

Electrical Properties of MWCNTs Nanocomposites in Microwave Frequency Range



Sixth Palestinian Conference on Modern Trends
in Mathematics and Physics PCMTMP-VI

Muna HajjYahya¹, Patrizia Savi², Mauro Giorcelli³

¹Department of Physics, An-Najah National University, Nablus, Palestine

²Department of Electronics and Telecommunications, Politecnico di Torino, Torino, Italy

³Department of Applied Science and Technology, Politecnico di Torino, Torino, Italy

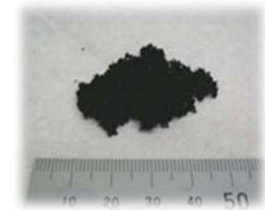
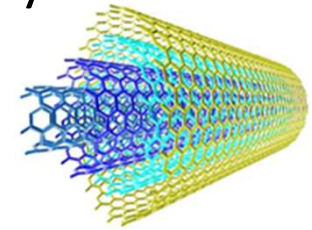
Research Activity Context

Benefits of CNTs to polymers

Increase of Electrical conductivity

Improvement of mechanical properties, especially strength

Enhancement thermal stability and conductivity



High aspect ratio



Effects at low CNT contents
< 5wt.%

MWCNTs polymer composites

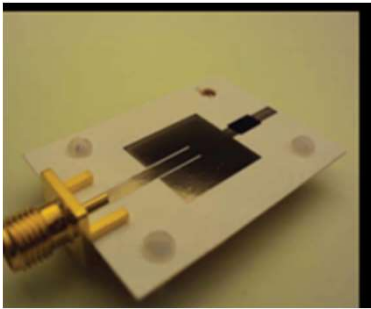


Involved

Many microwave applications

Potential Application Fields of CNTs

GROWING INTEREST IN NEW MATERIALS FOR:



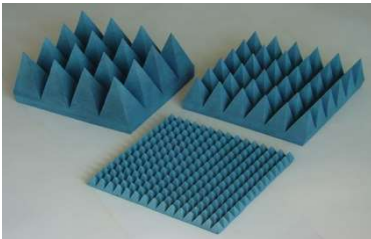
Antennas Design



Car enamel



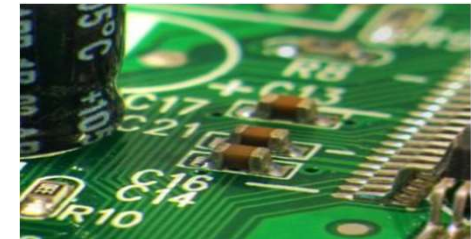
Electromagnetic Shielding



**Radar Absorbing Material
(RAM)**



**Satellite and automotive
applications**



Capacitors (charge storage)

Objectives of the Research Activity

Nanocomposites preparation

Goals:

