Measurement techniques for electromagnetic properties of nanostructured materials, available equipment, and service provision in Europe

(ECONAM FP7 project)

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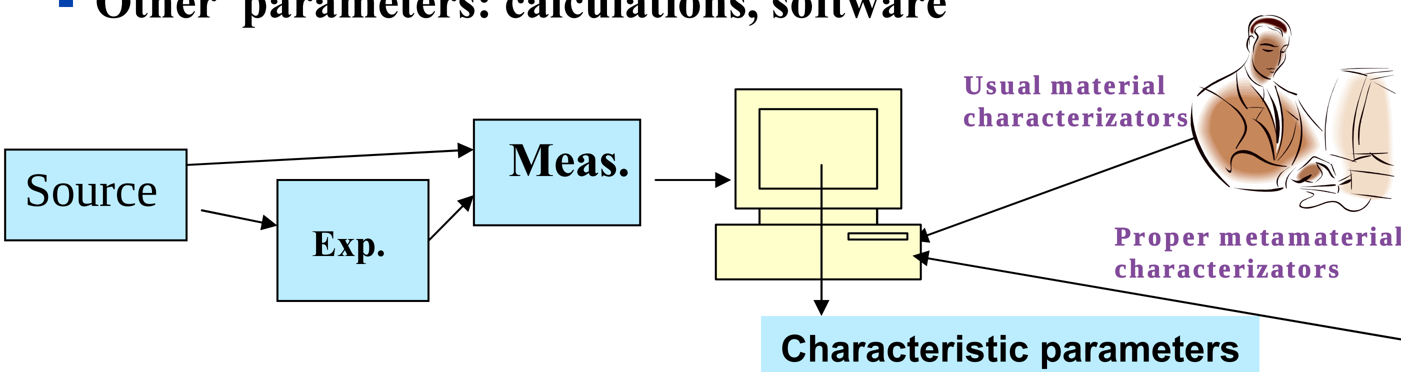
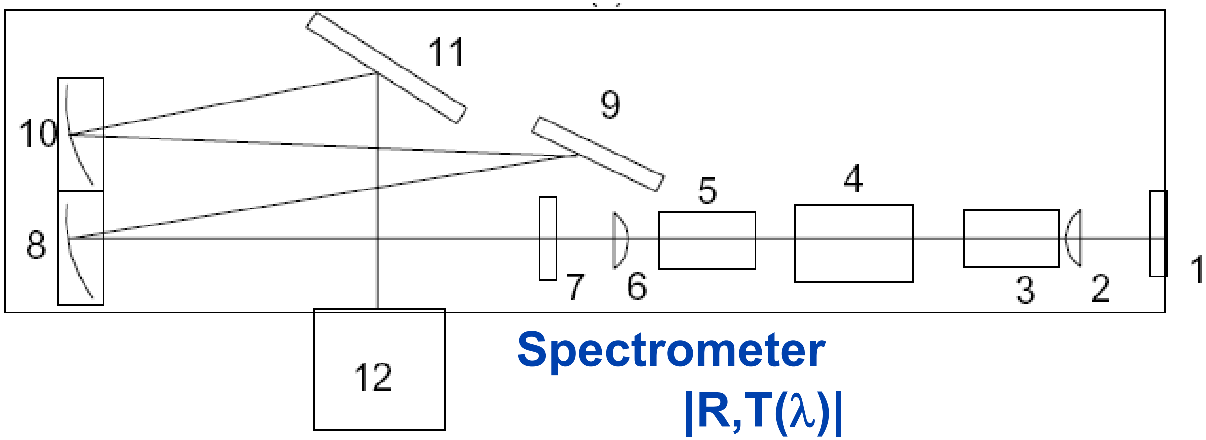
TKK

Problem formulation

- Measurements for describing electromagnetic properties ≡ electromagnetic characterization of the sample
- Characteristic parameters=adequate condensed description of the sample
- What is directly measured:
- Radio: pulses E(t), H(t), harmonic |E(ω)|, phase(E),|H|, phase(H),
- Optics: |E| (detectors), |E(ω)| (spectrometers, ellipsometers)
- All other parameters – strictly speaking retrieved!
- phase(E) – interferometers (retrieval within the tool)
- Other parameters: calculations, software



Spectrometers and interferometers

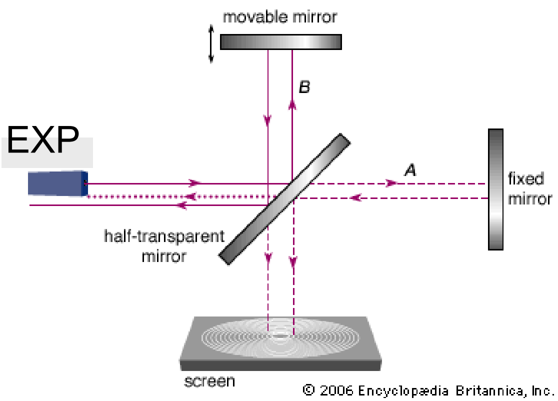


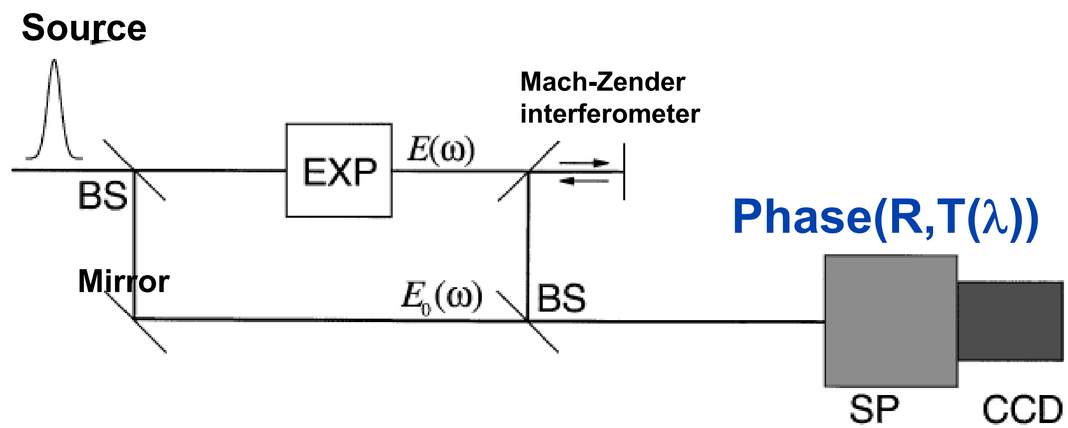
Spectrometer

|R,T(λ)|

(1) collimating lens,
(2) polarizer,
(3) AOTF, (5) polarizer,
(6) imaging lens,
(7) slit, (8) coll. mirror,
(1) input aperture,
(9) echelle grating,
(10) foc. mirror, (11) mirror,
(12) Detector matrix

Michelson interferometer





Phase(R,T(λ))

Spatial spectrum of a beam

[1]

Surface passive structures d<<λ
Art and Nanosensing.

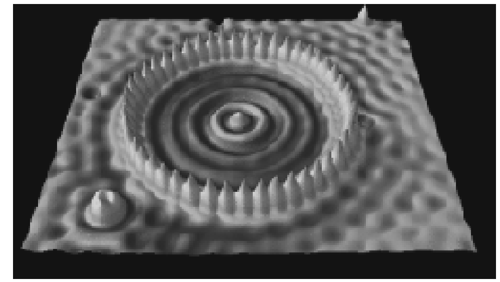
[2]

$\varepsilon = \varepsilon_{substrate}$

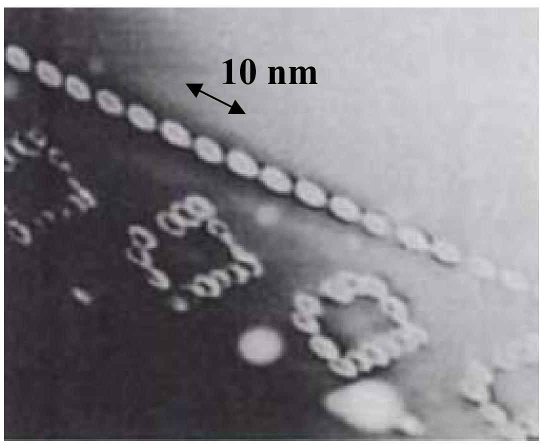
No EM material characterization needs

Molecular characterization!

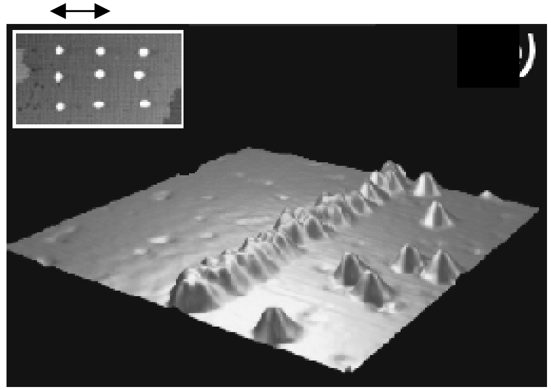
5 nm

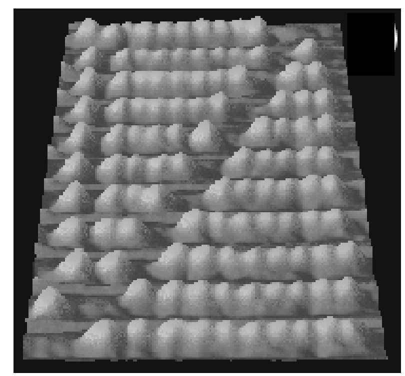


"Corral" of Fe atoms/Cu



2 nm





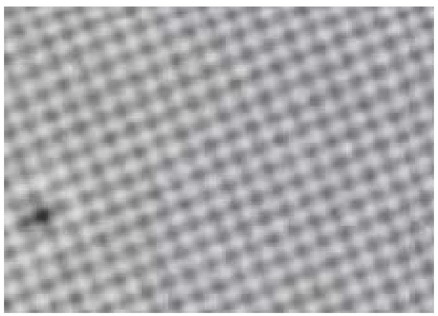
Si (Resist)

Grid (inset) C60 /Si, NanoWires C60 /Si Defect line C60 /Si

Surface-bonded molecules – molecular sensing

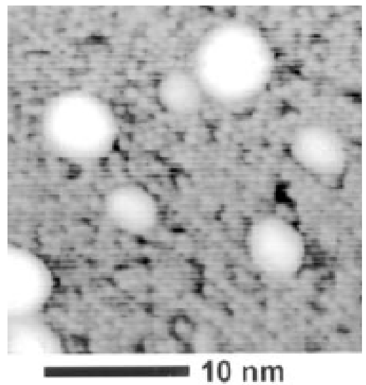
Bulk non-plasmonic nanocomposites. Transparent

[3]

