



Low-Loss Materials in High Frequency Electronics and the Challenges of Measurement

Glenn Oliver

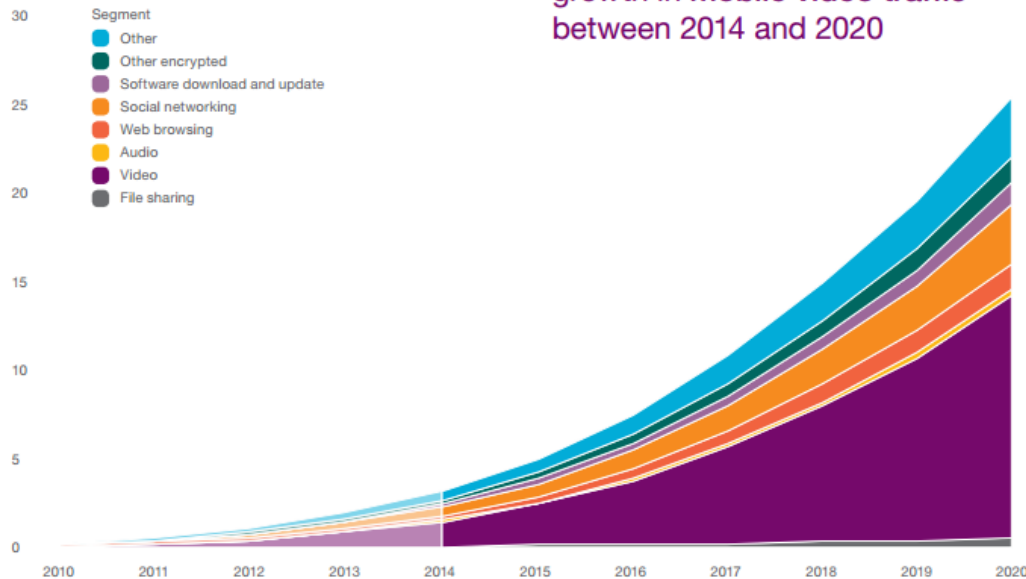
glenn.e.oliver@dupont.com

E.I. Du Pont de Nemours

- Company founded in 1802 – First Century: Gun Powder
 - Supplied US Military
- Trust Busted in 1912 – Second Century: Chemical Company
 - First polymers: Neoprene, Nylon
 - Ongoing brands: Teflon®, Tyvek®, Nomex®, Kevlar®, Kapton®, Corian®
- Third Century: Science to Feed, Protect, and Sustain
 - In: Danisco, Pioneer
 - Out: Commodity Polymers, Coatings, Chemours

Drivers of Higher Frequency - Mobile

Mobile data traffic by application type (monthly ExaBytes)



10X

growth in mobile video traffic between 2014 and 2020

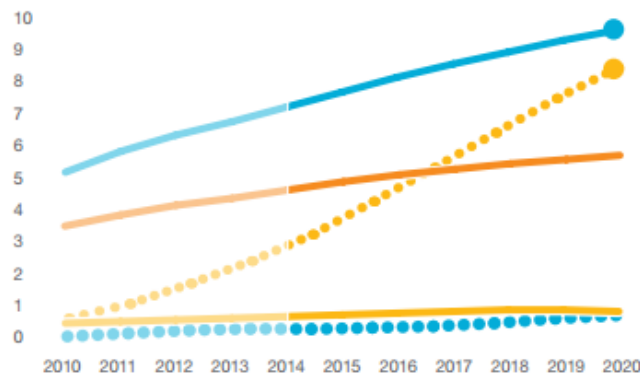
- People now expect high quality video content.
- Almost everyone will have mobile devices utilizing more and more bandwidth.

Ericsson Mobility Report – November 2014

<http://www.ericsson.com/ericsson-mobility-report>

NOVEMBER 2014 ERICSSON MOBILITY REPORT 15

Subscriptions/lines, subscribers (billion)



9.5 BILLION

mobile subscriptions by the end of 2020

90%

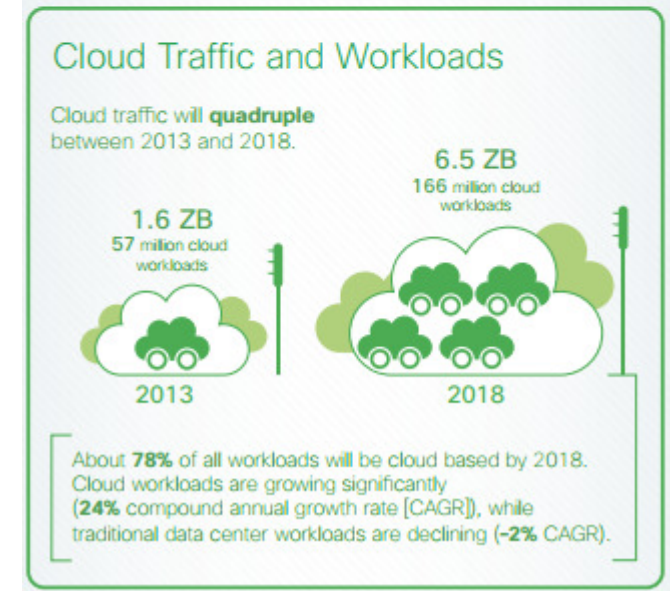
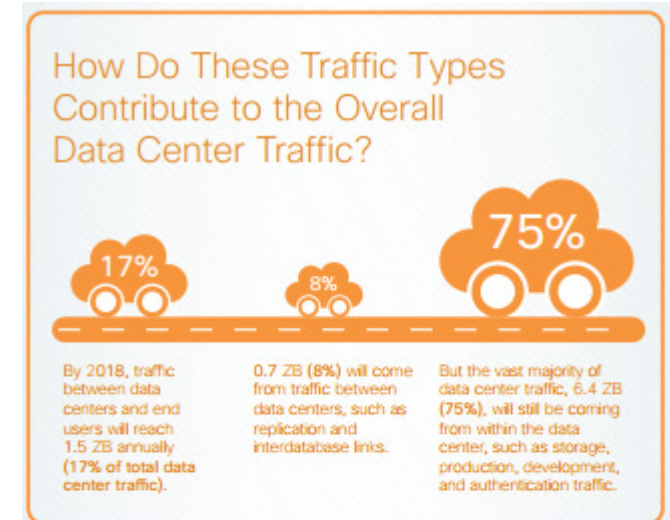
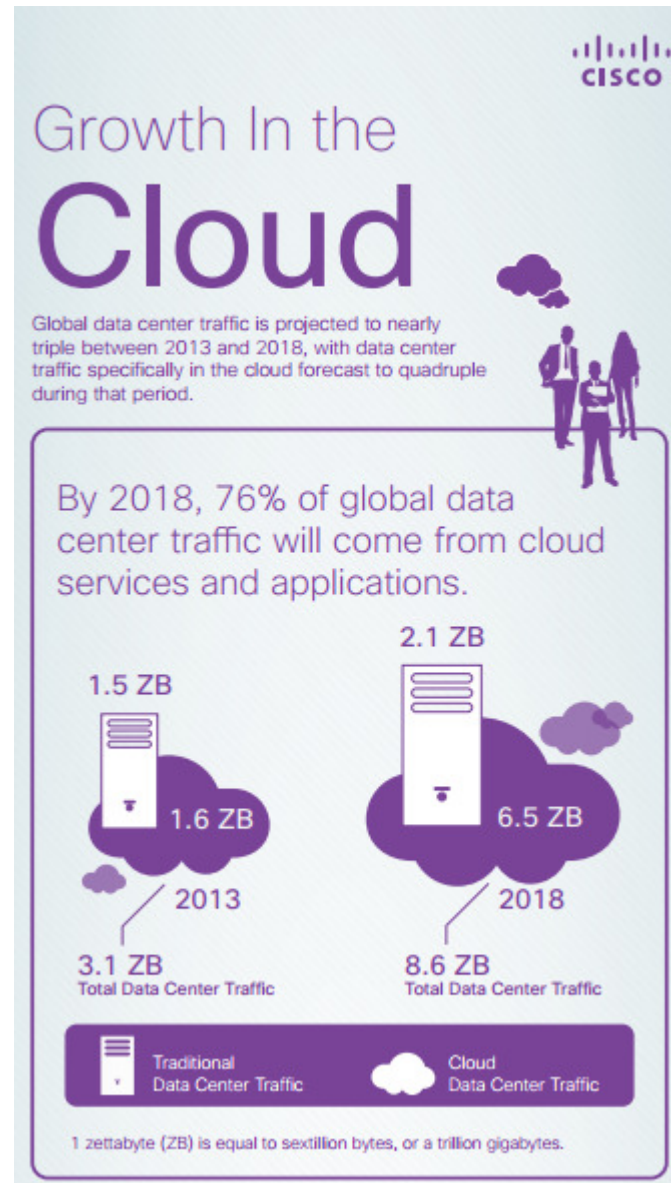
of the world's population over 6 years old will have a mobile phone by 2020



Drivers of Higher Frequency – Cloud Data

- Examples from Cisco Systems
- Not only does the video content need to be delivered, but it needs to be stored and connected.

<http://www.cisco.com/c/en/us/solutions/service-provider/global-cloud-index-gci/index.html>



Material Requirements for High Frequency

Most Cases: Low Dielectric Constant and Low Loss

Dielectric for Cables:

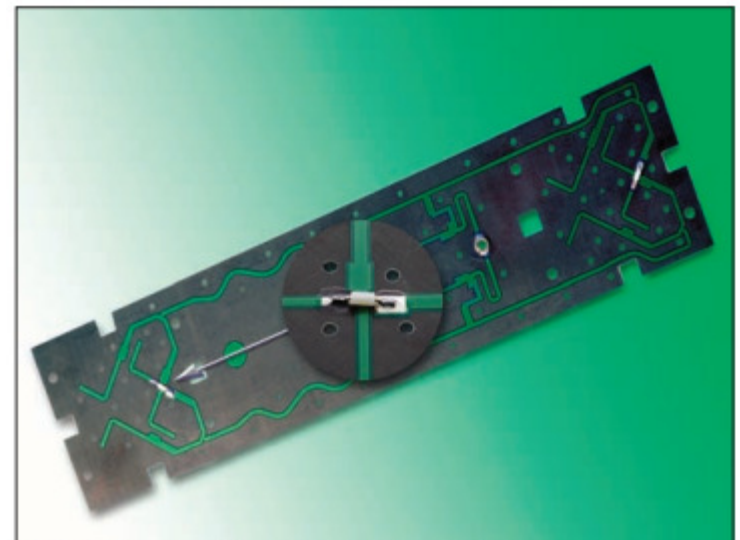
- Film, Tape or Melt Processible

Dielectrics for Printed Circuits:

- Adhesion of dielectric to metal
- Not absorb chemicals from wet processing (etching & plating)
- Able to reliably connect between layers (vias)
- Withstand solder assembly and thermal cycles during operation



Wireline extrusion with Teflon® FEP

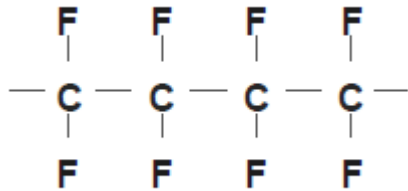


Printed wiring board for mobile telephony antenna

What is Special about Fluoropolymers?

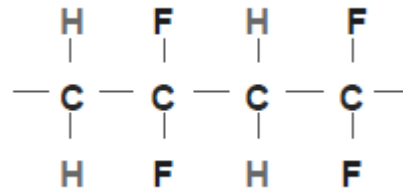
- Chemically inert and resistant to solvents
- Non-stick (self cleaning)
- Low friction (self lubricating)
- Broad operating temperature range (-200 C to +260 C)
- Does not degrade in humidity and UV light
- Non-toxic and non-flammable
- Low dielectric constant and dielectric loss

Classes of Fluoropolymers



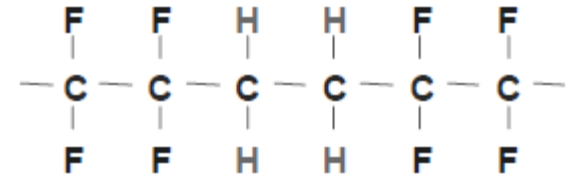
PTFE

PolyTetraFluoroEthylene



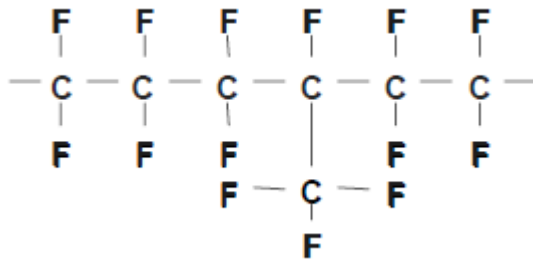
PVDF

PolyVinylideneFluoride



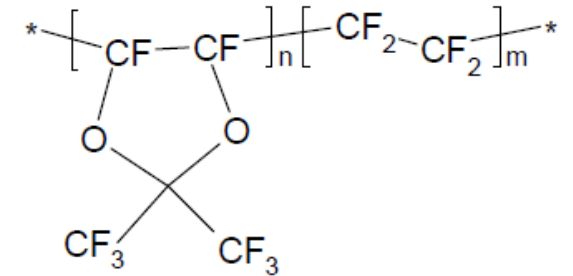
ETFE

Ethylene + TetraFluoroEthylene



FEP

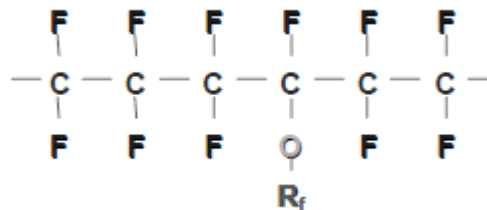
FlourinatedEthylenePropylene



Teflon® AF

PDD + TFE

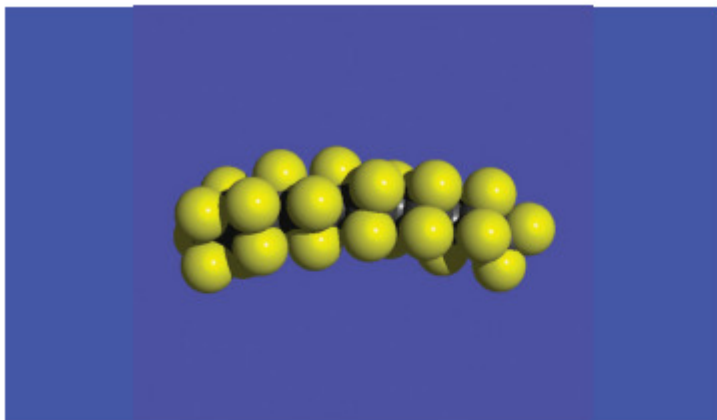
Perfluoro-2,2-Dimethyl-1,3-Dioxole



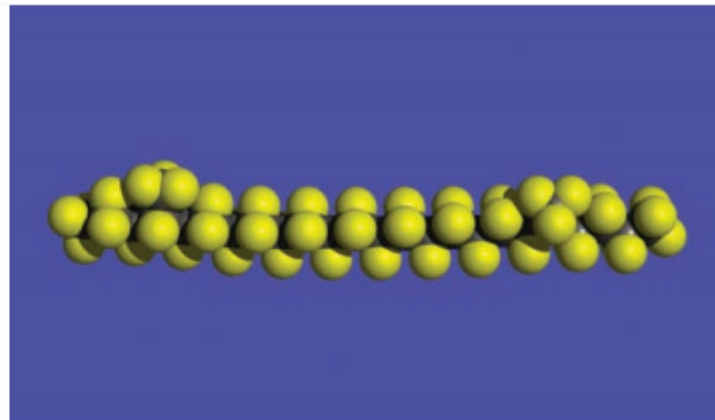
PFA

PerFluoroalkoxy Alkanes

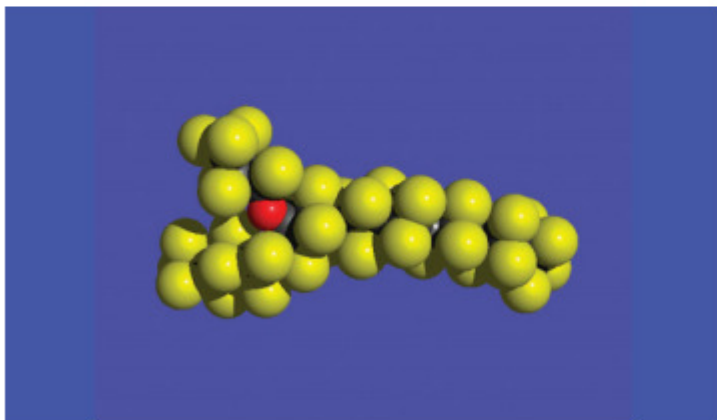
Fluoropolymers – Organic Models



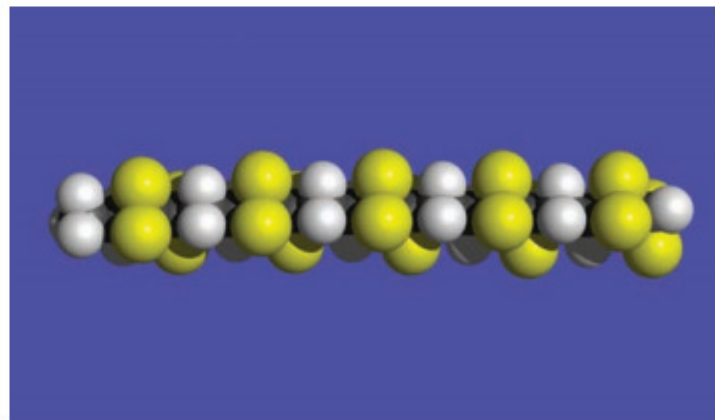
Polytetrafluoroethylene



Fluoroethylenpropylene

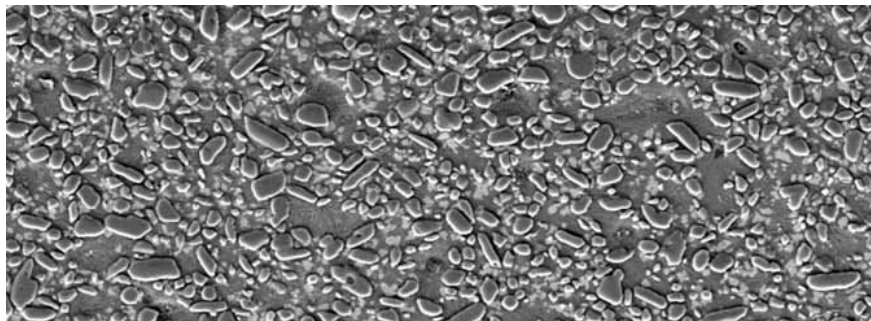
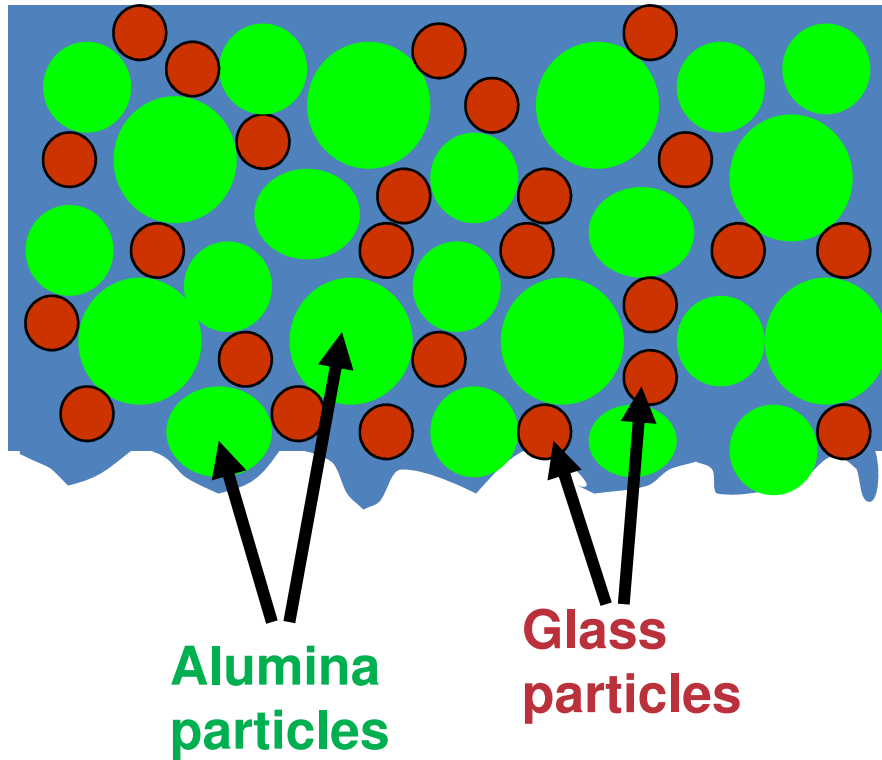


Perfluoroalkoxy



Ethylene - Tetrafluoroethylene

Low Temperature Cofired Ceramic (LTCC)



Roughly speaking, think of a unfired (green) ceramic cast into a tape consisting of Alumina particles (green), glass particles (red) and polymeric binder (blue).

