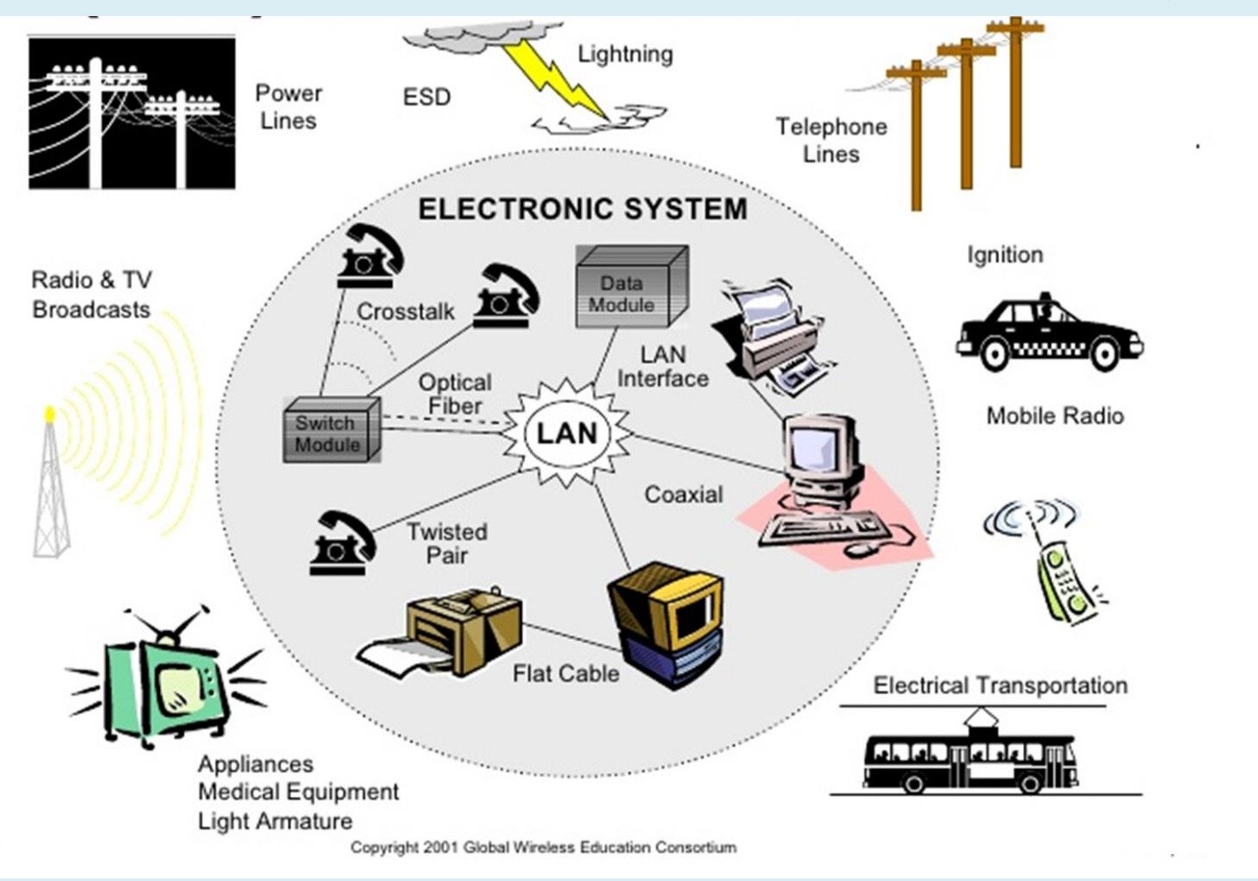
Electromagnetic Properties of Carbon based Polymer Nanocomposites for Shielding, Chaffing and Camouflage Applications

Joseph Vimal Vas, High Voltage Engineering Lab, Indian Institute of Science, Bengaluru

Electromagnetic Interference (EMI)



EMI induces noise in electronic systems

Modes of noise coupling

- Conductive coupling
- Common impedance coupling
- Coupling via Electric and Magnetic fields

Methods to reduce EMI

- Shielding
- Balancing
- Filtering
- Grounding

Electromagnetic Shielding

Materials interact with EM waves through:

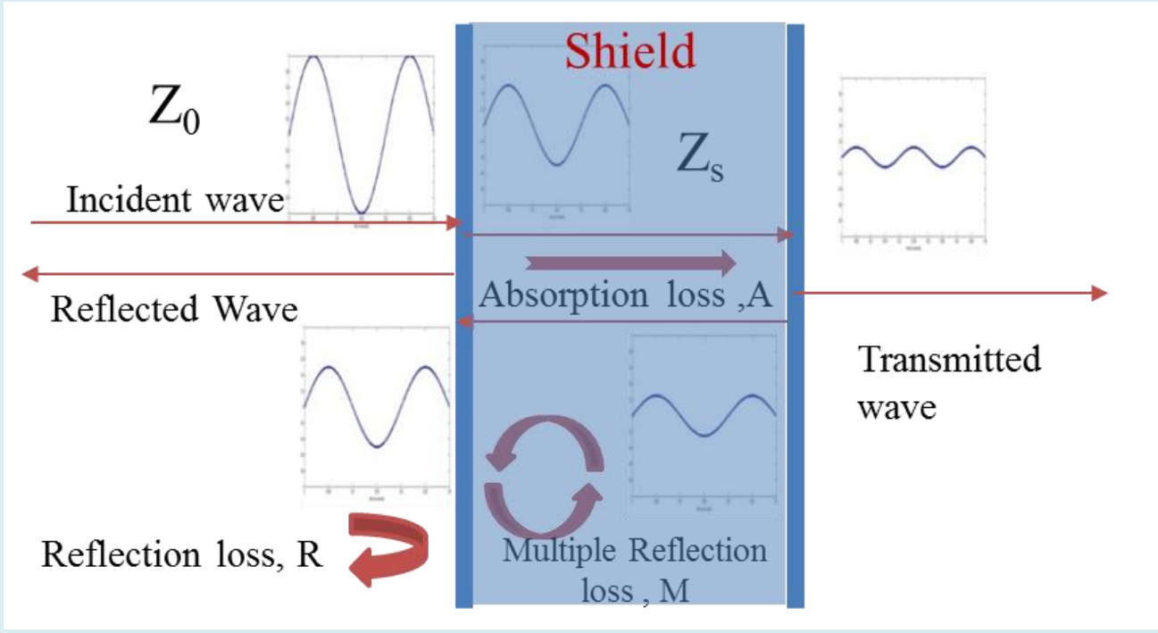
- Reflection
- Absorption
- Multiple reflections

Requirements of a good shield

- High conductivity
- High permittivity
- High permeability

Commercial Shielding Materials

- Highly Conducting Materials- Copper, Aluminium, Stainless Steel
- Shielding mainly through reflection



EM interaction with materials

Drawbacks of Metallic shields

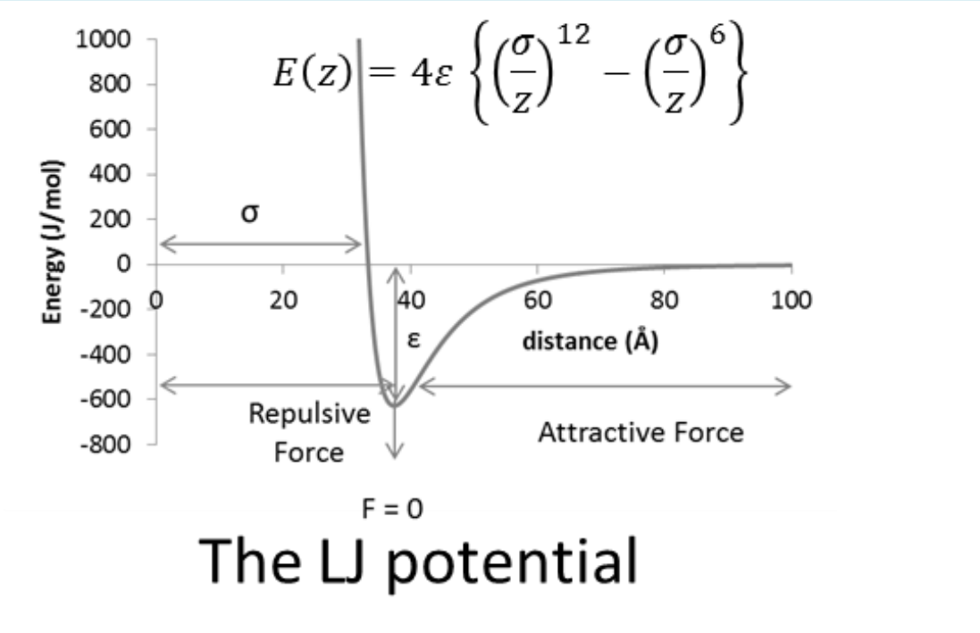
- Heavy and inflexible
- Prone to Corrosion
- Not cost effective – difficult to process

Microwave oven door

Casing of sensitive electronics

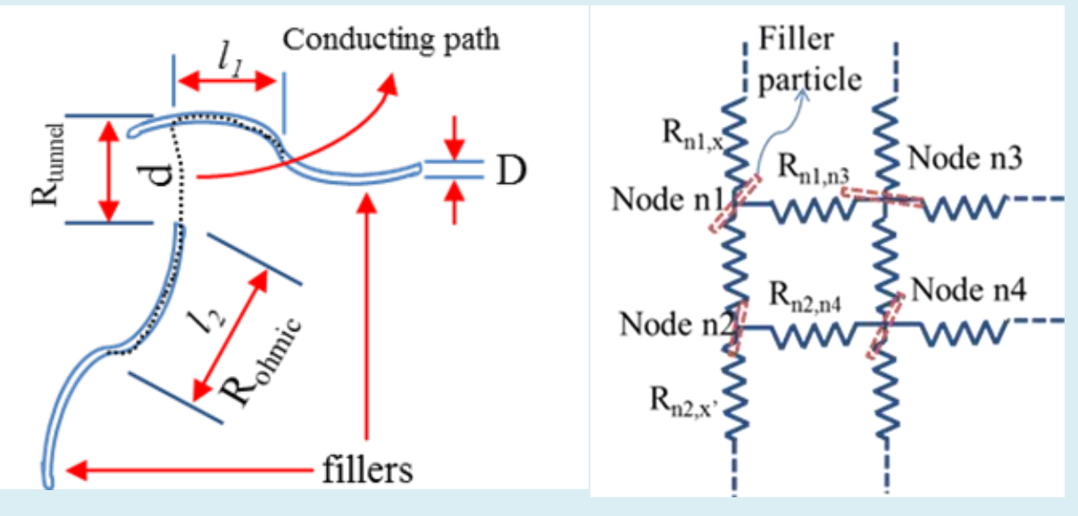
Monte Carlo Simulations

Step 1 – MC simulation performed to find the particle distribution using LJ potential.