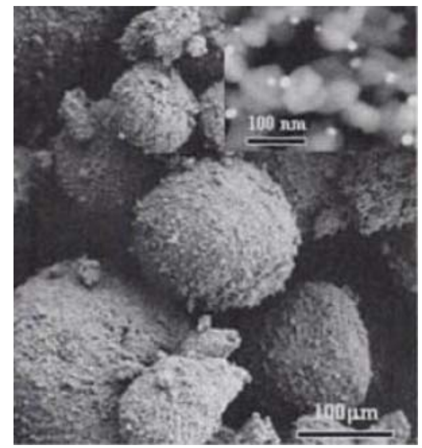


$Sr_2Ti_xRu_{1-x}O_4$

Molecular lattice d=3.5 nm

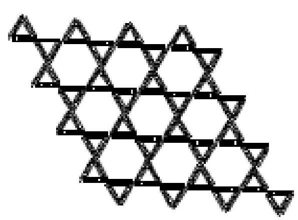


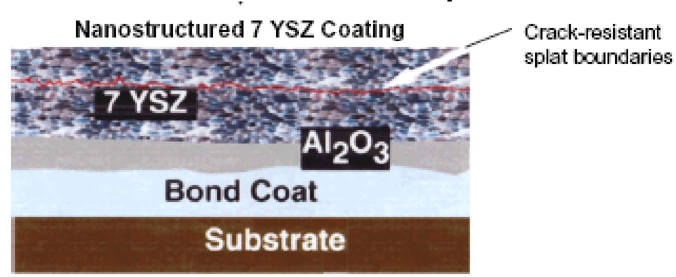
Colloid of Mn nanoparticles



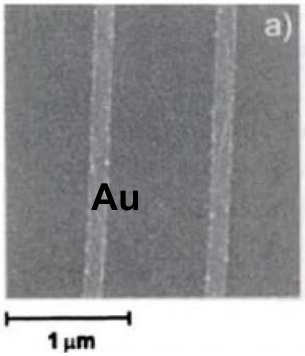
TiO_2 nanoparticles

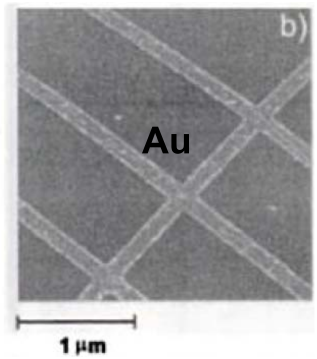
Knotted CNT

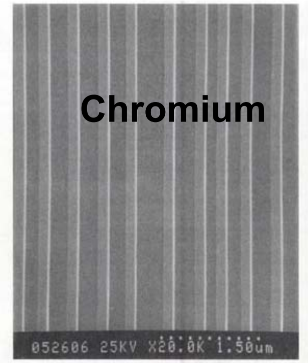


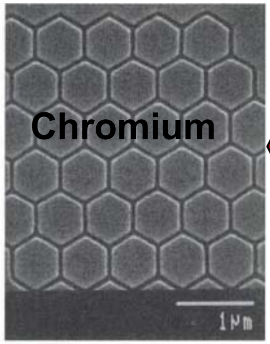


Diffraction gratings (d>λ), mesoscopic layers (d<λ)

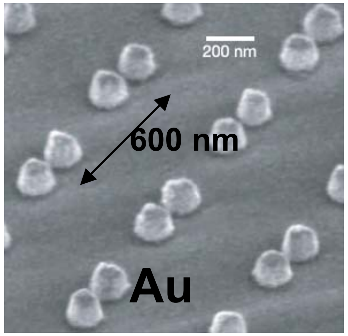


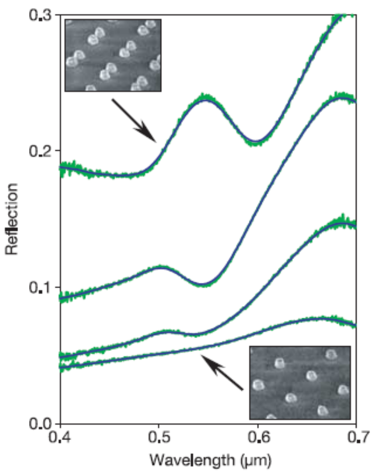




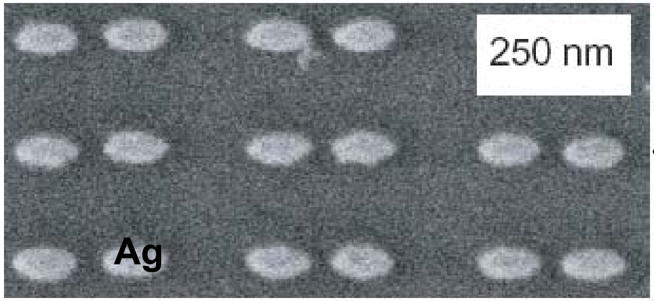


[4]





[6]



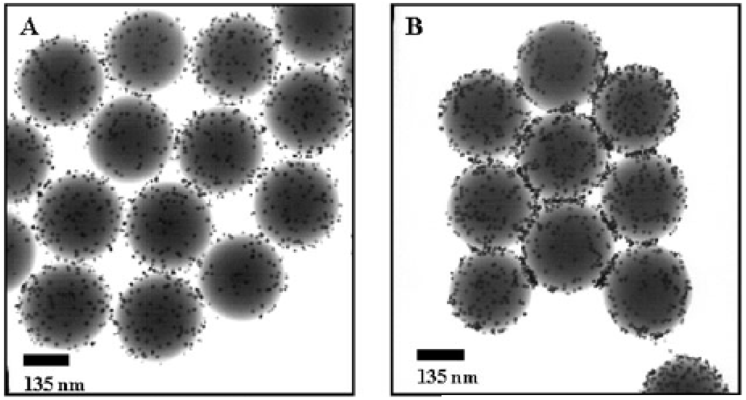
G2: 110 / 55 / 40 / 200 / 400

[5]

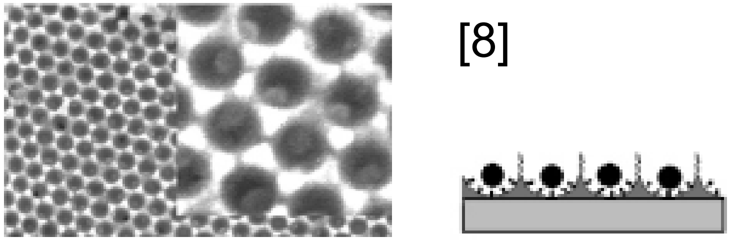
Nanoantenna array (2003)

Plasmonic mesoscopic layers. Examples

[7]

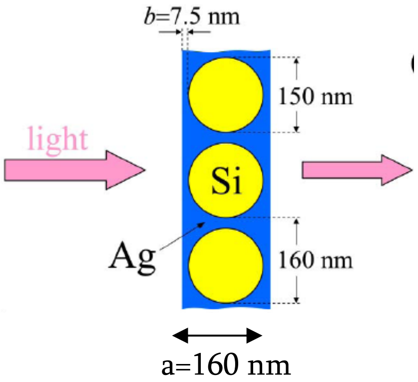


Resonant nanoclusters
/ Si substrate



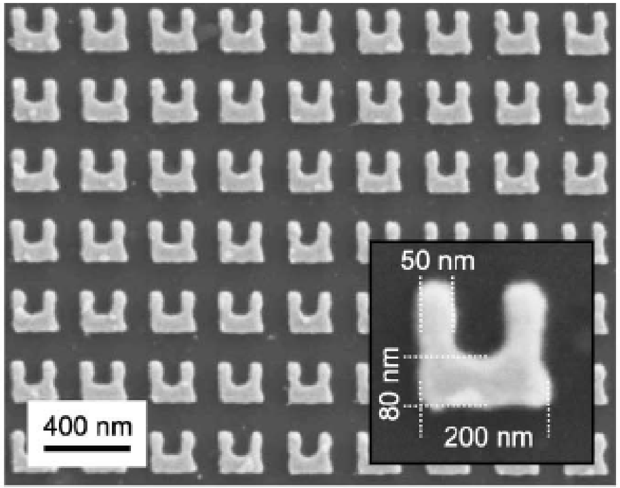
[8]

Au - Ag nanocavities
/Si substrate



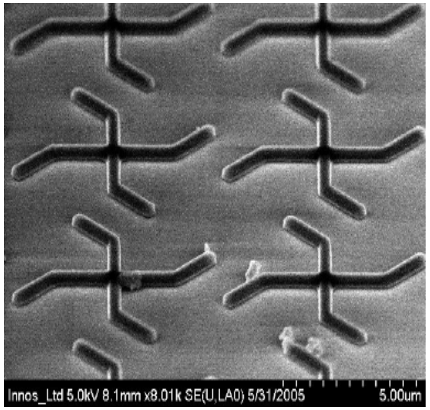
Porous plasmonic layer

[9]



U-shaped SRR layer

[10]

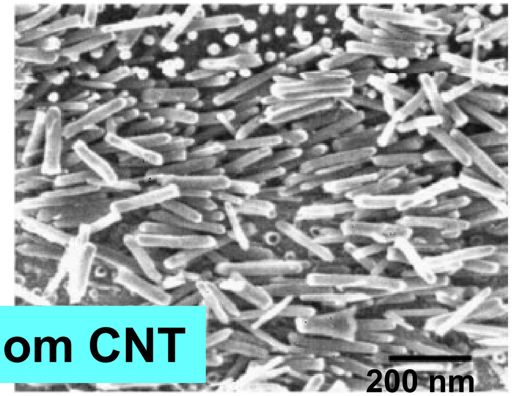


Plasmonic chiral film
(out of scope)

[11]

TKK

Scattering (non-transparent) media including plasmonic ones

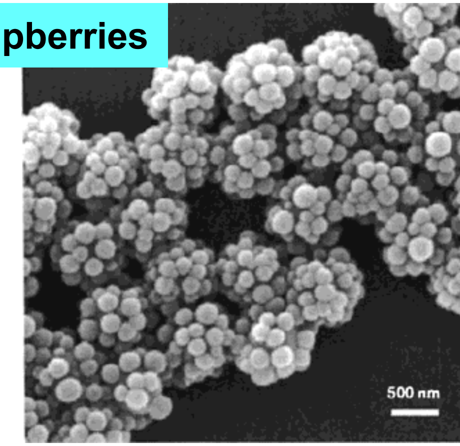


Random CNT

[12]

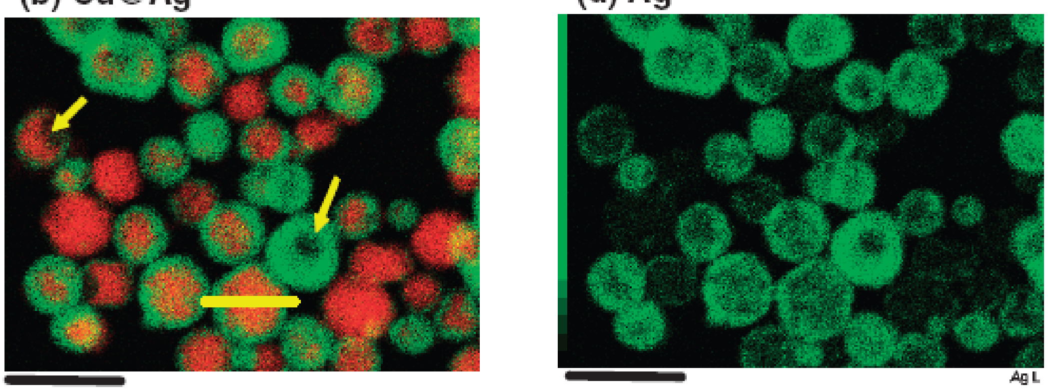
Clustered nano-raspberries

[13]