The packing density of the particles in Green Tape™ is very high, with binder occupying interstitial areas. This allows for screen printing of metal on the unfired tape.

The binder will burn out during firing

Dielectric Constant

DC Case: Very simple. κ' is real and just depends on capacitance. There is no loss because there is no current (open circuit). Electric field is static.

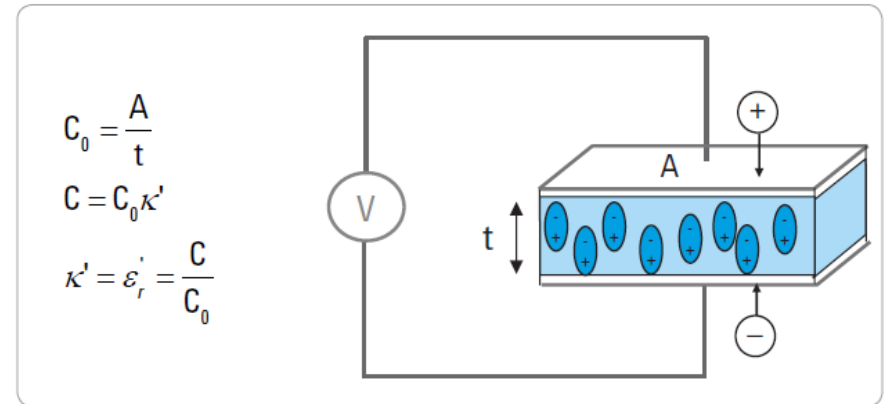


Figure 1. Parallel plate capacitor, DC case

AC Case: Now there is current. Electric field changes with frequency.

$$I = I_c + I_l = V(j\omega C_0 \kappa' + G)$$

If $G = \omega C_0 \kappa''$, then

$$I = V(j\omega C_0)(\kappa' - j\kappa'') = V(j\omega C_0)\kappa$$

$$\omega = 2\pi f$$

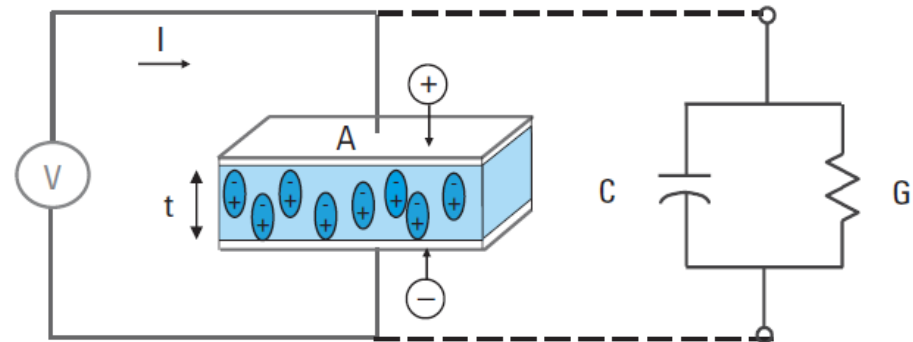


Figure 2. Parallel plate capacitor, AC case

Relative Permittivity and Loss Tangent

Permittivity is a complex number.

Real component = $\kappa' = \epsilon_r'$ = "D_k"

Imaginary component = ϵ_r''

$$\tan(\epsilon_r'' / \epsilon_r') = \tan \delta = \text{Loss Tangent}$$

$$\tan \delta = \frac{\epsilon_r''}{\epsilon_r'} = D = \frac{1}{Q}$$

$$= \frac{\text{Energy lost per cycle}}{\text{Energy stored per cycle}}$$

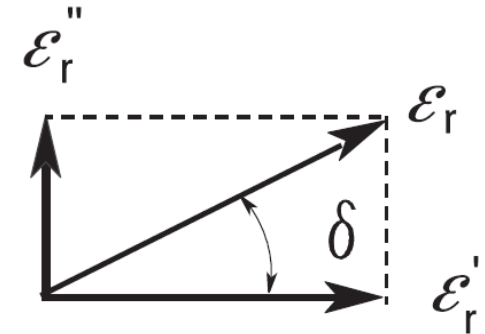


Figure 3. Loss tangent vector diagram

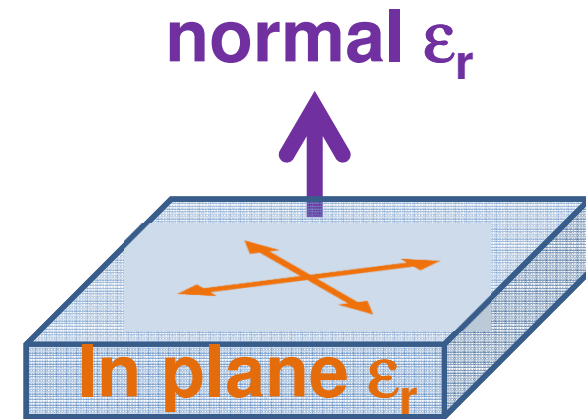
- Relative Permittivity is the more correct way to express "Dk" above 1 GHz.
- Magnitude of permittivity is fairly constant at low frequencies.
- As frequencies increase, more interaction with materials since wavelengths are on same scale as circuit features
 - at 1 GHz, $\lambda_{\text{air}} = 30 \text{ cm}$
 - at 10 GHz, $\lambda_{\text{air}} = 3 \text{ cm}$

Reference: Keysight Applications Note "Basics of Measuring the Dielectric Properties of Materials"

<http://cp.literature.agilent.com/litweb/pdf/5989-2589EN.pdf> (Note: Agilent is now Keysight)

Permittivity: Vector and Complex Property

- NOT CONSTANT WITH FREQUENCY!
- $\omega = 2\pi * \text{frequency}$
- More precisely, $\epsilon_r(\omega) = \epsilon_r(\omega)' + i \epsilon_r(\omega)''$
 - Real component $\epsilon_r(\omega)'$ associated with energy STORED
 - Imaginary component $\epsilon_r(\omega)''$ associated with energy ABSORBED
- These are **complex**, **vector** components
- Most circuit materials are planar, and regular in the x-y direction so the permittivity components are generally referred to as “**in plane**” and “**normal**”
- The difference between these two directions can be between 0%– 20 % for common commercial circuit materials.



Complex Permittivity

Basic Mechanisms that affect real and complex permittivity.

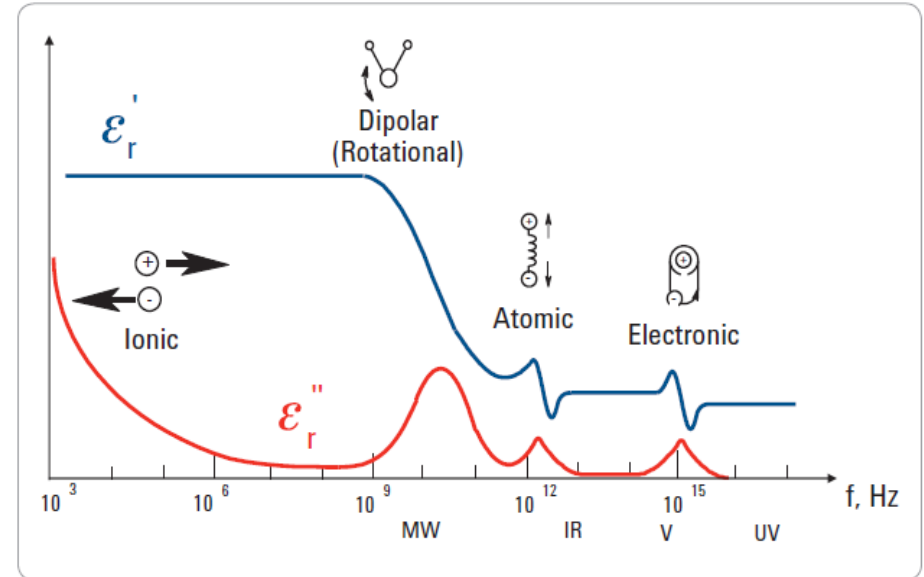


Figure 7. Frequency response of dielectric mechanisms

In the GHz range, the most common mechanism is Rotation or Precession of Dipoles. Rotation is shown here.

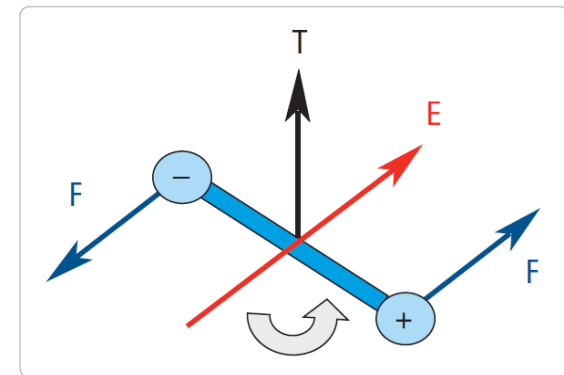
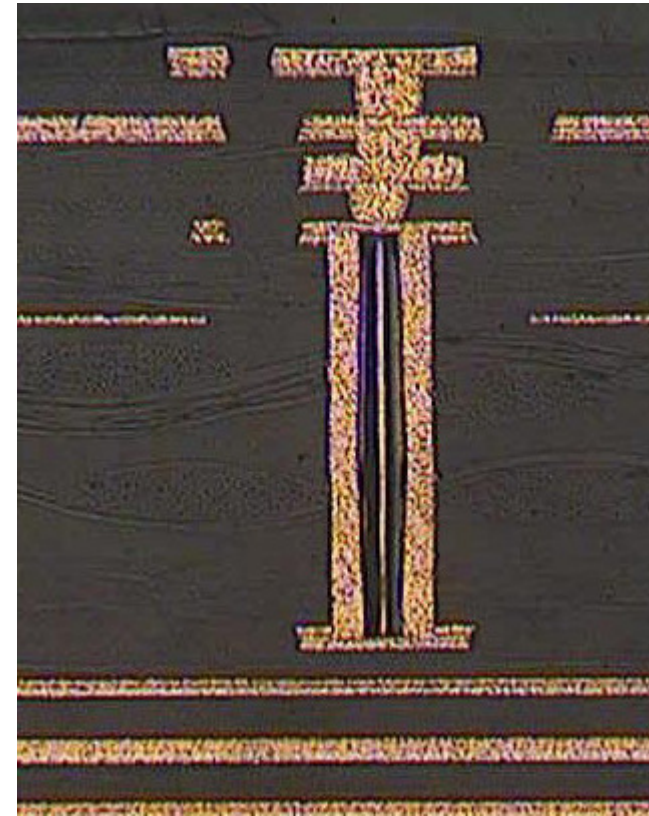


Figure 8. Dipole rotation in electric field

Note: Every material has different curves (different elements, bonds, etc.) AND how the materials are polymerized affect these curves.

Printed Circuit Materials are Usually Composites

- Almost all dielectrics $> 100 \mu\text{m}$ thick are composites of dielectric, glass, and fillers
- Glass fabric is used as reinforcement
- Some inorganic fillers are used to tune properties like dielectric constant or linear expansion
- Often organics are thermosets like epoxies that cross-link
- It is practically impossible to model all of the composite materials individually to build correct models of their high frequency behavior. Measurements are required

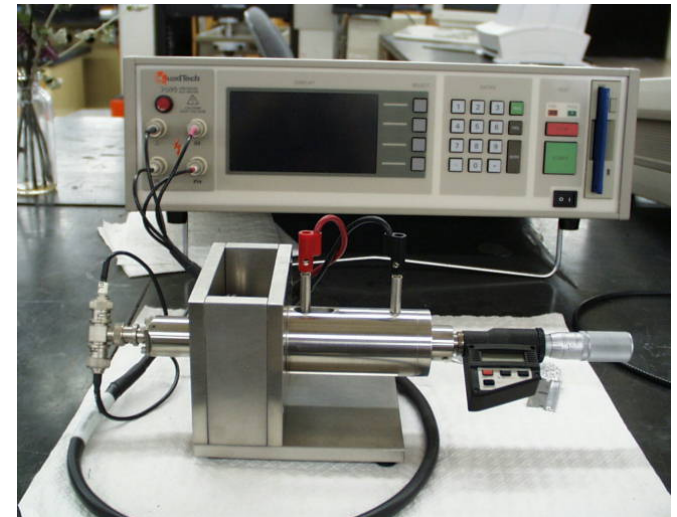


How to Measure Permittivity and Loss < 20 MHz

- Capacitance (IEC 60250 / ASTM D150)



Keysight Technologies
<http://www.keysight.com/>

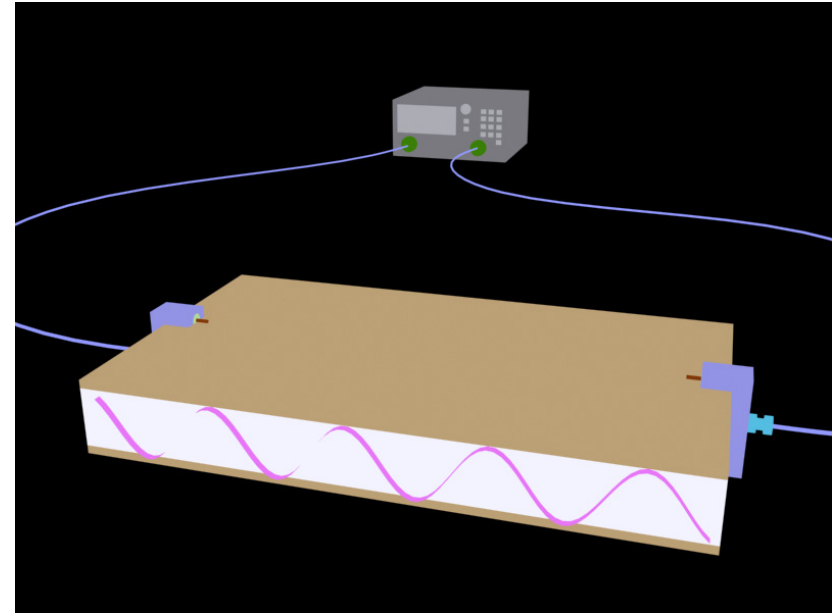


Intertek Plastics Technology Lab
<http://www.ptli.com/>

- Mature Technology – These techniques have not changed in many decades.