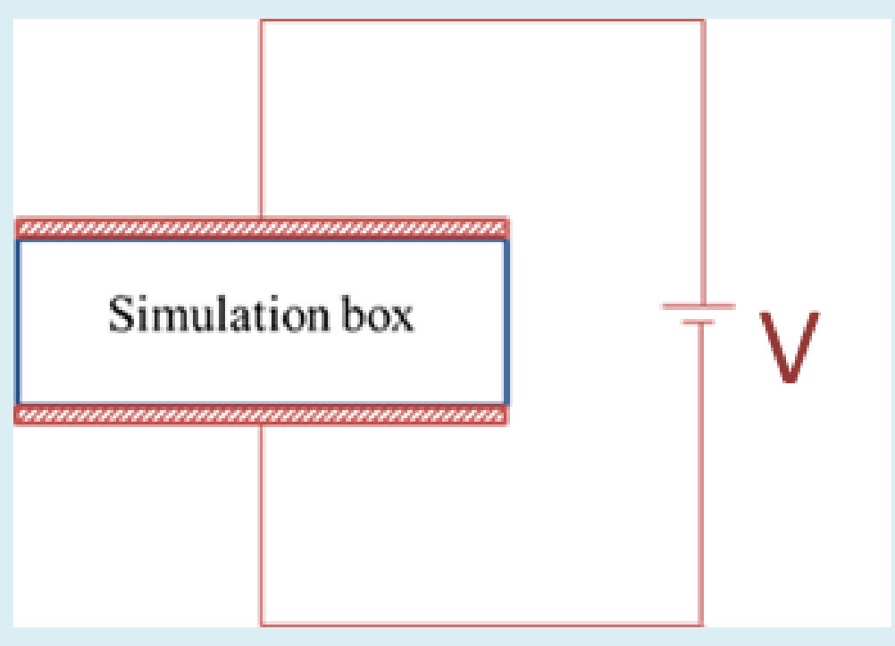


The LJ potential

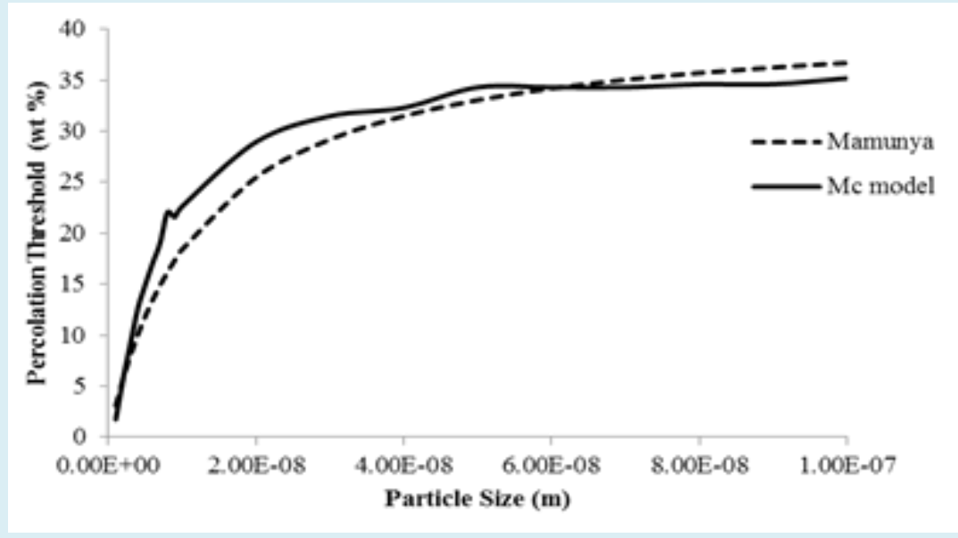
Step 2 – Calculation of the interparticle contact resistance using the particle orientation



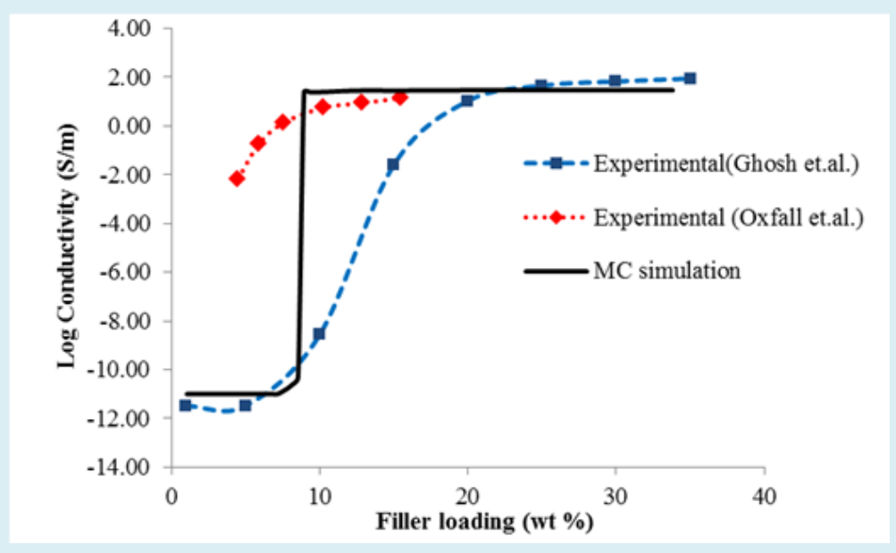
Step 3 - Modelling the resistive network using basic circuit theory and computing the composite conductivity.