- To obtain a good dispersion

DC conductivity measurements

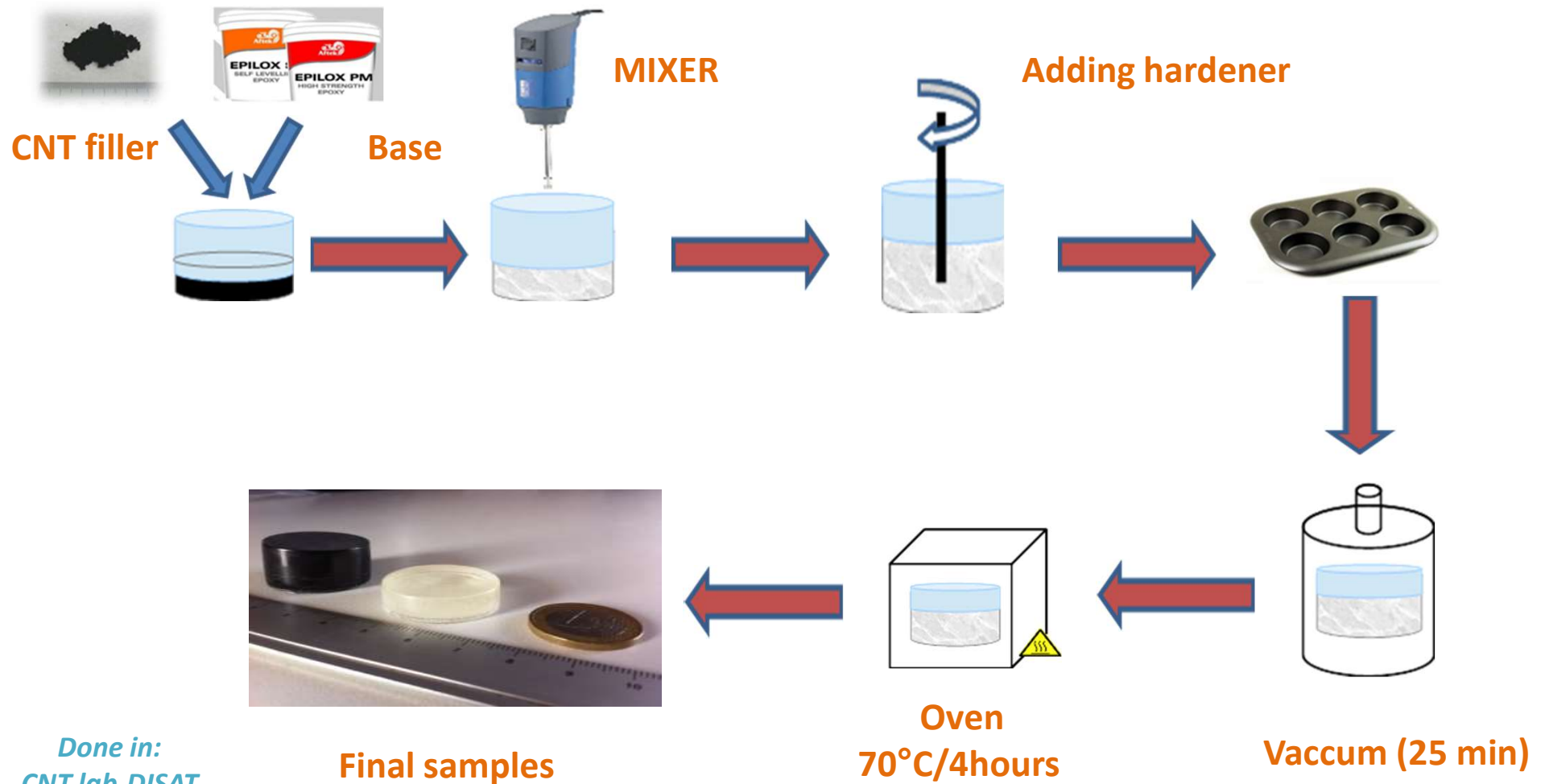
- Percolation threshold of MWCNTs

Complex permittivity measurements

Goals:

- Measure the complex permittivity with:
 - Small samples
 - Wide frequency band
 - Easy and fast method
- Statistical analysis independence of the samples

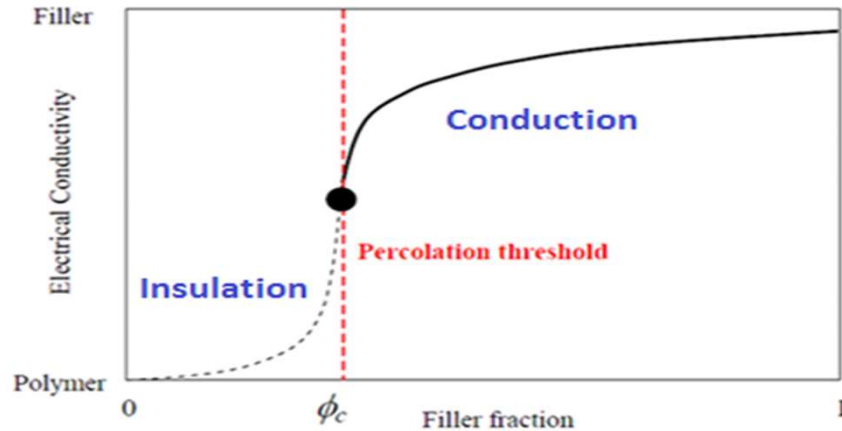
Nanocomposites Preparation - Polymer Based MWCNTs Reinforced Composites Preparation Procedure



Nanocomposites Preparation - Types of Multi-Walled Carbon Nanotubes Used

Acronym	Type	Diameter [nm]	Length [μm]	Aspect Ratio (AR) (Length/ Diameter)	Producer	Purity (weight %)
MW0	Short Multi Wall	30 – 50	0.5 – 2	17 - 40	Cheaptubes	> 95
MW3	Short Thin Multi Wall	9.5	1.5	150	Nanocyl	> 95
MWG-1	Graphitized MW	20-30	10-30	500 - 1000	Cheaptubes	>99.5
F4	-COOH Functional MW	9.5	1.5	150	Nanocyl	> 95
E15	Multi Wall	25 – 45	> 10	200 - 400	Nanothinx	=98.5
E16	Multi Wall	6 – 10	> 10	1000 - 1600	Nanothinx	>90
MW6	Multi Wall	18-35	> 10	285 - 555	Nanothinx	97

Percolation Threshold of MWCNTs



Nanotubes can be conducting or insulating depending on their properties:

- Diameter
- Length
- Chirality/twist
- Number of walls

The most widely used definition is

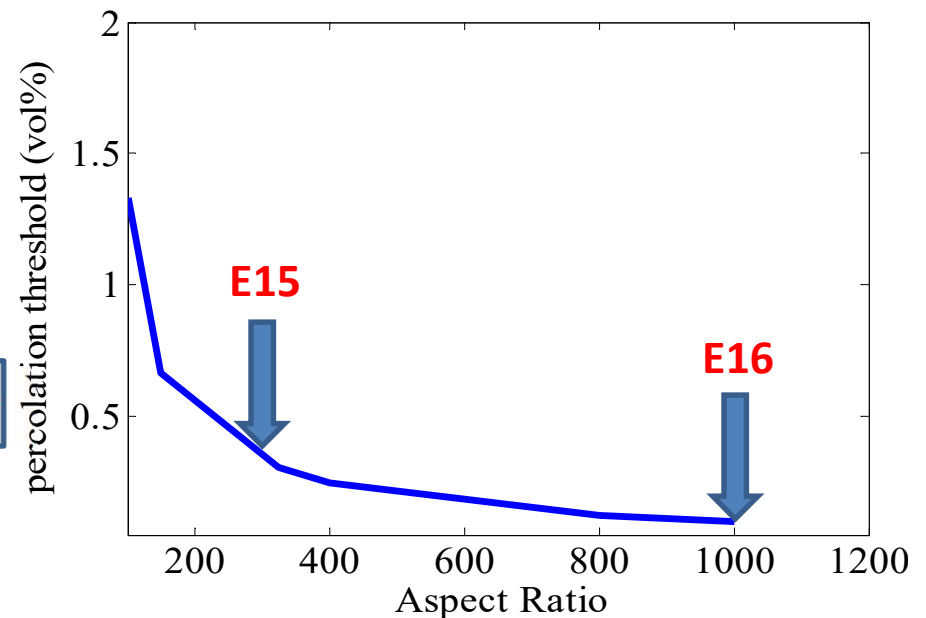
$$P_c = \frac{1}{AR}$$

Other formulas for evaluating percolation threshold :

$$P_c = \frac{1}{2AR}$$

$$P_c = \frac{0.7}{AR}$$

When CNT Vol % > P_c → Reaching conductive behavior



- Bauhofer W, Kovacs JZ: A review and analysis of electrical percolation in carbon nanotube polymer composites. *Compos Sci Technol* 2009, 69:1486–1498.
- Balberg I, Anderson CH, Alexander S, Wagner N. Excluded volume and its relation to the onset of percolation. *Phys Rev B* 1984;30(7):3933-43.
- Celzard A, McRae E, Deleuze C, Dufort M, Furdin G, Marêchê JF. Critical concentration in percolating systems containing a high-aspect-ratio filler. *Phys Rev B* 1996;53(10):6209-14.

