



Italian National Committee

Commission A: Electromagnetic Metrology, Electromagnetic measurements and standards

Dielectric characterization of materials at microwave frequencies

DR. ERIKA PITTELLA



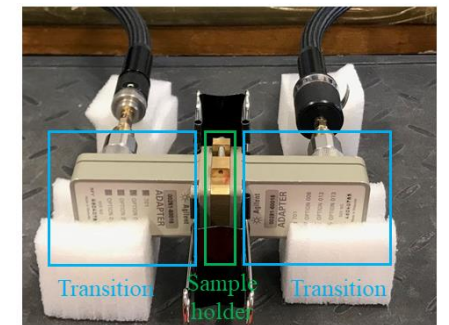
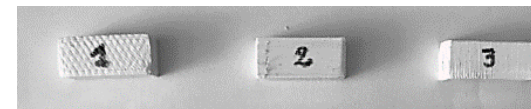
SAPIENZA
UNIVERSITÀ DI ROMA



*Department of Information Engineering, Electronics and Telecommunications
(DIET)*

Microwave systems for material characterization and monitoring

- Cultural Heritage Monitoring
- Health Monitoring in Concrete Structures
- Composite Materials Characterization

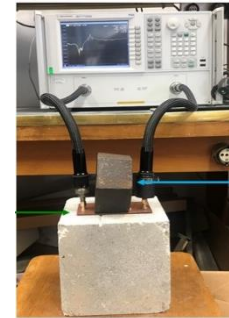


Microwave systems for material characterization and monitoring

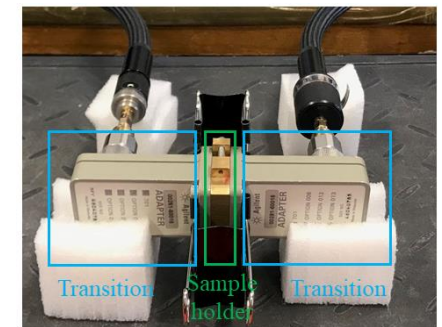
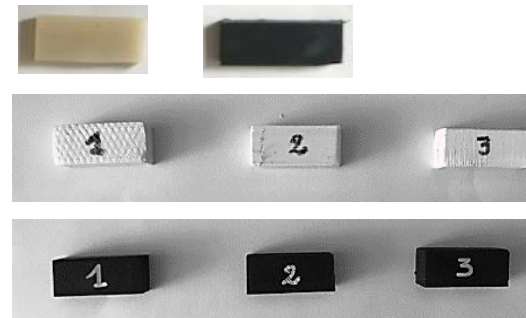
- **Cultural Heritage Monitoring**



- Health Monitoring in Concrete Structures



- Composite Materials Characterization



Cultural Heritage Monitoring

Aim:

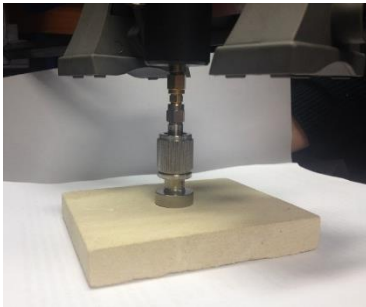
non-invasive microwave technique for moisture monitoring in historical buildings



Cultural Heritage Monitoring

Different methods and probes were comparatively assessed to infer, noninvasively, the **water content** of stone materials starting from **dielectric properties at microwave frequencies**

Open Ended Coaxial
(OEC)



Open Ended Waveguide
(OEW)



Patch Resonator
(PR)



Agilent E8363C
VNA

Campbell Scientific
TDR100 reflectometer

- allow noninvasive approach
- easy to use in the field
- can be realized in low cost versions

Cultural Heritage Monitoring

$$\varepsilon_{r,stone} \cong 5 - 6 \quad (\text{dry stone})$$

$$\varepsilon_{r,water} \cong 78 \quad (25^\circ \text{ C})$$

GENTILE
STONE



3.0 cm × 7.7 cm × 10.1 cm

LECCESE
STONE



1.5 cm × 8.2 cm × 10.1 cm

SAMPLE MOISTENING PROCEDURE:

- (1) drying of the sample (volume V_{stone} in cm^3) in a microwave oven;
- (2) weighing of the dry sample, W_{dry} (in g);
- (3) bath in deionized water until saturation (which, for the considered samples, took less than two hours);

(4) weighing of the sample, W_i (in g);

(5) assessment of the gravimetric moisture θ_v :

$$\theta_v = \frac{W_i - W_{dry}}{V_{stone} \cdot \rho_w} \times 100;$$

(6) measurements through the different probes and instruments;

(7) oven drying for a limited amount of time, in order to remove part of the moisture.

Open-ended coaxial (OEC) probe

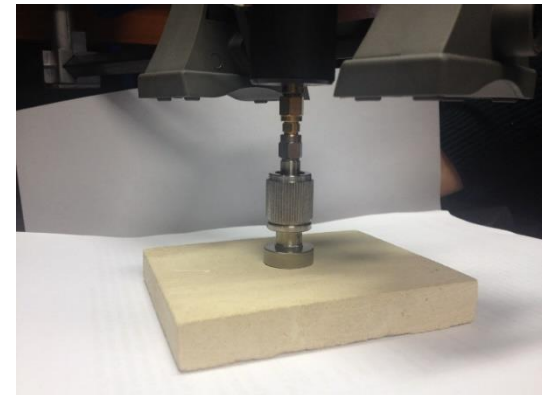
KEYSIGHT HIGH TEMPERATURE PROBE



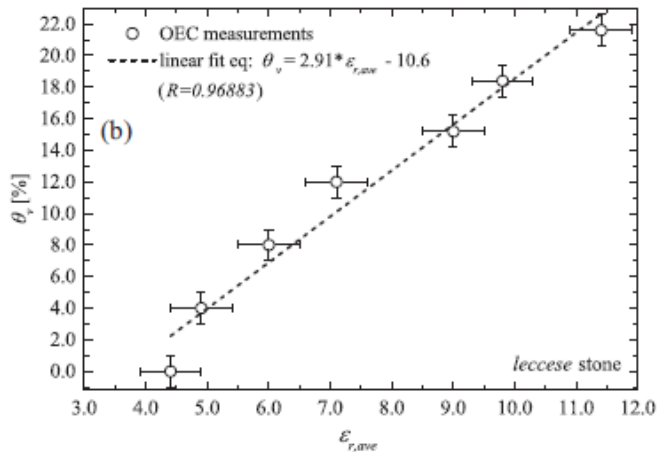
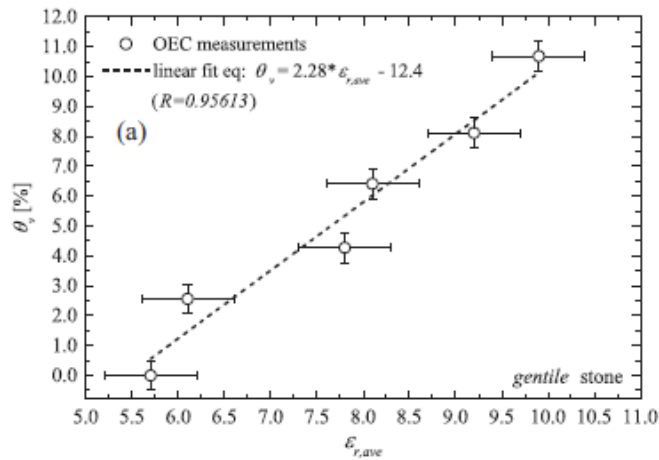
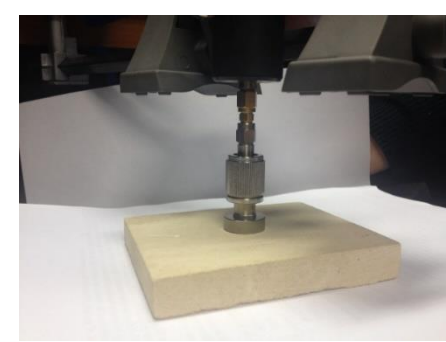
- range: 200 MHz to 20 GHz
- suitable for permittivity measurements on liquids and solid materials with flat surfaces
- Diameter sample > 2 cm and thickness $> \frac{2}{\sqrt{\epsilon_r}}$ cm

1. System calibration using air, shorting block and de-ionized water
2. S_{11} measured through a VNA
3. $S_{11} \rightarrow \epsilon_r'$ (custom software [Piuizzi et al. 2013])
4. $\epsilon_{r,ave}$ on the measurement band

$$\epsilon_{r,ave} \longleftrightarrow \theta_v$$

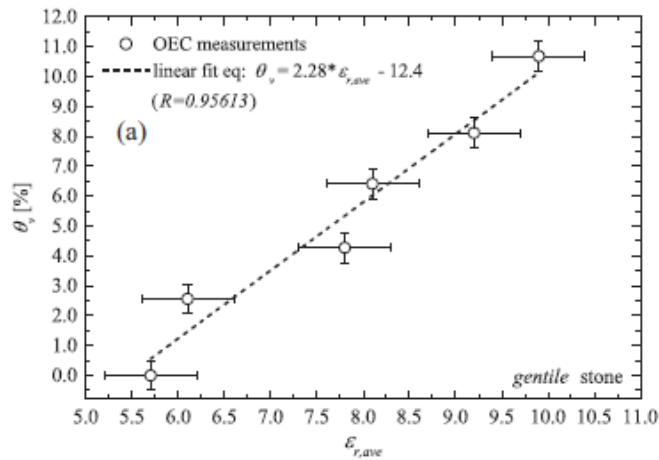
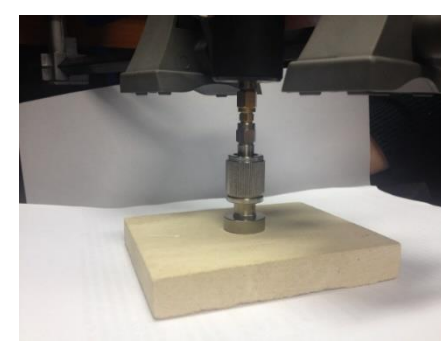


OEC



- Good linearity θ vs $\epsilon_{r,ave}$
- Vertical error bars: due to water evaporation during measurement, it can be made negligible by avoiding oven drying
- Horizontal error bars: **scarse measurement repeatability**
 1. the VNA port cable was kept still, through a mechanical vice; the inevitable mechanical pressure resulted in slight movements, which (although very little) led to movements of the cable changing its phase response
 2. the sensing volume of the probe is limited, the stone non homogeneous and the probe is positioned in different portions of the sample.