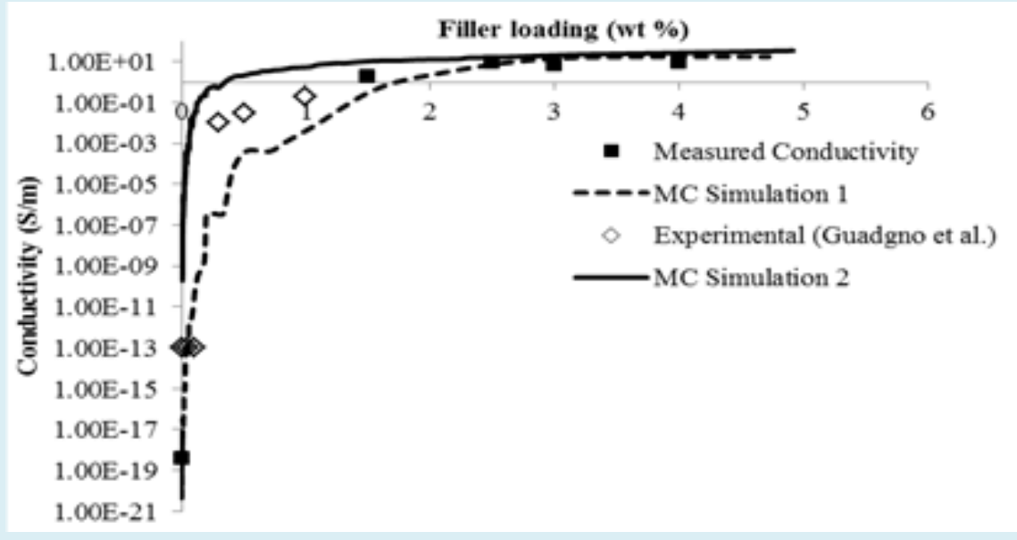
Validation of the MC model



Comparison with theoretical model for spherical particles



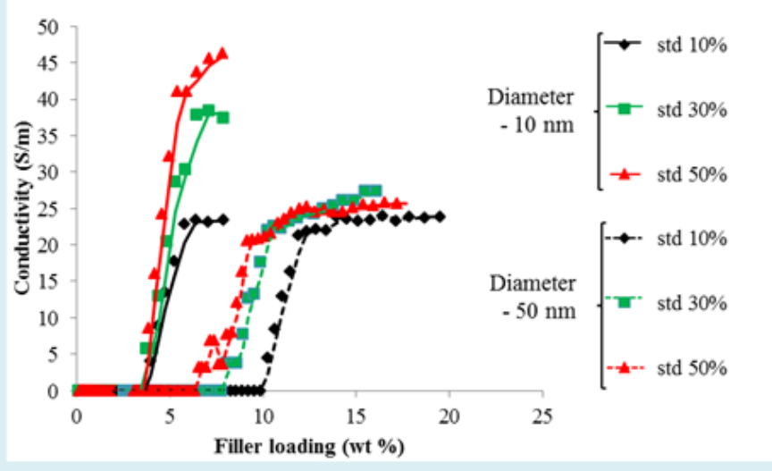
Comparison with experimental data – spherical fillers



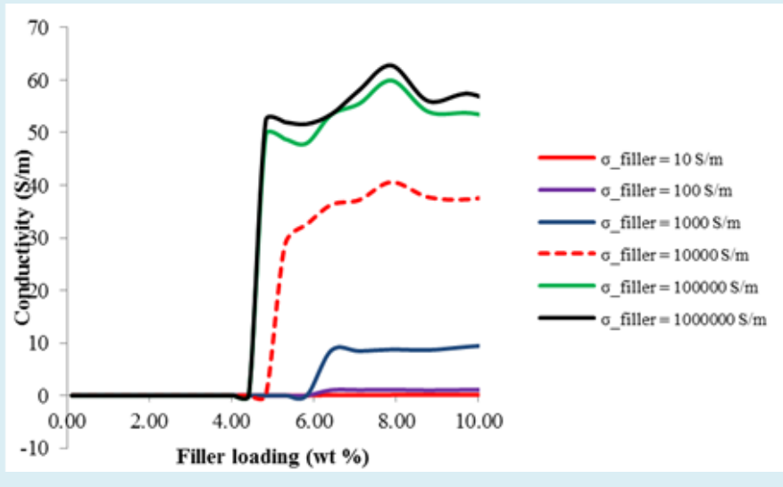
Comparison with experimental data – rod like fillers