Resistance Measurements

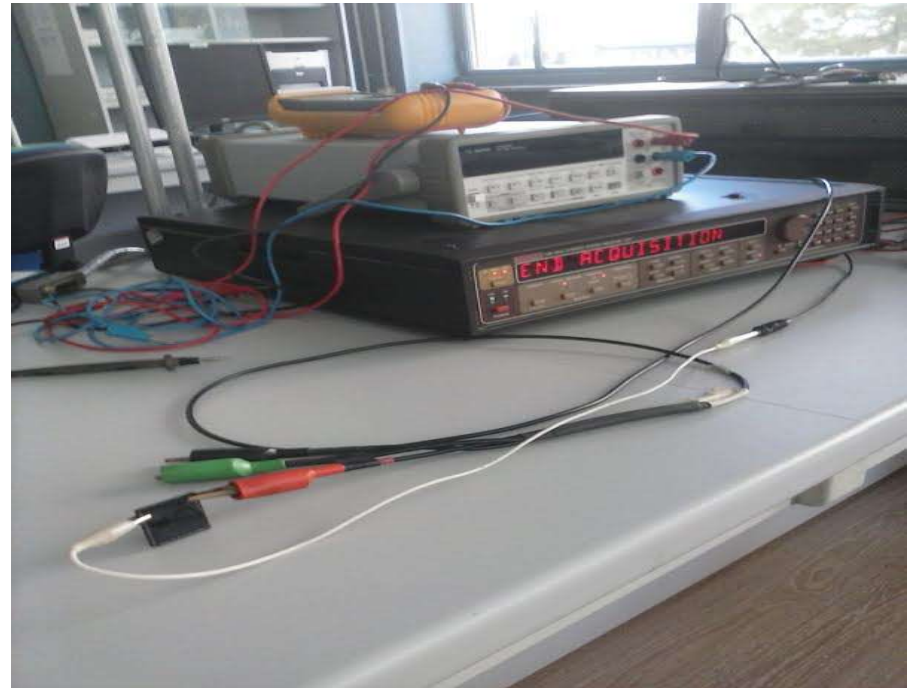
A two point probe (TPP) method

- Stable electrical contact
- Using A Keithley-238 High Current Source

Goals:

- Average value for the resistance
- Test sample uniformity

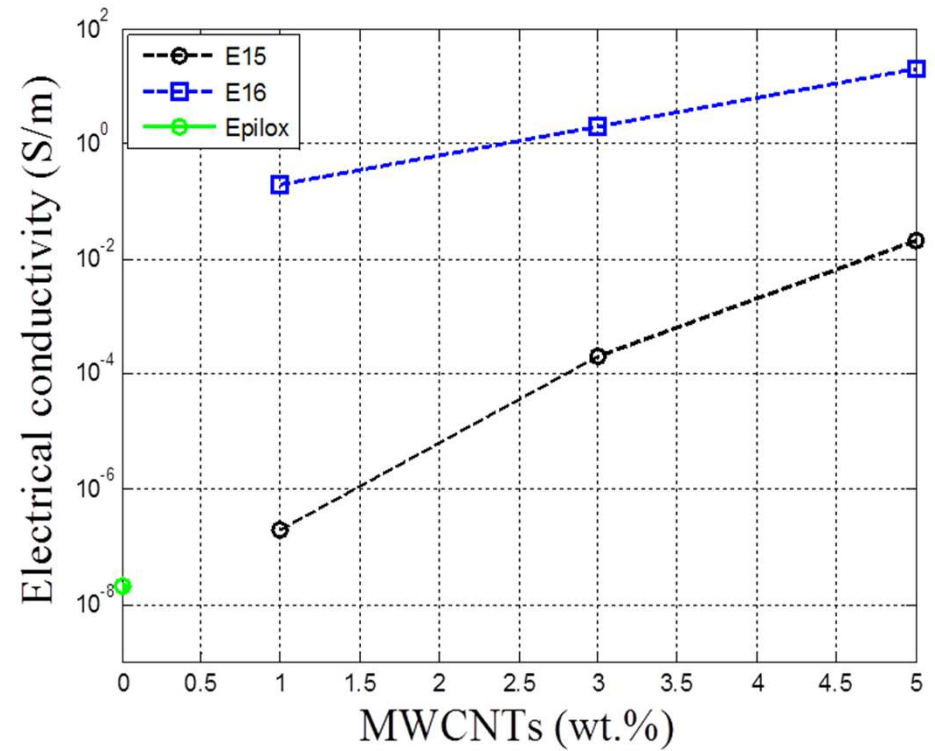
*In cooperation with:
Mauro Giorcelli*



DC Conductivity

Electrical conductivity:

$$\sigma = \frac{I}{V} \frac{l}{A}$$



Higher aspect ratio → Higher conductivity

Increasing wt.% → Conductivity increasing

CNTs type	Diameter [nm]	Length [μm]	Aspect ratio	Density (g/cm ³)
E15	25 - 45	>10	200 - 400	2.05
E16	6 - 10	>10	1000 - 1600	2.12

