.5 GHz / µm



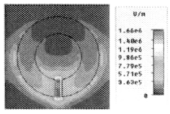
2) Film deposited under rings : 6.75 GHz / µm



3) Film deposited in gap : 1.45 GHz / µm

Less than 0.1 % fill factor !!!

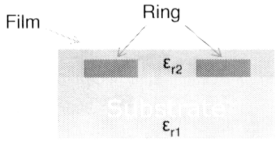
Electric field at f_0



Thin-film sensing

-Developed LC equivalent model for the case of depositing film on the SRR array:

$$C = \frac{\varepsilon_0 L}{2}\left[\frac{K(k_0')}{K(k_0)} + (\varepsilon_{r1}-1)\frac{K(k_1')}{K(k_1)} + (\varepsilon_{r2}-1)\frac{K(k_2')}{K(k_2)}\right]$$



-Characterising SU8-photoresist:

Substrate	SU-8 thick (μm)	Measured f_0 (GHz)	Simulated f_0 (GHz)	Calculated f_0 (GHz)	Measured shift (GHz)
Quartz	1.6	135	132.03	135.11	4
Quartz	2.1	136	133.2	136.5	4.25

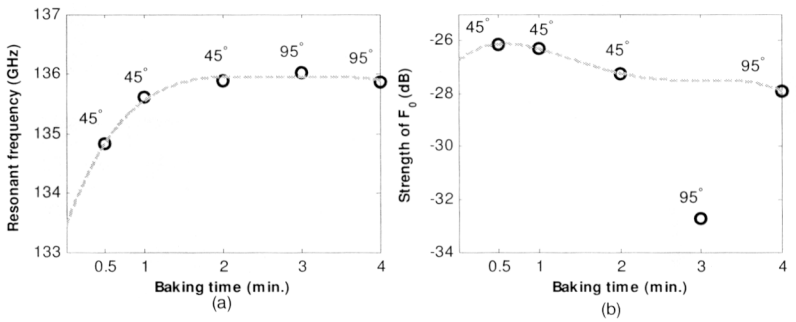
✓ Maximum error is < 1%

-The extracted ε is 1.545±0.14 (*Published value is 1.725±0.08*)

Monitoring the evaporation of a component in a mixture

E.g. Controlling the baking time of photoresist solvent during the Photolithography process:

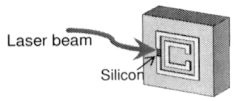
✓The shift of f_0 reflects the change of the refractive index.
✓The strength of the resonant frequency is directly related to the loss change (evaporation).

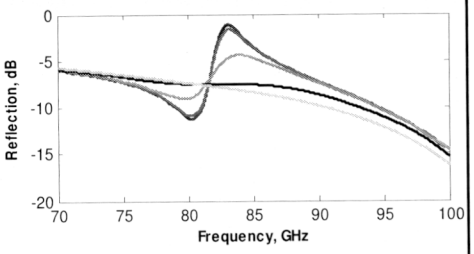


a) The resonant frequency and b) its strength in function of the baking time. The dotted curves are guides to the eye.

Tunable ring resonators

The electric resonance is illuminated with focused laser beam localized at the gap region.

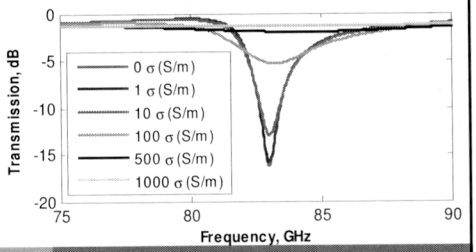




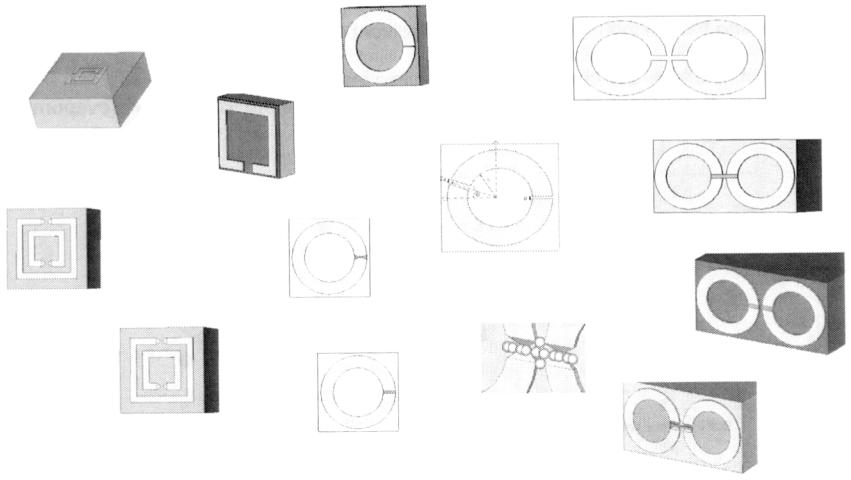
• 2 μm thick Silicon is localized in the gap region of the outer ring.