Results

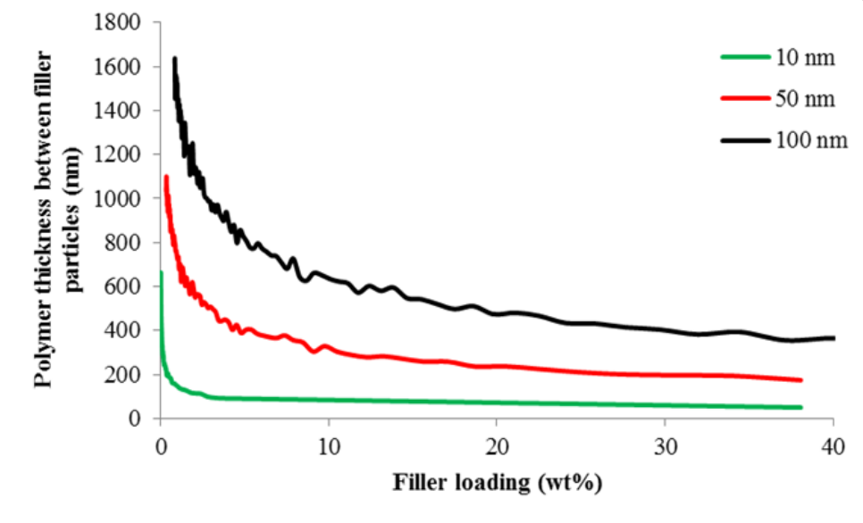
Spherical Particles



Effect of variation of filler loading and particle size

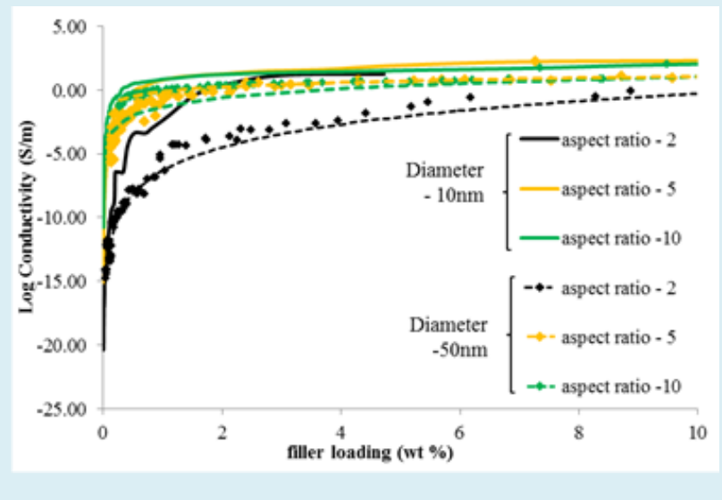


Effect of variation of filler conductivity

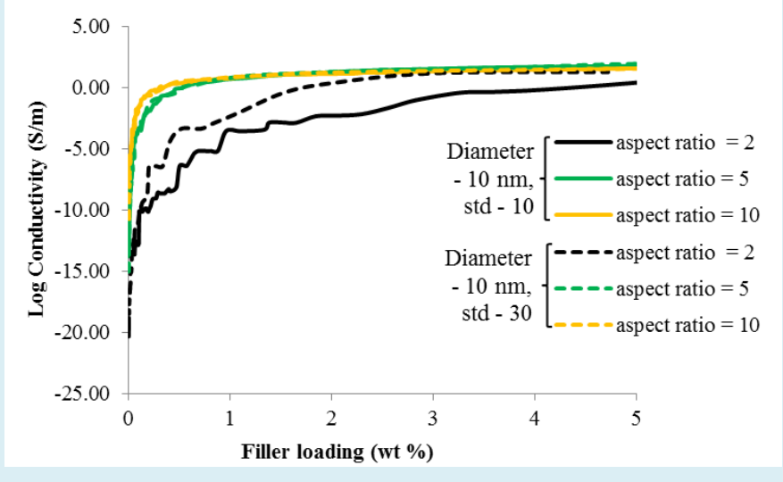


Interparticle distance vs filler loading

Rod like particles



Effect of variation of filler loading and particle size



Effect of variation of standard deviation of filler size

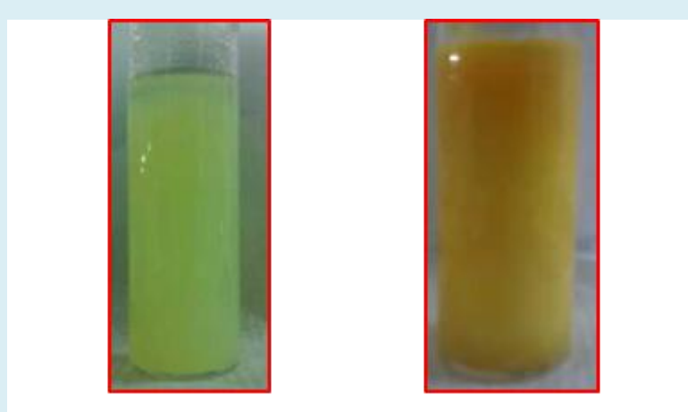
- Composites with rod like fillers achieve conductivity at lower filler loadings than those with spherical fillers.
- The conductivity is limited owing to lack of contact between fillers.
- Conductivity is set up by tunneling of electrons through the polymer layer.