OEC

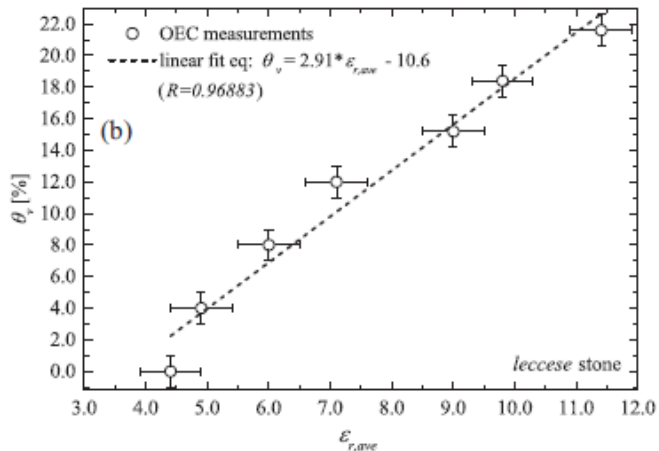


GENTILE

$$\vartheta_v = 2.28\epsilon_r - 12.4$$

LECCESE

$$\vartheta_v = 2.91\epsilon_r - 10.6$$



SENSITIVITY
0.35-0.45

MEASUREMENT
REPEATABILITY

$$WCU_{\epsilon,rep} = 0.5$$

INSTRUMENTAL
MEASUREMENT
UNCERTAINTY

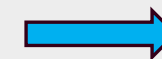
$$WCU_{\epsilon,syst} = 0.05$$

negligible

$$WCU_{\vartheta_{v,G}} = 1\%$$

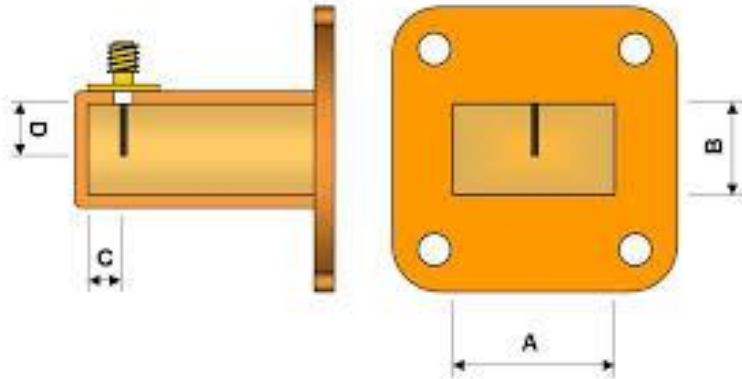
$$WCU_{\vartheta_{v,L}} = 1.5\%$$

Care in the positioning
Scarce repeatability
Costs of the system (VNA + probe)
On-site calibration needed (complex)



Not suitable
for in-the-field
applications

Open-ended waveguide (OEW) probe



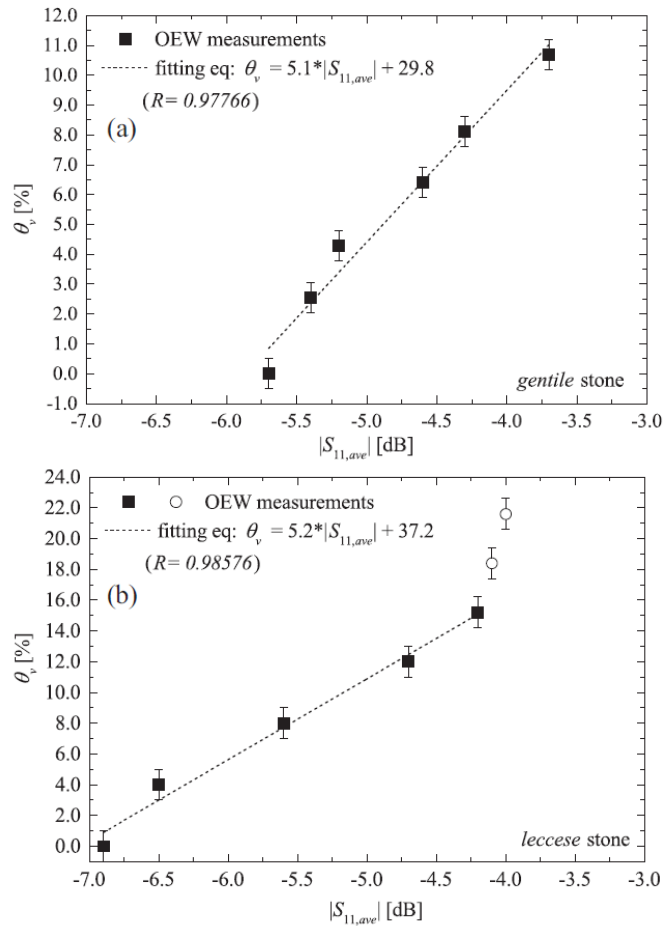
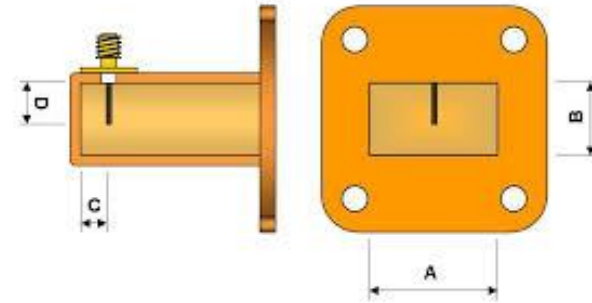
- frequency range: 8.2 GHz to 12.4 GHz (WR90 waveguide)
- dimensions: $A = 22.86 \text{ mm}$
 $B = 10.16 \text{ mm}$

1. System calibration using a TRL mechanical kit
2. S_{11} measured through a VNA
3. $|S_{11,ave}|$ over the measurement band

$$|S_{11,ave}| \longleftrightarrow \theta_v$$

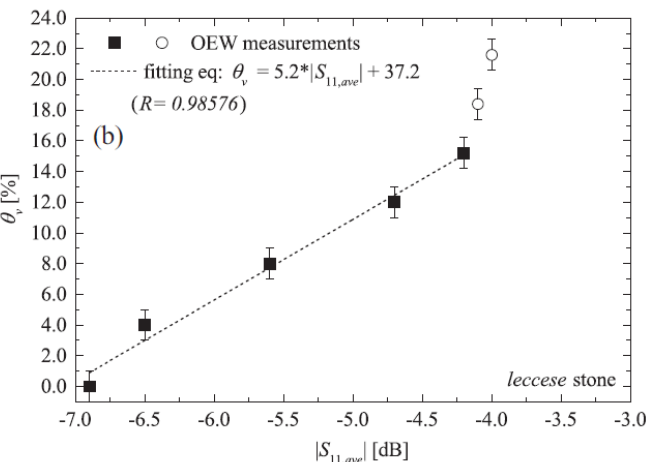
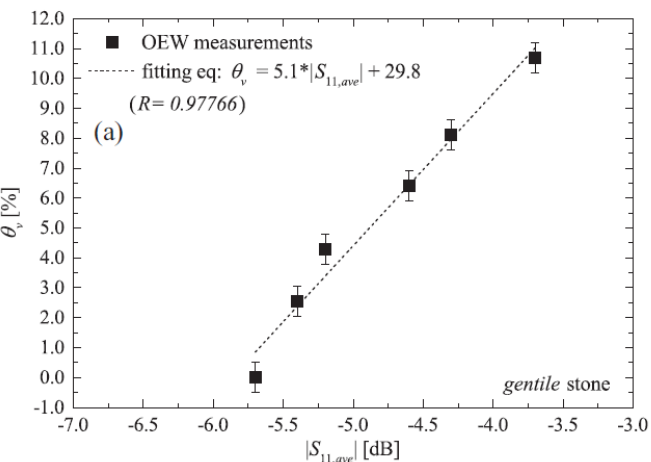
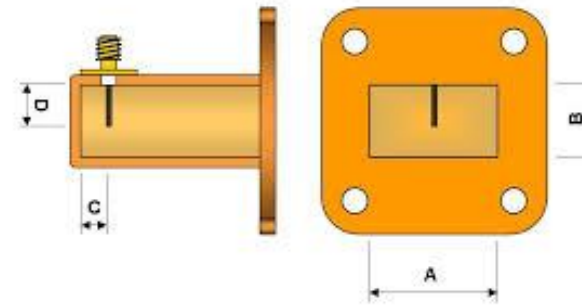


OEW



- $|S_{11,ave}|$ increases linearly with the water content, except for $\vartheta_v > 15\%$ (value well beyond commonly experienced moisture levels)
- almost the same angular coefficients (similar S_{11} behaviour for the 2 stones)

OEW



GENTILE

$$\vartheta_v = 5.1|S_{11,ave}| + 29.8$$

LECCESE

$$\vartheta_v = 5.2|S_{11,ave}| + 37.2$$

SENSITIVITY
0.2 dB

MEASUREMENT
REPEATABILITY

$$WCU_{S_{11},rep} = 0.18 - 0.23 \text{ dB}$$

INSTRUMENTAL
MEASUREMENT
UNCERTAINTY

$$WCU_{S_{11},syst} = 0.1 \text{ dB}$$

COMBINED STANDARD UNCERTAINTY

$$\sigma_{S_{11},rep-G} = 0.13 \text{ dB} \quad \sigma_{S_{11},rep-L} = 0.14 \text{ dB}$$

$$u_{S_{11}C-G} = \sqrt{\sigma_{S_{11},syst}^2 + \sigma_{S_{11},rep,G}^2} = 0.14 \text{ dB}$$

$$\sigma_{S_{11},syst} = \frac{WCU_{S_{11},syst}}{\sqrt{3}} = 0.058 \text{ dB} \quad (\text{uniform distribution})$$

$$u_{S_{11}C-L} = \sqrt{\sigma_{S_{11},syst}^2 + \sigma_{S_{11},rep,L}^2} = 0.17 \text{ dB}$$

EXPANDED UNCERTAINTY

hp: gaussian distribution

$$k_P = 2$$

$$U_{S_{11},95\%-G} = 0.28 \text{ dB}$$

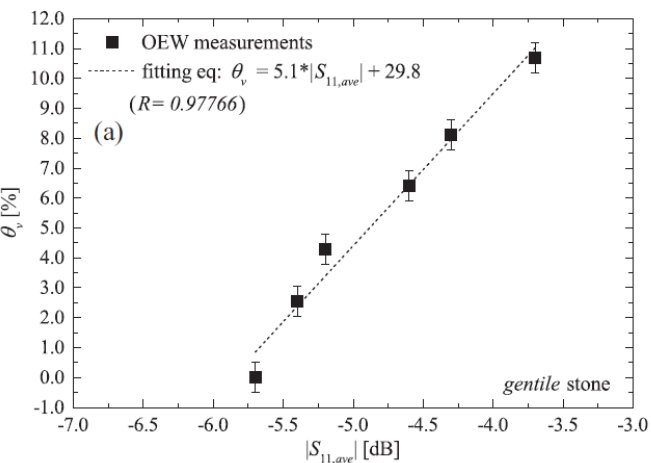
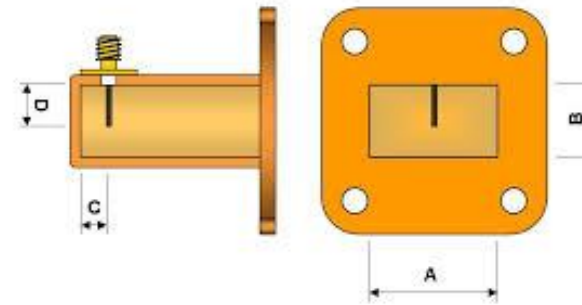
$$U_{S_{11},95\%-L} = 0.34 \text{ dB}$$



$$U_{\theta-G,95\%} = 1.4\%$$

$$U_{\theta-L,95\%} = 1.7\%$$

OEW



GENTILE

$$\theta_v = 5.1 |S_{11,ave}| + 29.8$$

LECCESE

$$\theta_v = 5.2 |S_{11,ave}| + 37.2$$

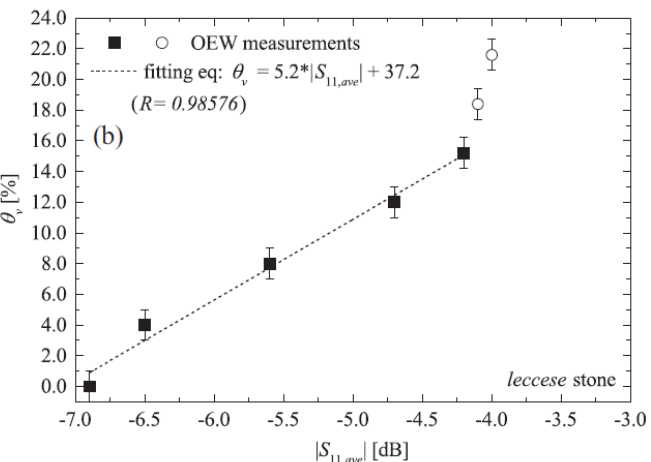
SENSITIVITY
0.2 dB

**MEASUREMENT
REPEATABILITY**

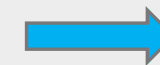
$$WCU_{S_{11},rep} = 0.18 - 0.23 \text{ dB}$$

**INSTRUMENTAL
MEASUREMENT
UNCERTAINTY**

$$WCU_{S_{11},syst} = 0.1 \text{ dB}$$

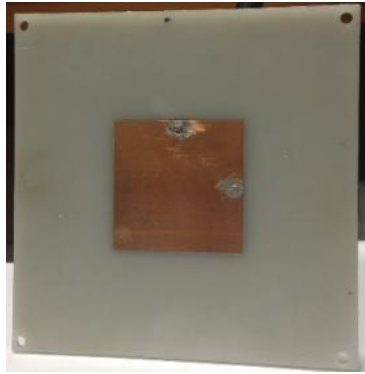


Some care in the positioning
 Scarce repeatability
 Costs of the system (VNA)
 On-site calibration needed (easy)



Does not seem
 a good solution
 for in-the-field
 applications

Patch resonator (PR) probe



Dielectric and geometric characteristics of the patch resonator.