Permittivity Measurements Setup

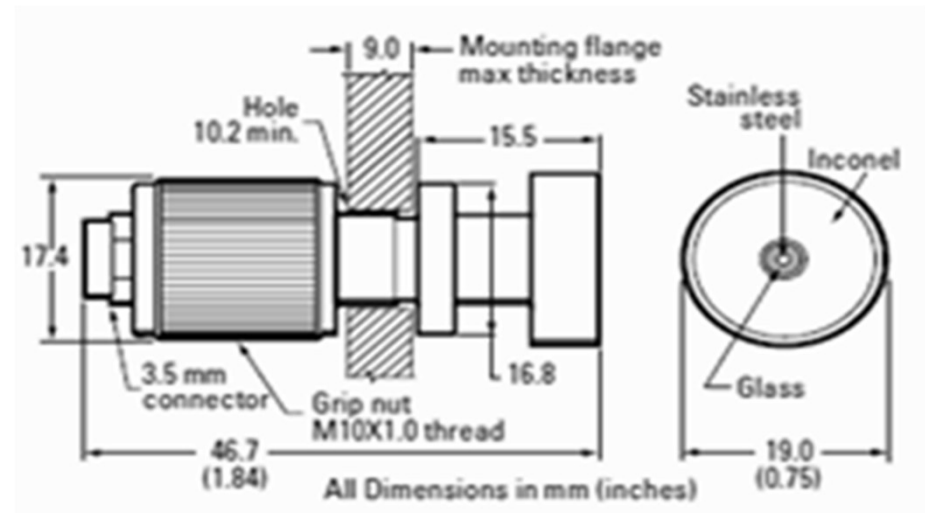
A Network Analyzer (E8361A) + A commercial dielectric probe (Agilent 85070D)

Advantages:

- ☺ Frequency band 200 MHz – 20 GHz
- ☺ Fast response
- ☺ Samples of small dimensions

Drawback:

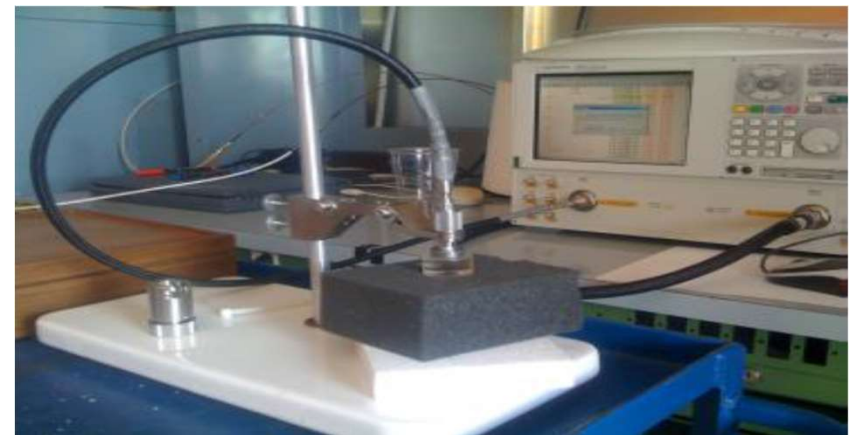
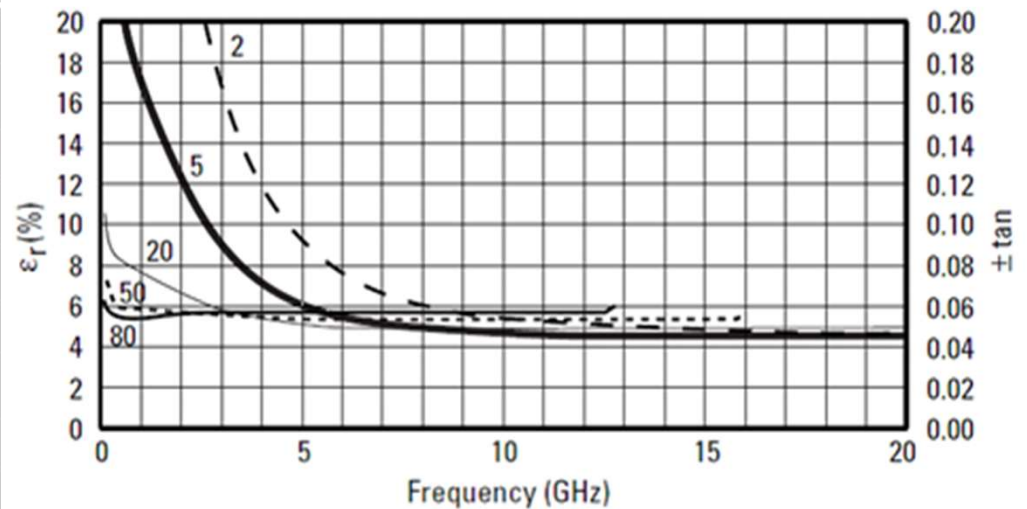
- ☹ Roughness and flatness of the surface are critical
- ☹ Thick samples



A commercial dielectric probe
(Agilent 85070D)

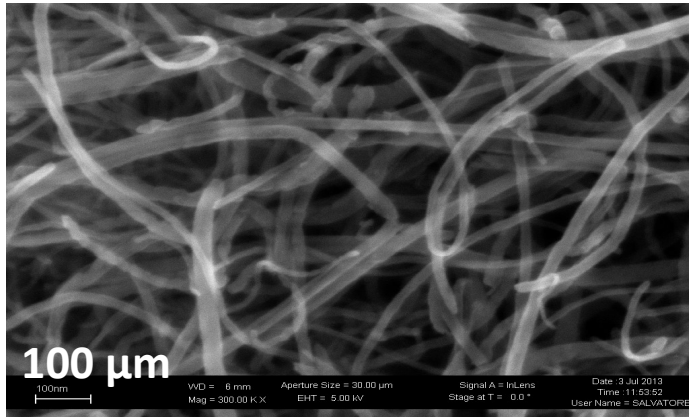
Performance Characteristics of Dielectric Probe

Frequency range	200MHz - 20GHz (nominal)
Temperature	-40°C to 200°C
Homogeneity	The sample should be homogeneous
Surface roughness	The sample should be flat
Diameter of sample	>20 nm
Thickness of sample	$> \frac{20}{\sqrt{\epsilon_r}}$ nm
Granule size	< 0.3 mm
Maximum recommended ϵ_r	<100
Minimum recommended $\tan\delta$	>0.05
Accuracy in real permittivity	$\pm 5\%$
Accuracy in $\tan\delta$	$\pm 5\%$