How to Measure Permittivity and Loss < 1 GHz

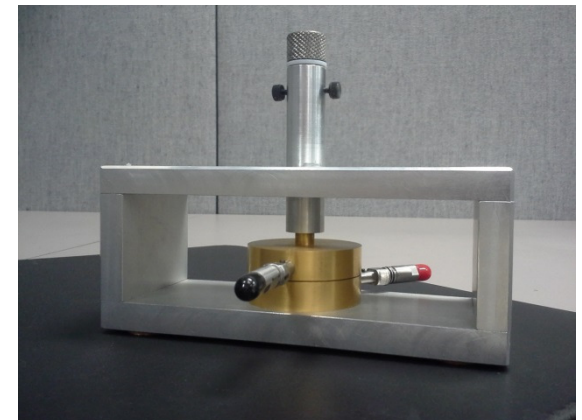
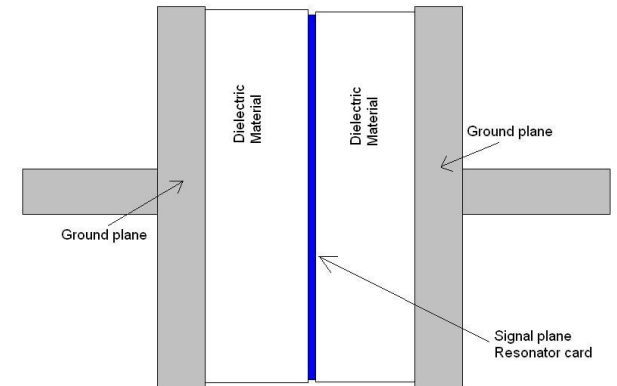
- FSR (Full Sheet Resonance)
 - IPC-TM-650 2.5.5.6
 - Copper clad panel acting as a parallel plate waveguide
 - Resonance peaks are established within the panel
 - Resonates at frequency where a $\frac{1}{2}$ wave is within the panel
 - Multiples of $\frac{1}{2}$ waves will have resonance peaks too
 - This is a low microwave frequency test, because the panels are relatively large (18"x12" or more) and a $\frac{1}{2}$ wave which is that long, will be low frequency



Resonator Methods from 2-20 GHz

Three Common Approaches:

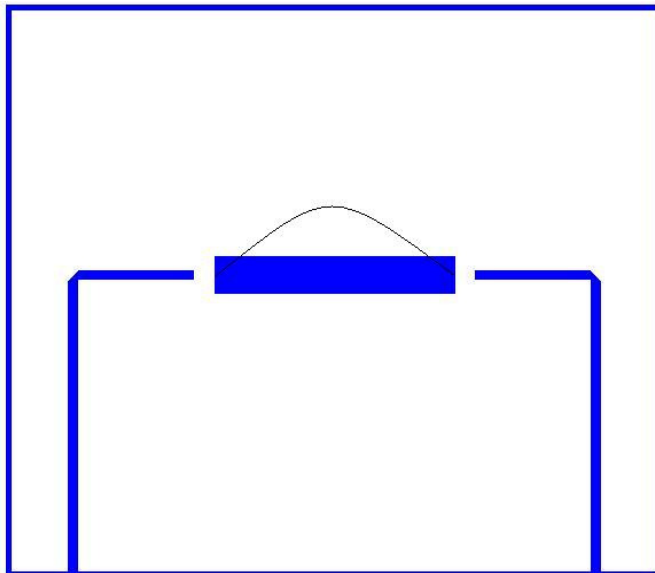
1. Dielectric Resonators
2. Waveguide Cavity Resonator Perturbation
3. Cylindrical Cavity Resonator Perturbation



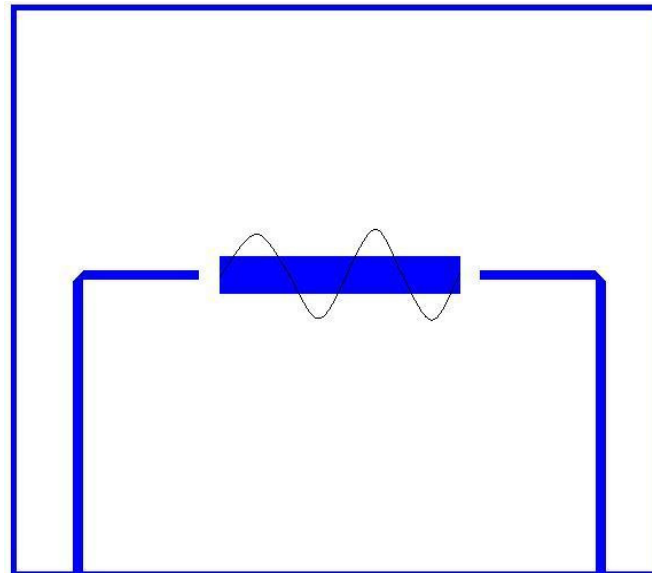
Clamped Stripline Dielectric Resonator

X-band Clamped stripline test

- The resonator circuit is based on $\frac{1}{2}$ wavelengths too
- The resonator length is appropriate for the relative permittivity of the material being tested
- There are different nodes in this test too, based on how many have $\frac{1}{2}$ wavelengths are on the resonator



2.5 GHz testing with one $\frac{1}{2}$ wavelength or node 1



10 GHz testing with 2 wavelengths, 4 half wavelengths or node 4

The resonator lengths determine the frequency. The standard test fixture is optimized for 10 GHz.

Clamped Stripline Dielectric Resonator



IPC-TM-650		
Number 2.5.5.5	Subject Stripline Test for Permittivity and Loss Tangent (Dielectric Constant and Dissipation Factor) at X-Band	Date 3/98
Revision C		

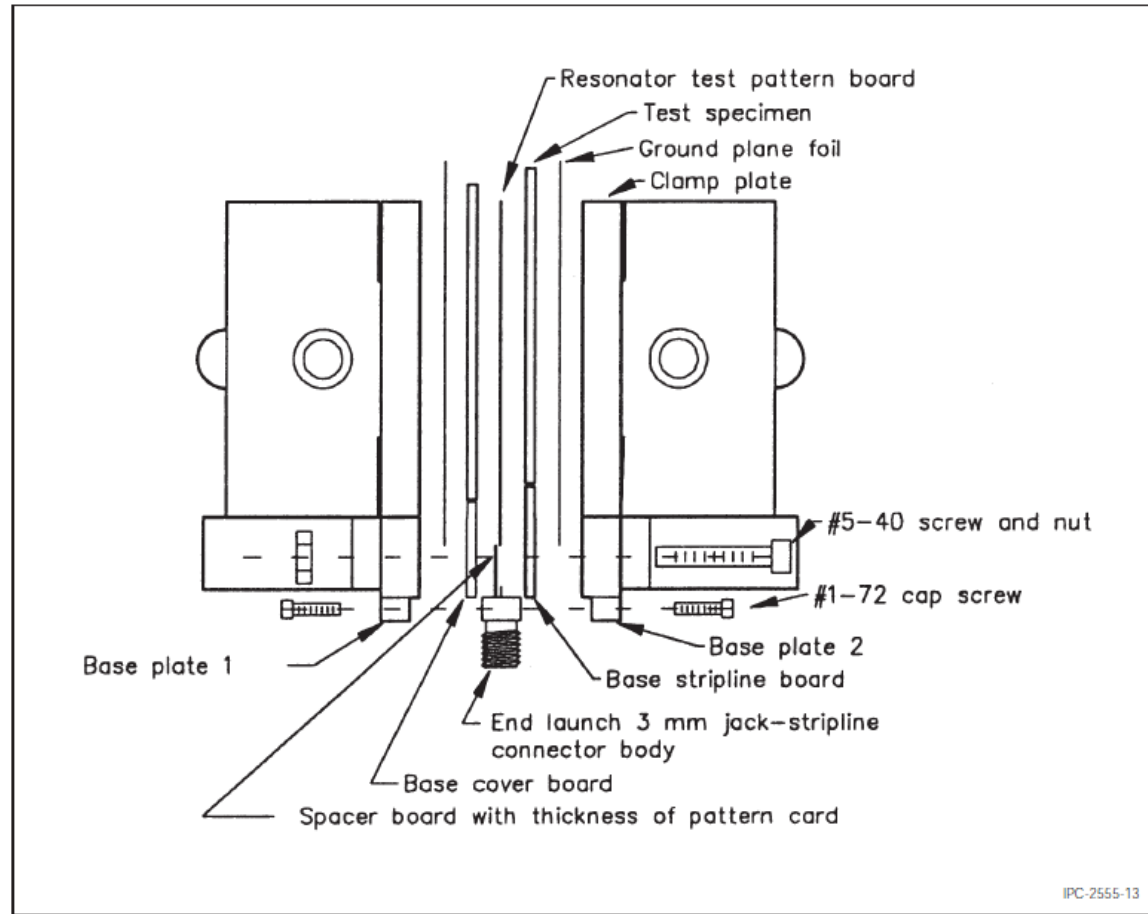


Figure 13 Exploded Side View of Assembly

Waveguide Cavity Resonator Perturbation

Basic principle is to define a resonant structure of a constant volume and compare the changes in resonant frequency and Q resulting from adding the material under test.

V_c = Volume of Cavity
 Q_c = Q of Empty Cavity
 f_c = Resonant Frequency of Empty Cavity

V_s = Volume of Sample
 Q_s = Q of Cavity with Sample
 f_s = Resonant Frequency of Cavity with Sample

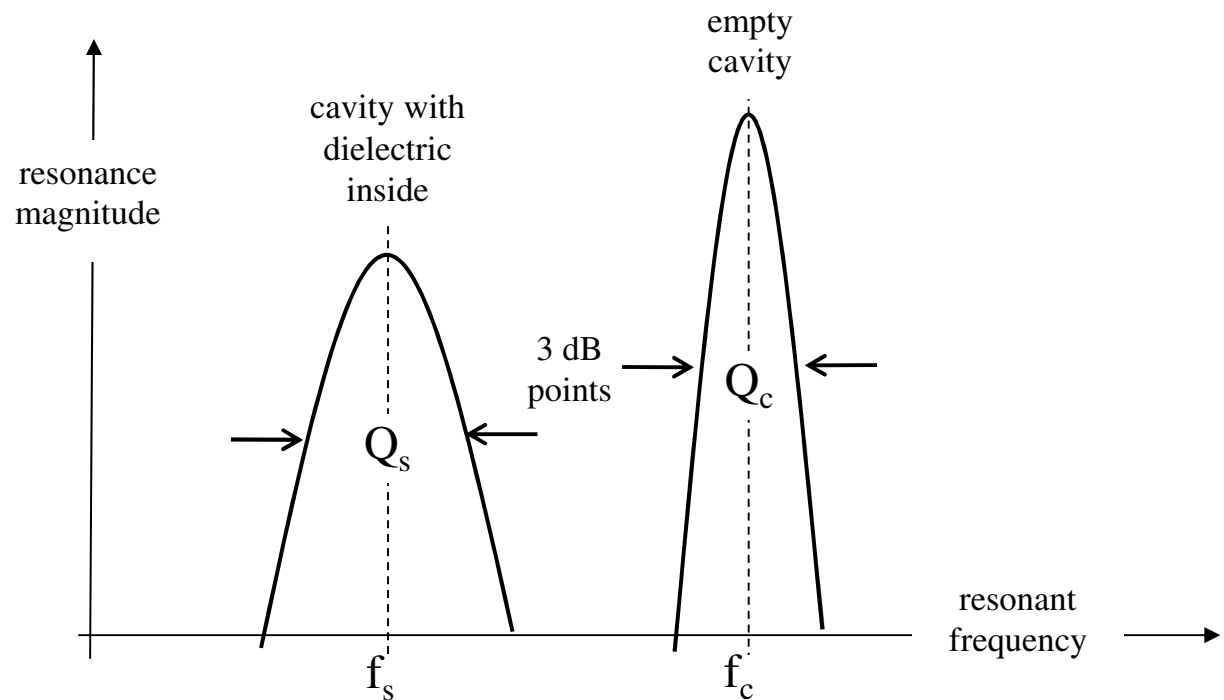
$$\epsilon_r' \approx \frac{V_c(f_c - f_s)}{V_s f_s} + \epsilon_{r(air)}$$

$$\epsilon_r'' \approx \frac{V_c}{4V_s} \left(\frac{1}{Q_s} - \frac{1}{Q_c} \right)$$

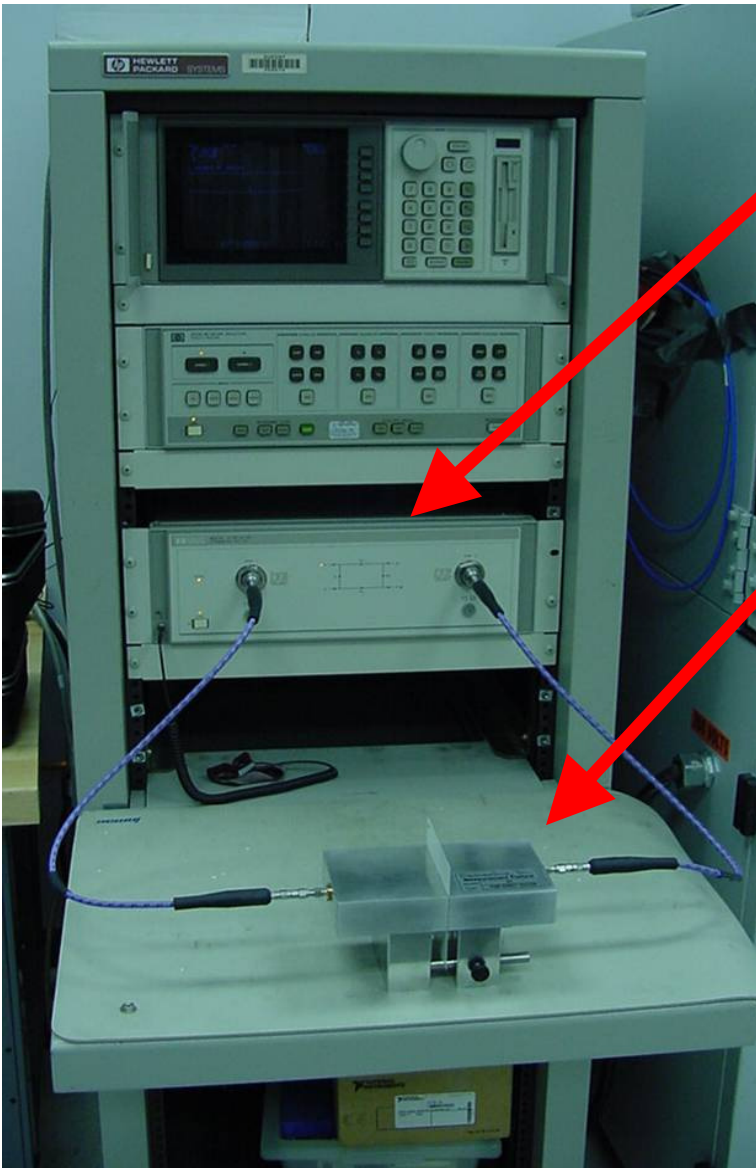
Approximate equations shown to illustrate the main factors and how they relate.

In practice, transcendental equations solved numerically.

In a rectangular waveguide cavity the electric field is oriented in the same plane as the dielectric.



Waveguide Cavity Resonator Perturbation



Network Analyzer

Resonant Cavity

First six odd-mode resonances measured. For this geometry, these are at 2.2, 3.4, 5, 6.8, 8.6 and 10.4 GHz.

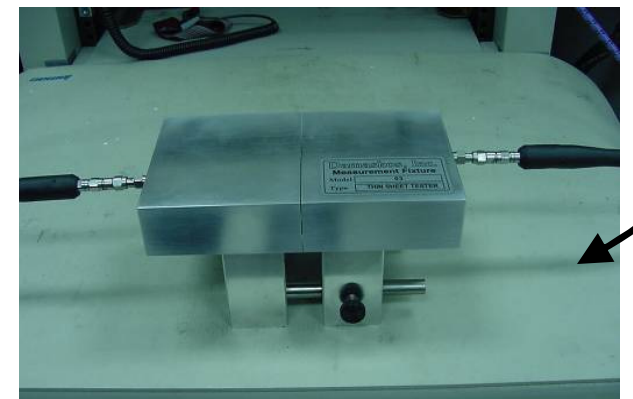
Two sets of measurements done for each sample, one rotated 90 degrees from the other (to see if the two planar directions are equal).



Resonant Cavity measured with film inside and data is stored....

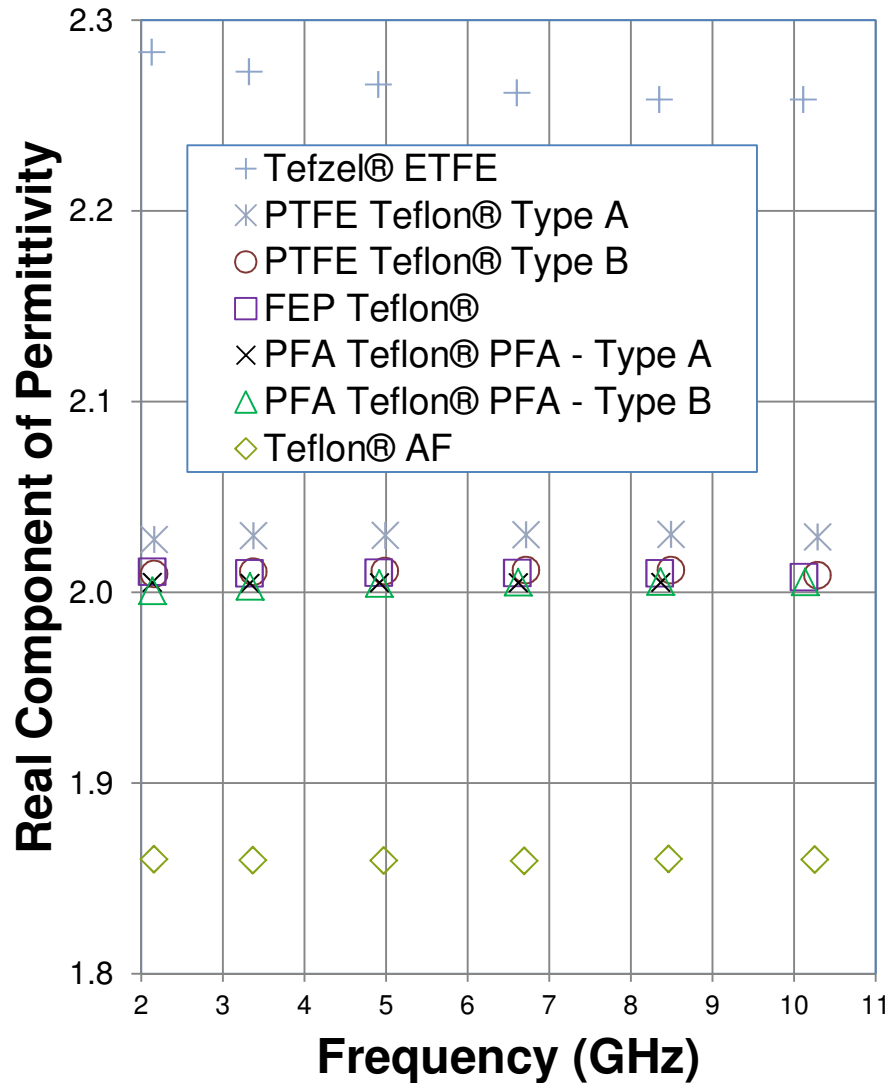
Then the sample is removed without changing cavity dimensions...

The effect of the cavity is calibrated out and you are left with the frequency response of the material.