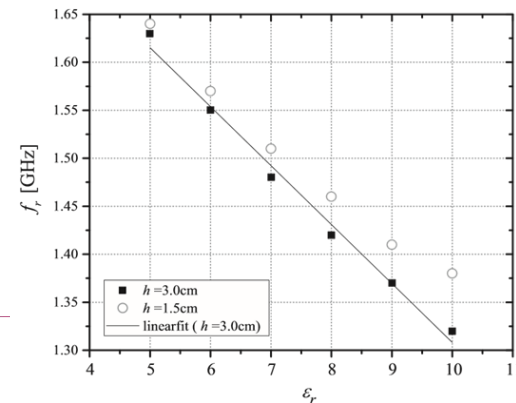
Parameter	Description
Substrate material	Polyflon
Substrate dielectric characteristics	$\epsilon_{r,sub} = 2.32$ $\text{tg } \delta = 0.0002$
Substrate thickness	3.175 mm
Metallization thickness	35.6 μm (copper)
Patch dimensions	4.8 \times 4.8 cm
Resonant frequency (in air)	$f_r \cong 2 \text{ GHz}$

1. S_{11} measured through VNA or TDR
2. $S_{11} \rightarrow f_r$

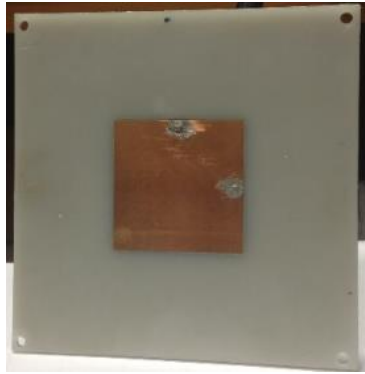
$$\begin{array}{l} f_r \rightarrow \epsilon_r \\ \epsilon_r \leftrightarrow \theta_v \end{array}$$



Preliminary full-wave simulations CST MWS



Patch resonator (PR) probe



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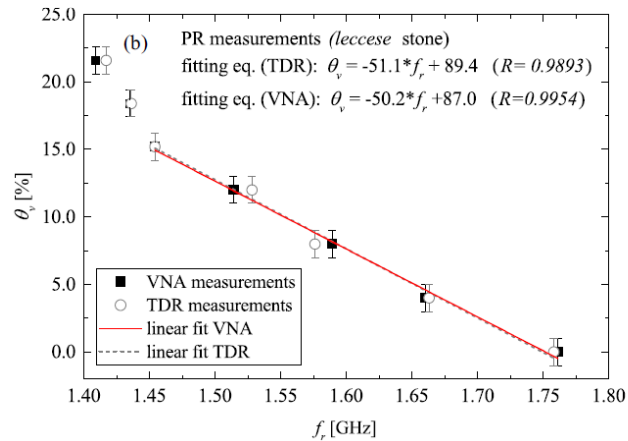
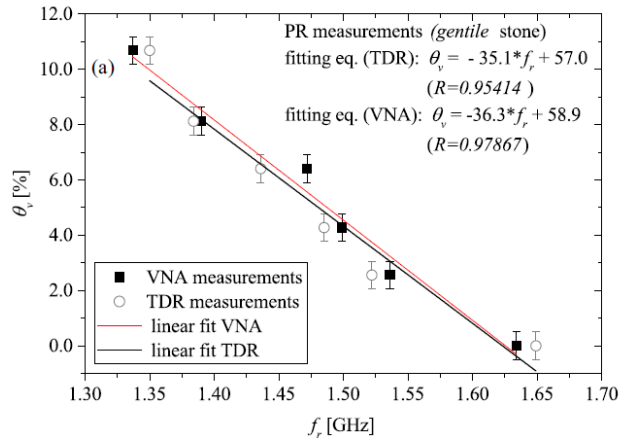
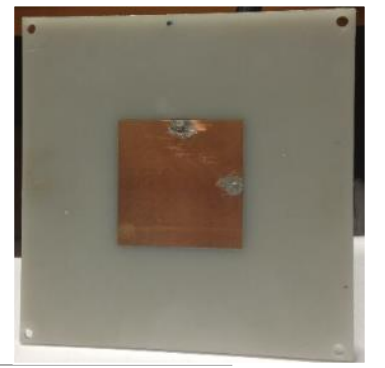
1. S_{11} measured through VNA or TDR

2. $S_{11} \rightarrow f_r$

$$f_r \longleftrightarrow \theta_v$$

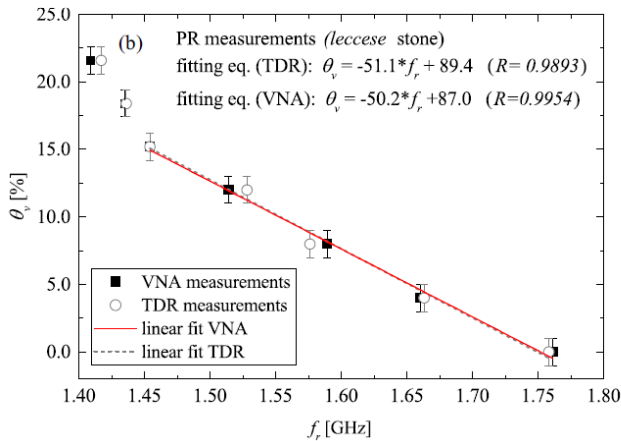
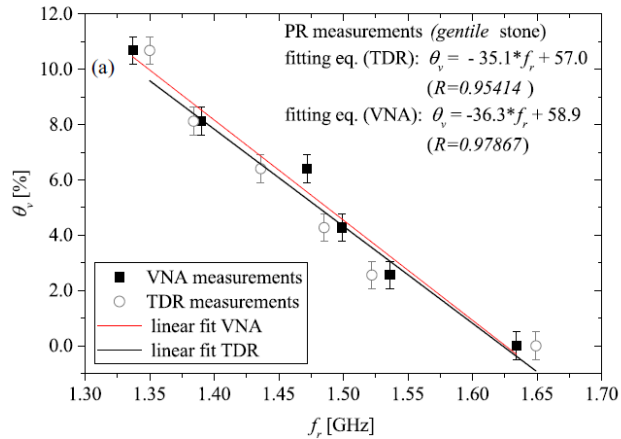
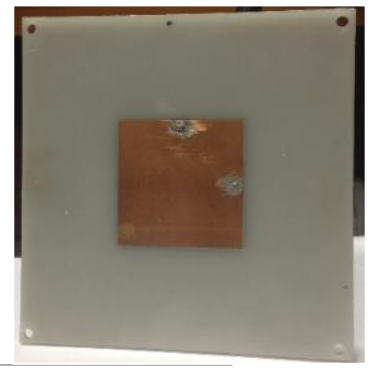


PR



- linear relationship between the two quantities
- f_r decreases linearly with the water content, except for $\vartheta_v > 15\%$ (value well beyond commonly experienced moisture levels)

PR



GENTILE

$$\vartheta_{v\%} = -40f_r + 59$$

LECCESE

$$\vartheta_{v\%} = -50f_r + 88$$

SENSITIVITY
0.020–0.025 GHz

**MEASUREMENT
REPEATABILITY**

**INSTRUMENTAL
MEASUREMENT
UNCERTAINTY**

$$WCU_{f_r, rep} = 3.8 \text{ MHz}$$

$$WCU_{f_r, syst} = 2 \text{ kHz}$$

negligible

$$WCU_{\vartheta_v} \cong 0.2\%$$

Repeatability

Ease in positioning

Cheap (TDR solution)

Calibration **not needed**



Suitable
for in-the-field
application

Comparative analysis

	MEAS.	INSTR.		CAL.	REPEAT.	POSITIONING	COST	
OEC	$\varepsilon_r \rightarrow \theta_v$	VNA		LIQUID	SCARSE	DIFFICULT	HIGH	
OEW	$S_{11} \rightarrow \theta_v$	VNA		TRL	SCARSE	SOME CARE	MEDIUM	
PATCH	$f_r \rightarrow \theta_v$	VNA	TDR	NO	HIGH	EASY	MEDIUM	LOW

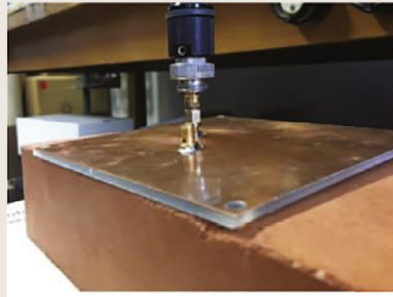
Low cost of the sensor, low-cost instrumentation (TDR), simple positioning, high repeatability, no need for calibration

**PATCH PROBE
SUITABLE FOR
IN THE FIELD APPLICATION**

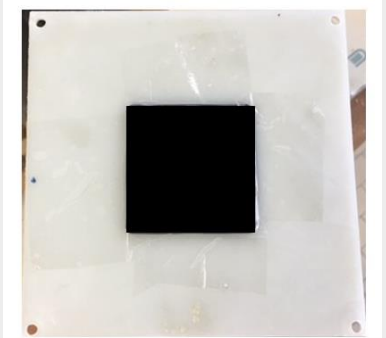
Patch resonator

Types of stones:

- **Gentile**
- **Leccese**
- **Carparo**
- **Red brick**
- **Heat Resistant Red brick**