• The effect of photoexcitation was investigated by changing the conductivity (σ) of the Silicon

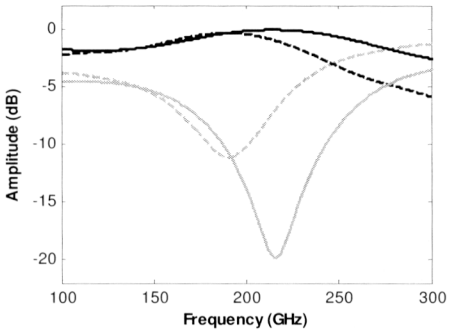
➢ The resonance is switched off at conductivity of about 500 S/m,



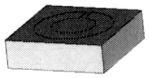
Future: Further Optimization of alternative structures



Recently designed structure

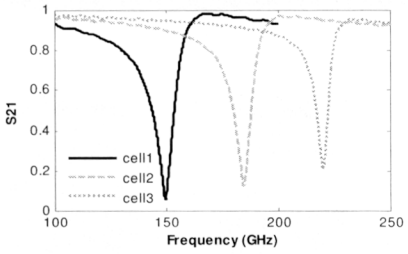


Classic SRR with Film on rings : 2.5 GHz / µm

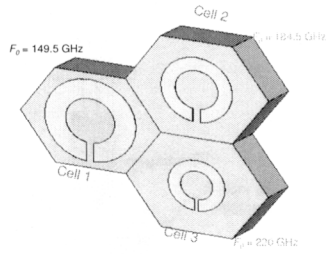


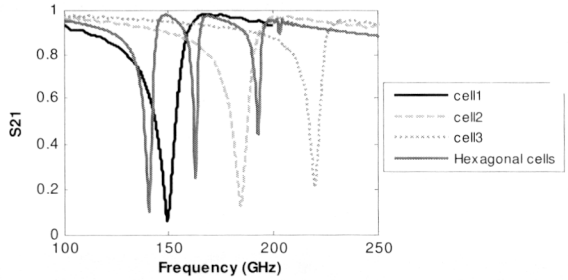
New design: 30 GHz / µm !!! (not yet published)
=> deep sub-micron sensitivity possible

Multiple integrated SRRs: improved identification



Individual cells





Near-Field Sensing:

•Increase substantially the horizontal resolution: → λ/100… → λ/1000

•Different Solutions under Study

Aperture-less: Dithering tips

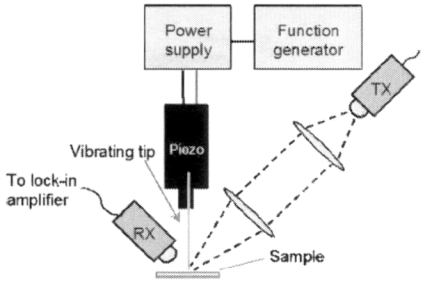
Dielectric focusing probes

- connected as extension horn antennas

- connected as extension to waveguides

META-MATERIALS (soon....

Aperture-less: dithering tips



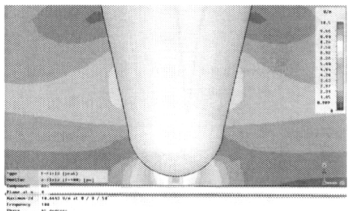
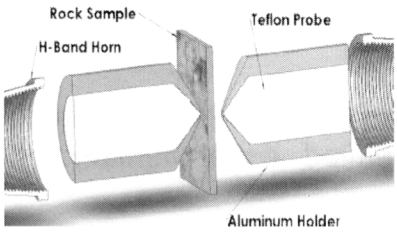
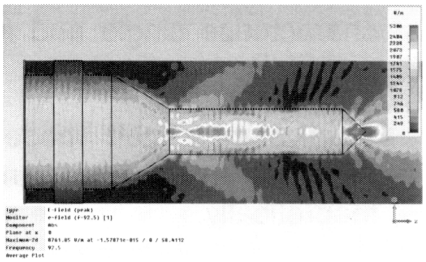


FIG. 1. A schematic of the experimental setup for terahertz ANSOM.

FWO-project: ETRO-VUB, INTEC-UGENT:, Belgium
J. Stiens, R. Vounckx, D. De Zutter, B. Zhu, S. Goris

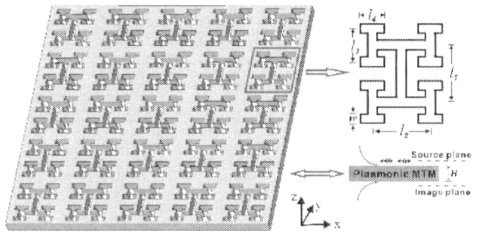
Tapered probes

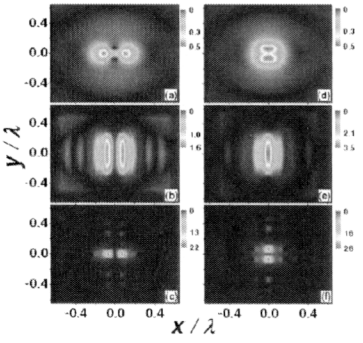




FWO-project: ETRO-VUB, INTEC-UGENT:, Belgium
J. Stiens, R. Vounckx, D. De Zutter, B. Zhu, S. Goris

Fractal-based Meta-Materials





REF:

Conclusions

•Direct and iterative algorithms are developed to characterize single and multilayer right- and left-handed materials.

•Equivalent circuit model for dielectric loaded circular SRR is investigated and verified experimentally and theoretically.

•Three coating approaches of thin film sensing using SRR are implemented.

CONCLUSIONS