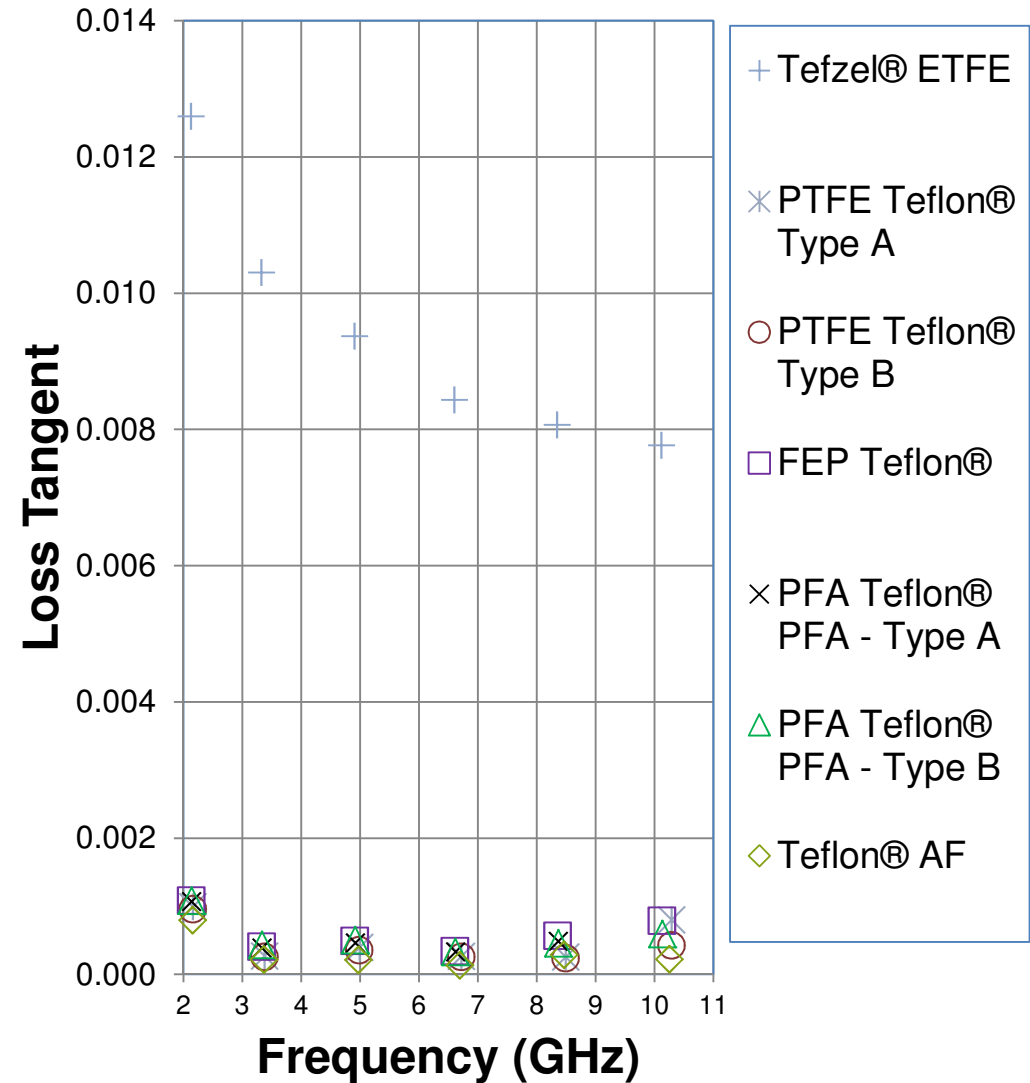


Measurement Summary (sans LTCC)

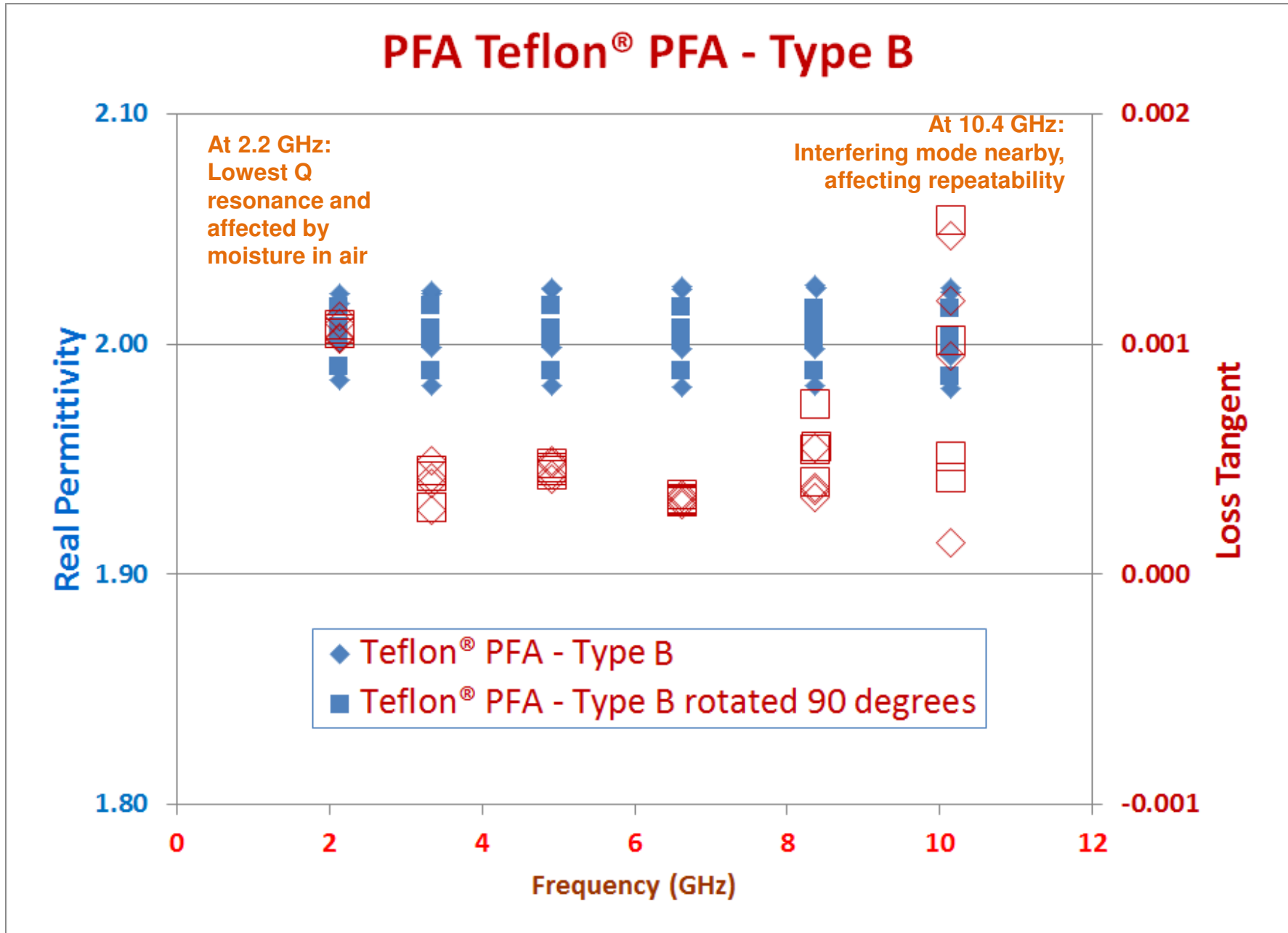
Rectangular Waveguide



Rectangular Waveguide



Raw Data Example



Split Cylinder Resonator Perturbation

- Standard Method developed by NIST
- Very precise method
- Size of cavity depends on the thickness of the sample so...
 - Sample must be almost perfectly flat
 - Thickness measurement accuracy and precision is critical
- Electric Field oriented IN THE PLANE of the dielectric.

IPC-TM-650 TEST METHODS MANUAL

1 Scope This method describes the nondestructive measurement of the relative permittivity and loss tangent of unclad dielectric substrates at microwave frequencies using a split-cylinder resonator (see Figure 1).

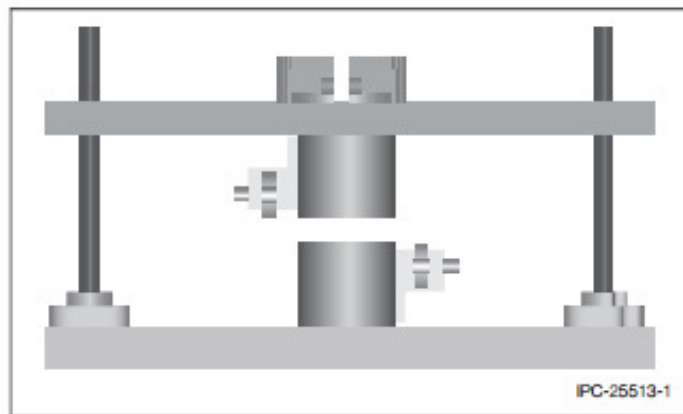


Figure 1 Split-Cylinder Resonator

This test method is directly applicable for measuring the in-plane (the plane parallel to the surface of the specimen) permittivity of the specimen because the electric field is in-plane. The permittivity of isotropic dielectrics can also be

Number 2.5.5.13	
Subject Relative Permittivity and Loss Tangent Using a Split-Cylinder Resonator	
Date 01/07	Revision
Originating Task Group High Frequency Resonator Test Method Task Group (D-24c)	

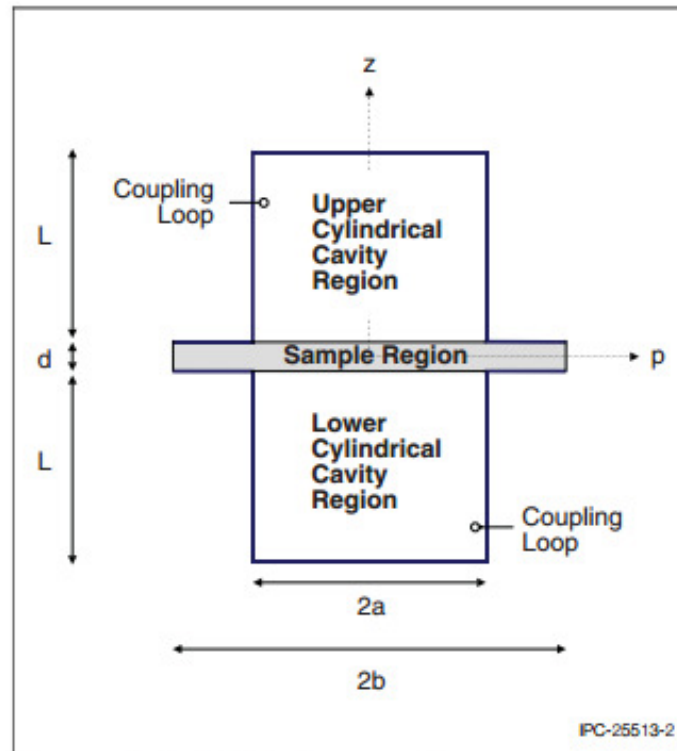
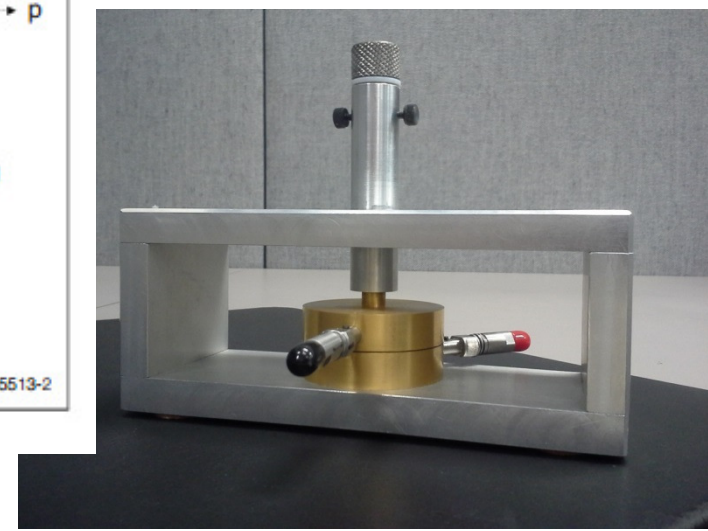


Figure 2 Split-Cylinder Resonator Diagram

Cavity used in our lab pictured below:

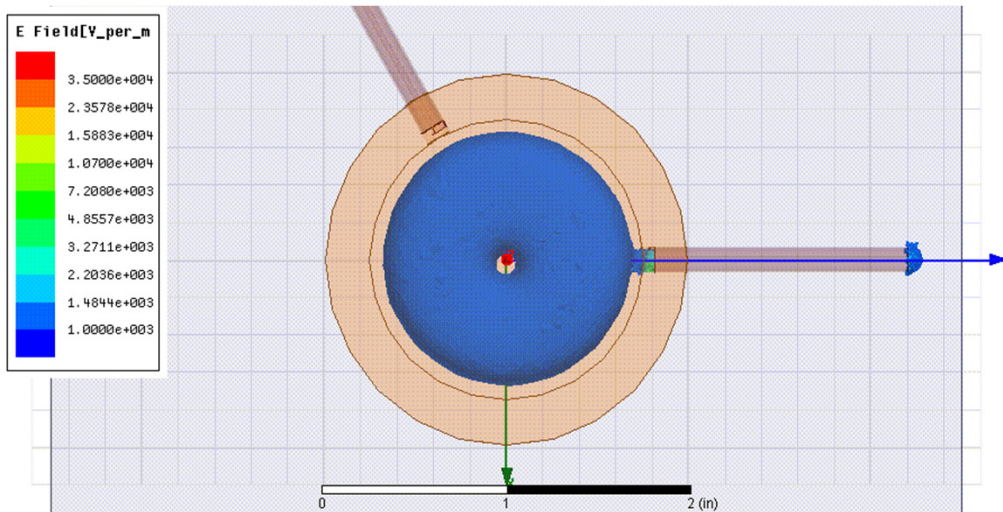
Inside radius about 19 mm

Inside cavity depth about 10 mm

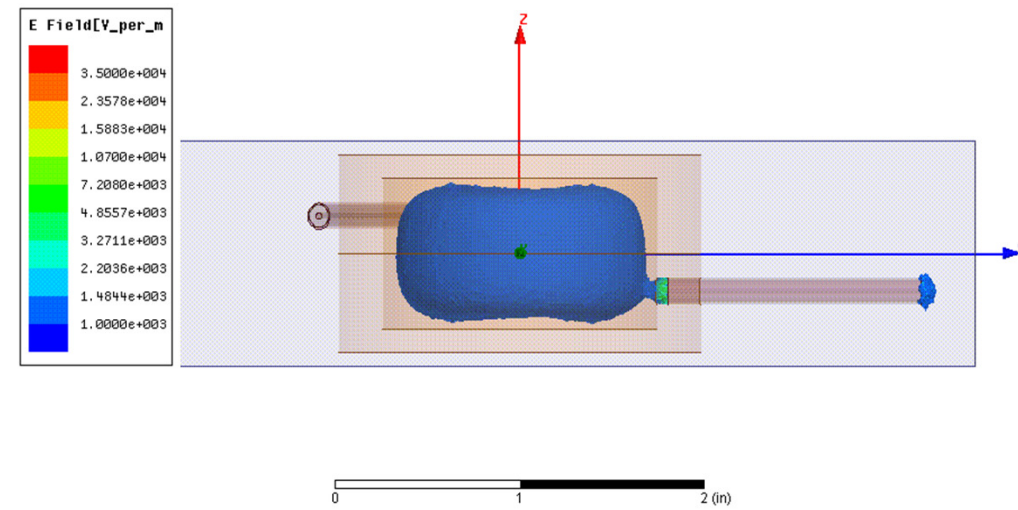


Animation of E Fields: TE011

TOP VIEW



SIDE VIEW



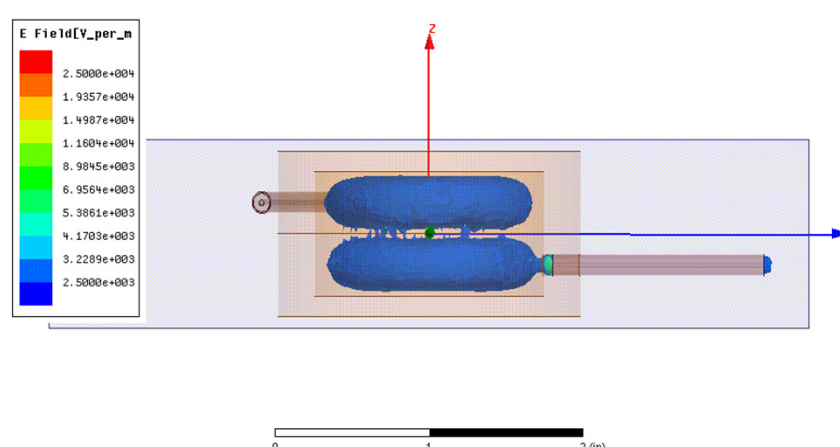
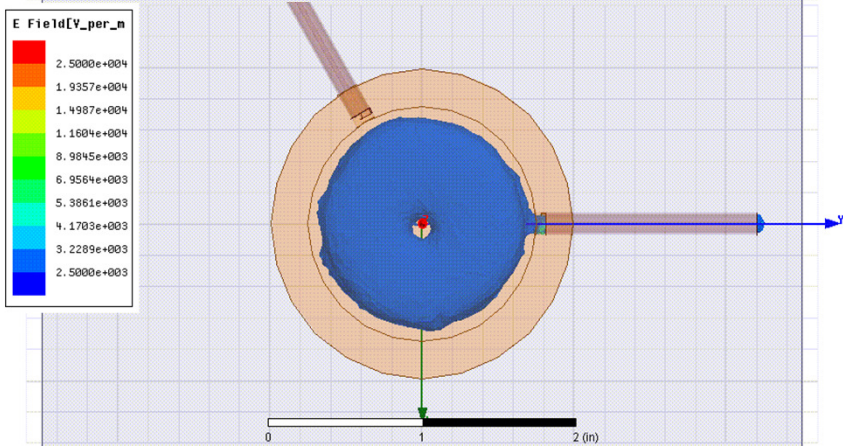
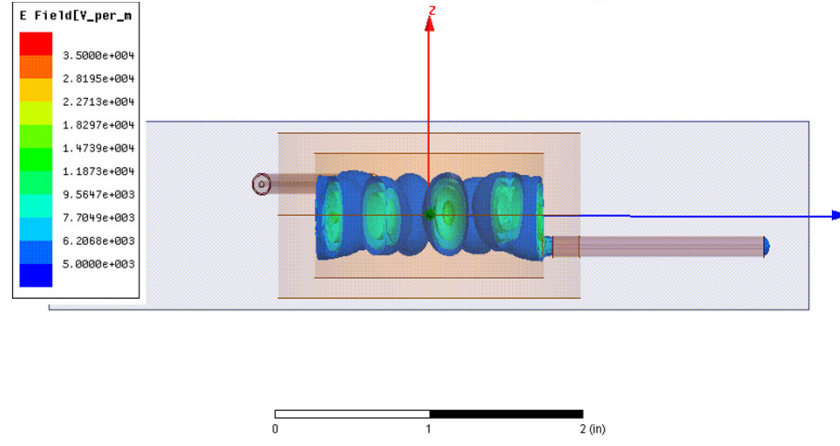
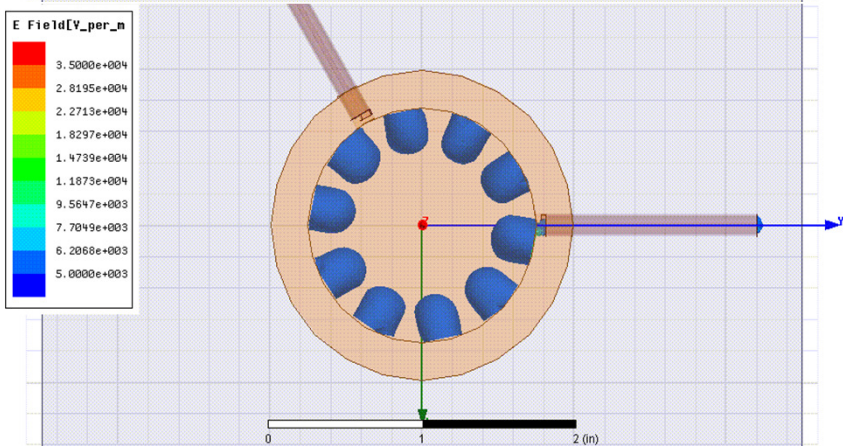
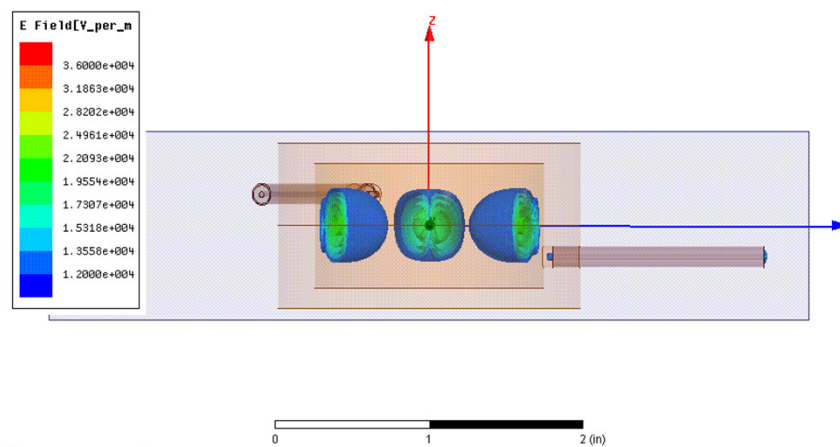
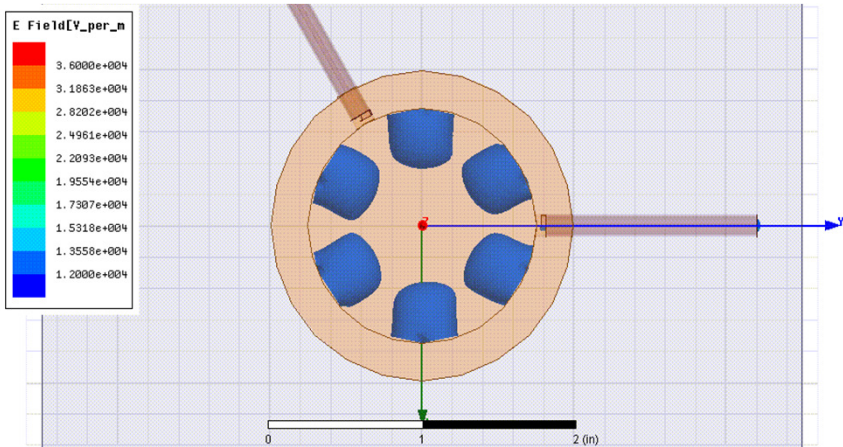
Big thanks to Brad Thrasher at DuPont for helping generate these HFSS models.

Split Cylinder Resonator – Higher Order Modes

OK

OK

NO!!



Split Cylinder – Summary Results

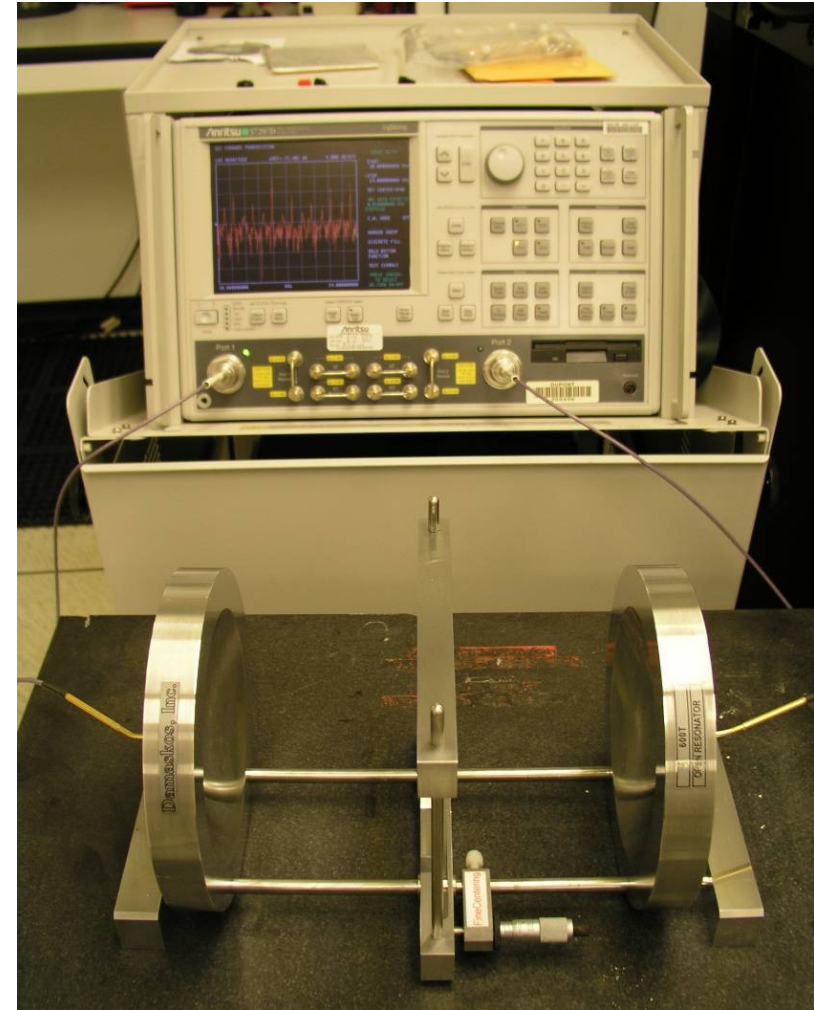
Sample	F(GHz)	Thickness (mm)		Permittivity		Loss Tangent	
		mean	StDev	Er	+/-	Tan δ	+/-
Teflon® AF	11.10	1.475	0.033	1.871	0.032	0.00024	0.00014
PTFE Teflon® Type A	11.29	1.032	0.019	2.027	0.028	0.00028	0.00013
PTFE Teflon® Type B	11.27	1.034	0.022	2.037	0.031	0.00026	0.00010
PFA Teflon® Type A	10.36	2.474	0.076	2.049	0.041	0.00036	0.00021
PFA Teflon® Type B	10.39	2.476	0.069	2.024	0.038	0.00037	0.00019
Teflon® FEP	10.30	2.554	0.074	2.067	0.039	0.00036	0.00020
Tefzel® ETFE	10.10	2.457	0.038	2.311	0.025	0.00668	0.00053
9K7 LTCC	9.42	0.876	0.014	7.042	0.021	0.00085	0.00009

Jim Parisi of DuPont took these measurements.

Resonator Method for 15-65 GHz

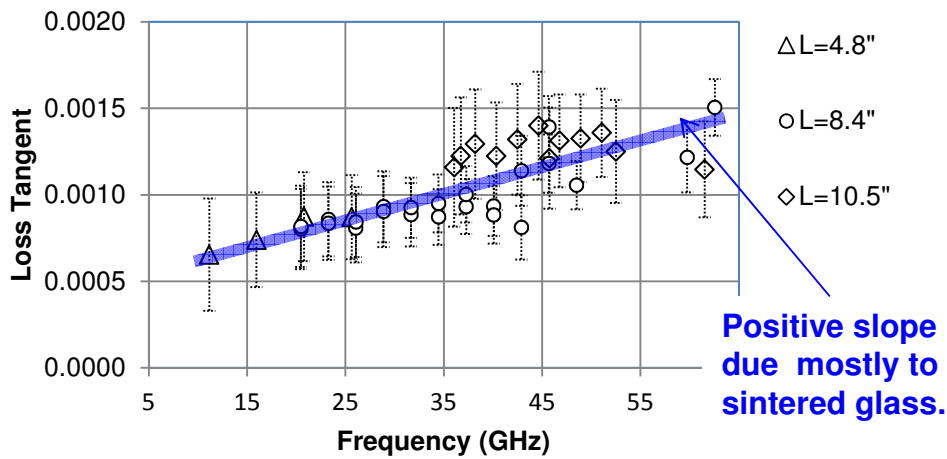
Open Resonator (Fabry-Perot)

- Hemispherical Mirrors
- Distance between mirrors allow for different useable bandwidths
 - Close together good at 20 GHz
 - Far apart good at 60 GHz
- Very precise ($Q > 10^5$ common)
- Quite tedious – thermal expansion causes cavity length to change due to very slight temperature changes