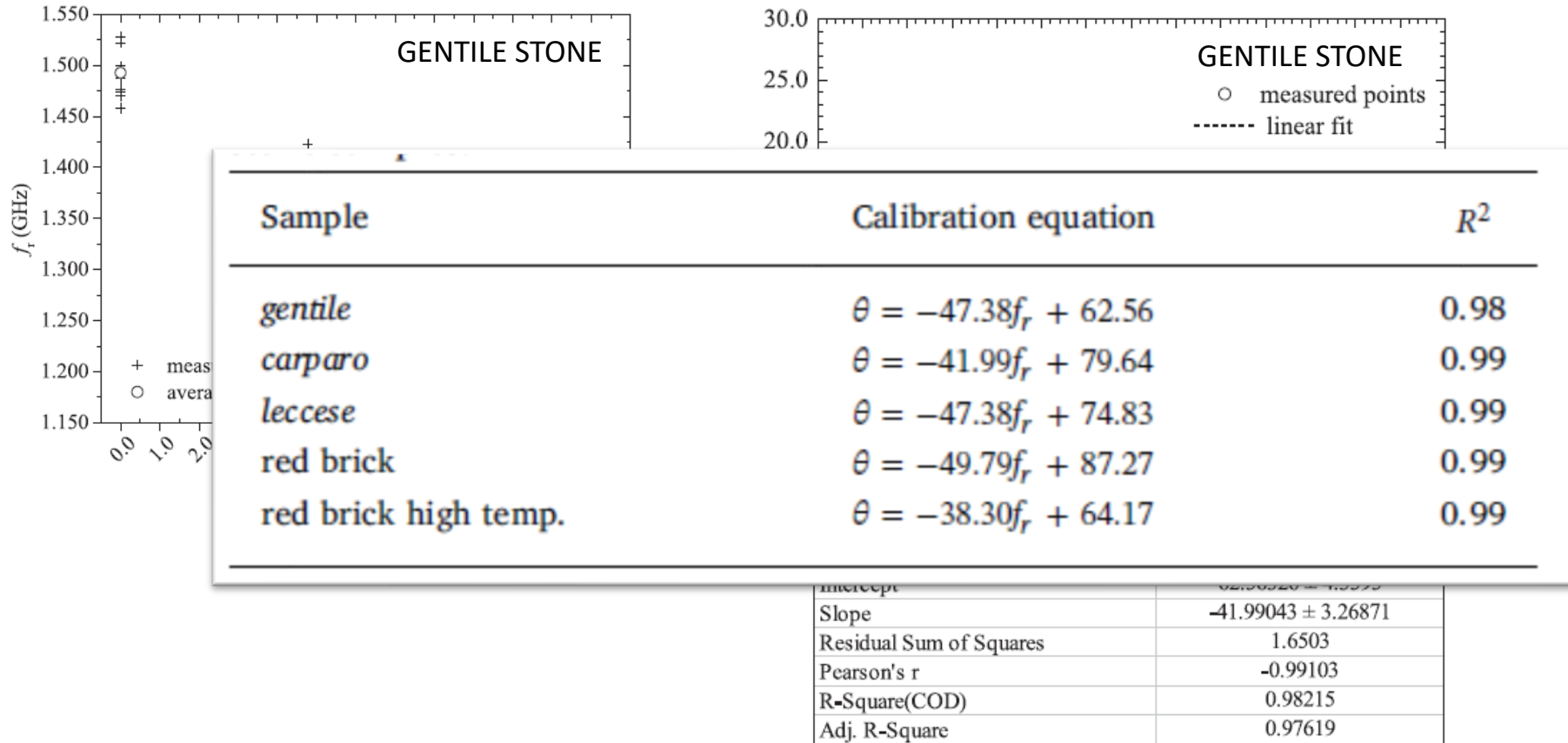


To improve the contact between the planar resonator and the rough surface of the stone, the copper patch was covered with a layer of conductive silicone



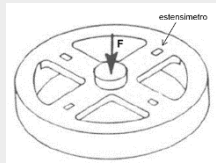
1. For each ϑ_{ref} , ten repeated measurements of $S_{11}(f)$
2. From $S_{11}(f)$ curves, evaluation of the resonance frequency ($f_{r,i}$, $i = 1, \dots, 10$)
3. Evaluation of the average f_r and standard deviation
4. f_r linked to the reference moisture ϑ_{ref}

Stone Results



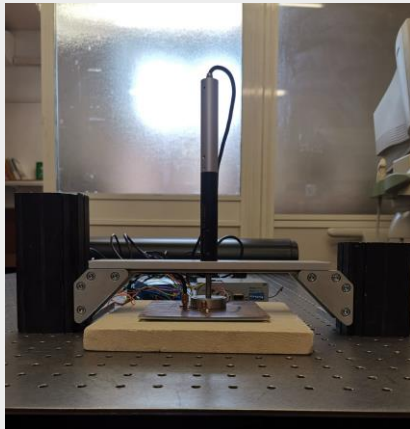
Load cell and portable reflectometry set-up

LOAD CELL

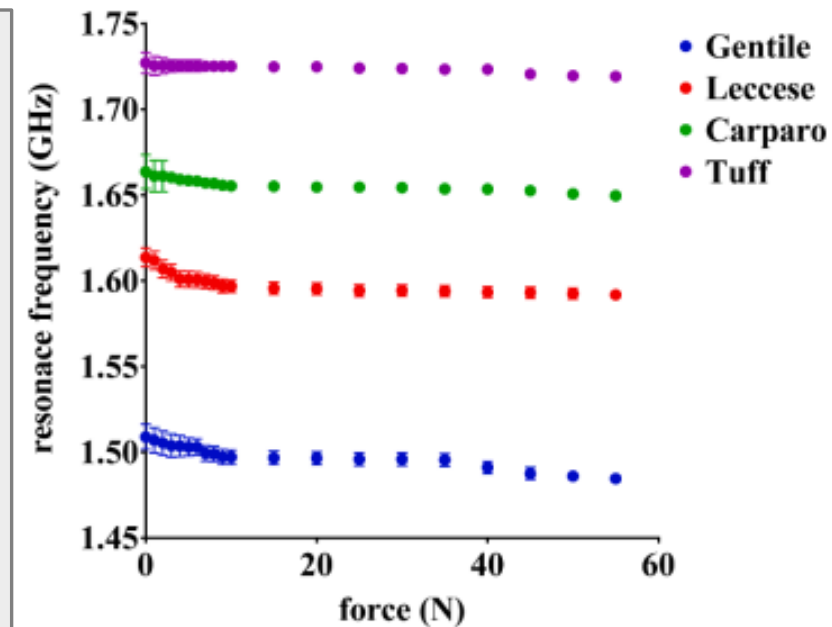


Pancake Cell Features:

- Elastic element subjected to bending
- Ease of implementation and miniaturization
- Patch shape compatibility
- Measuring range: 5 g - 5 kg



miniVNA-TINY



Relationship between the applied force (0 N to 55 N range) and the resonance frequency.

miniVNA-TINY results

Gentile Stone. Comparison between the resonance frequency and the θ_v for the force applied with load cell and without control

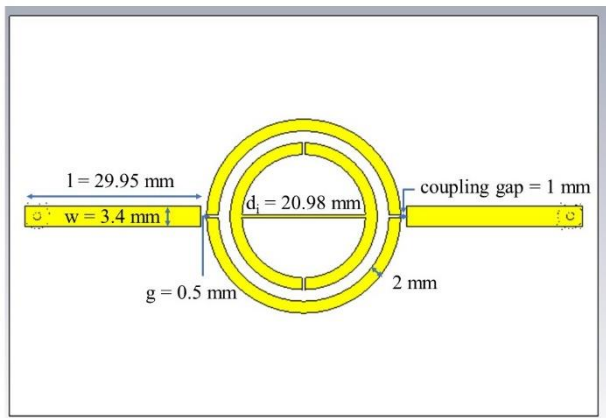
Gentile Stone				
humidity	Load cell		Hand	
	f_r (GHz)	2σ (GHz)	f_r (GHz)	2σ (GHz)
0.0%	1.469	0.061	1.465	0.091
1.8%	1.371	0.053	1.370	0.075
3.6%	1.327	0.025	1.325	0.038
5.5%	1.295	0.036	1.288	0.083
7.3%	1.259	0.014	1.261	0.071
9.1%	1.227	0.029	1.228	0.098
10.9%	1.215	0.028	1.213	0.079
12.8%	1.192	0.043	1.196	0.080
14.6%	1.155	0.021	1.154	0.054

Leccese Stone. Comparison between the resonance frequency and the θ_v for the force applied with load cell and without control

Leccese Stone				
humidity	Load cell		hand	
	f_r (GHz)	2σ (GHz)	f_r (GHz)	2σ (GHz)
0.0%	1.590	0.042	1.591	0.089
3.4%	1.532	0.015	1.547	0.025
8.3%	1.438	0.010	1.417	0.027
10.1%	1.360	0.024	1.373	0.032
13.5%	1.321	0.009	1.327	0.028
16.8%	1.217	0.022	1.211	0.061
20.2%	1.180	0.009	1.175	0.085
23.6%	1.127	0.039	1.128	0.073
26.9%	1.089	0.014	1.086	0.091

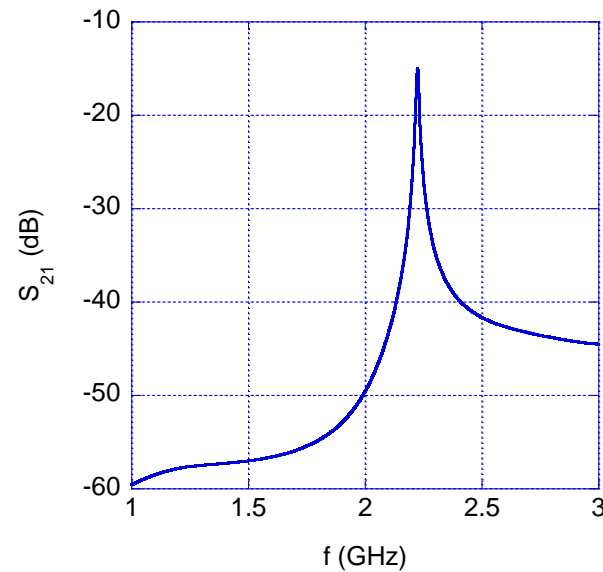
Split Ring Resonator (SRR)

SRR



Resonators characteristic:

- ✓ high sensitivity
- ✓ high quality factor
- ✓ small size
- ✓ do not require a particular sample preparation



Test of the SRR on the following materials:

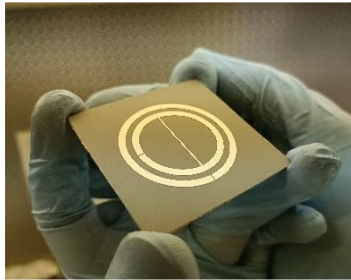
- low density polyvinyl chloride (LDPVC)
- polytetrafluoroethylene (PTFE)
- polymethyl methacrylate (PMMA)
- polycarbonate (PC)

These samples were characterized with the SRR and, for comparison, with the standard reflection/transmission technique, based on the NIST method, through WR430 (at 2 GHz) and WR90 (at 12 GHz) waveguides.

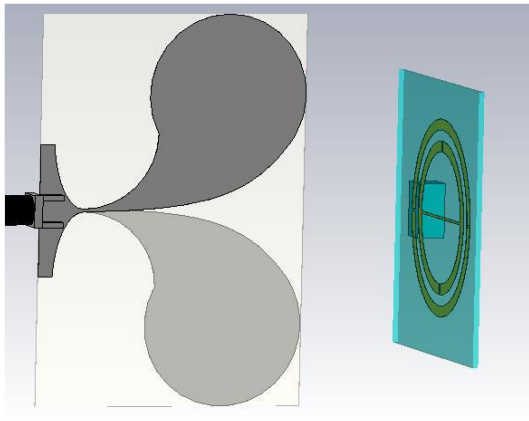
material	ϵ'_r - SRR	ϵ'_r - WR430	ϵ'_r - WR90
LD-PVC	1.6(1)	1.62(3)	1.61(2)
PTFE	1.9(1)	2.06(4)	2.04(2)
PMMA	2.4(1)	2.59(5)	2.59(3)
PC	2.7(1)	2.82(6)	2.81(3)

Passive SRR

used to monitor the dielectric characteristics of a MUT excited through an antipodal Vivaldi antenna