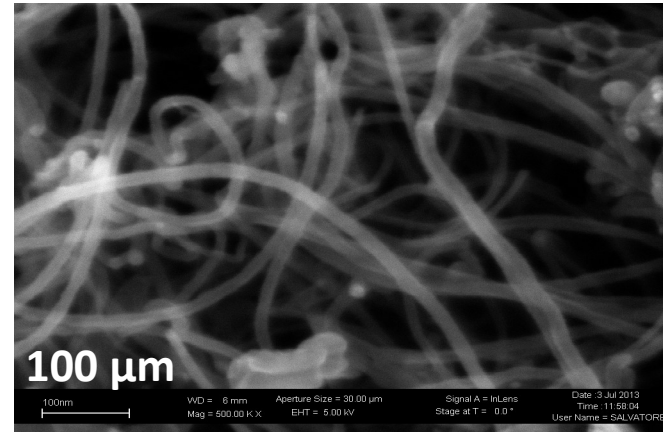


Calibration $\rightarrow$ Air\Short\Water
 $\rightarrow$ Removing systematic errors

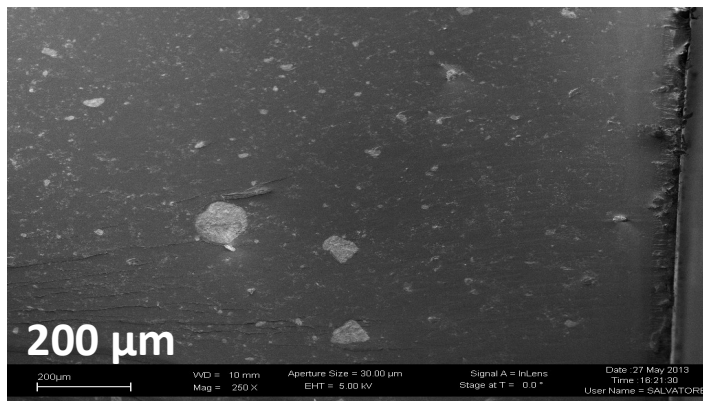
FESEM Analysis



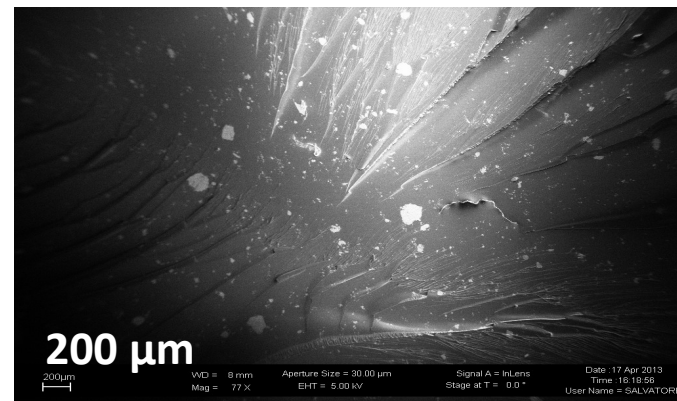
E15 material



E16 material



Crio-fractured E15 surface of composite

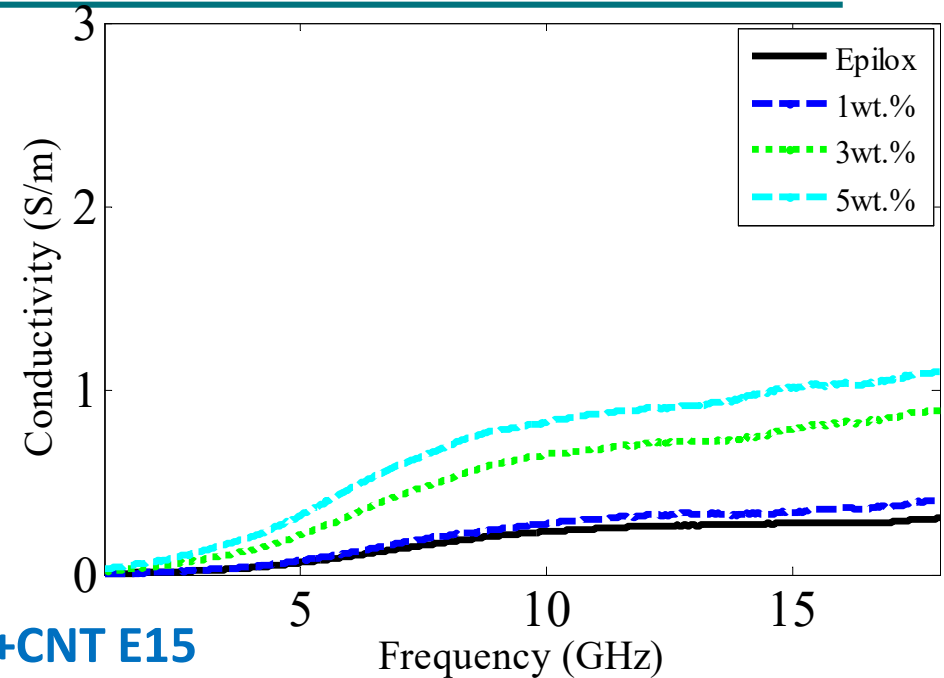
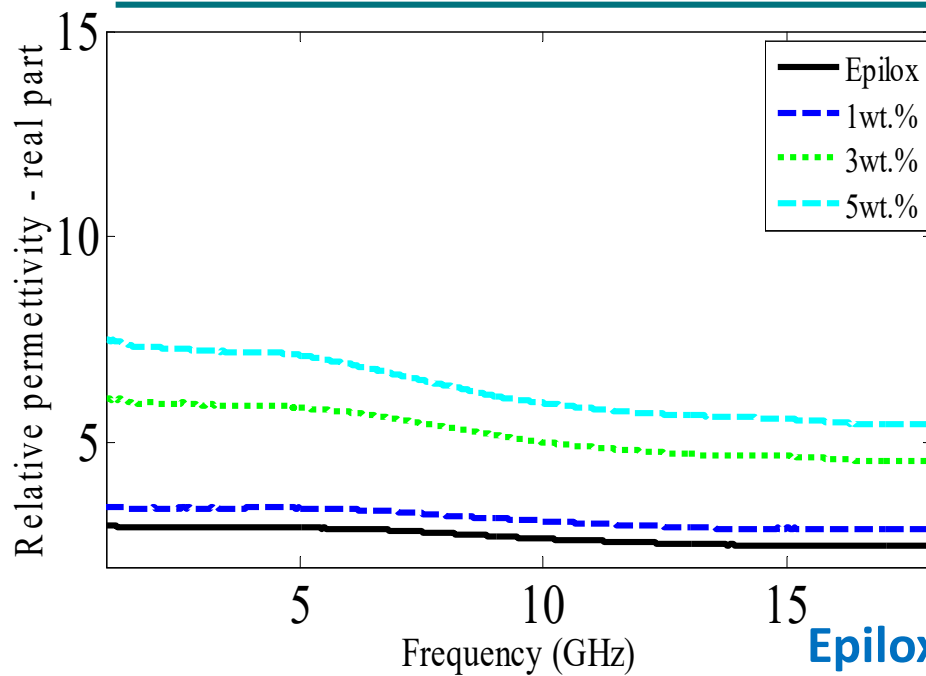