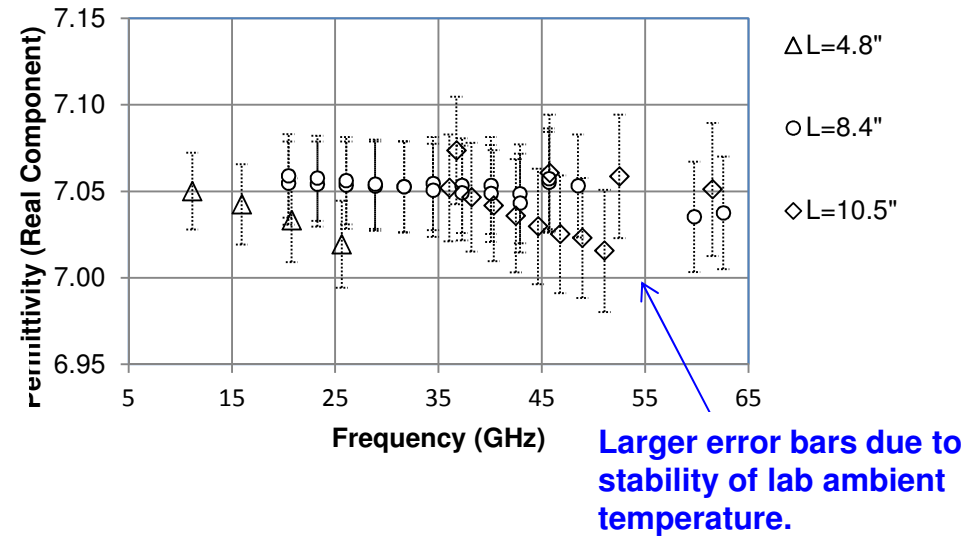


Open Resonator Measurements

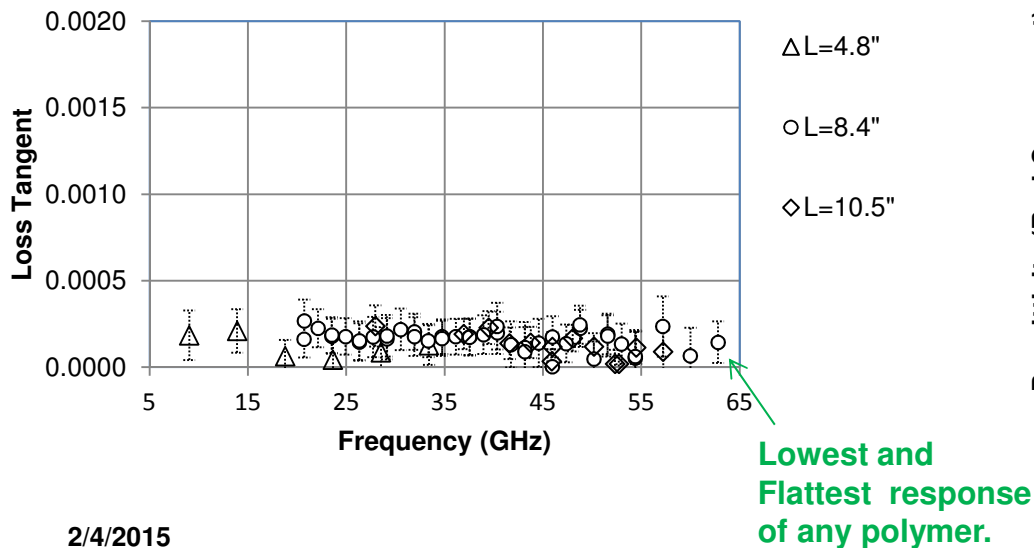
Green Tape™ 9K7 LTCC



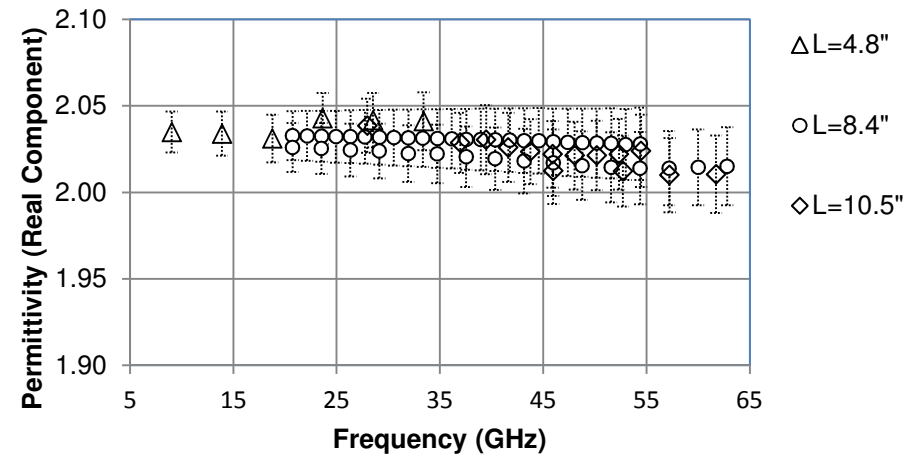
Green Tape™ 9K7 LTCC



PTFE Teflon® Type A

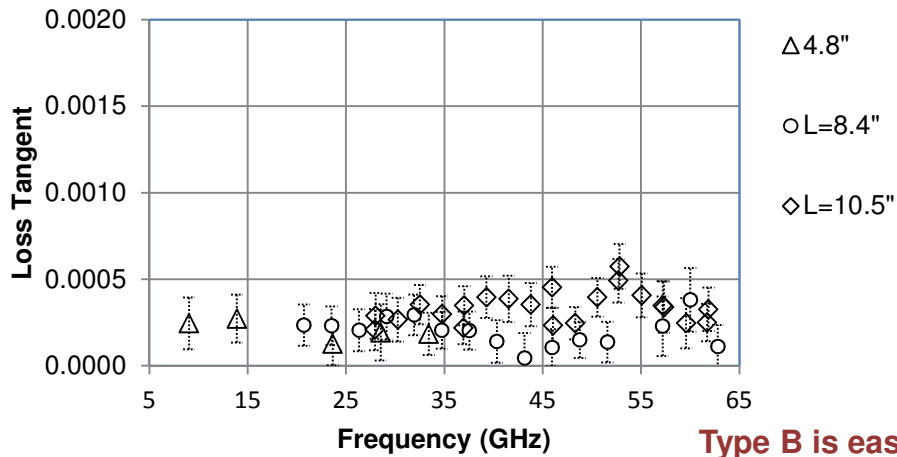


PTFE Teflon® Type A



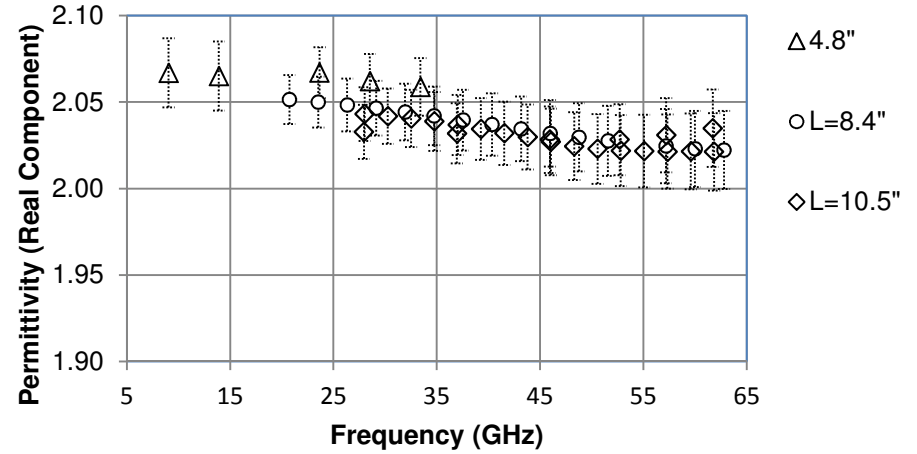
Open Resonator Measurements

PTFE Teflon® Type B

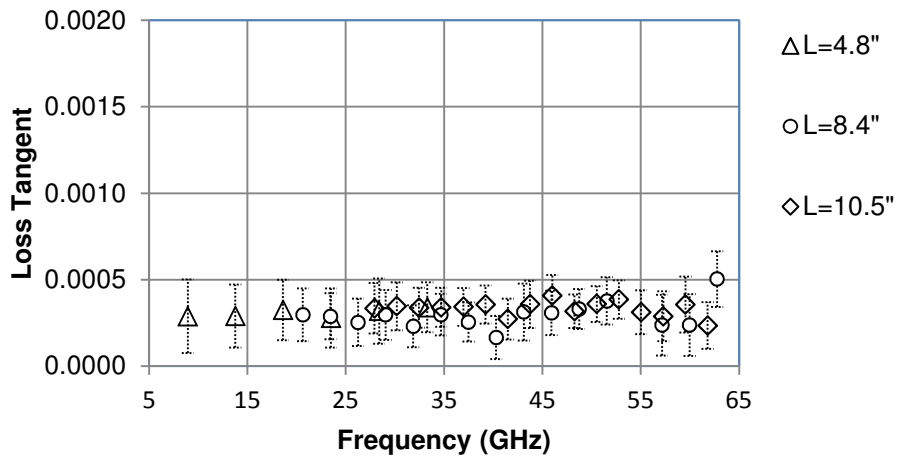


Type B is easier to process than Type A

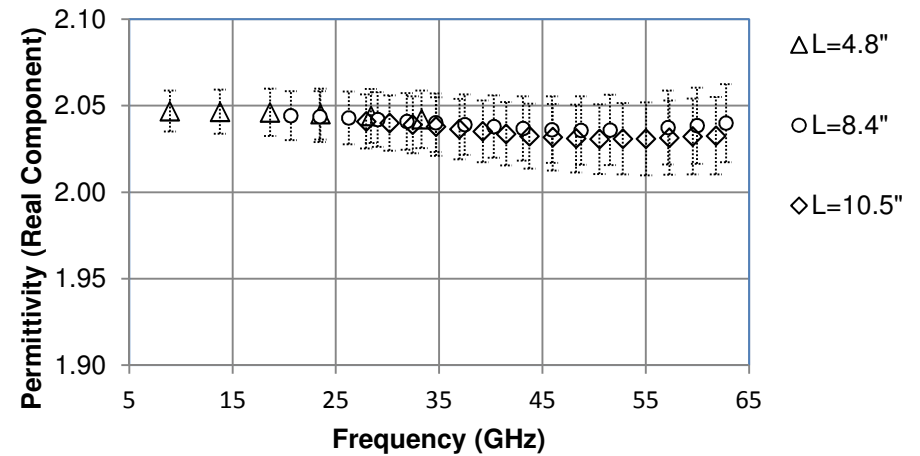
PTFE Teflon® Type B



Teflon® FEP

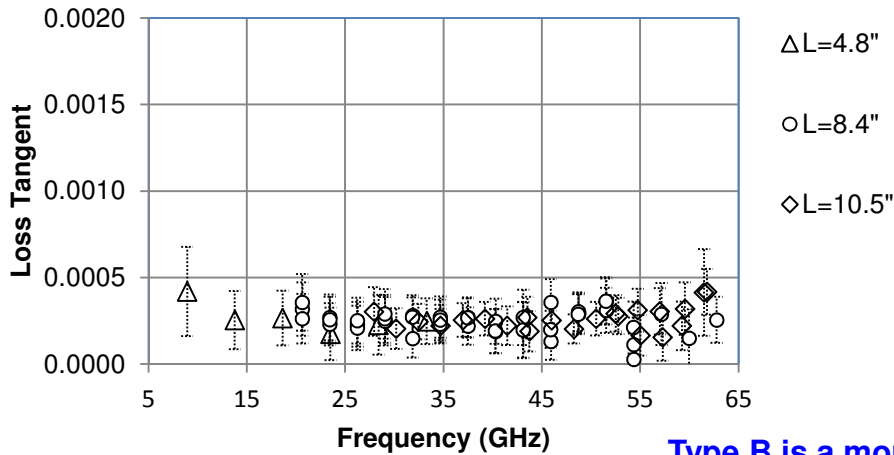


Teflon® FEP



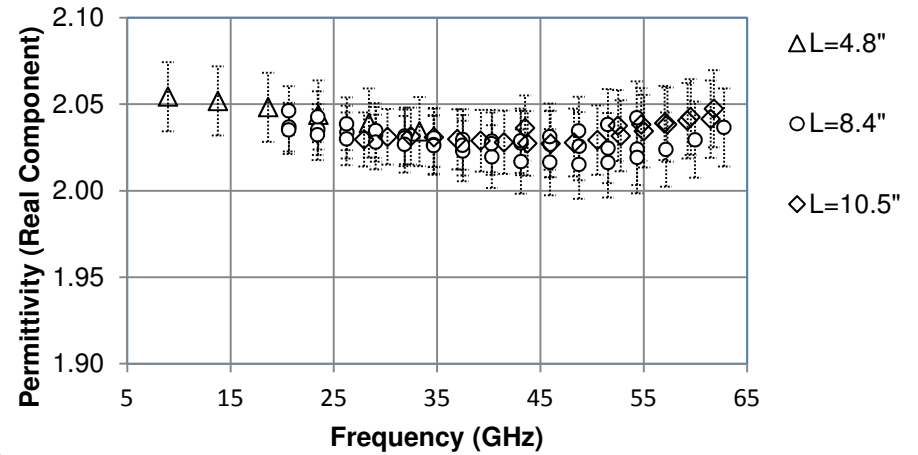
Open Resonator Measurements

Teflon® PFA – Type A

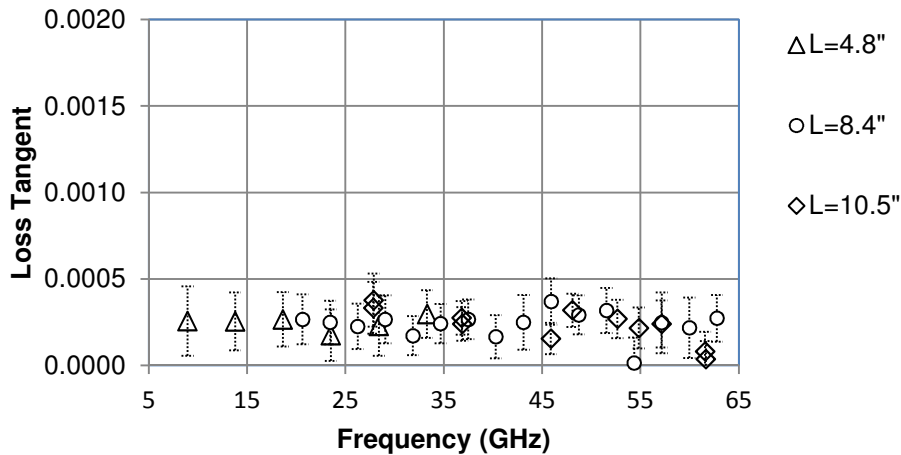


Type B is a more pure grade of PFA than Type A

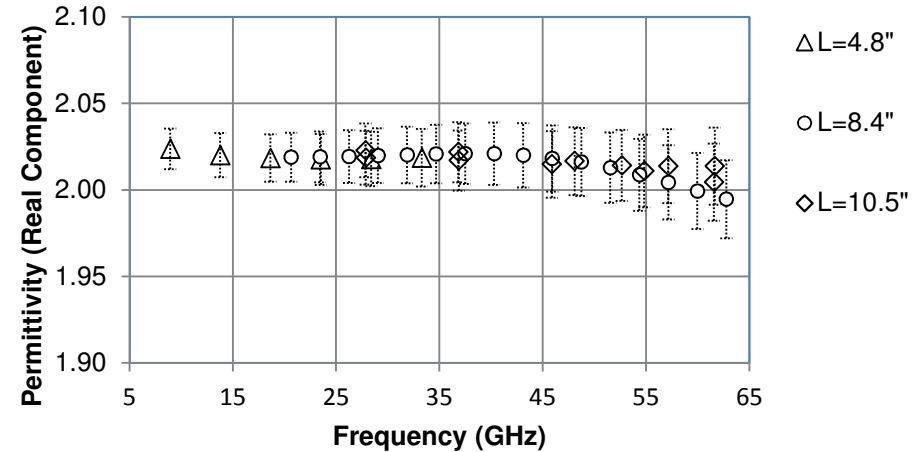
Teflon® PFA – Type A



Teflon® PFA – Type B

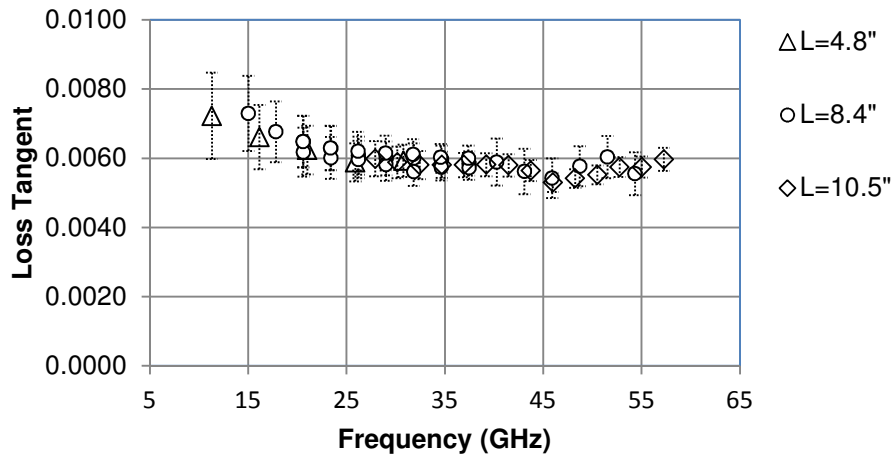


Teflon® PFA – Type B

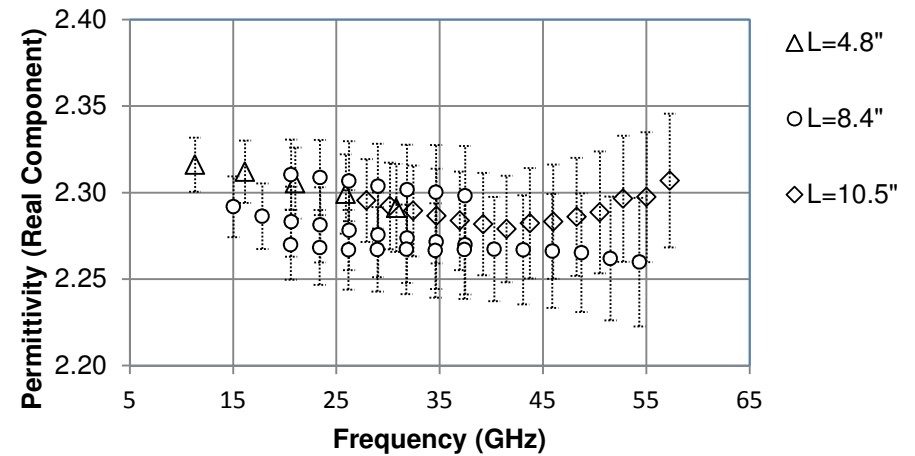


Open Resonator Measurements

Tefzel® ETFE

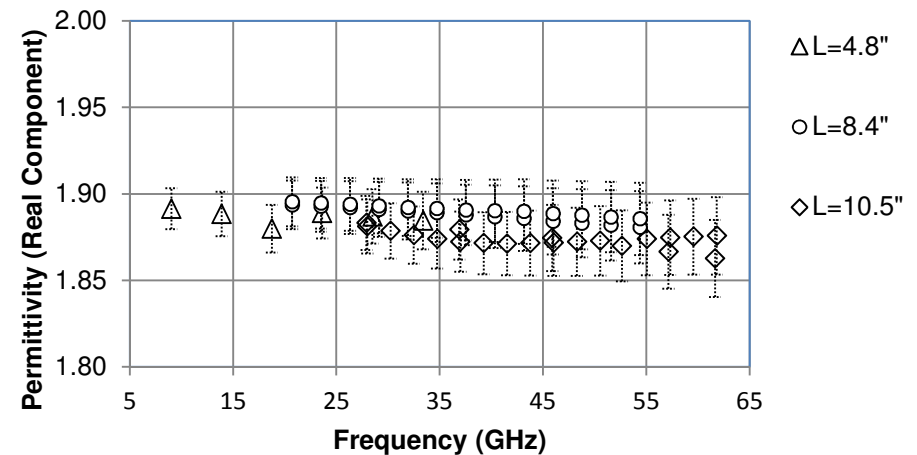
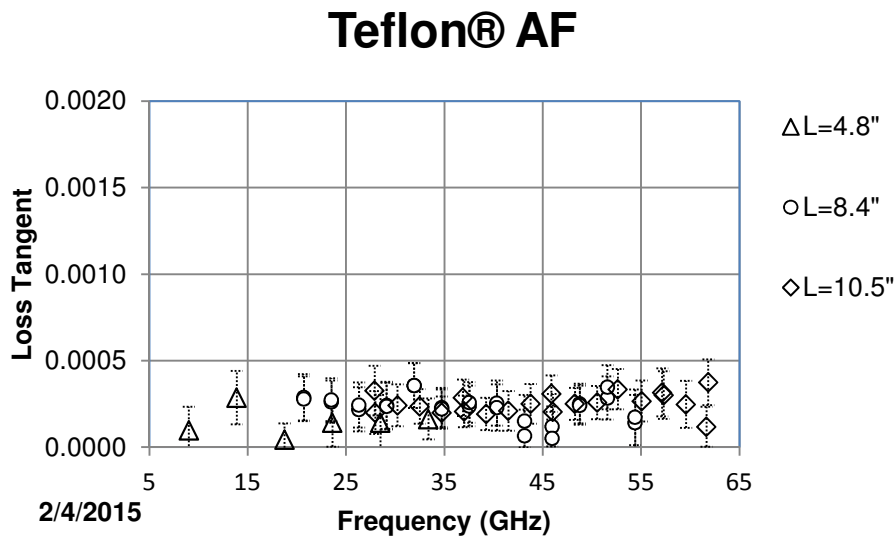


Tefzel® ETFE



Note: order of magnitude difference in loss tangent between Teflon® and Tefzel®

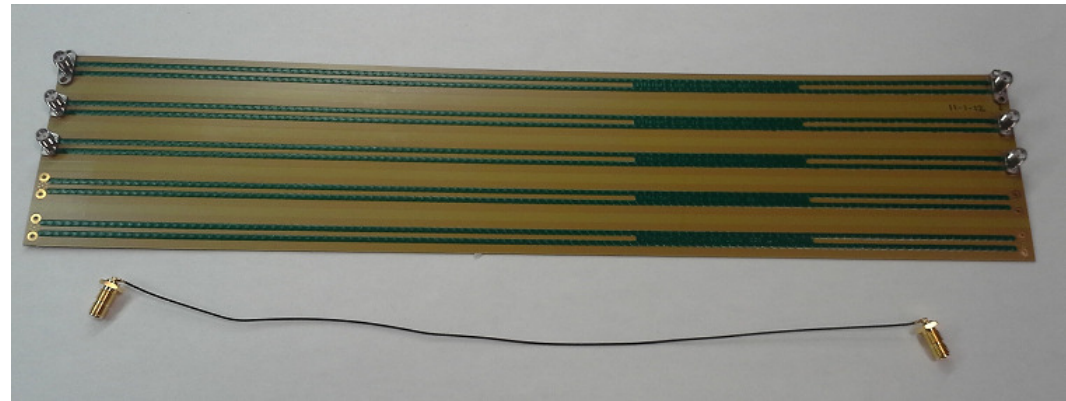
Teflon® AF



Transmission Lines

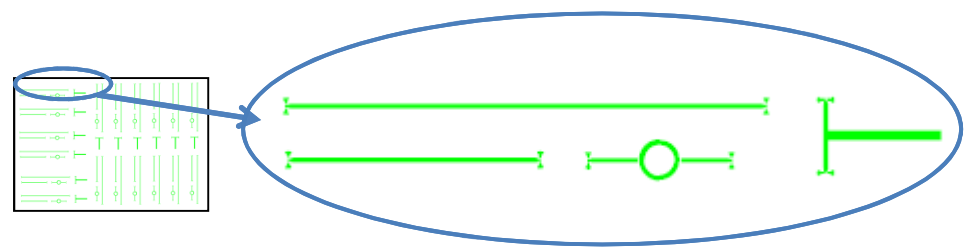
Connector Based

- Can measure up to 65 GHz with standard connectors.
- Does not require special test fixtures or probes.



Probe Based

- Up to 110 GHz capability is common. Some systems are available into the THz regime, but are mostly confined to Research institutions..
- Requires both great expense and expertise to correctly measure.