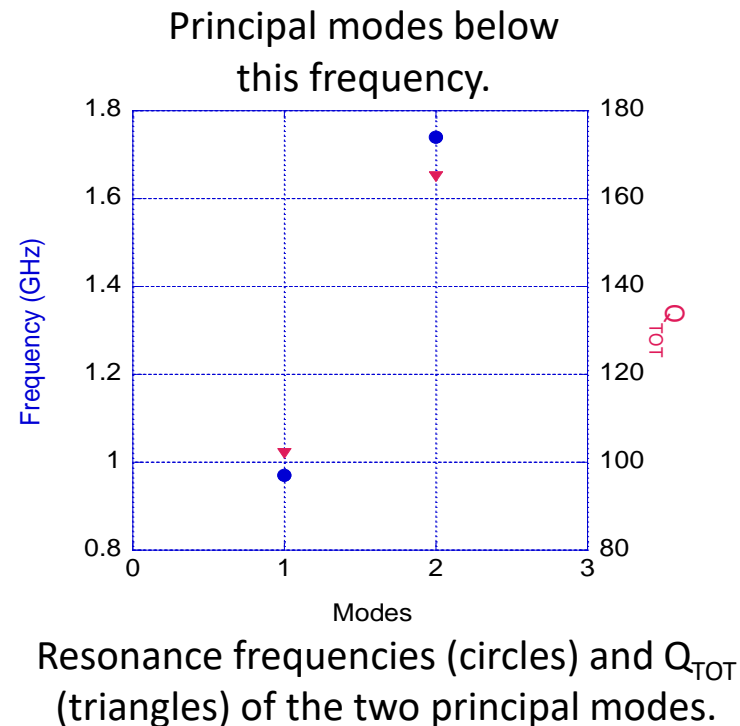
Passive SRR



Antipodal Vivaldi
antenna

Passive
SRR

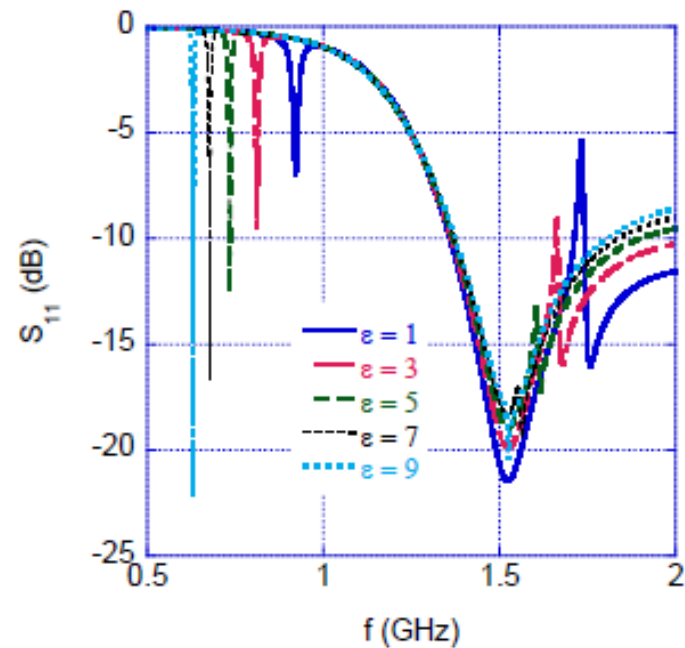
Idea: to use the system with a portable and low-cost VNA with a typical band not exceeding 3 GHz



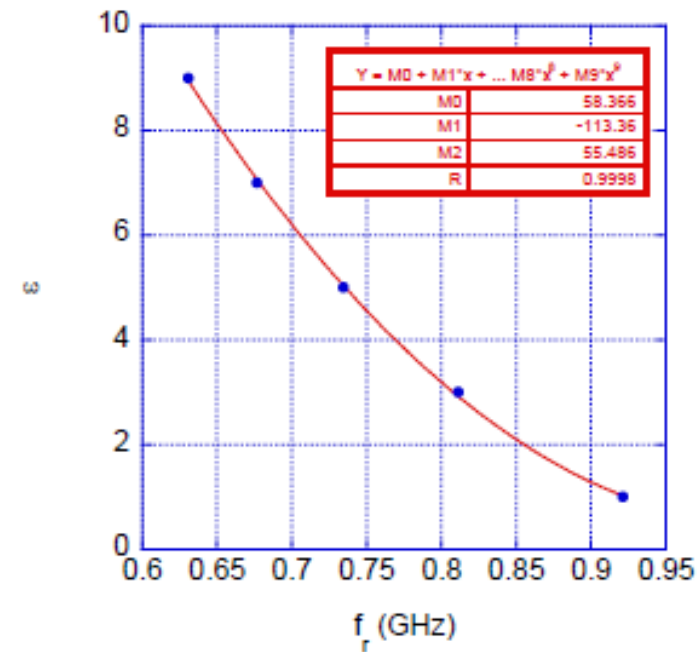
Two principle modes:

- 0.9 GHz ($Q_{TOT} = 102$)
- 1.7 GHz ($Q_{TOT} = 165$).

Simulation results



S_{11} of the antenna in front of the SRR placed on a MUT with varying permittivity.

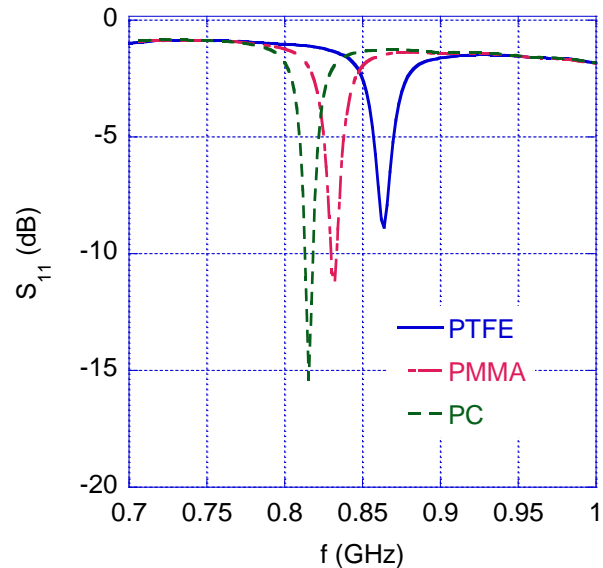


$f_r \rightarrow \epsilon_r$ relationship.

$$\epsilon = 58.366 - 113.36 \cdot f_r + 55.486 \cdot f_r^2$$

Test of the calibration curves

On well-known materials, previously characterized [1]: PTFE, PMMA, and PC



Material	f_r (GHz)	$\epsilon_r(f_r)$	$\epsilon_r [1]$
PC	0.81543	2.82	2.82
PMMA	0.83225	2.46	2.59
PTFE	0.86402	1.84	2.06

Table. Permittivity of test materials computed with the proposed system and comparison with reference data.

maximum relative error is below 10%

[1] A. Alimenti, **E. Pittella**, K. Torokhtii, N. Pompeo, E. Piuze, E. Piuzzi and E. Silva, "A Dielectric Loaded Resonator for the Measurement of the Complex Permittivity of Dielectric Substrates," in *IEEE Trans. Instr. Meas.*, 2023.

E. Pittella, A. Cataldo, M. Cavagnaro, L. D'Alvia, F. Fabbrocino, E. Piuzzi, "Wireless Sensing of Permittivity for Cultural Heritage Monitoring Using a Passive SRR", IMEKO TC4 International Conference on Metrology for Archaeology and Cultural Heritage, MetroArchaeo 2023.

Measurement results on wood samples

Wood types:



FIR

POPLAR

BEECH

OAK

All samples have the following
transversal dimensions:
22.86 mm × 10.16 mm

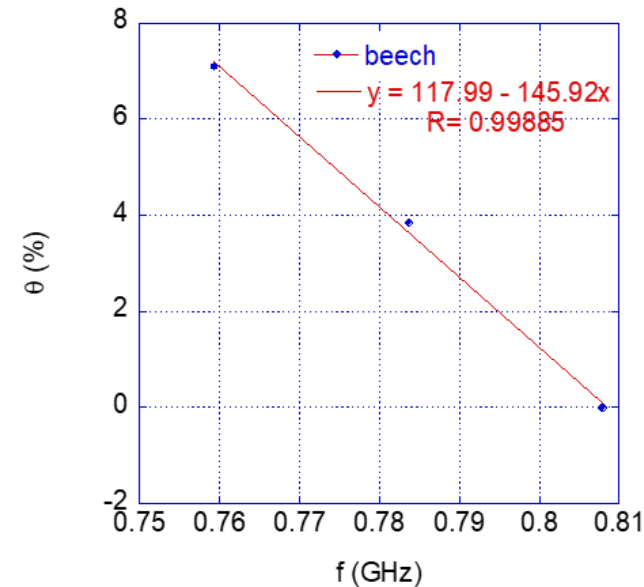


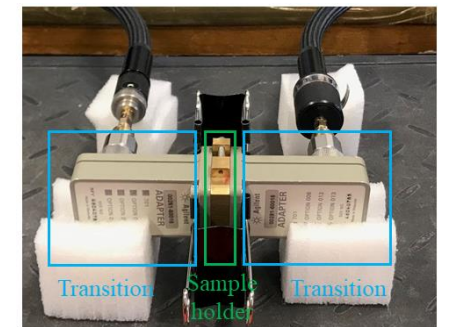
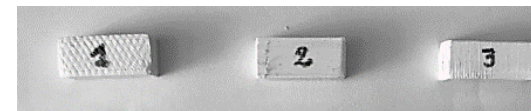
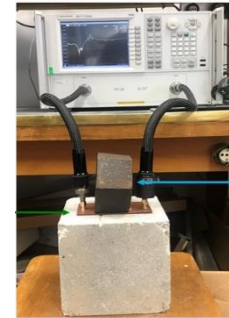
Fig. Calibration curve for θ
measurements on beech
samples.

Material	θ_2 (%)	θ_3 (%)
beech	3.840	7.09
fir	22.72	25.00
oak	4.09	7.37
poplar	8.13	14.29

Table. Gravimetric water content
of the tested samples.

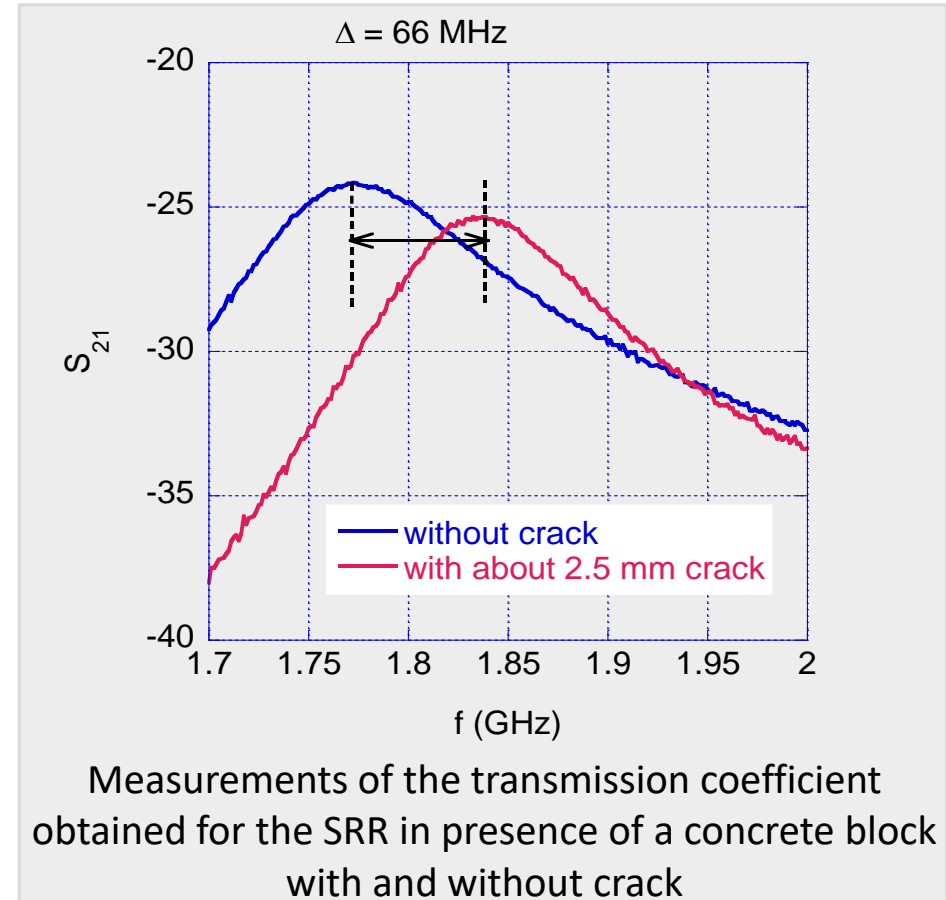
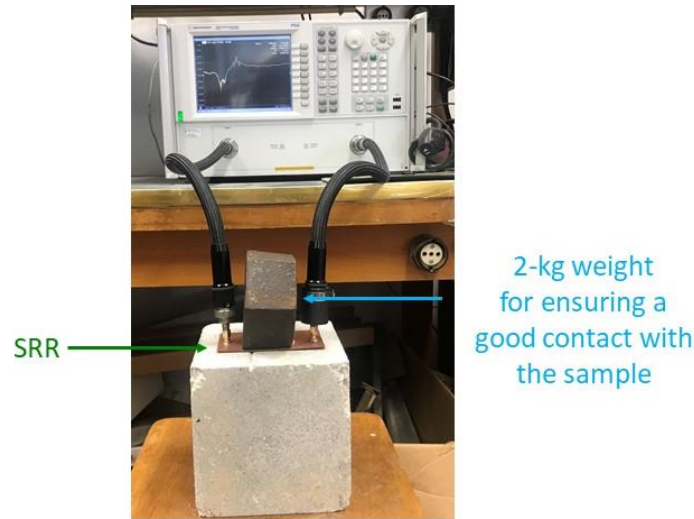
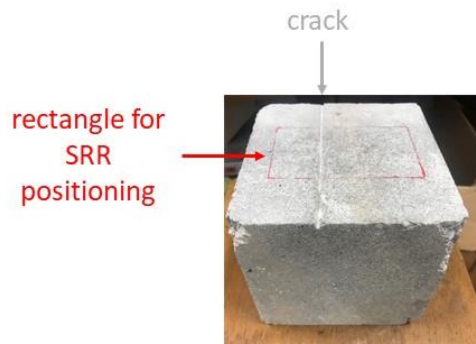
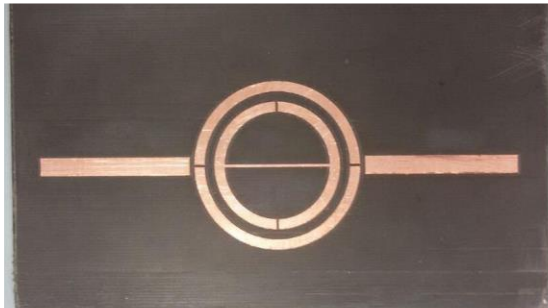
Microwave system for material characterization and monitoring