(b) Cu@Ag

(d) Ag

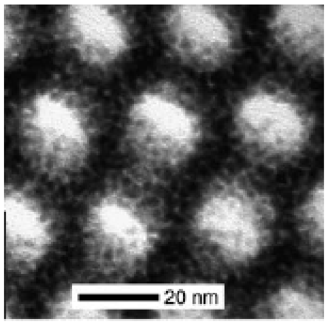


[14]

Clusters of plasmonic nanoparticles in liquid > 500 nm

Bulk plasmonic arrays, d<<λ

1)g~d Dipole materials. 2 g<<d Photonic crystals

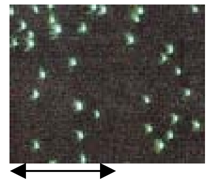


Regular

← Dipole MTM →

[15]

Bulk lattices: Au particles 4 nm



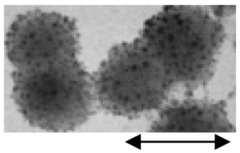
200 nm

[16]

Colloids of Au (Ag) NanoParticles

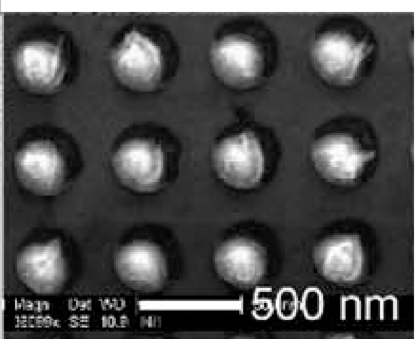
Random

Colloids of Au Core-shell Nanoclusters

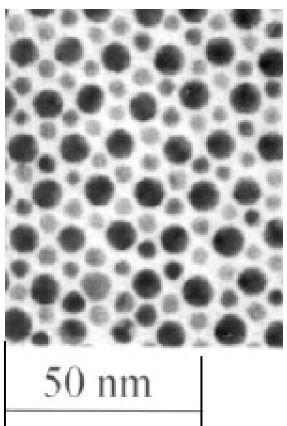


[17]

100 nm



Ag Particles 200 nm

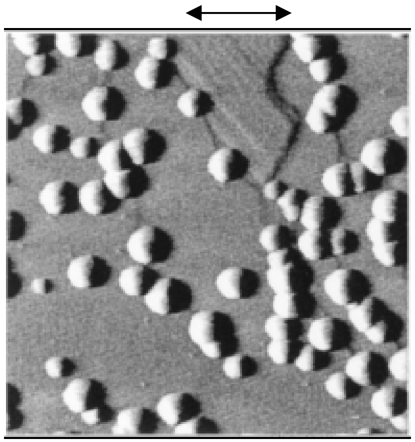


Alternating Ag Particles 10+5 nm

Plasmonic Photonic Crystal

[15, 67]

Scattering Media with Resonant Absorption

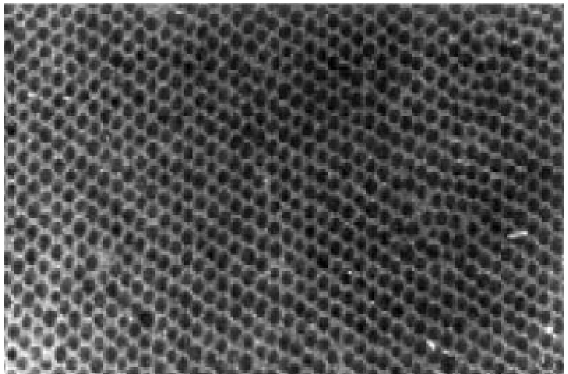


Multilayer/random Ag or Au NanoParticles

[18]

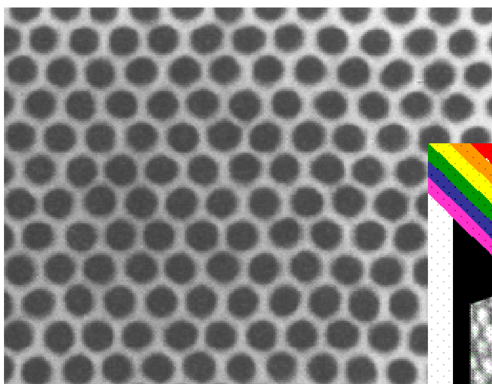
Nanostructured photonic crystals ($d \sim > \lambda$)

[19]

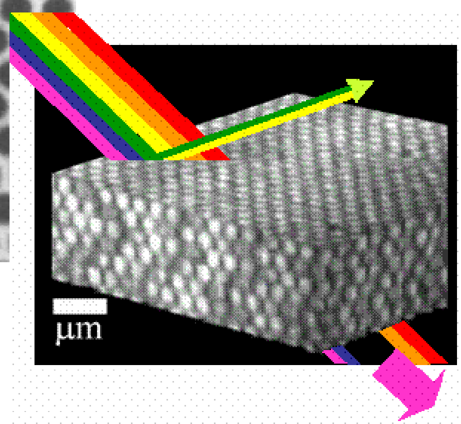


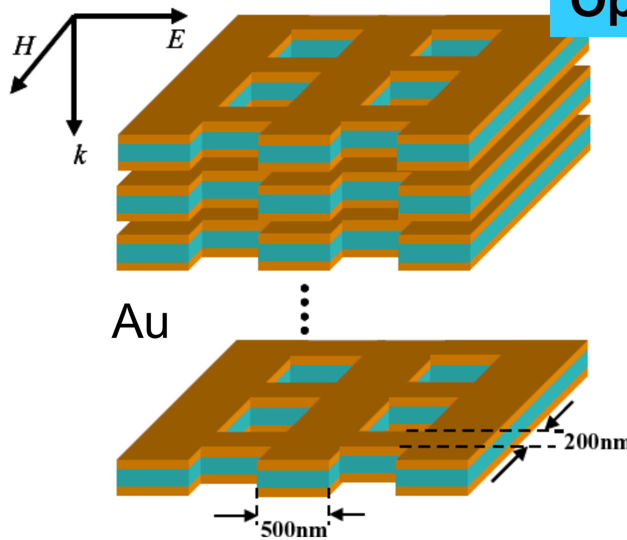
Inverse opals SiC **1 μm**

[19]

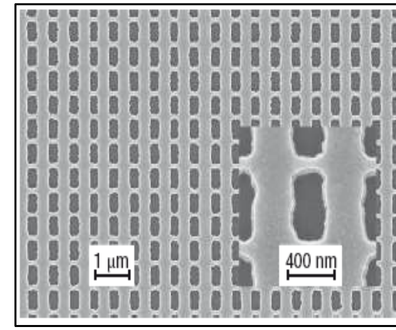


Opals TiO_2 **1 μm**

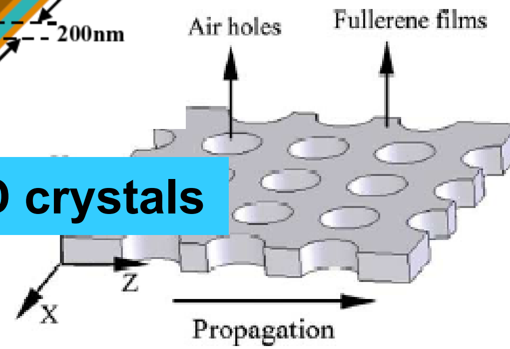




Opt. Fishnets [20]

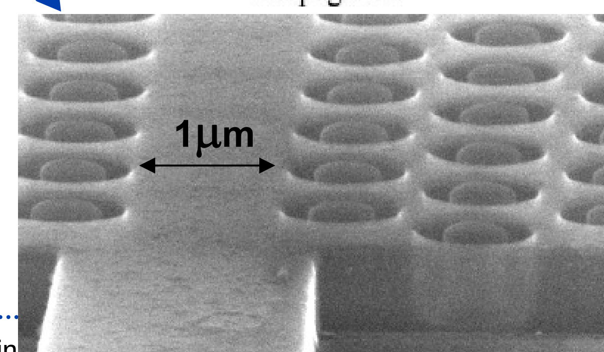


Ag

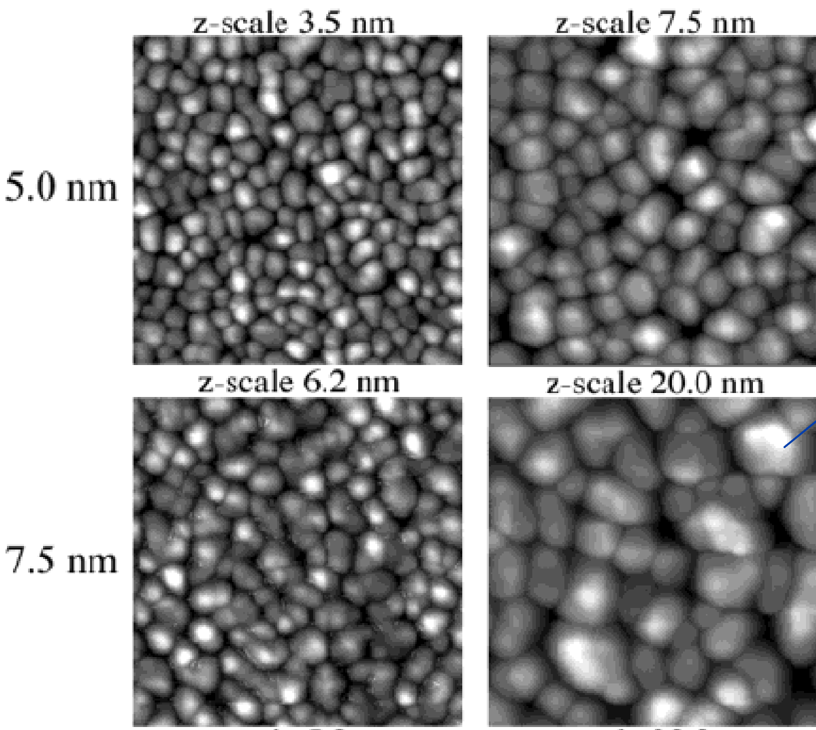


Usual 2D crystals

[21]



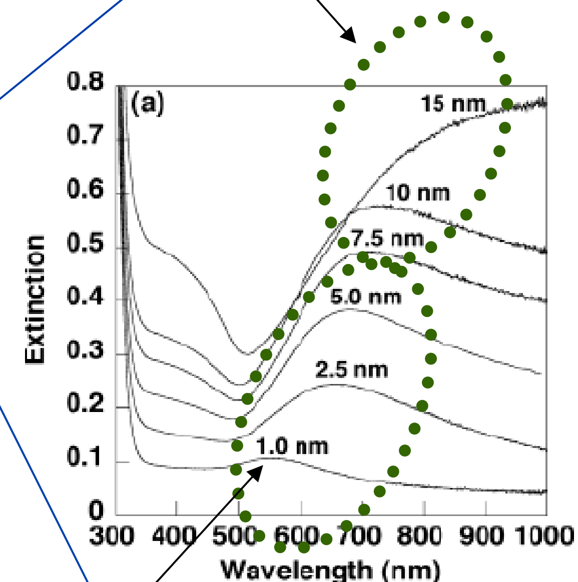
Ultrathin island films



Au /silanized glass

[66]