Crio-fractured E16 surface of composite

Done by :
Salvatore Guastella – DISAT Department

FESEM: Field Emission Scanning Electron Microscopy

Investigation on the Effects of Increasing of CNT Loading



Increasing wt%



- Permittivity increase
- AC conductivity increase

All the volume fractions used of E15 > Pc **BUT** good results starts from 3wt.% (1.92vol.%)

E15-1wt.% (0.64 vol.%) results approximately the same of Epilox

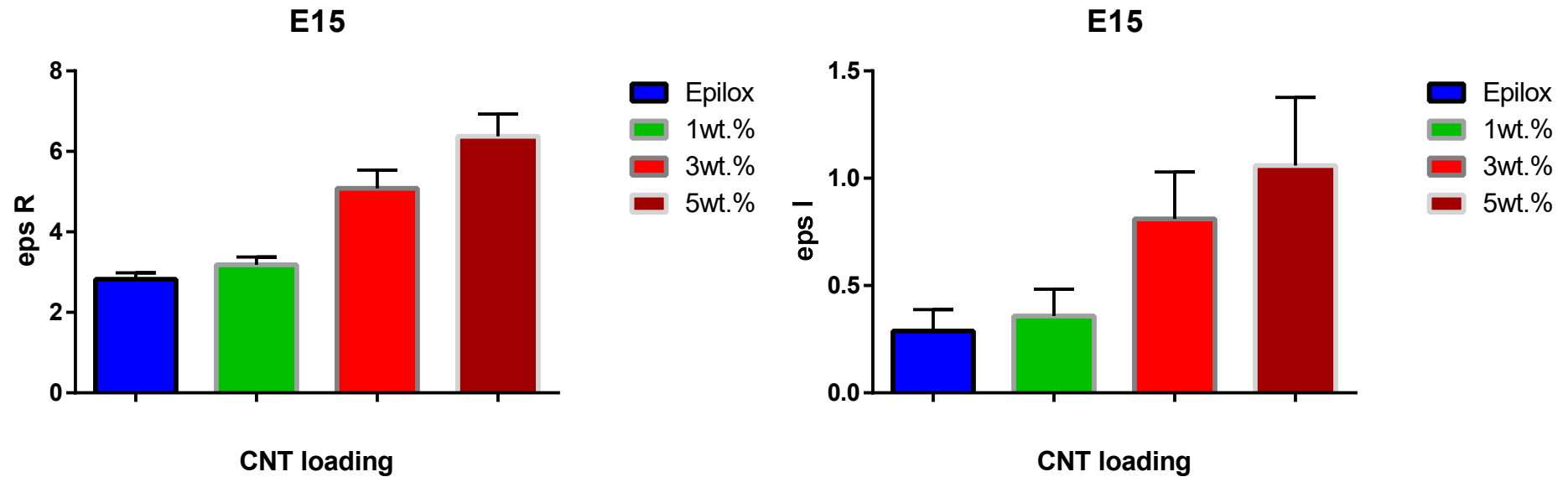
BECAUSE OF

Problems in Dispersion of prepared composites and existence of bubbles .

E15

Diameter [nm]	25 – 45
Length [μm]	>10
Aspect ratio	200 - 400
Density (g/cm ³)	2.05
Pc	0.1 vol.%