- Cultural Heritage Monitoring
- **Health Monitoring in Concrete Structures**
- Composite Materials Characterization



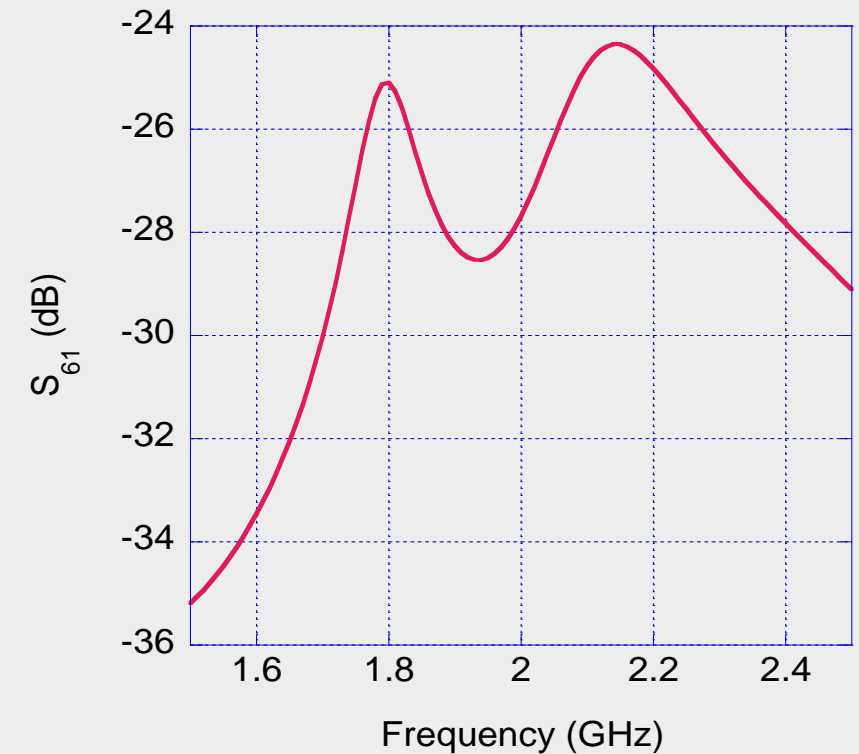
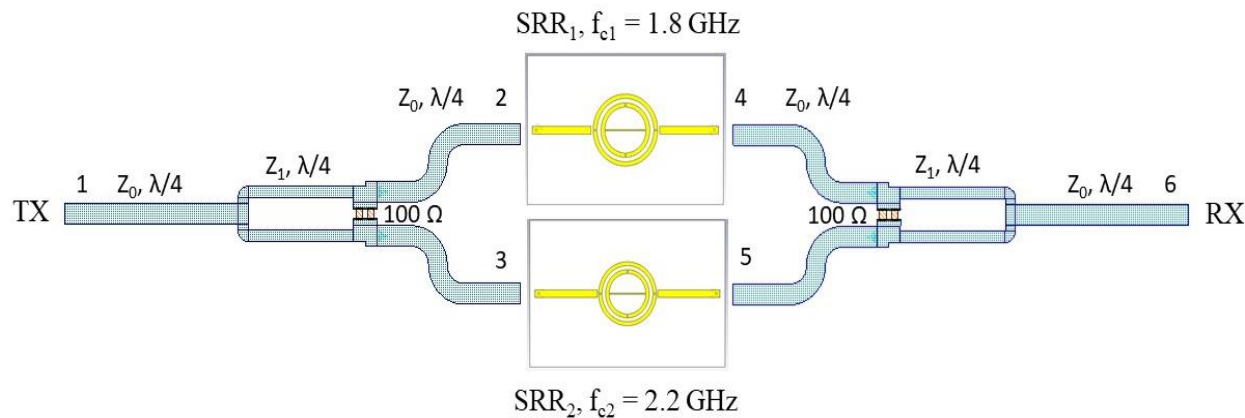
SRR for Health Monitoring in Concrete Structures

SRR



SRR Network for Health Monitoring in Concrete Structures

Simplified scheme of the proposed monitoring system



Transmission coefficient of the
proposed system

cadence

AWR
RF/MICROWAVE
DESIGN SOFTWARE

SRR Network and Diffused Sensing Element (SE) Embedded in a Concrete Beam for Structural Health Monitoring

Two different and complementary sensors:

- SE → able to detect the variation of the concrete dielectric properties along the whole beam length for a diffuse monitoring
- SRRs → work punctually, in their surroundings, allowing an accurate evaluation of the permittivity

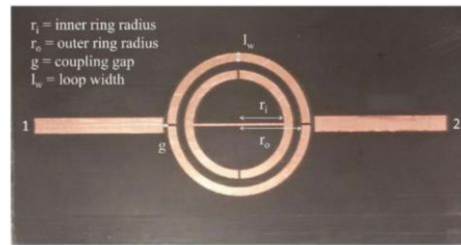
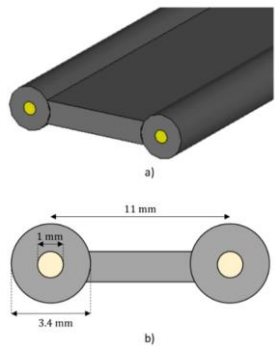


Fig. SRR geometry and its principal parameters.

Table 1. Geometric dimensions of Split Ring Resonators.

SRR	r_i (mm)	r_o (mm)	g (mm)	l_w (mm)
1	14.0	18.0	0.44	2.0
2	12.4	16.4	0.44	2.0
3	11.0	15.0	0.44	2.0
4	10.0	14.0	0.44	2.0

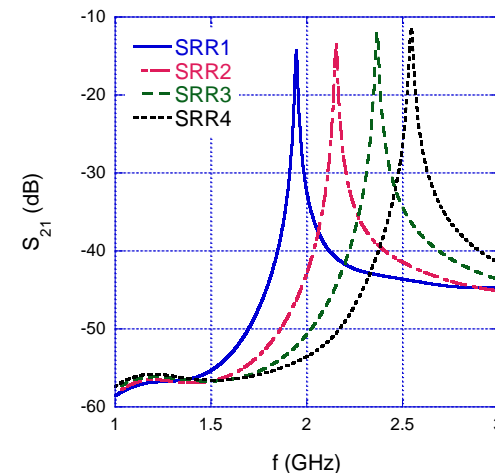


Fig. Transmission coefficients of SRRs in air.

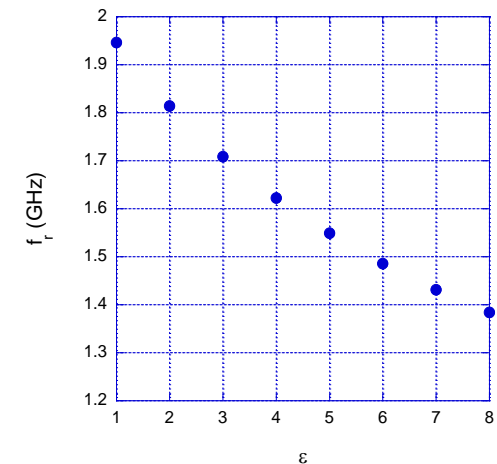


Fig. Relationship between the SRR1 resonance frequency and the permittivity of the surrounding material.

4 Split Ring Resonator Network

Resonance frequency of the four split ring resonators embedded in a material with permittivity ϵ .

ϵ	1	2	3	4	5	6	7	8
$f_{r,SRR1}$	1.9461	1.8141	1.7082	1.6224	1.5489	1.4856	1.4313	1.3842
$f_{r,SRR2}$	2.1531	2.0091	1.8894	1.7910	1.7109	1.6407	1.5771	1.5222
$f_{r,SRR3}$	2.3670	2.2101	2.0772	1.9710	1.8819	1.8039	1.7364	1.6737
$f_{r,SRR4}$	2.5464	2.3733	2.2347	2.1231	2.0268	1.9431	1.8690	1.8015

To feed the four SRRs, the design of a power divider has been conducted with Microwave Office by AWR

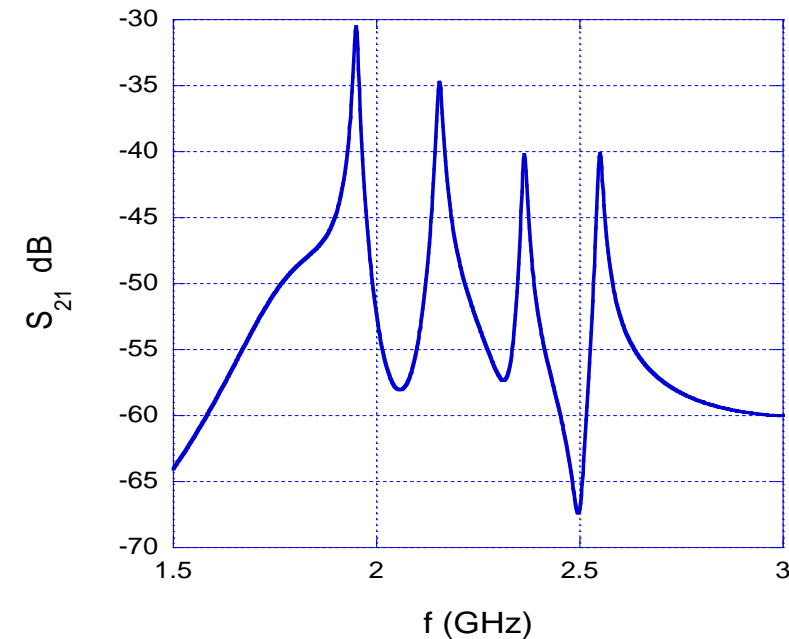
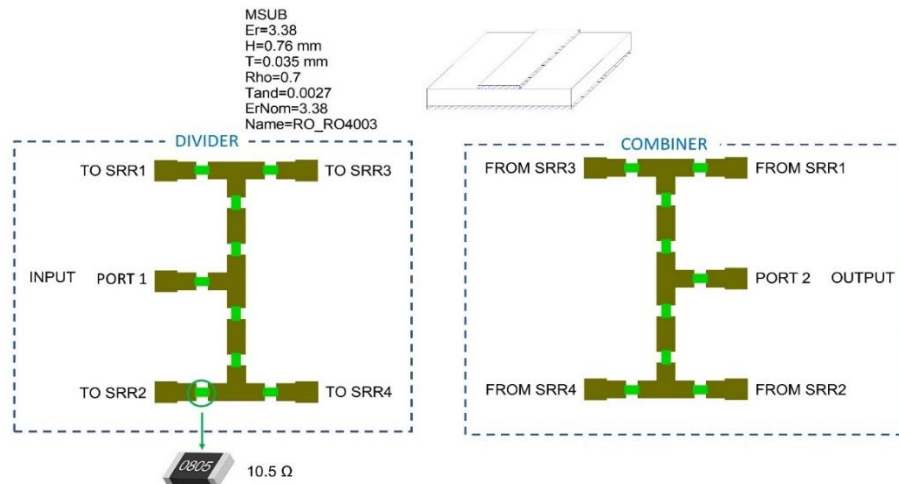


Fig. S_{21} MWO simulation of the designed network.