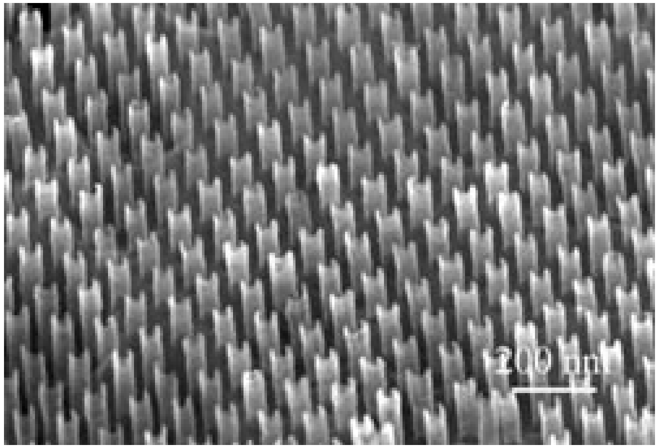
N>5: Plasmonic scattering medium



N<5: Mesoscopic material

films. In the present work, ultrathin, island-type gold films were prepared by evaporation of 1.0–15.0 nm (nominal thickness) gold at a rate of 0.005–0.012 nm s^{-1} onto glass substrates

Vertically aligned nanorods(nanotubes)



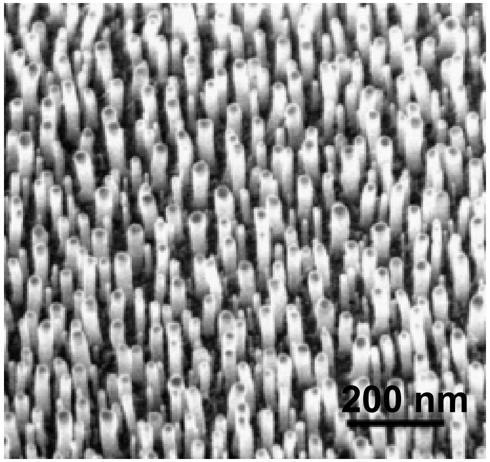
Plasmonic (gold) nanorods

Modest slow-wave factor

Uniaxial dielectric

(no spatial dispersion)

[22]



Carbon NT

Huge slow-wave factor (>100)

Wire medium

(a kind of photonic crystal)

[23]

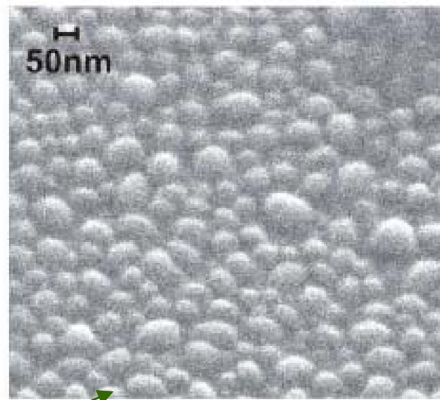
Other vertically aligned nanorods (InP, TiO2 etc)

[24]

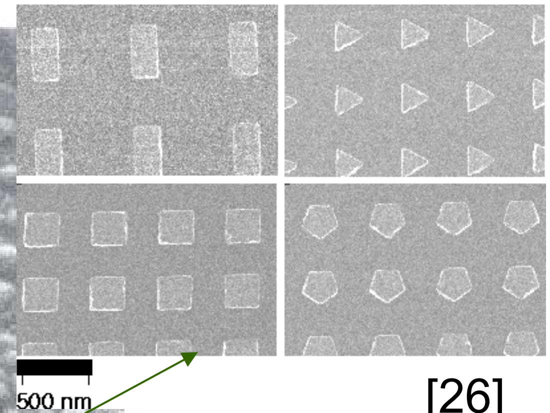
Bulk magnetic nanostructures. Examples

Optical Range: Sufficient transparence (similar to crystalline hexaferrite)

1. **FM multilayer/polymer**
2. ***NiZn* particles and other ferrite colloids**
3. **Nanostructured ferrites (*Co* island films, *Bi*-doped garnets)**



[25]



[26]

Optics (1-3):

$H_{dc} \neq 0$: $\varepsilon \neq \varepsilon_2$

$$\varepsilon = \begin{pmatrix} \varepsilon_1 & +ig_z & 0 \\ -ig_z & \varepsilon_1 & 0 \\ 0 & 0 & \varepsilon_2 \end{pmatrix}$$

Radio (2+3):

1. $H_{dc} \neq 0$: $\mu \neq 1$

$$\overline{\overline{\mu}} = \mu_0 \begin{pmatrix} \mu & 0 & -j\kappa \\ 0 & \mu_y & 0 \\ j\kappa & 0 & \mu \end{pmatrix}$$

2. $H_{dc} \neq 0$: **Spin waves**

4. **Nanomagnets**

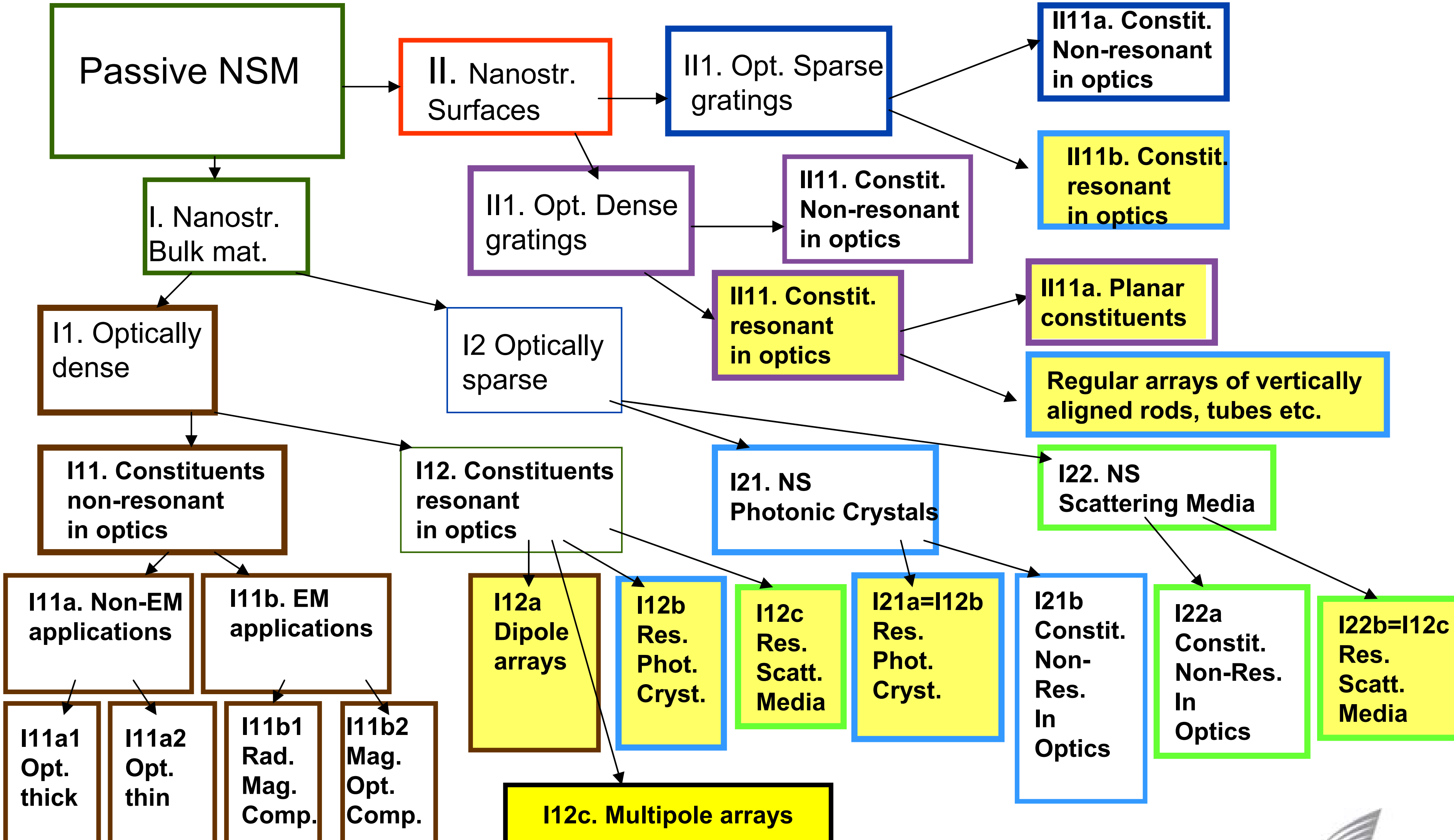
$H_{dc}=0$ Radio: $\mu \neq 1$.

Optics: $\varepsilon = \varepsilon_h$

Classification of nanostructured materials (NSM) by their linear EM properties. Text

- Bulk passive structures **(N>4-5 Unit Cells)**
- **Optically dense bulk structures d<<λ**
- **Non-resonant materials:**
- **Non-EM applications, EM applications,**
- **Thick films, optically large samples, Thin films and island films,**
- **Radiofreq. Mag. Med. and Nanomagnets, Magneto-Optical Media**
- **Plasmonic and polaritonic MTM:**
- **Dipole arrays, Multipole arrays, Resonant Photonic Crystals, Resonant scattering media**
- **Optically sparse bulk structures d~>λ**
- **Nanostructured Photonic Crystals, Scattering media (resonant and non-resonant)**
- Surface passive structures **(N<4-5 Unit Cells)**
- **d<<λ Dense gratings d~>λ Diffraction gratings,**
- **Non-resonant, Resonant**
- **Planar MTM, Vertically Aligned Nanorods**
- Active nanostructures **(of quantum dots and wires, dye-doped nanoporous and liquid crystals matrices, etc). Out of scope**

Classification of NSM by their linear EM properties. Chart



Explanation of the chart

Metamaterials

Scattering (non-transparent) media

Sample parameters: QE, QA, QS

Bulk uniform concentration media

Material parameters: ε,μ.

(Bianisotropic and bulk multipole arrays are out of scope)

Photonic crystals/EBG

Material parameters: stopbands (bandgaps).

Additionally: Brillouin dispersion diagram, Fresnel isofrequency surfaces.