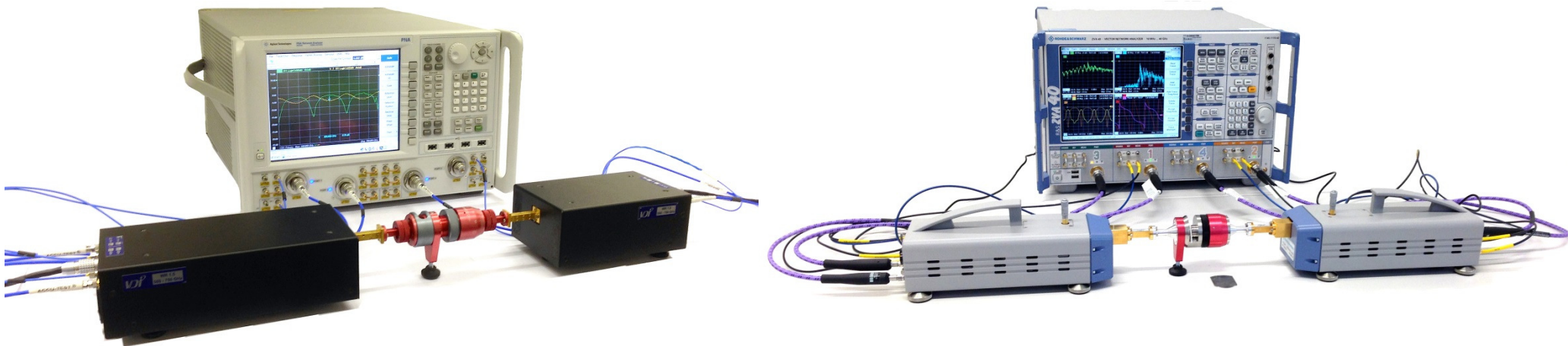


Measurements of du Pont materials by SWISSto12
using its MCK product

January 2015

SWISSto12 Material Characterization Kit (MCK)

- SWISSto12 MCK enables mm-wave to THz Materials Measurements:
 - Fast (Real Time)
 - Calibrated
 - Simplified Set up
 - Banded solution from WR15+ (47-77 GHz) up to WM250 (750-1100GHz)
 - No Sample preparation
 - Software supplied for data Analysis
- Measurements require a Vector Network Analyzer possibly with millimeter wave frequency extenders/converters

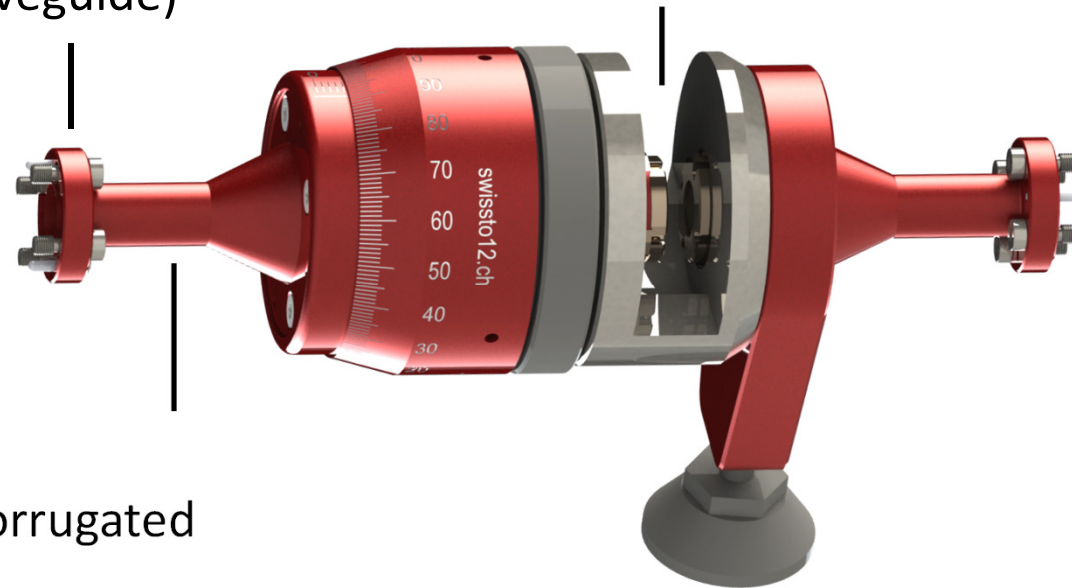


SWISSto12 Material Characterization Kit (MCK)

Concept: 2-Port configuration (S11, S21)

Connection to VNA
(Rectangular waveguide)

Gap for sample

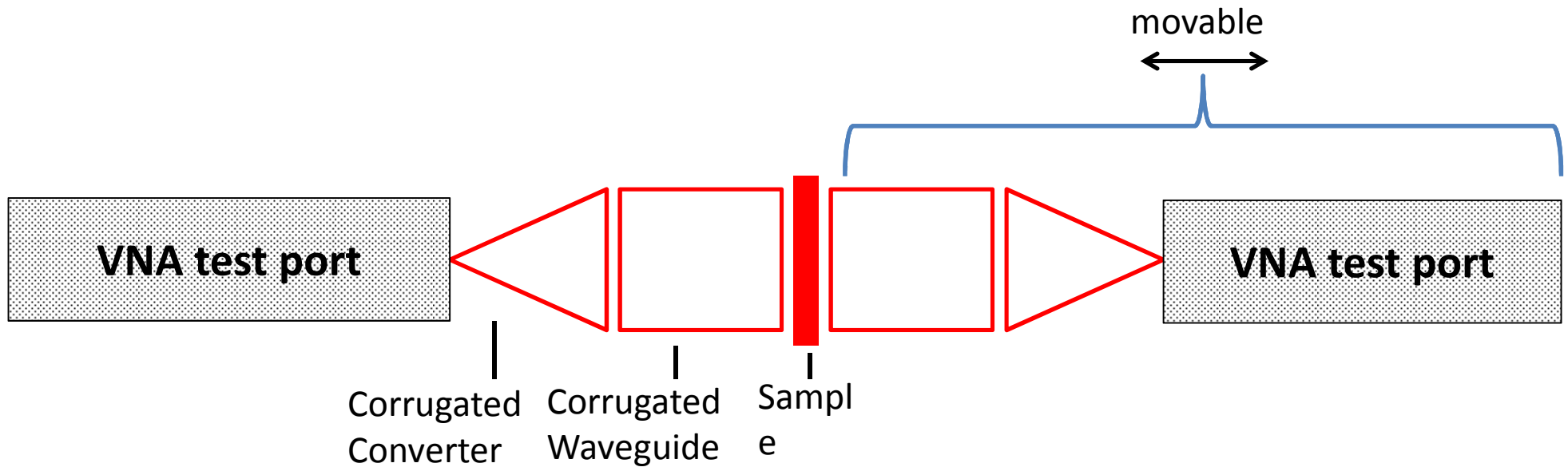


Converter from
rectangular to corrugated
waveguide

- The **sample is clamped into a gap** between two Corrugated waveguides
- The gap does not perturb signal propagation: **“guided free-space”** approach
- Samples are exposed to a beam with a **plane phase front**
- Minimum measurement configuration needs **only S21 and S11 data**

SWISSto12 Material Characterization Kit (MCK)

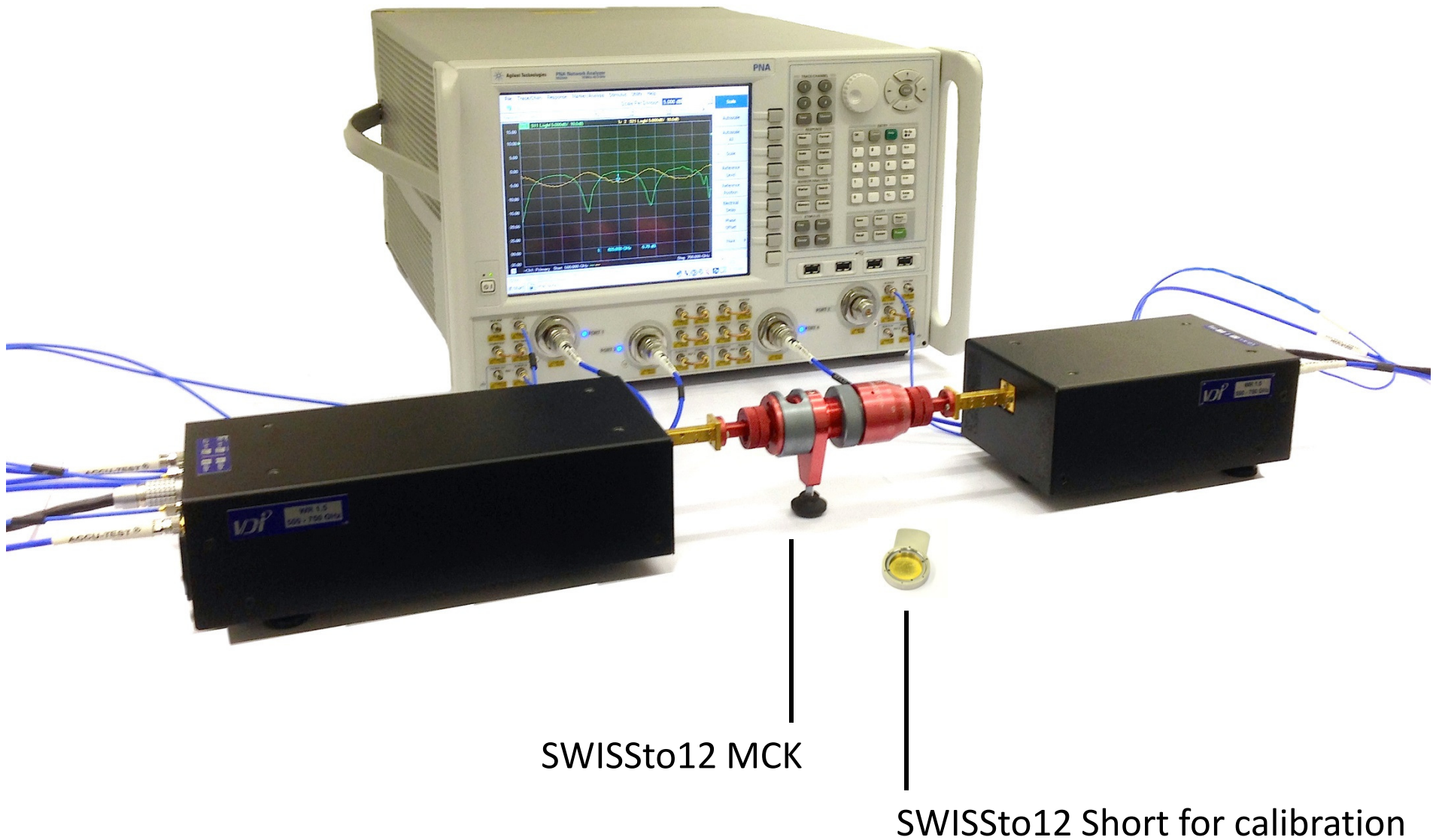
Fast Measurement Sequence



1. Re-normalize S21 data by measuring a **“through”** (no sample and no gap in the waveguide line)
2. Re-normalize S11 data by measuring a **“short”** (Reflecting mirror clamped in the gap)
3. Clamp the sample, measure S21 and S11 time gated **data**
4. Post-process the S parameter data with the SWISSto12 materials measurements **software**

Material Characterization KIT (MCK)

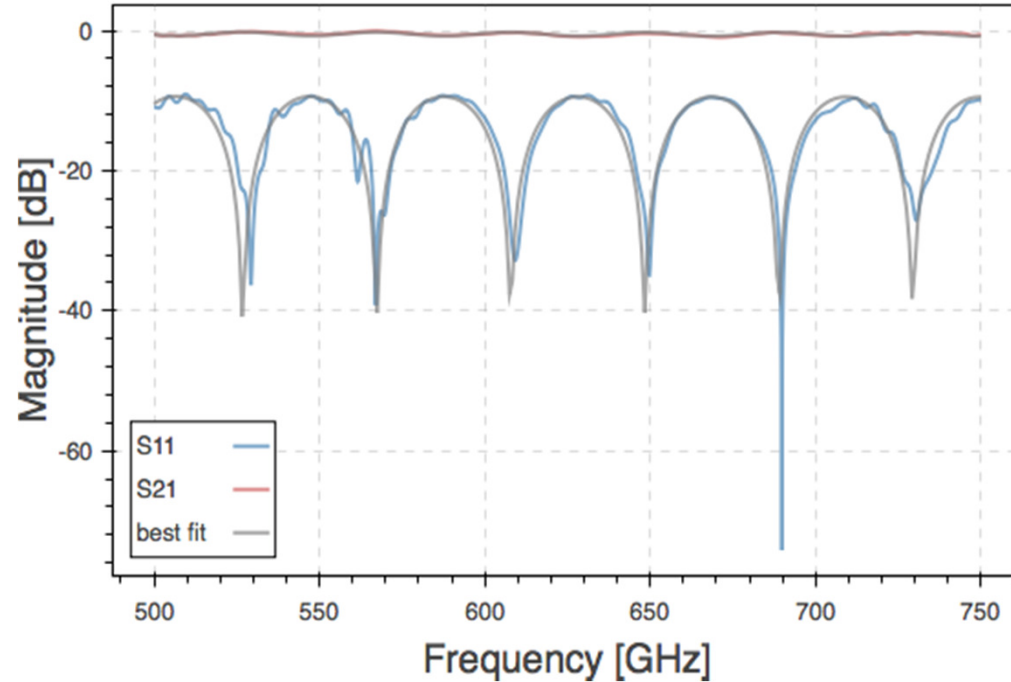
Example: setup in WR-1.5 band (500-750 GHz)



DuPont Teflon[®] FEP (constant permittivity assumed)

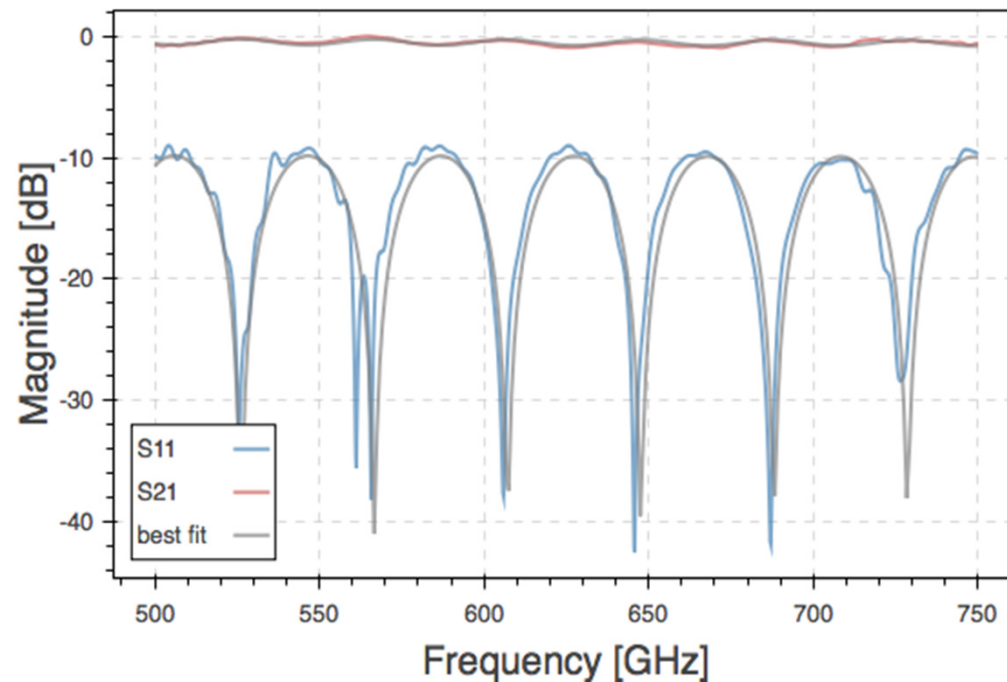
Measurement 1

- Thickness = 2.58 mm
- $\epsilon = 2.06$
- $\text{Tan}\delta = 1.08 \text{ E-}3$



Measurement 2

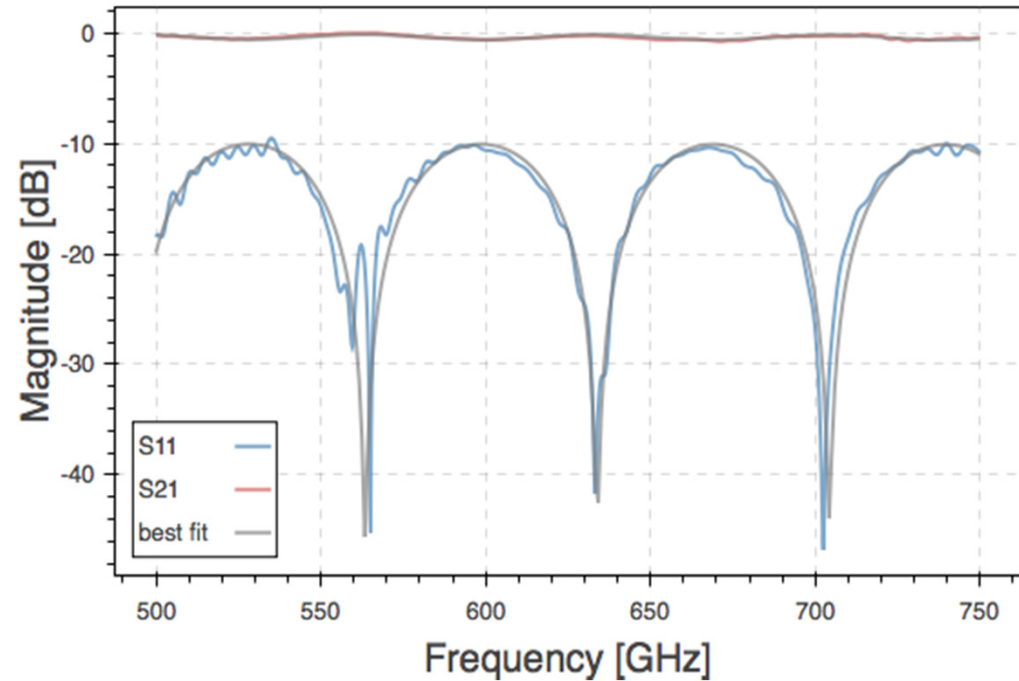
- Thickness = 2.63 mm
- $\epsilon = 1.98$
- $\text{Tan}\delta = 1.19 \text{ E-}3$



DuPont Teflon[®] AF (constant permittivity assumed)

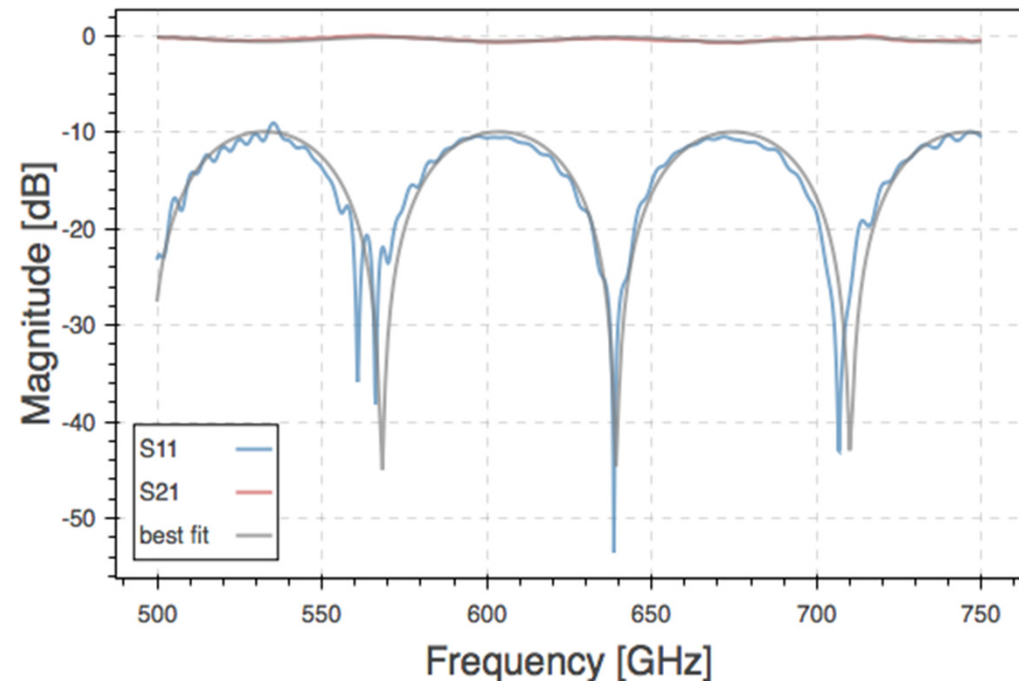
Measurement 1

- Thickness = 1.53 mm
- $\epsilon = 1.94$
- $\text{Tan}\delta = 1.21 \text{ E-}3$