SRR Network and Diffused SE Results



Fig. View of the four SRRs embedded in the mold.

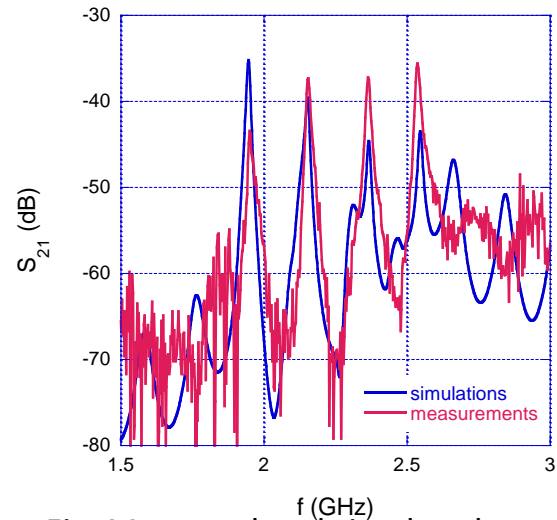


Fig. Measured and simulated network transmission coefficient in air.

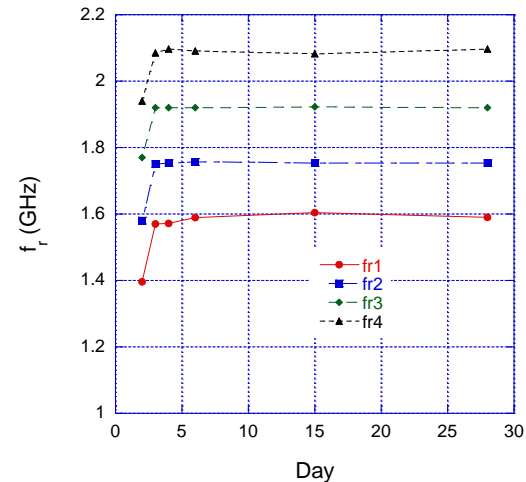


Fig. Computed network transmission coefficient resonance frequency during the curing phase.

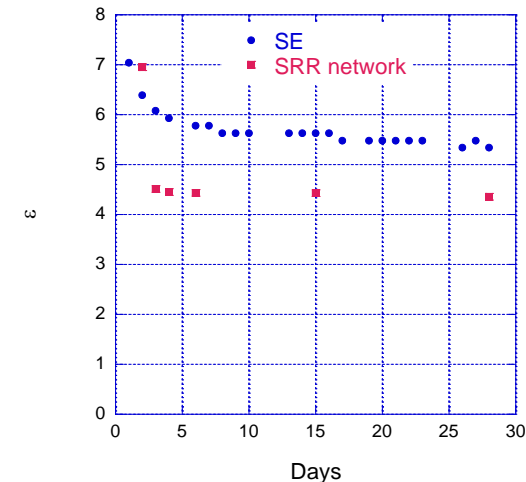


Fig. Measured permittivity during the curing of the concrete

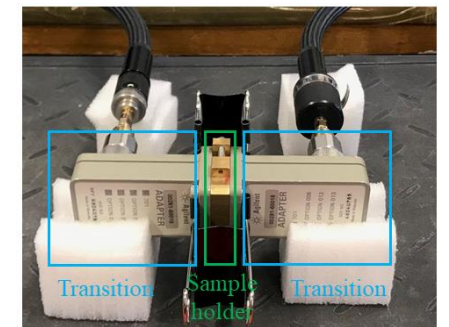
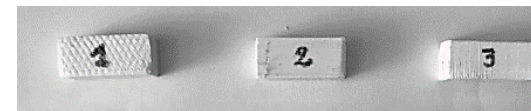
NB:

1. The first day, when the concrete beam contain a considerable quantity of water, the S_{21} as a bell-shaped trend and the four resonances of the SRR network are not clearly visible; they begin to be clearly visible on the second day, when a good amount of water has evaporated
2. difference in concrete permittivity was deliberately made in fact a different mixture of cement was placed around the SRRs

✓ control the presence of water inside the beam & to locate any internal cracks in the beam

Microwave system for material characterization and monitoring

- Cultural Heritage Monitoring
- Health Monitoring in Concrete Structures
- **Composite Materials Characterization**



Material Characterization

- Composite materials (GNPs)

PA6

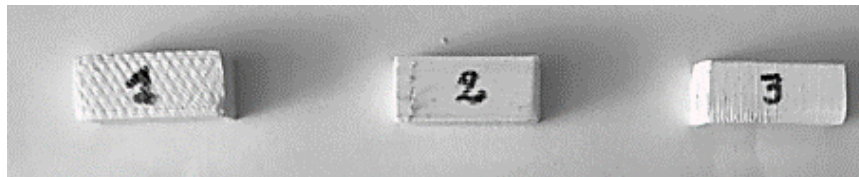


PA6/GNP



- 3D Printed PLA and PLA/CNT Composites

3D printed PLA

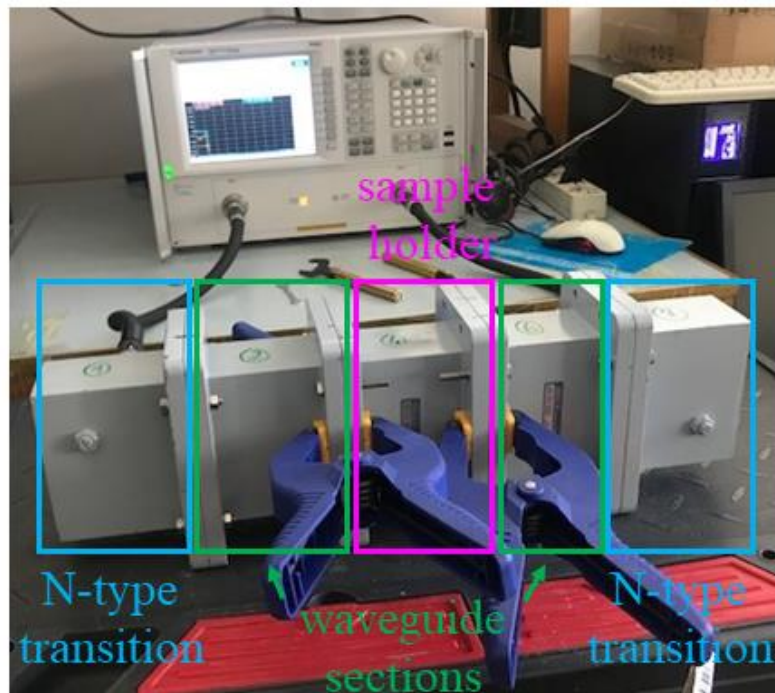


3D printed PLA with CNT

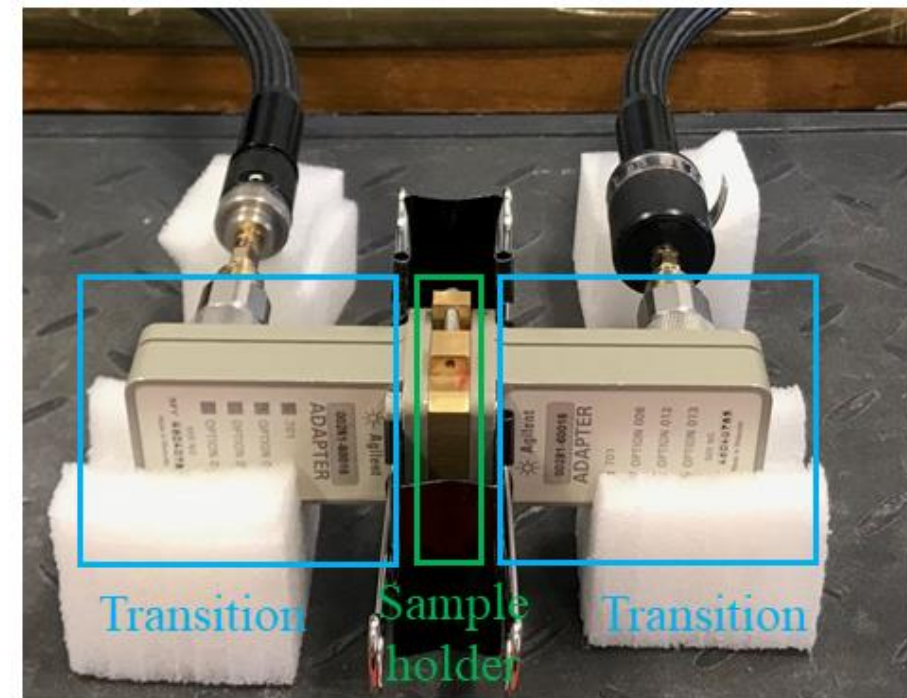


Experimental set-up

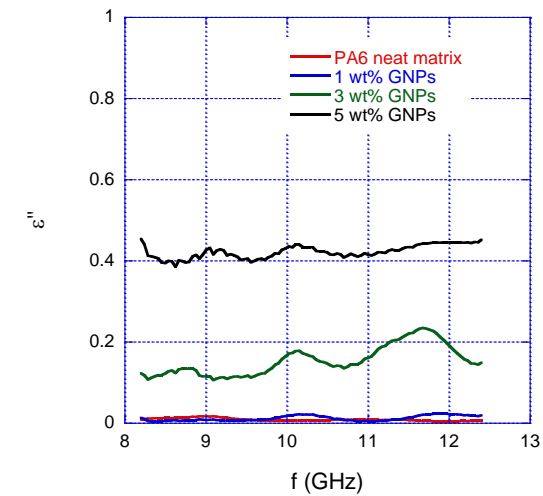
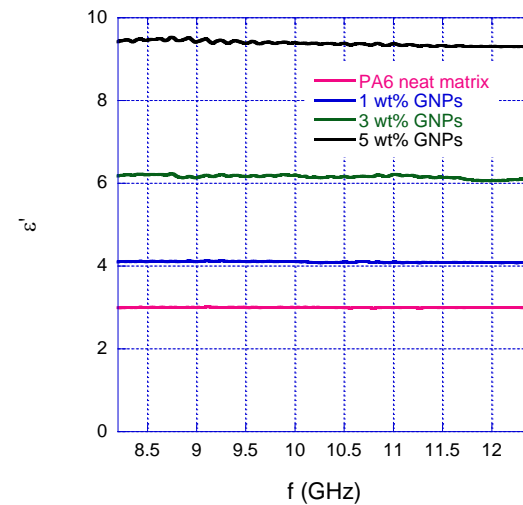
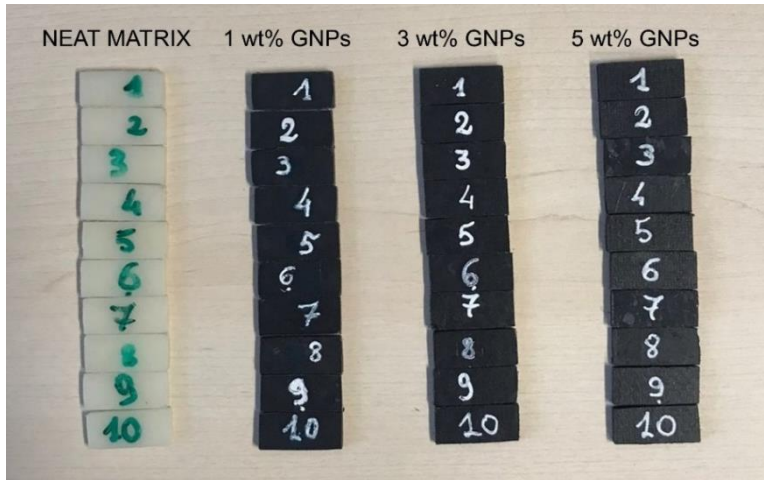
WR430 1.7 – 2.6 GHz