Diffraction gratings

Characteristic parameters: **D**(λ,m), $\Delta\lambda$(m), $I_{norm.}$(λ,m)

Mesoscopic layers

Sample parameters: QE, QA, |R(λ,θ)|, |T(λ,θ)|

(many-layer structures are out of scope)

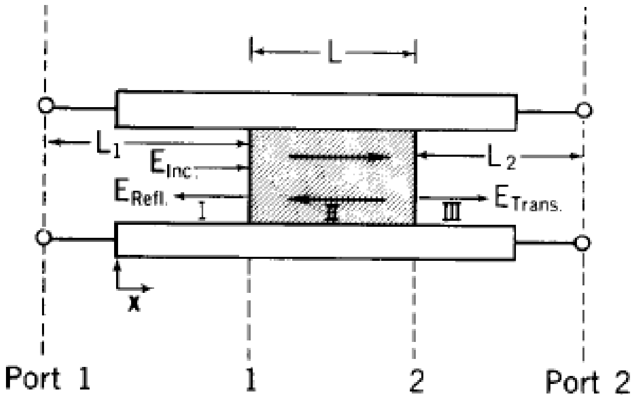
Bulk layers and bars characterization. Nicholson-Ross-Weir (NRW) technique

Network analyzer

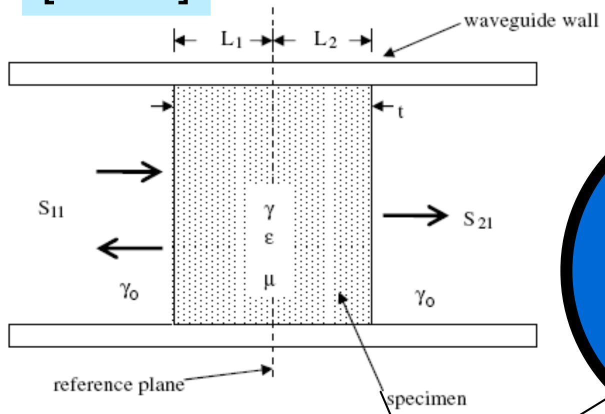
S11, S21 ➡ $n, \gamma = \sqrt{\mu/\varepsilon}$

[27-29]

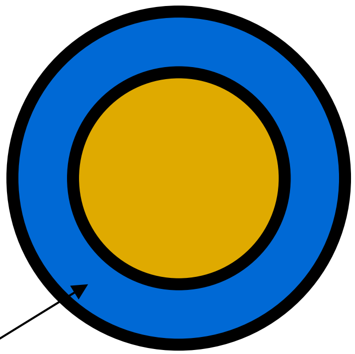
Radio range



Bar or layer sample

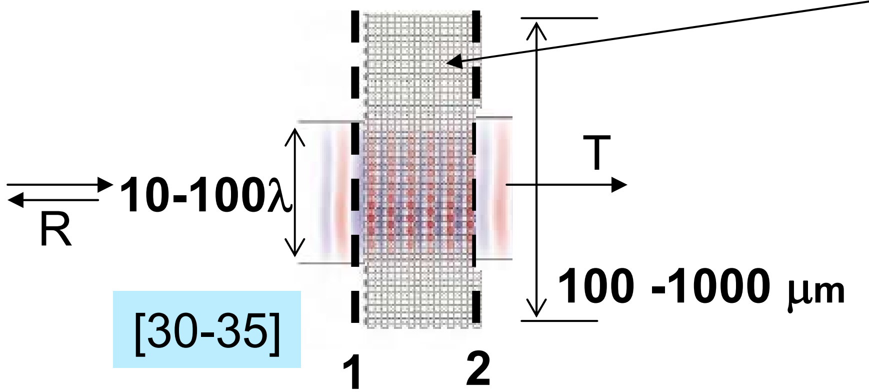


Ring sample



Detector (Spectrometer) + Interferometer

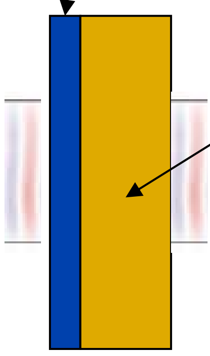
Layer sample



[30-35]

Nanostructured material

Optics

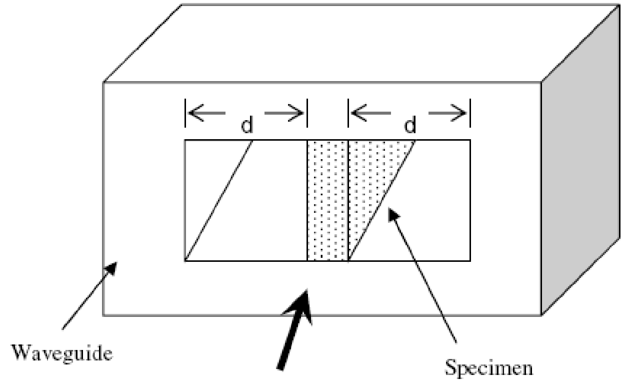


History: [36]

TKK

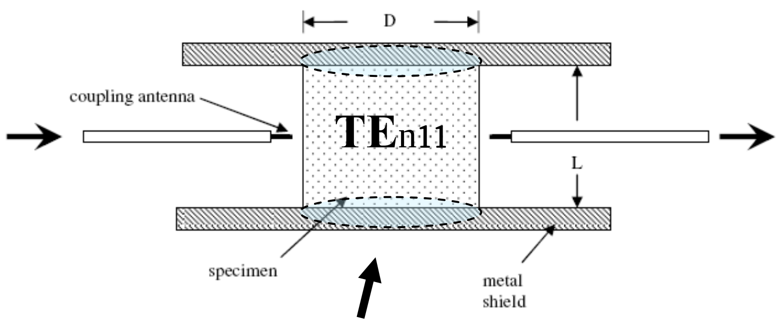
Bulk samples characterization. Other radio techniques

Dube-Lanagan (1984)
Complements NRW for Anis. Magnetodiel.



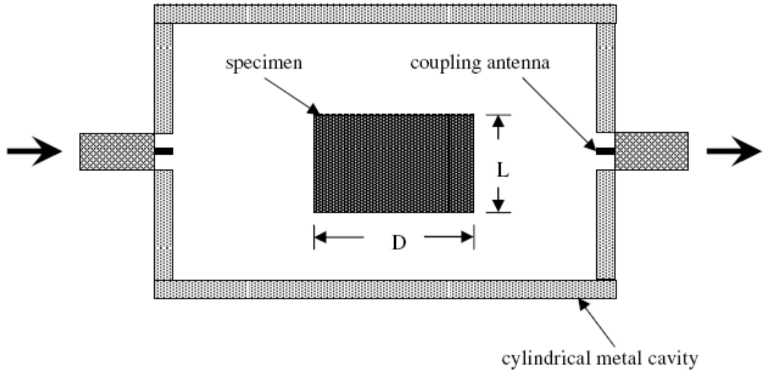
[37-40]

Resonator techniques: only permittivity (precisely)



1. **Hakki-Coleman method (1946)**

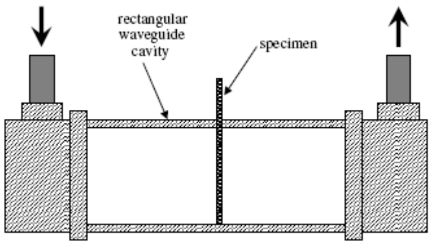
Specimen-resonator

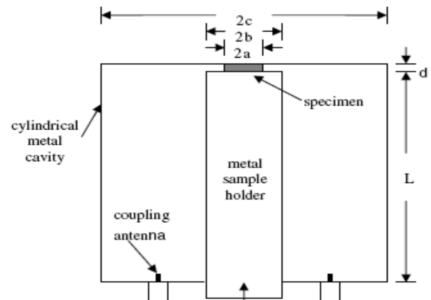


2. **Cylindrical cavity method (specimen-rod)**

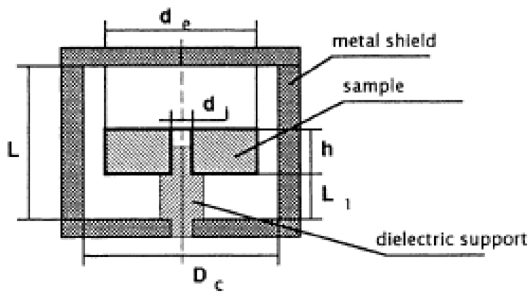
3. **Rectangular cavity method (specimen- bar)**
(many people in 1950s)