Material Synthesis

Silver Nitrate + Benzyl mercaptan + Solvent → Magnetic Stirrer / Ultrasonication → Nano AgS + CNF → Ultrasonication / Vacuum filtration → CNF wafer

Nano AgS Synthesis

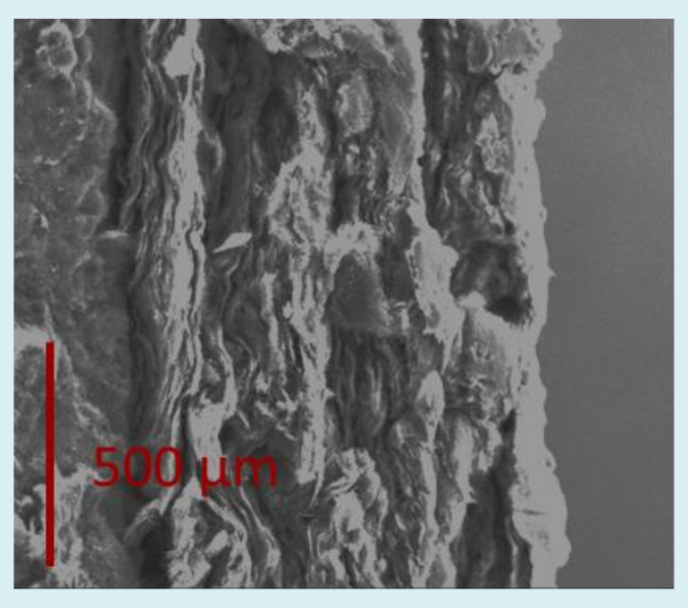
Layered structure in SR composites layered with CNF wafers