WR90 8.2 – 12.4 GHz



Results



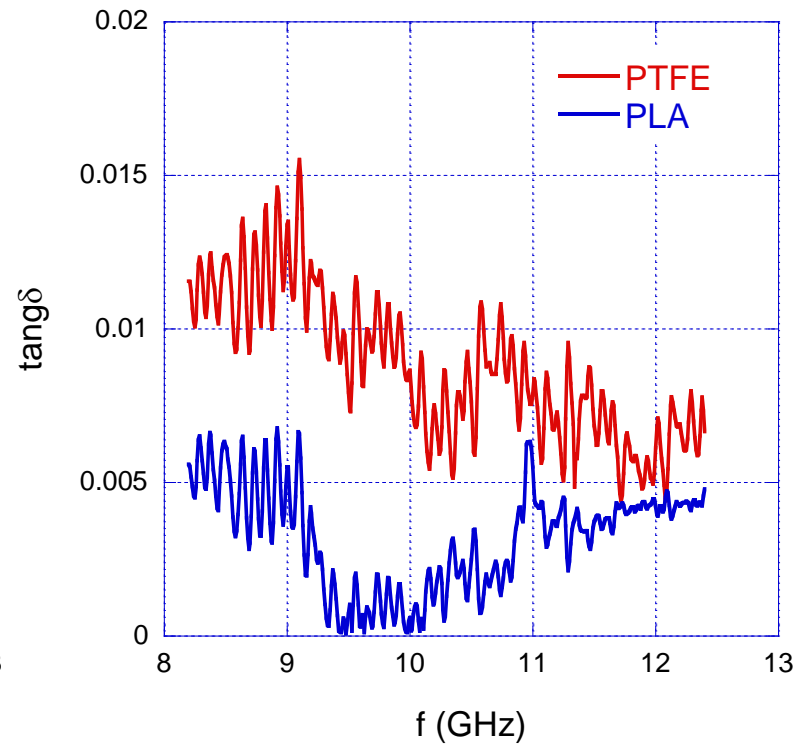
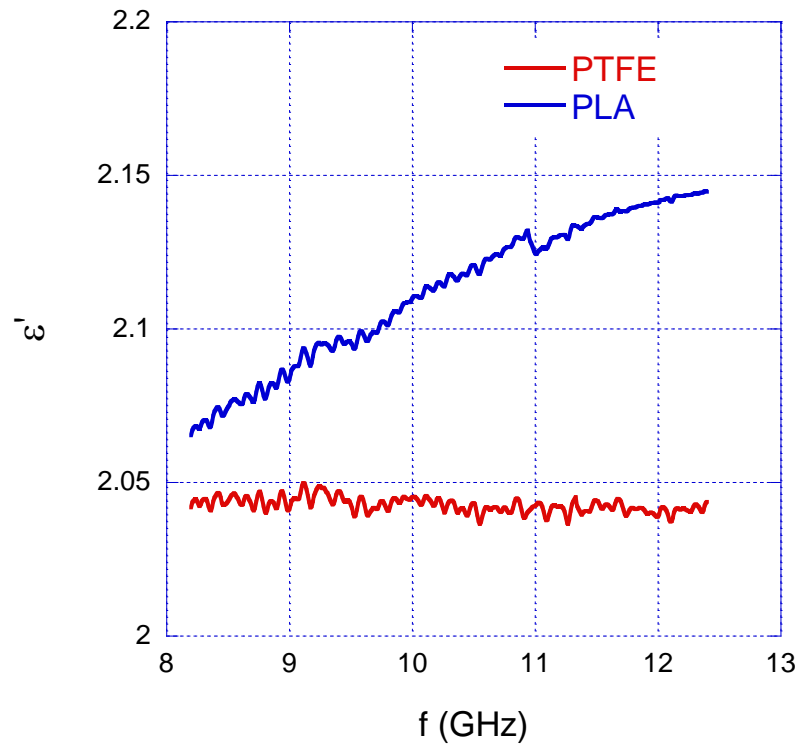
	ϵ'		ϵ''	
	WR430	WR90	WR430	WR90
PA6	3.0723 ± 0.0095	2.999 ± 0.0059	0.0107 ± 0.0083	0.0091 ± 0.0045
1%	4.195 ± 0.019	4.100 ± 0.014	0.0227 ± 0.016	0.0120 ± 0.0083
3%	5.846 ± 0.038	6.159 ± 0.043	0.106 ± 0.042	0.155 ± 0.043
5%	10.172 ± 0.069	9.381 ± 0.060	0.493 ± 0.039	0.422 ± 0.030

$$\sigma = \omega \epsilon_0 \epsilon''$$



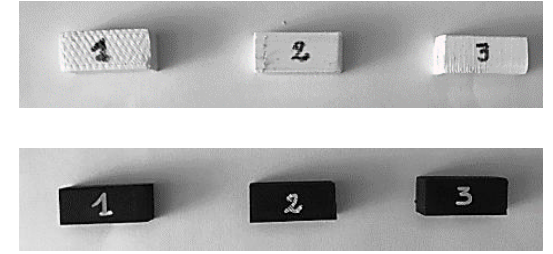
Results

Measured complex permittivity for the 3D printed PLA and PTFE

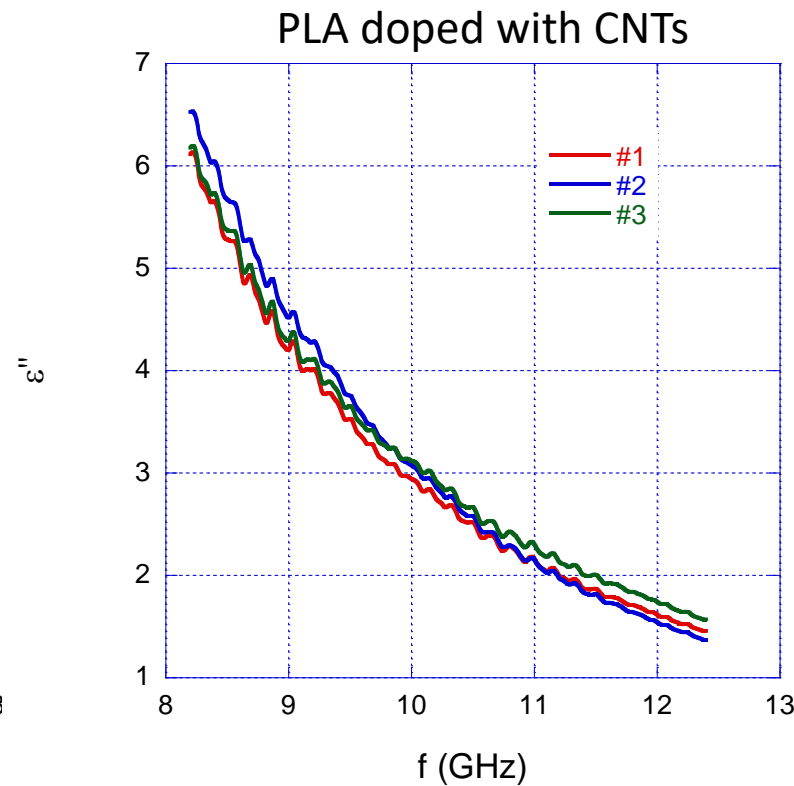
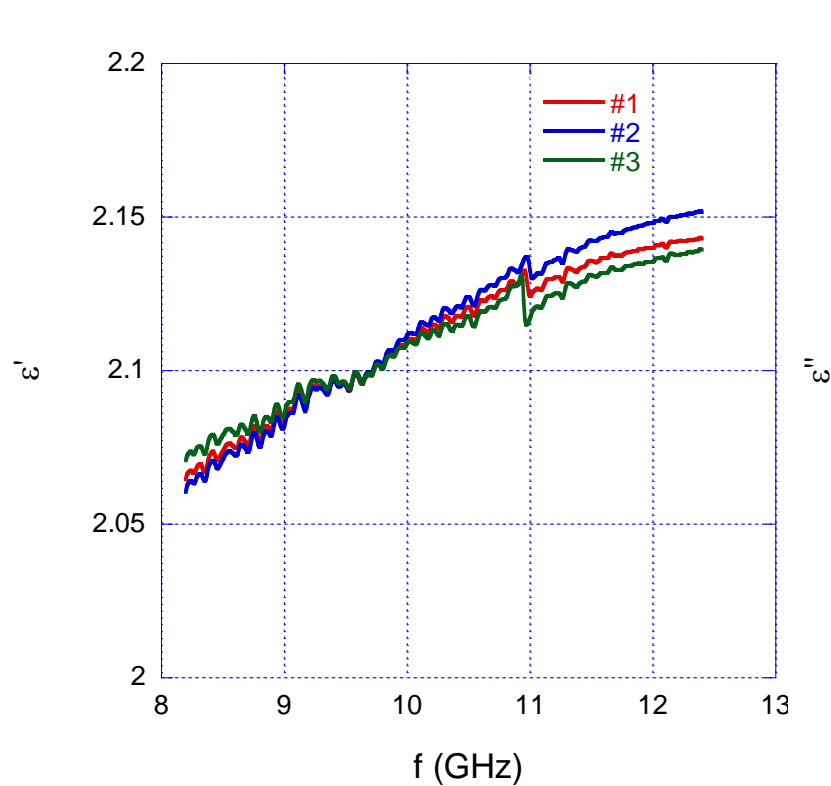


- discrepancy between the two curves can be considered negligible, as the maximum variation is within 3%
- losses for PLA are even lower than PTFE
 - PLA can be used to build microwave components

Results



Measured complex permittivity for the 3D printed PLA and PLA doped with CNTs



- measured values are consistent with the literature
- no substantial differences between the three ϵ_c samples \rightarrow anisotropy **not** significative
- small discontinuity around 11 GHz \leftarrow non-perfect planarity of the sample resulting from defects in the 3D-printing process
- quite high value of the imaginary part of the complex permittivity \rightarrow losses are not negligible (EMI shielding or EM absorber)

Conclusions and Future Developments

- **Cultural Heritage Monitoring**

- ✓ possibility of monitoring the moisture content in materials largely used in artistic artifacts
- ✓ measurements of the SRR resonance frequency, directly related to material dielectric characteristics
- Expanding the set of tested materials, improving the calibration curves, including also the effect of sample size, and assessing whether using both the resonance frequency and the Q value as input data, the accuracy of water content evaluation can be improved

- **Health Monitoring in Concrete Structures**

- ✓ possibility of monitoring concrete during the important curing phase
- ✓ highlight the presence of water that could cause deterioration, especially in cases of dams, bridges and structures that are constantly subjected to an important presence of water
- It is planned to test the possibility of monitoring the presence of water near one of the SRRs and the localization of artificial cracks, that will be created with mechanical stress in some points of the beam

- **Composite Materials Characterization**

- ✓ a material with GNPs thanks to the high electrical conductivity yields a good shielding efficiency against microwave radiation
- ✓ 3D printed PLA can be used to build microwave components
- It would be very interesting to investigate these materials in broader band experiments



Italian National Committee

Commission A: Electromagnetic Metrology, Electromagnetic measurements and standards

Dielectric characterization of materials at microwave frequencies

DR. ERIKA PITTELLA

erika.pittella@uniroma1.it

THANK YOU



SAPIENZA
UNIVERSITÀ DI ROMA



*Department of Information Engineering, Electronics and Telecommunications
(DIET)*