Bulk samples radio characterization. Unusual resonator techniques

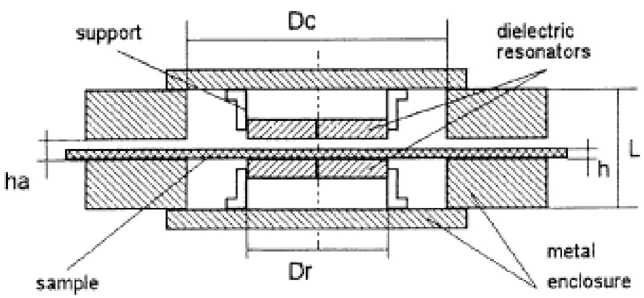




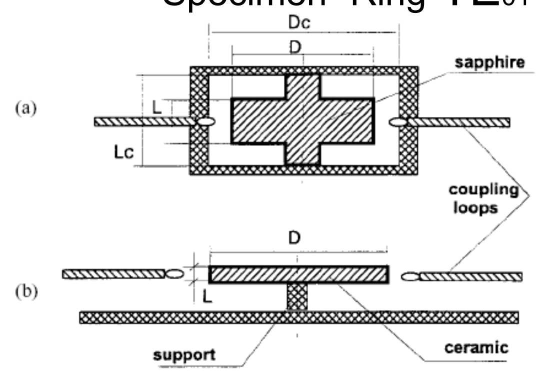
Modified cavity methods



Specimen -Ring TE011 resonator



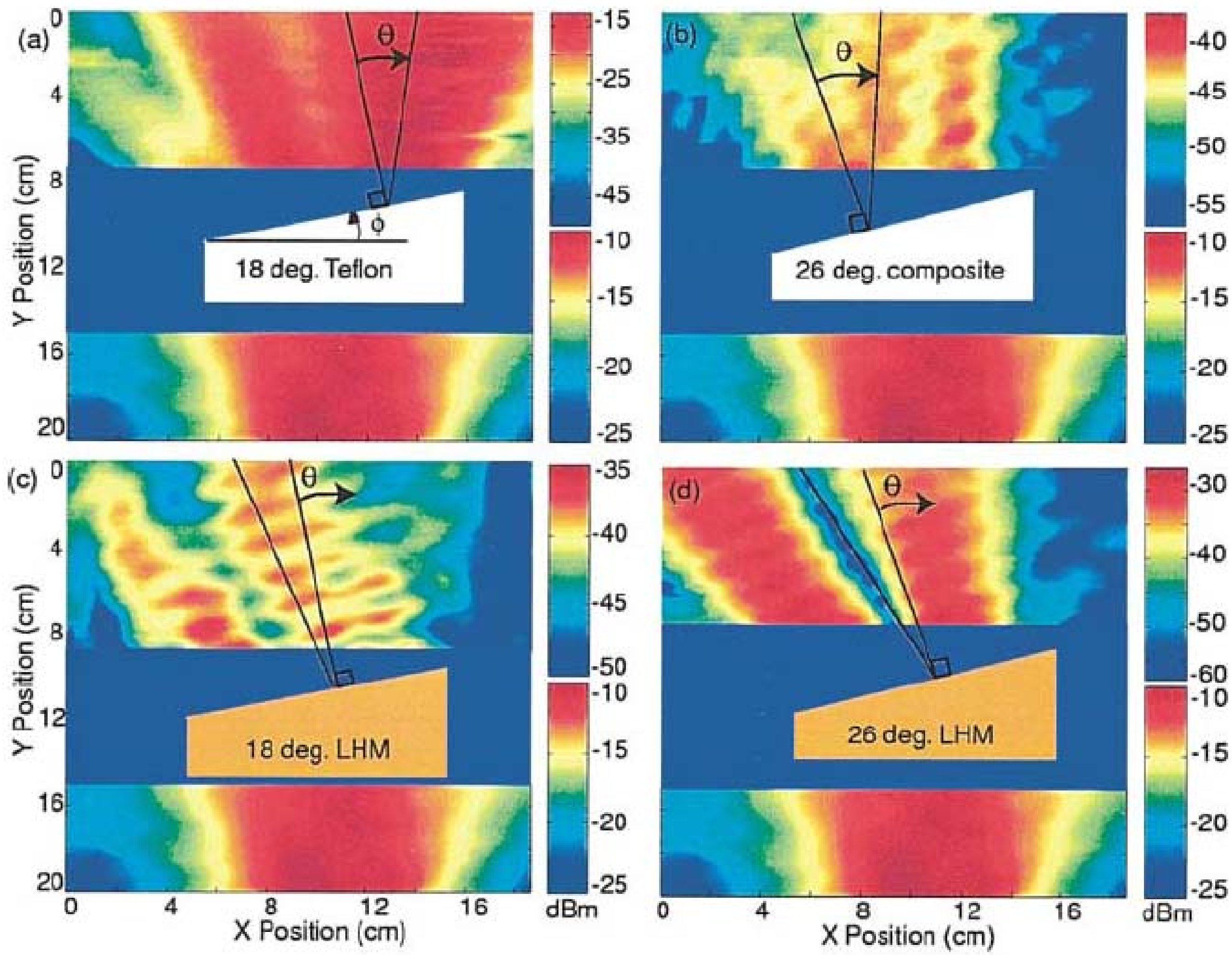
Specimen - Layer in a split-disk (quasi-TE011) resonator



Specimen – Whisp. Gal. resonator

[37-40]

Characterization of bulk media using wedges



Deviation angle shows the Energetic Velocity $\mathbf{V_E}$.

Low loss: $\mathbf{V_E = V_g}$

Only detectors need to detect the Negative Refraction.

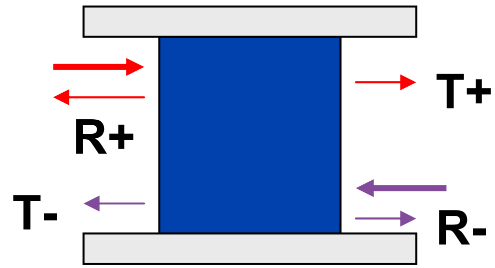
ε and μ can be extracted from **R** and **T**

[41]

TKK

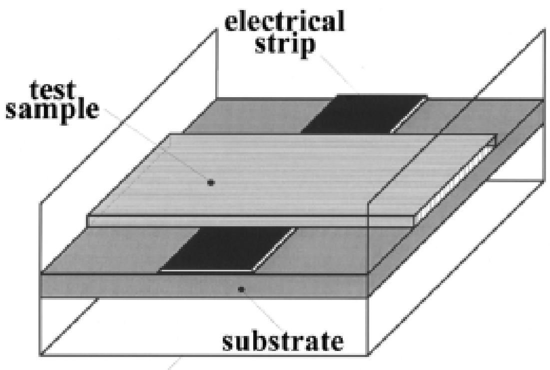
Magnetic NSM: characterization in the radio range

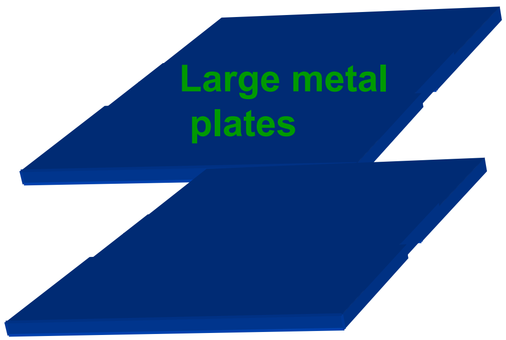
$$\overline{\overline{\mu}} = \mu_0 \begin{pmatrix} \mu & 0 & -j\kappa \\ 0 & \mu_y & 0 \\ j\kappa & 0 & \mu \end{pmatrix}$$



"Two-side NRW"

[separate list]

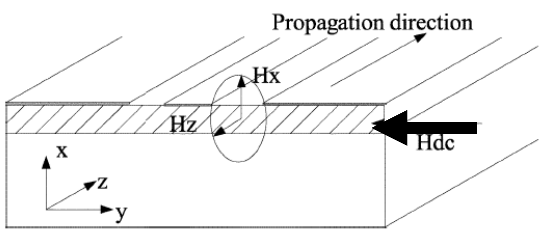


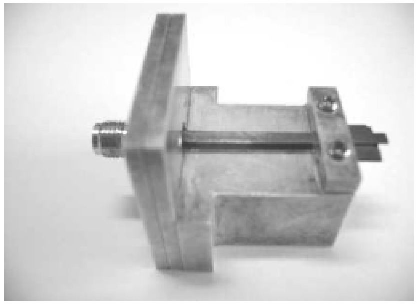


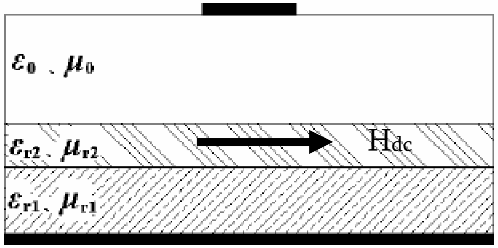
Modifications of two-side NRW

[separate list]

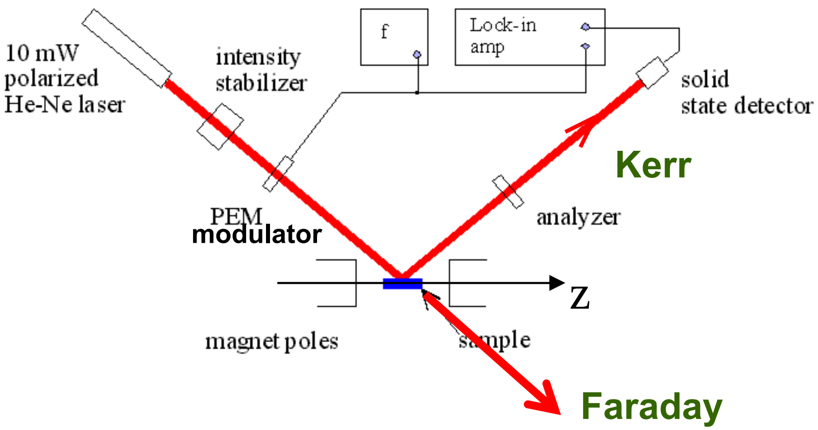
Characterizations *in situ*, e.g. coplanar and microstrip isolators [42-46]



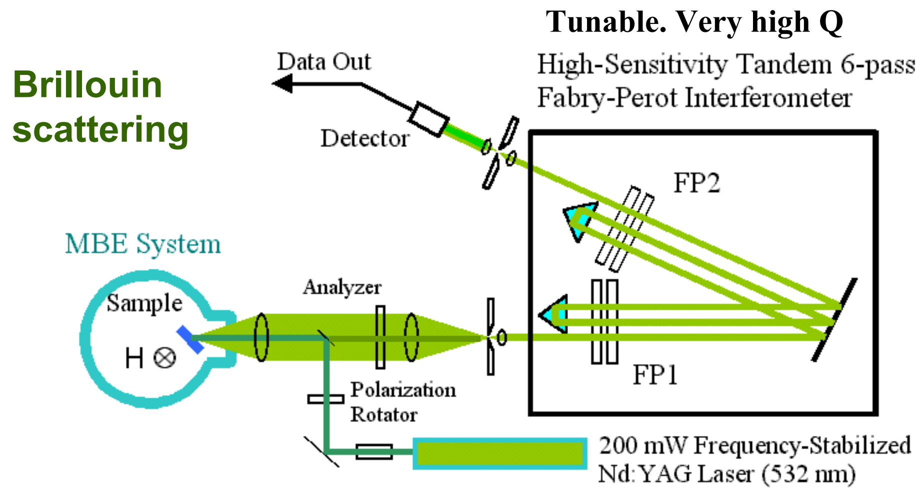




Magnetic samples: experimental characterization in the visible



1.



2.

1. Magnetic Kerr constant – (ε_2-ε_1) [47]

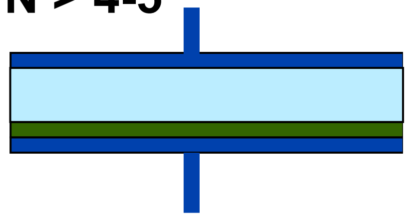
Faraday-Verde constant - g_z [48]

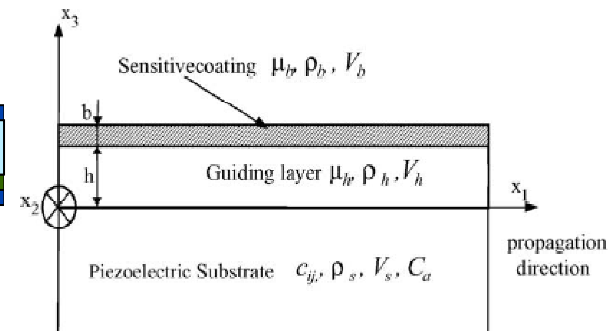
2. Brillouin – microwave characteristics: spin waves frequencies [47]

Non-magnetic films experimental characterization

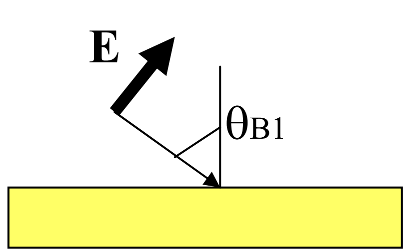
Thin (Nd~<λ) film=bulk medium: N > 4-5

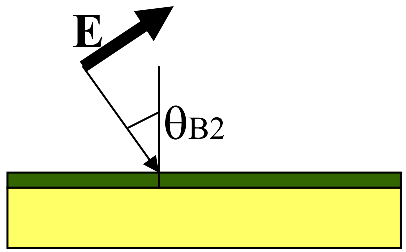
Radio range
1. Quasi-static method
2. WaveGuide methods [49]





Optical range [50-56, 40]:
a). Known thickness:
1. Ellipsometry (Drude, 1889)
2. Abelè method (1950)