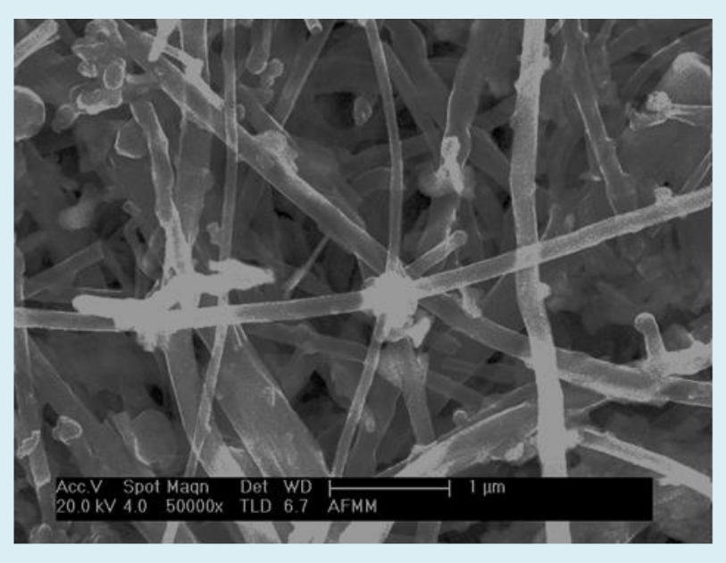
Initial mixture Nano AgS

CNF wafer
σ = 1320 S/m

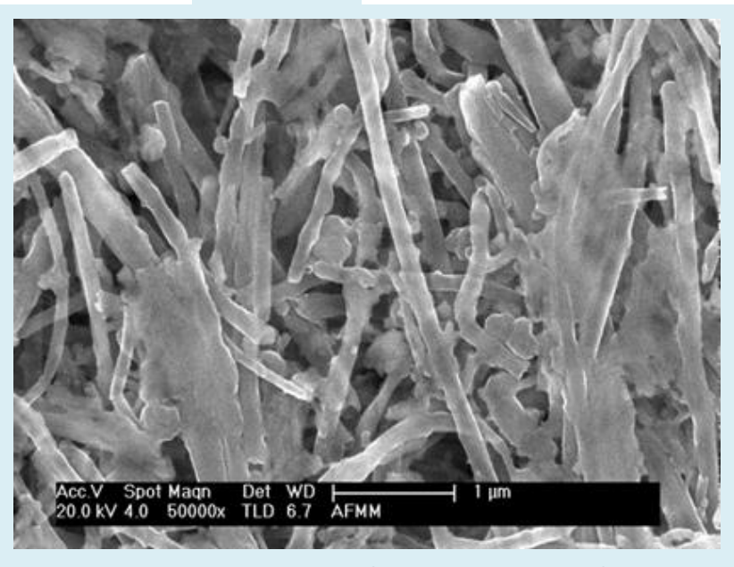
SR composites layered with CNF wafer



Cross section of CNF wafer

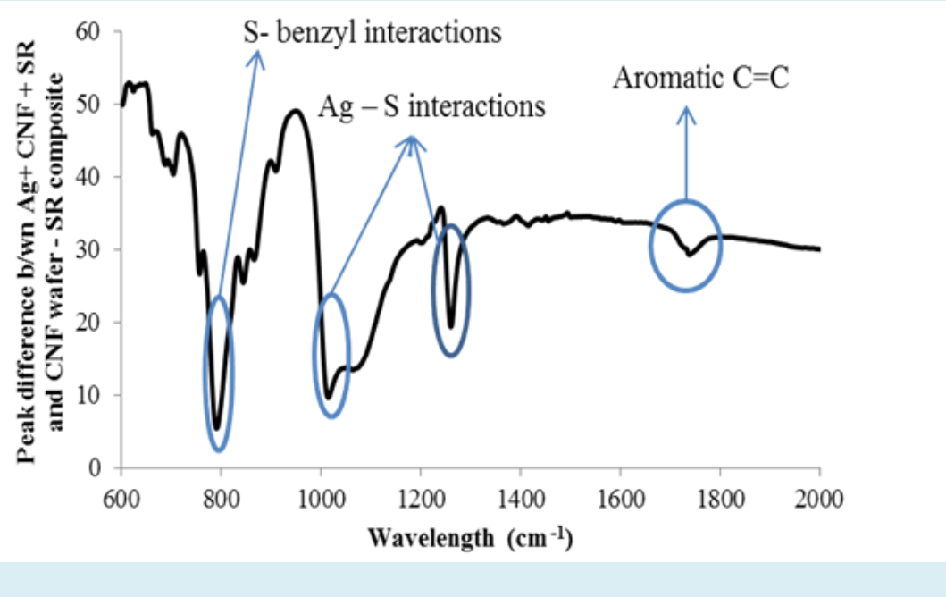


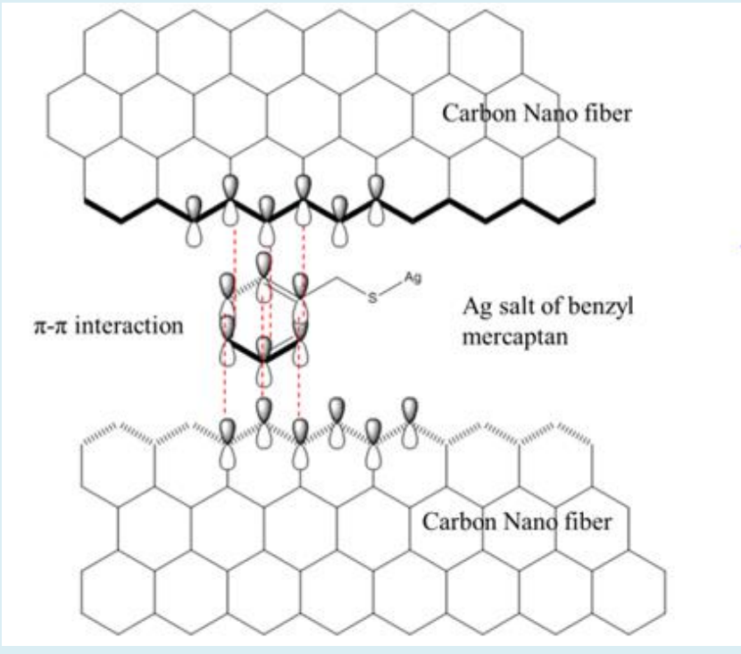
Top view of CNF wafer showing Ag- S nodes



Ag- S nodes binding the CNF fibers

FTIR spectra of SR composites with CNF wafers showing Ag- S as well as π-π interactions between Ag- S and CNF particles



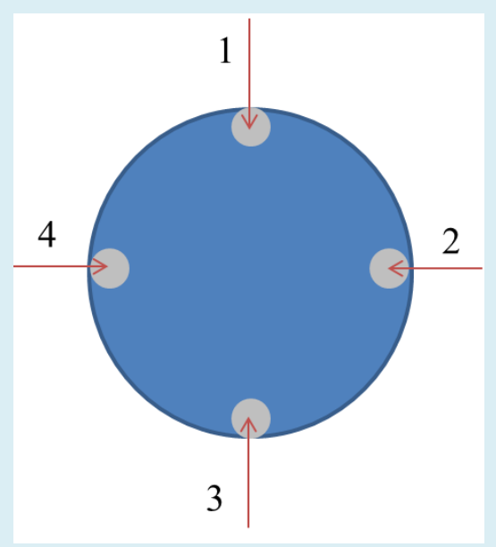


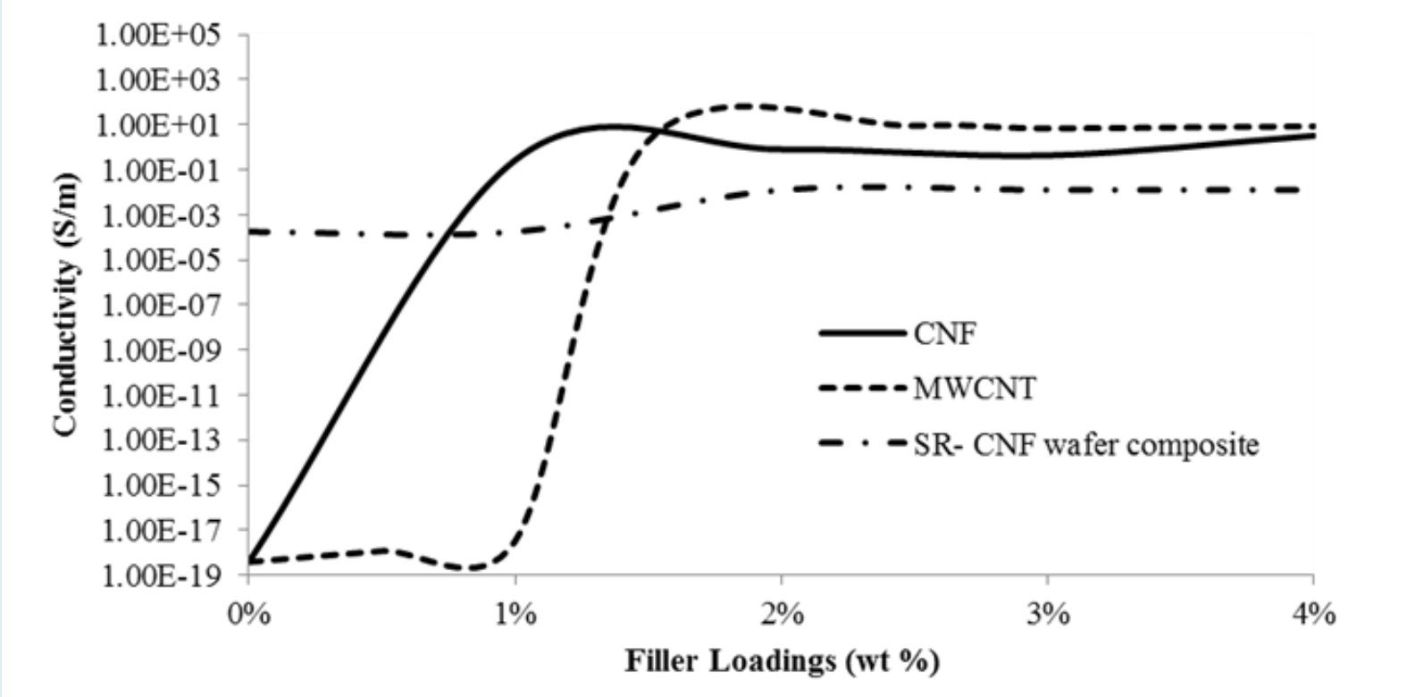
Structure of Ag- S nodes: Bonding between different CNF particles in CNF wafer