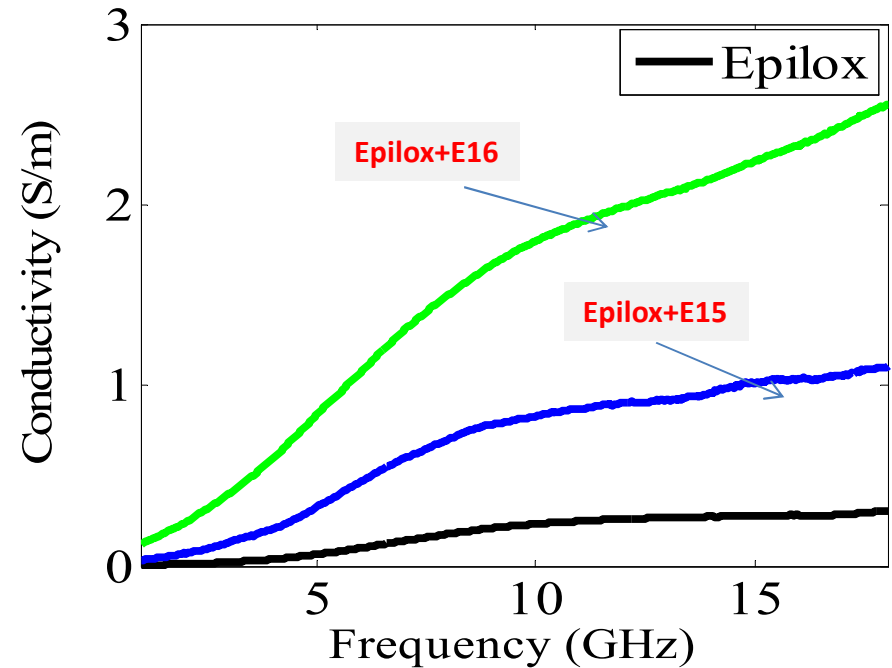
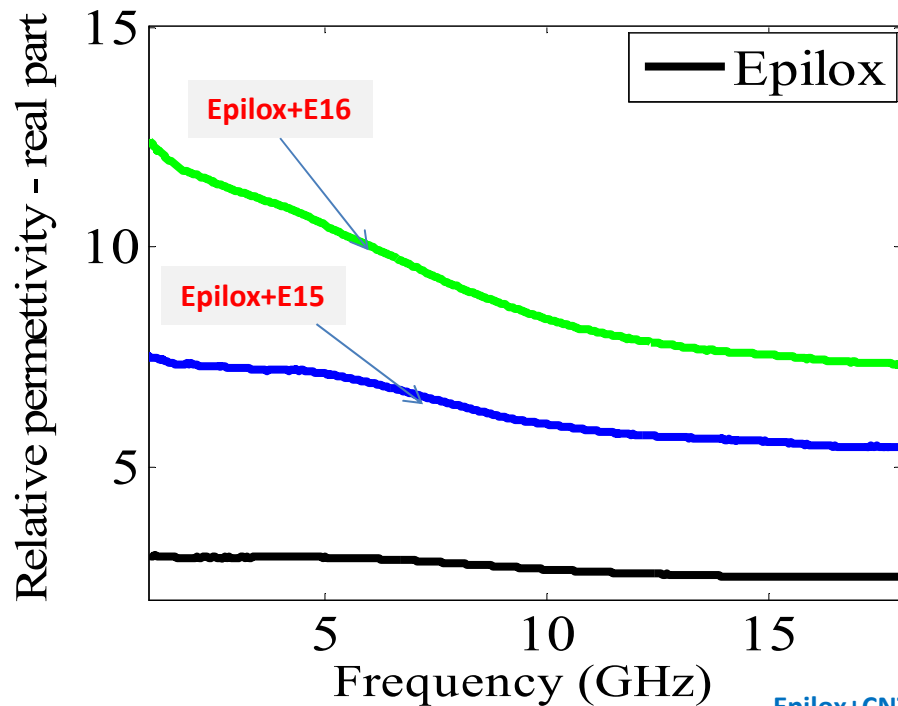
Statistical Analysis



Comparing E15 - 1 wt.% samples to Epilox → The variation is not significant

Increasing the CNT concentration → Imaginary permittivity increase → Electrical conductivity increase

Comparison of Different Types of CNT with Different Aspect Ratio



Epilox+CNT E15-5wt.%
Epilox+CNT E16-5wt.% } comparison

- Number of walls
- Aspect Ratio
- Density

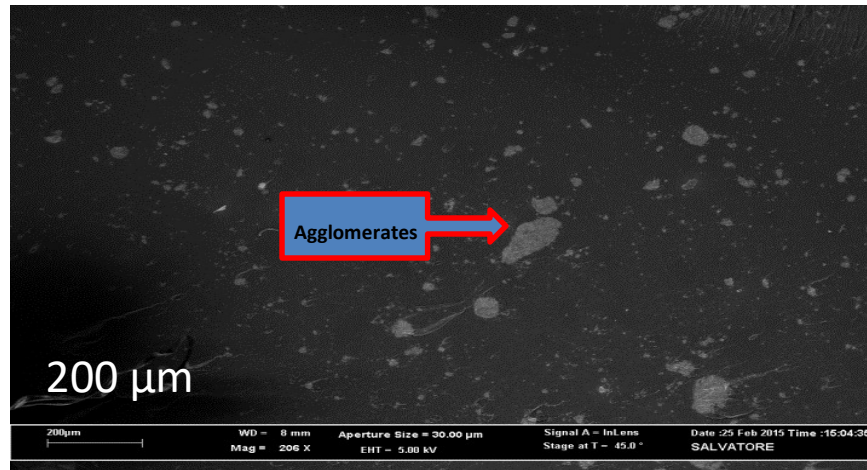


Play an
important role

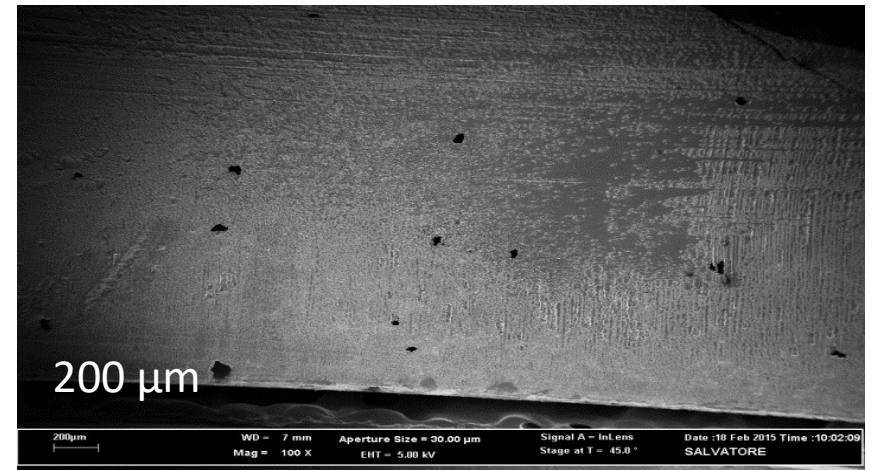
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Analysis of Dispersion of Nano-fillers in Polymer Matrix