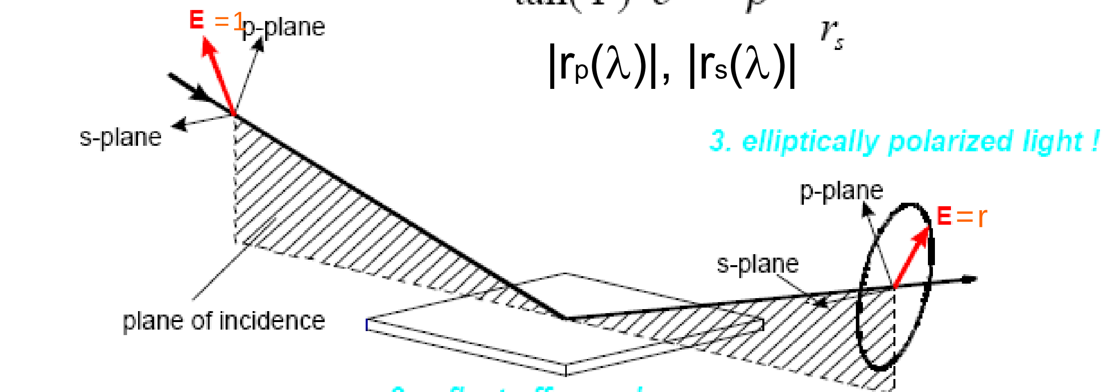




b) Unknown thickness h (especially island films):
1. Schopper method (1952) – the same as NRW where h and ε to be found (μ=1)
2. Malé method (1950) – low-loss films: ε and h can be found from |R| and |T|
3. Modern ellipsometry [51-56, 40]

Modern film ellipsometry

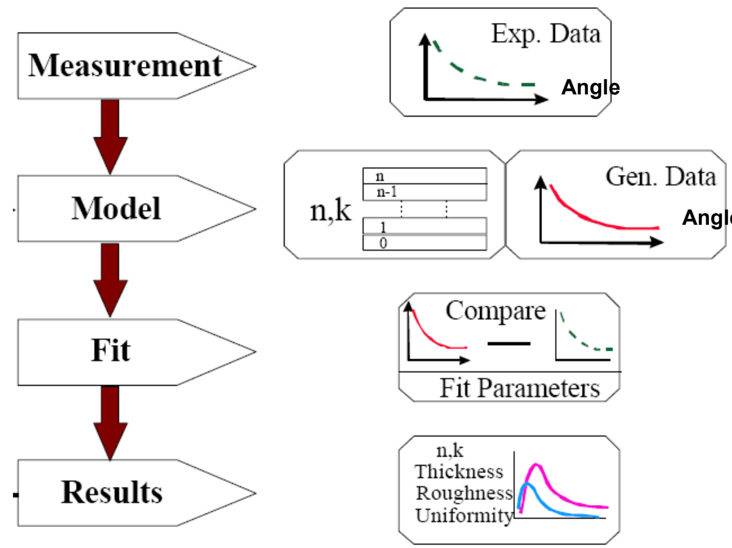
Most advanced: Variable-Angle-Spectrometric Ellipsometry (VASE) **[40]**



$$\tan(\Psi)\cdot e^{i\Delta} = \rho = \frac{r_p}{r_s}$$

$$|r_p(\lambda)|,\ |r_s(\lambda)|$$

Basic configuration for reflection ellipsometry.



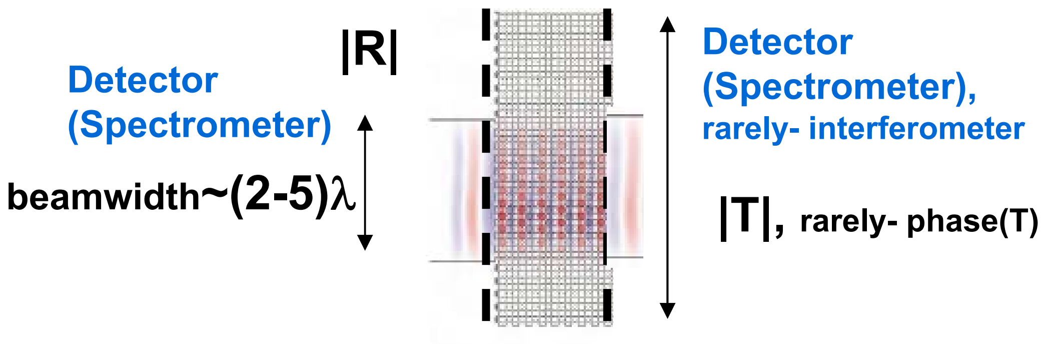
Known h –complex ε_t, ε_n for uniaxial films

Transmission ellipsometry [51, 52].

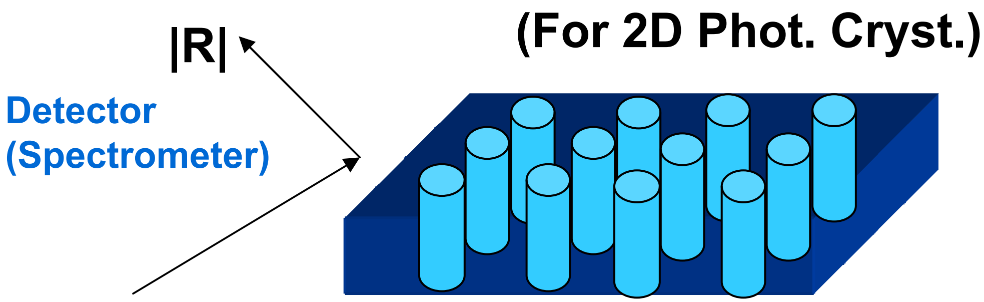
Generalized ellipsometry (fully anisotropic specimen, unknown h) - both schemes [54-56]

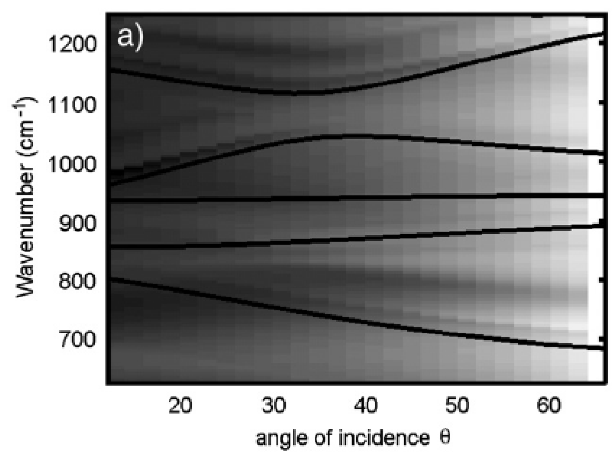
Photonic crystals experimental characterization

Usually - validation of simulations!



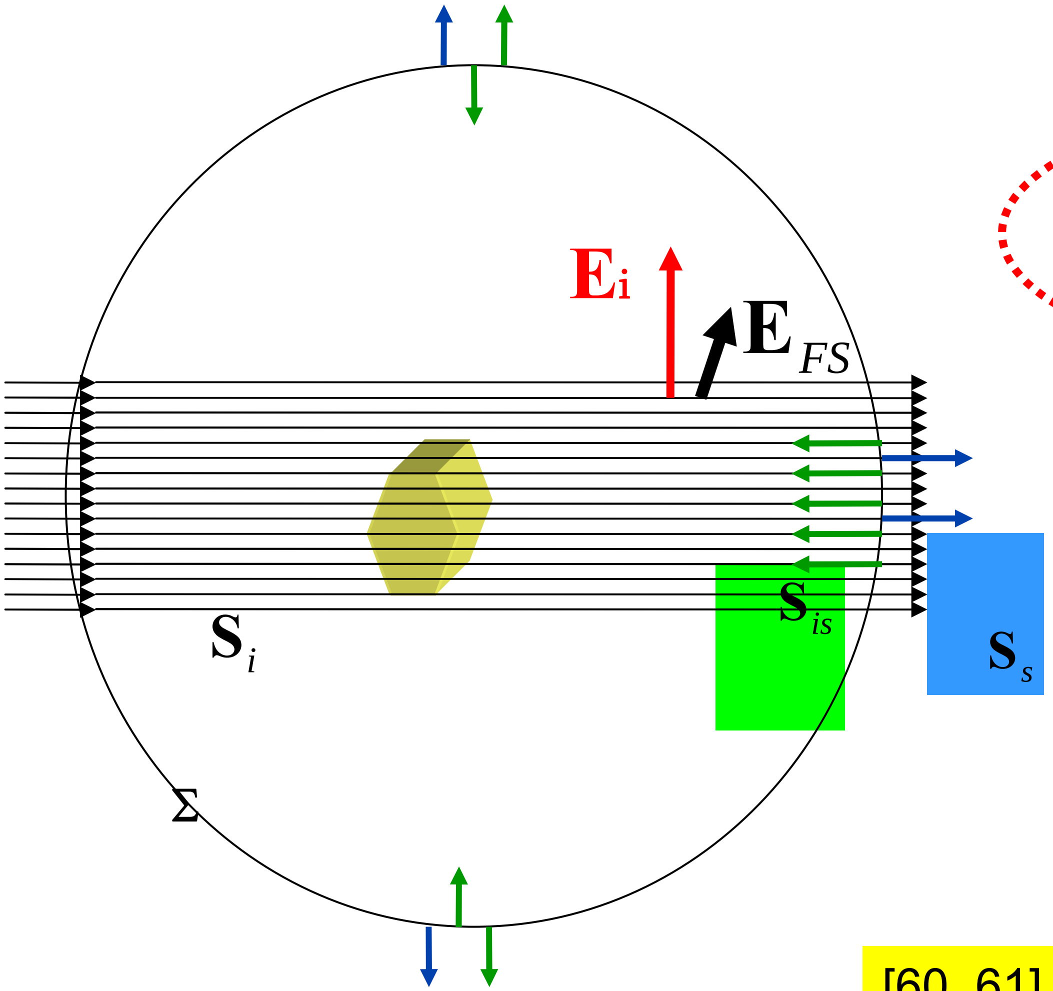
1. **Band-gaps detection [57].**
2. **For low-loss structures: phase(T) – dispersion along ΓX [58]**





3. Dispersion diagaram - almost complete retrieval [59]

Scattering sample's experimental characterization: absorption and extinction coefficients



$|\mathbf{E}_s + \mathbf{E}_i|$, $|\mathbf{E}_i|$.