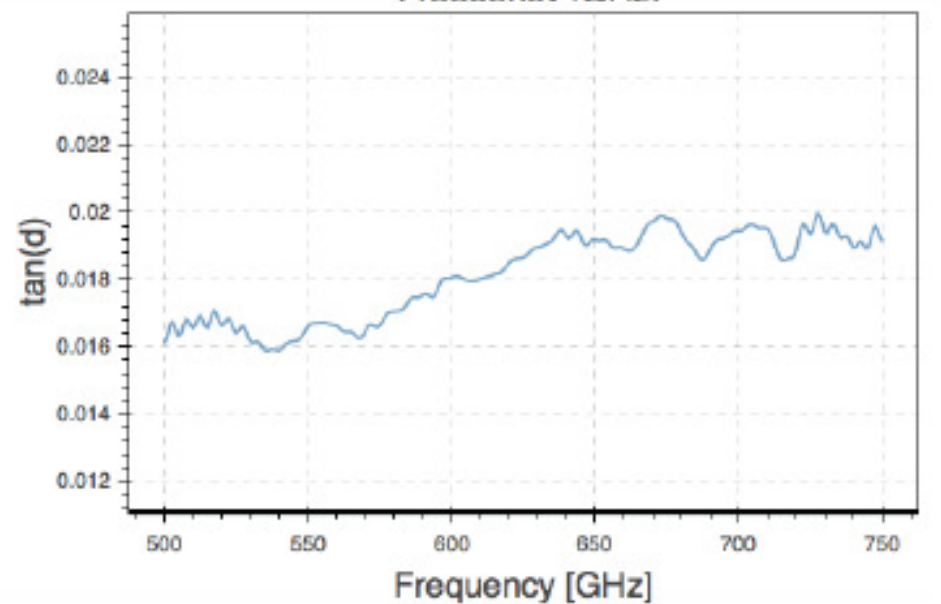
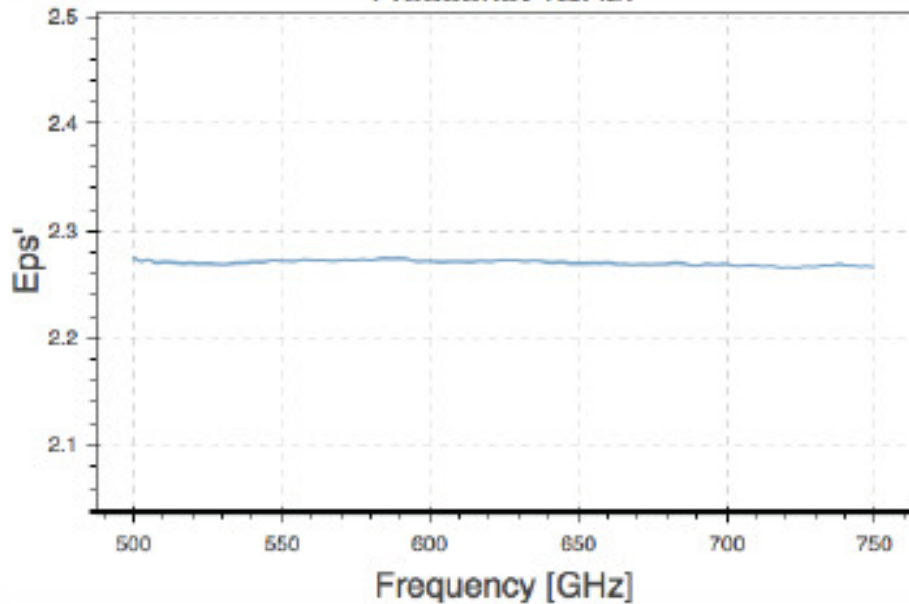
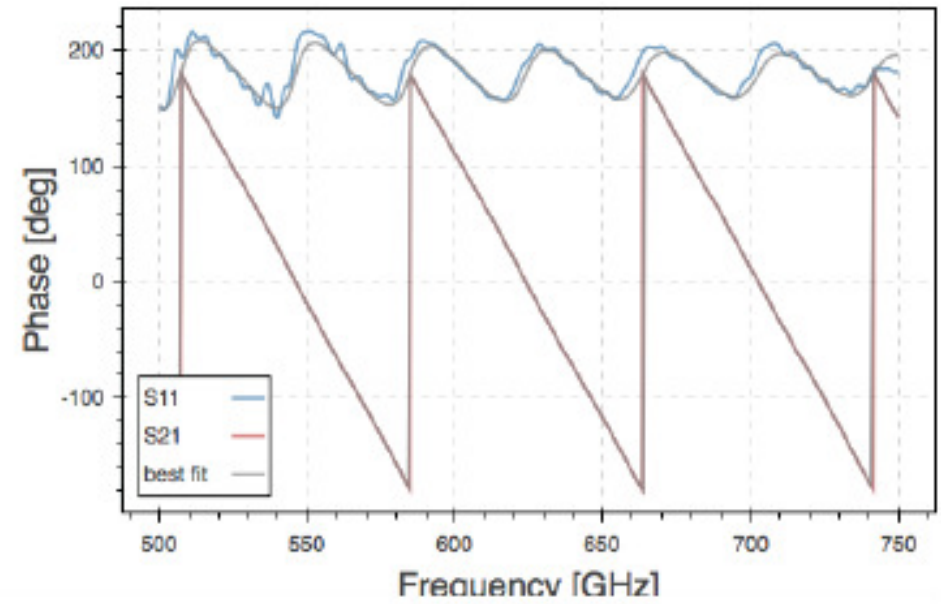
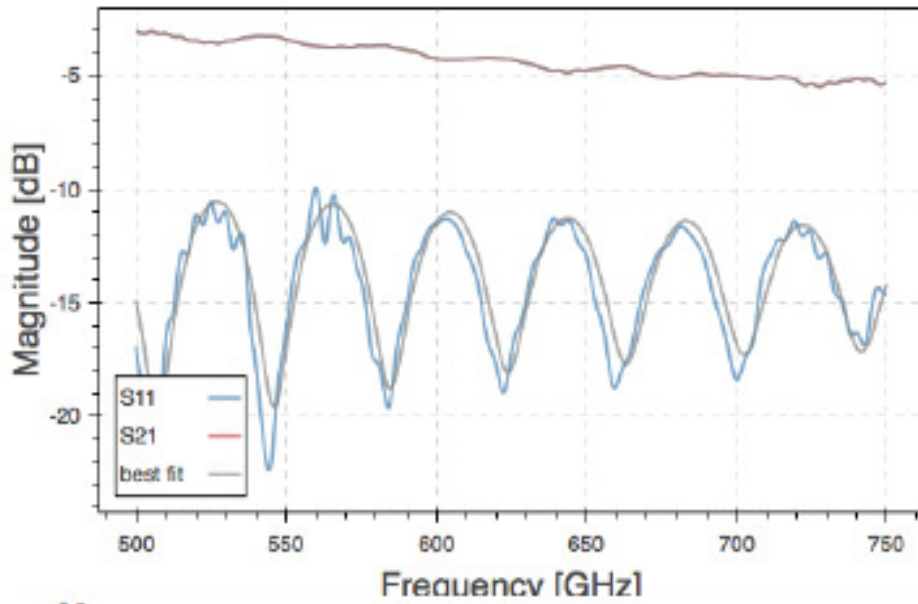
Measurement 2

- Thickness = 1.51 mm
- $\epsilon = 1.95$
- $\text{Tan}\delta = 1.25 \text{ E-}3$

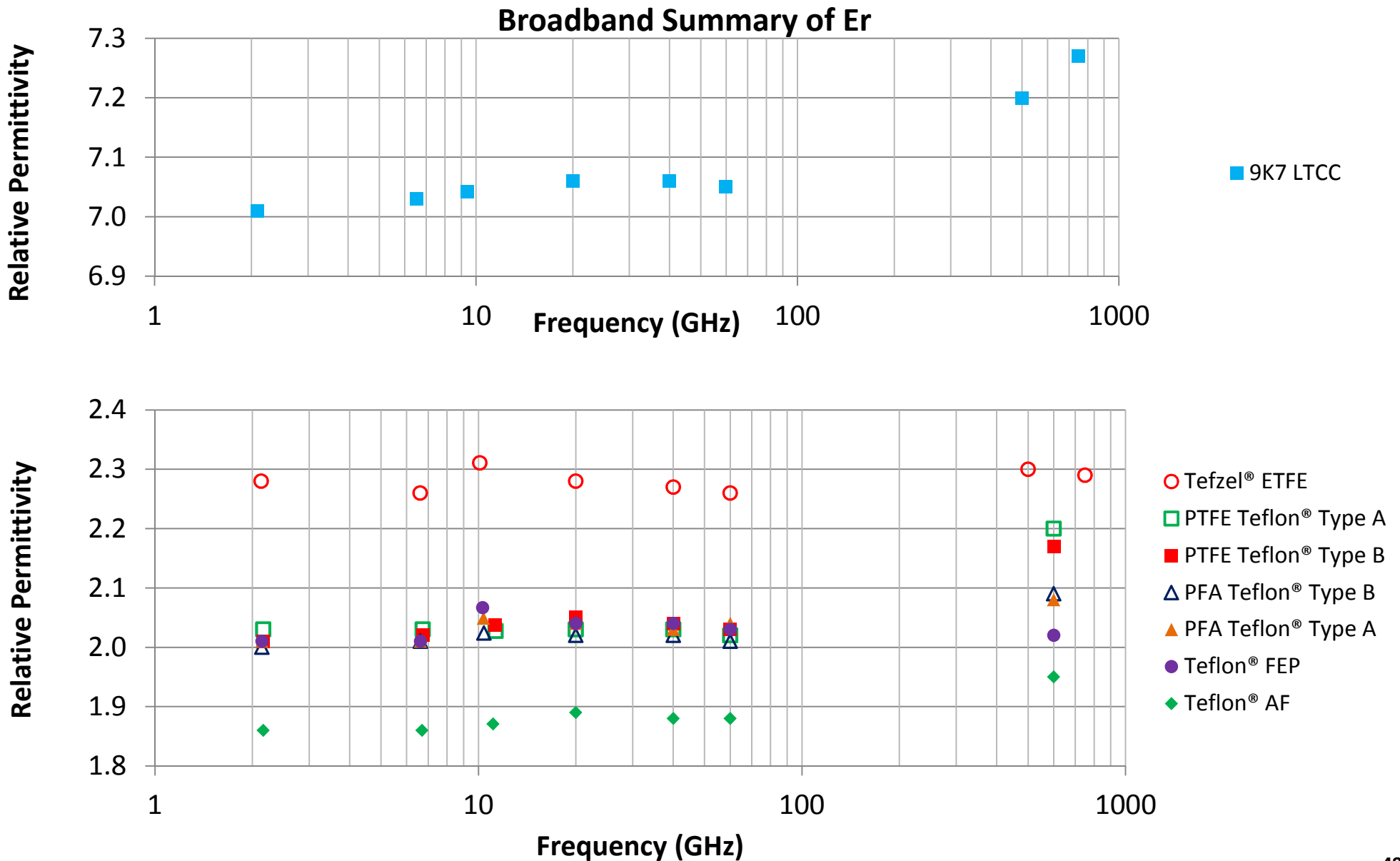


DuPont Tefzel® ETFE Frequency Dependent Model

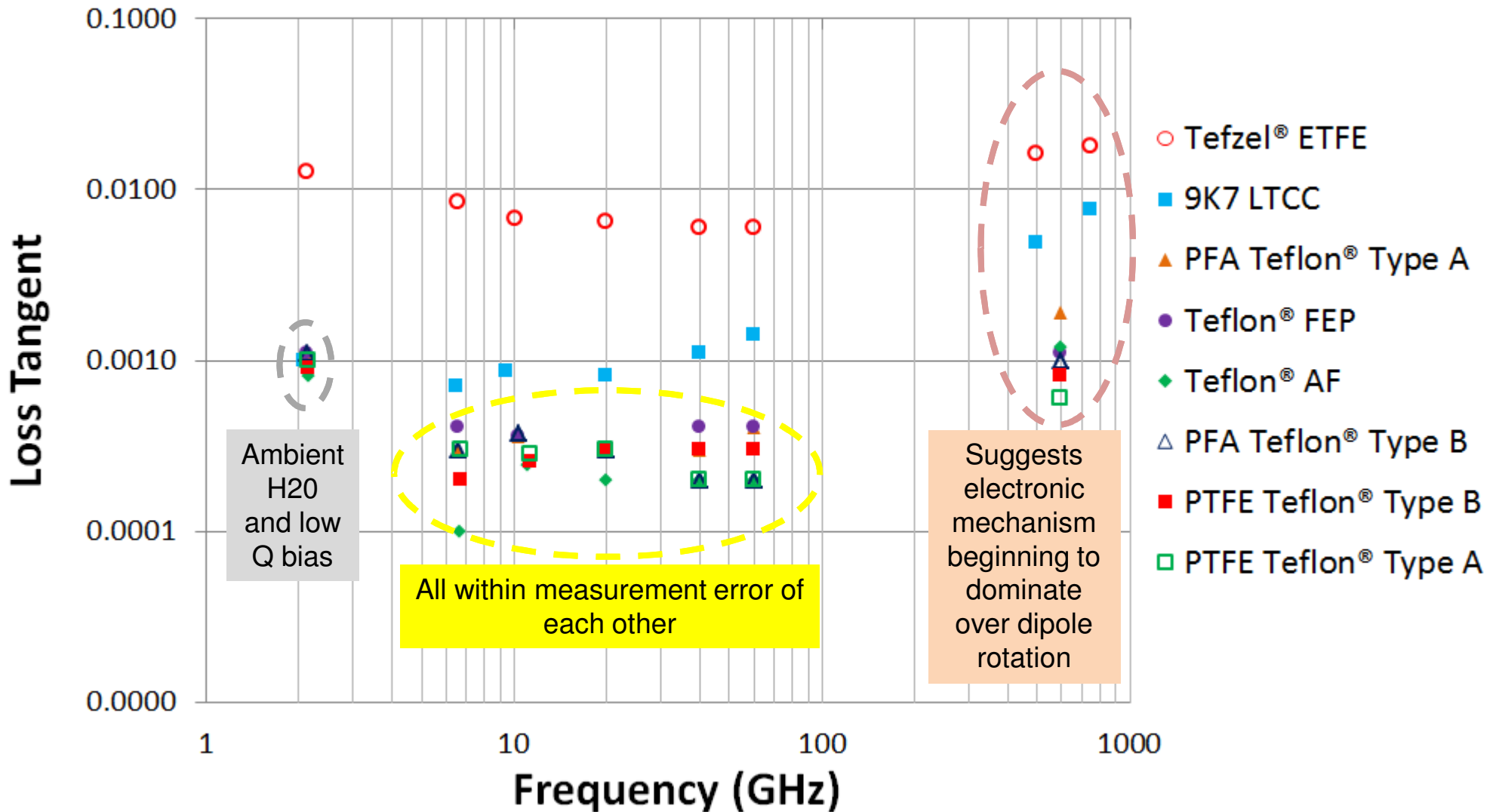
2.55 mm thick sample



Summary of All Measurements - Er



Broadband Summary of Loss Tangent



Summary Table with Uncertainty

Teflon® AF			Tan d	
F(GHz)	Er	+/-	x10 ⁻³	+/-
2.16	1.86	0.07	0.8	0.7
6.69	1.86	0.04	0.1	0.3
11.10	1.871	0.032	0.24	0.21
20	1.89	0.03	0.2	0.2
40	1.88	0.03	0.2	0.2
60	1.88	0.03	0.2	0.3
600	1.95	2%	1.2	10%

PTFE Teflon® Type A			Tan d	
F(GHz)	Er	+/-	x10 ⁻³	+/-
2.16	2.03	0.06	1.0	0.8
6.72	2.03	0.04	0.3	0.3
11.29	2.027	0.028	0.28	0.13
20	2.03	0.02	0.3	0.2
40	2.03	0.03	0.2	0.2
60	2.02	0.04	0.2	0.3
600	2.20	2%	0.6	10%

PTFE Teflon® Type B			Tan d	
F(GHz)	Er	+/-	x10 ⁻³	+/-
2.16	2.01	0.07	0.9	0.8
6.72	2.02	0.04	0.2	0.3
11.27	2.037	0.031	0.26	0.10
20	2.05	0.03	0.3	0.2
40	2.04	0.03	0.3	0.3
60	2.03	0.04	0.3	0.3
600	2.17	2%	0.8	10%

PFA Teflon® Type A			Tan d	
F(GHz)	Er	+/-	x10 ⁻³	+/-
2.14	2.01	0.08	1.1	0.8
6.62	2.01	0.05	0.3	0.3
10.36	2.049	0.041	0.36	0.19
20	2.04	0.04	0.3	0.3
40	2.03	0.03	0.3	0.3
60	2.04	0.04	0.4	0.3
600	2.08	2%	1.9	10%

PFA Teflon® Type B			Tan d	
F(GHz)	Er	+/-	x10 ⁻³	+/-
2.14	2.00	0.08	1.1	0.8
6.62	2.01	0.05	0.3	0.4
10.39	2.024	0.038	0.37	0.20
20	2.02	0.03	0.3	0.3
40	2.02	0.03	0.2	0.2
60	2.01	0.04	0.2	0.2
600	2.09	2%	1.0	10%

Teflon® FEP			Tan d	
F(GHz)	Er	+/-	x10 ⁻³	+/-
2.14	2.01	0.08	1.1	0.8
6.61	2.01	0.05	0.4	0.4
10.30	2.067	0.039	0.36	0.14
20	2.04	0.03	0.3	0.2
40	2.04	0.03	0.4	0.3
60	2.03	0.04	0.4	0.3
600	2.02	2%	1.1	10%

Tefzel® ETFE			Tan d	
F(GHz)	Er	+/-	x10 ⁻³	+/-
2.13	2.28	0.07	12.6	0.8
6.60	2.26	0.04	8.4	0.4
10.10	2.311	0.025	6.68	0.53
20	2.28	0.07	6.5	0.8
40	2.27	0.06	6.0	0.4
60	2.26	0.06	5.9	0.4
500	2.30	2%	16	10%
750	2.29	2%	18	10%

9K7 LTCC			Tan d	
F(GHz)	Er	+/-	x10 ⁻³	+/-
2.09	7.01	0.05	1.0	0.6
6.53	7.03	0.03	0.7	0.3
9.41	7.042	0.021	0.85	0.09
20	7.06	0.04	0.8	0.3
40	7.06	0.04	1.1	0.5
60	7.05	0.05	1.4	0.4
500	7.20	2%	4.8	10%
750	7.27	2%	7.6	10%