FESEM analysis – two mixing procedures



By: Ultra turrax



By: Three roll mills

Epilox+CNT MW6 – 1wt.%

MW6

Diameter [nm]	18 - 35
Length [μm]	>10
Aspect ratio	285 - 555

Three roll mills
mixing



**Better dispersion of nano-fillers
in polymer matrix**

Done by:

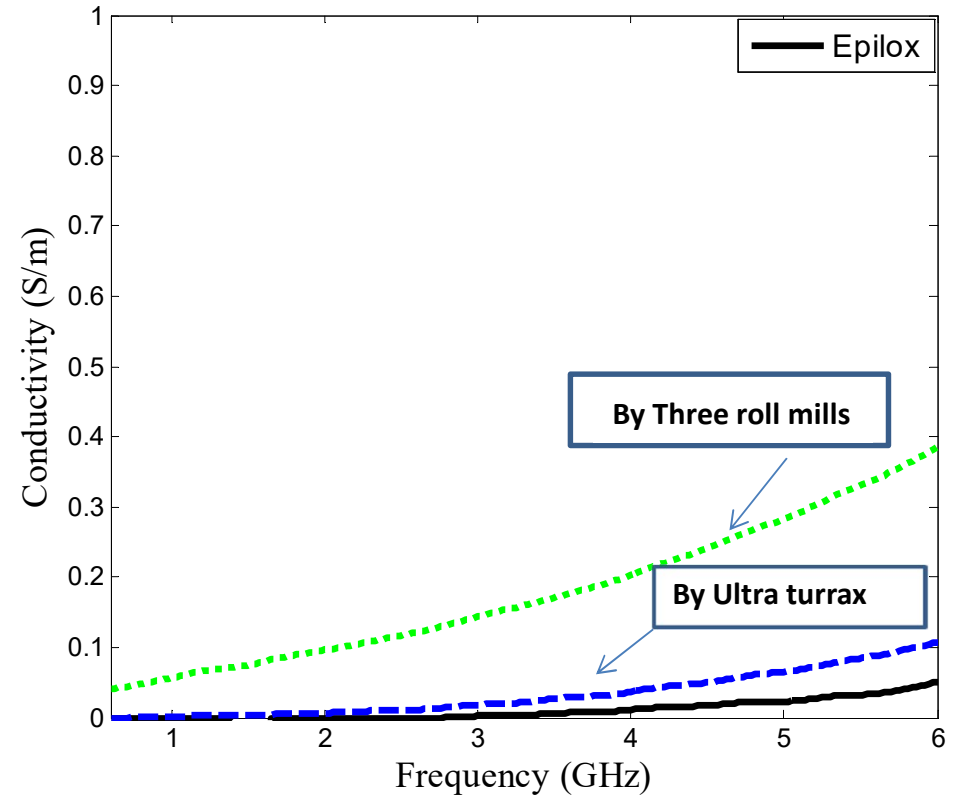
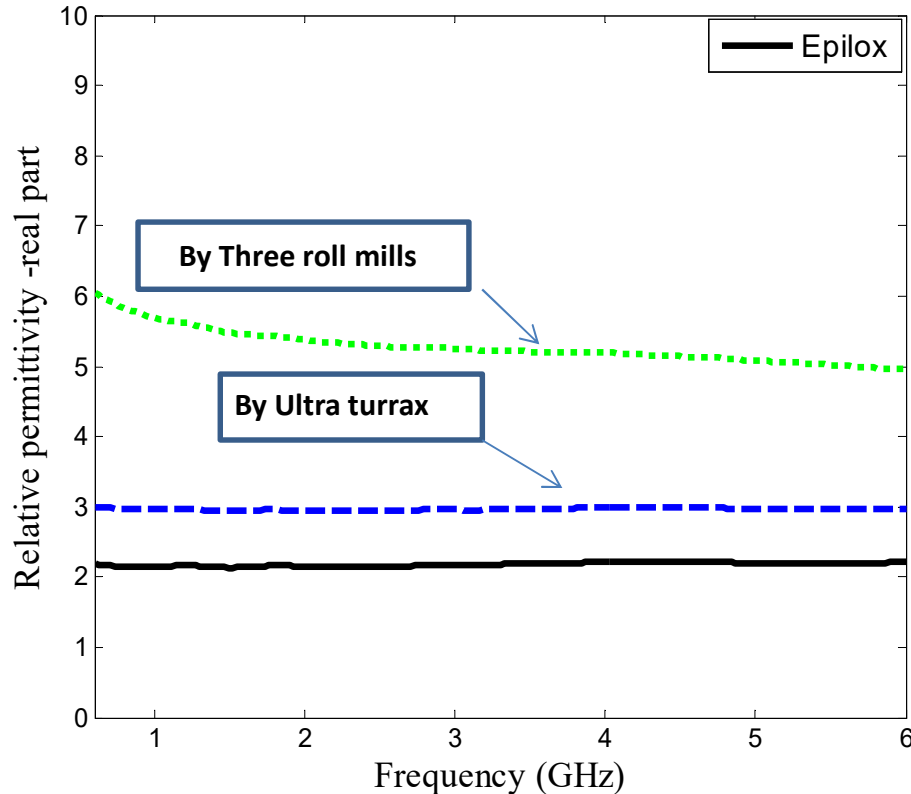
Salvatore Guastella – DISAT Department

FESEM: Field Emission Scanning Electron Microscopy

Analysis of Dispersion of Nano-fillers in Polymer Matrix

Complex permittivity - two mixing procedures

Epilox+CNT MW6 – 1wt.%



Three roll mills mixing procedure



Higher permittivity and AC conductivity

Thanks for Your Attention