Spectrometer

Sometimes additionally:

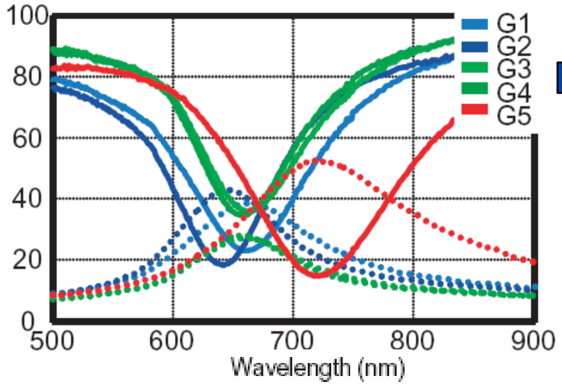
Phase($\mathbf{E}_{FS} + \mathbf{E}_i$), Phase($\mathbf{E}_i$),

Interferometer

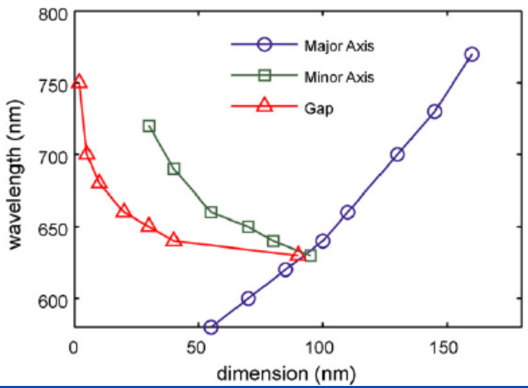
[60, 61]

Experimental characterization of mesoscopic layers

|R|, |T|



λ_{res} (d, *g*)



Spectroscopic (not complete) characterization:

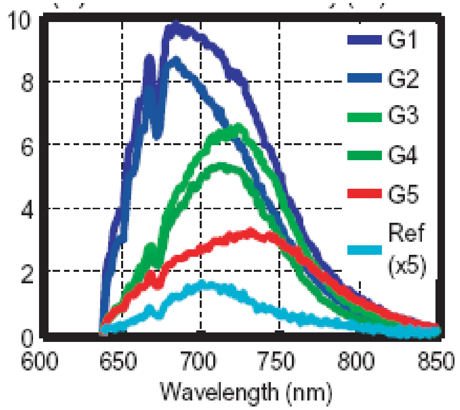
[61-63]

Nnonlinear characterization

[64]

Fluorescent layer

Fluorescence enhancement

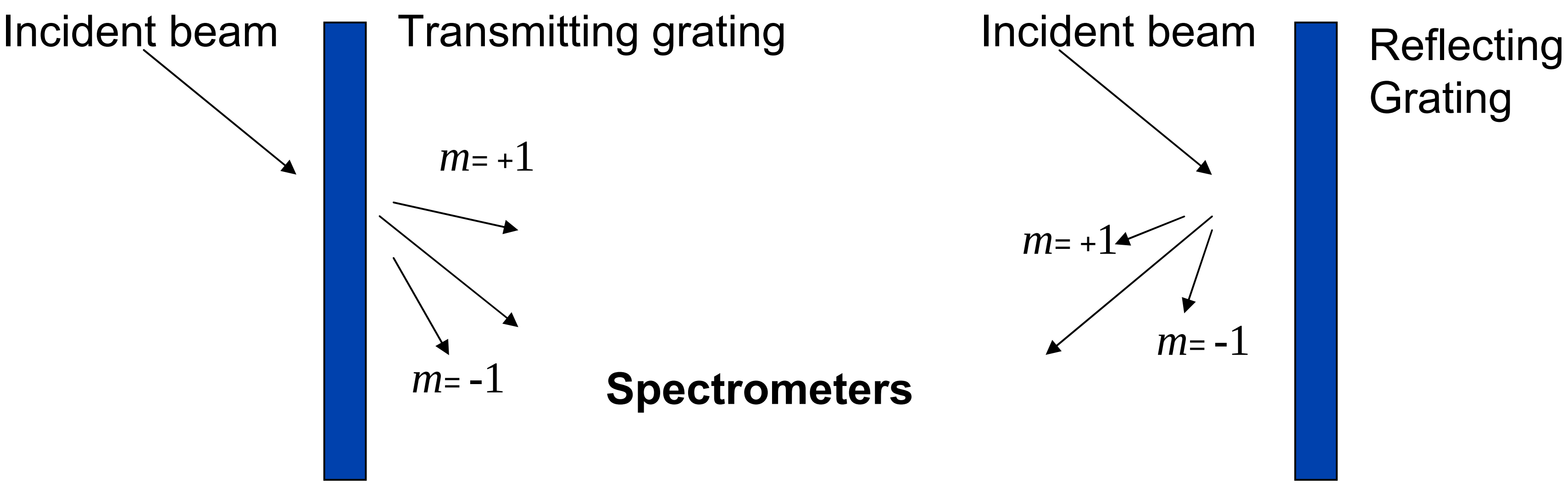


Output parameters:
1) Averaged Field Enhancement vs λ,
2) Local Field Enhancement vs λ

Experimental characterization of diffraction gratings

- **Normal incidence λ>d, oblique incidence λ>2d:**
- **|R| or |T| (λ). Plasmonic gratings: absorption coefficient (at Wood anomalies)**
- **Normal incidence λ<d, Oblique incidence λ<2d:**
- **Angular dispersion D(λ, m), where m=±1.. ±[d/λ] grating spectral orders.**
- **3. Free intervals of dispersion $\Delta\lambda$(m). 4. Normalized intensity distribution $I_{max}(\lambda,m)$.**

[60]



What should you take into consideration

Estimate: the class to which your NSM belongs

Availability of the needed equipment

Previous experience of the equipment owners and lab staff in EM characterization

Access conditions

What kind of equipment?

- Radio range: **network analyser**
- Optics: Spectrometers: **|R,T| (ω)**
- Ellipsometers: **polarisation ellipse (VASE – also |R, T| (ω))**
- Interferometers: **phase(R,T)**
- Optical radiation sources: **laser, emitter + tunable filter,**
- Special microscopes (SEM, TEM, AFM, aSNOM etc) : **internal geometry**
- Other: **chemical analysis tools (elemental characterization)**

EM charact. parameters are *derivative parameters*

- A list of the equipment (with some technical data) and a list of these facilities hosting institutions can be found at
- Disclaimer: The information has been collected taking into account the expertise of the facilities owners in EM characterization and their interest.

What kind of expertise is available?

Table 1. Samples (classes and measurement techniques)

Materials Types	Slabs	Wedges	Bulk samples or bars	Substrate	Layer(s) on a substrate	Sub-wave length samples	Other?
Isotropic materials							
Photonic crystals							
Quasicrystals							
Mesoscopic samples							
Bianisotropic							
Anisotropic inversion symmetrical							
Active materials							
Controllable materials							
Diffraction gratings							
Scattering media							
Other?							

This interactive table with filled cells is available at

http://econam.metamorphose-vi.org/facilities/by-materials-and-samples-types

Claimed expertise

See on these laboratories at http://econam.metamorphose-vi.org/ facilities/by-laboratories

Materials types	Slabs	Complex shape object	Bulk samples or bars	Substrate	Layer(s) on substrate	Sub-wave length samples	Thin films
Isotropic materials	LPC AMOLF USPI JENA INT ORC	AMOLF USPI INT ORC	LPC USPI INT ORC	LPC RWTH USPI INT ORC	KIT AMOLF RWTH USPI INT JENA ORC	AMOLF NBTG RWTH USPI INT ORC	LPC AMOLF RWTH USPI INT ORC
Photonic crystals	LPC AMOLF USPI JENA INT ORC	AMOLF USPI INT ORC	LPC USPI INT ORC	LPC RWTH USPI INT ORC	KIT AMOLF RWTH USPI JENA INT ORC	AMOLF NBTG RWTH USPI INT ORC	LPC AMOLF RWTH INT ORC
Quasicrystals	RWTH USPI JENA INT ORC	USPI INT ORC	USPI INT ORC	RWTH INT ORC	KIT RWTH USPI INT ORC JENA	NBTG USPI RWTH INT ORC	RWTH INT ORC
Mesoscopic samples	USPI JENA INT ORC	USPI INT ORC	USPI INT ORC	INT ORC	USPI JENA INT ORC	USPI INT ORC	INT ORC

Bianisotropic	AMOLF USPI JENA INT ORC	USPI INT ORC	USPI INT ORC	INT ORC	AMOLF USPI JENA INT ORC	AMOLF NBTG USPI INT ORC	AMOLF INT ORC
Anisotropic inversion symmetrical	AMOLF JENA INT ORC	INT ORC	INT ORC	INT ORC	AMOLF JENA INT ORC	AMOLF NBTG INT ORC	AMOLF INT ORC
Active materials	INT ORC	INT ORC	INT ORC	INT ORC	INT ORC	TUI NBTG INT ORC	INT ORC
Controllable materials	INT ORC	RWTH INT ORC	INT ORC	INT ORC	KIT RWTH USPI ORC INT	NBTG INT ORC	RWTH INT ORC
Diffraction gratings	USPI INT ORC	INT ORC	USPI INT ORC	INT ORC	INT ORC	INT ORC	INT ORC
Scattering media							
Other		RWTH	RWTH	RWTH	KIT RWTH USPI	TUI RWTH USPI	

Service rules

The rules for use of the facilities in terms of expenses reimbursement and profit sharing differ from lab to lab:

1. Non-for-profit use only or /and
2. Non-for-profit use for national institutions or other bodies or /and
3. Commercial use for any external customer

No ready contracts templates.

Owners prefer to shape contract agreements for each particular case.

We recommend to use DESCA agreement template as a starting point to prepare such contracts.
These recommendations and links can be found at http://econam.metamorphose-vi.org/facilities/access-rules

Equipment

	Elipsometers	*Interferometers*	*Spectrometers*	*Microscopes*	*Radiation sources*	*Other*
JENA		+	+	+	+	+
USPI	+		+	+	+	
RWTH	+	+	+	+	+	
NBTG			+	+	+	
TUI			+			
AMOLF	+		+	+	+	+
LPC		+		+	+	
KIT			+	+		+
INT		+	+	+	+	+
ORC			+	+	+	

Related expertise

	Elipsometry	*Interferometry*	*Spectrometry*	*Microscopy*	*Fabrication*	*Other*
JENA		+	+	+		+
USPI	+		+	+	+	
RWTH			+	+		
NBTG			+	+	+	
TUI			+	+		
AMOLF	+		+	+		
LPC						
KIT		+	+			
INT		+	+	+	+	
ORC			+	+		

Statistics on the equipment and facilities Information collected at the ECONAM website

- **Number of the referred equipment items:**
- **Spectrometers:……………………………….24**
- **Ellipsometers: polarisation rotation…………3**
- **Interferometers: phase………………………..6**
- **Radiation sources: ……………………………33**
- **Microscopes: internal geometry………………36**
- **Other: 4**
-
- **Number of the contact points: 10**
- **Number of the samples types combination: 68**
- **Frequency ranges of expertise: THz, Optical (IR, Visible)**

Suggested experimental characterization procedure

1. Check the external geometry and guess the internal geometry of your sample;
2. Choose the equipment owners with the corresponding expertise (or expected expertise) (Table 1.: Samples map on the ECONAM website);
3. Contact the owners and agree the conditions for possible cooperation ("Contacts and other information" database on the ECONAM website);
4. Decide what kind of parameters do you want to derive;
5. Agree the procedure of measuring (|R,T|, phases, polarization etc.) for your particular sample and source location;
6. Get the measured data and do post-processing
7. Apply the recommended technique to get desired derivative parameters (if there is such a technique).
8. Redo measurements (e.g. in case of iterative techniques) and make verification experiment if needed.

Some cautions for non-EM experts

- **Some groups do not reveal the important information how do they determine proper characteristic parameters and how retrieve them. Usually to protect their know-how. Our approach: do not try to reproduce! Determine and post-process characteristic parameters yourself. Ask our experts to follow the scientifically recommended characterization procedures**

- **http://econam.metamorphose-vi.org**

- **Uf-f! My respect to those who survived this talk**

- **Do not blame those who has aslept**

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- There is also a long additional references' list