Electrical Characterisation

Conductivity measurements using van der Pauws method

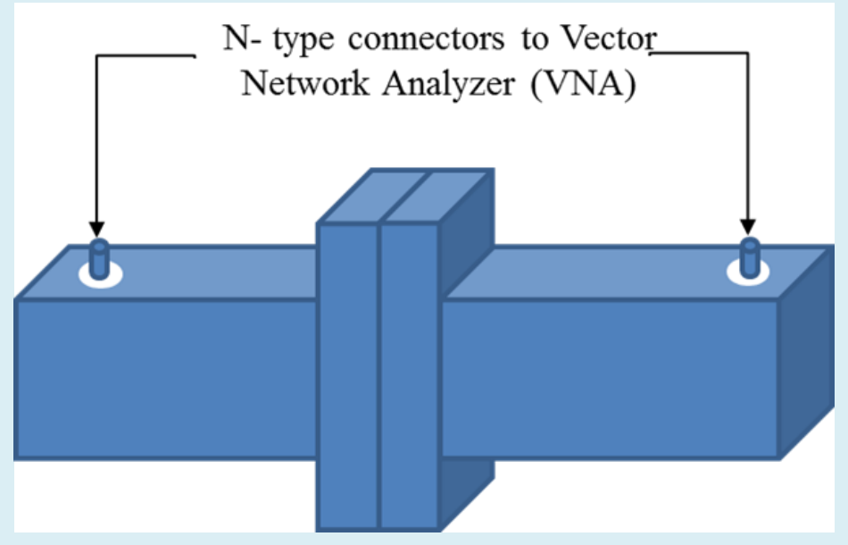
4 probe dc conductivity measurements



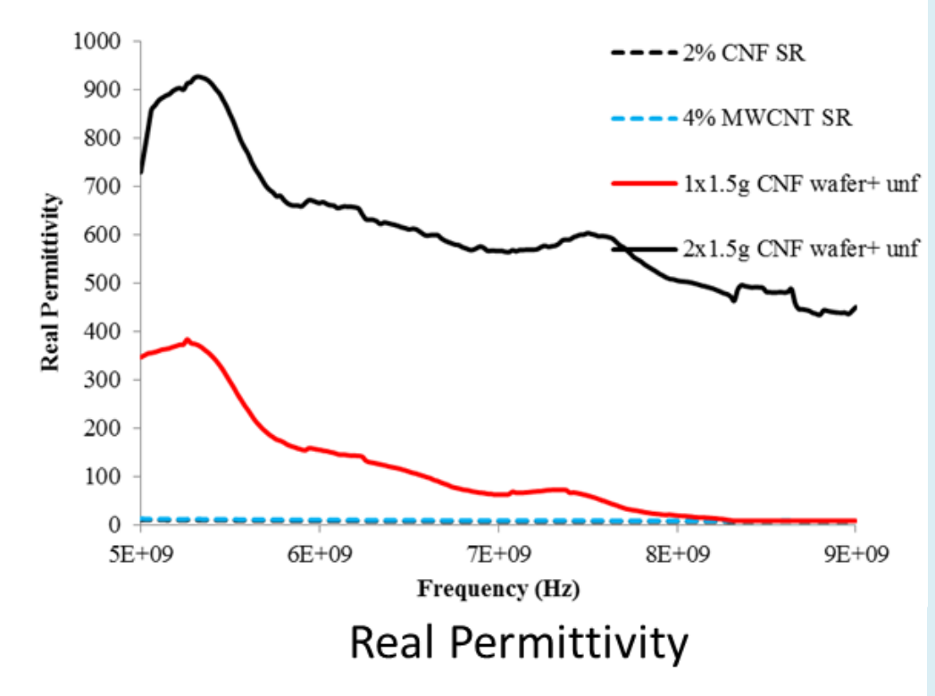


Conductivity vs filler loading of different composites

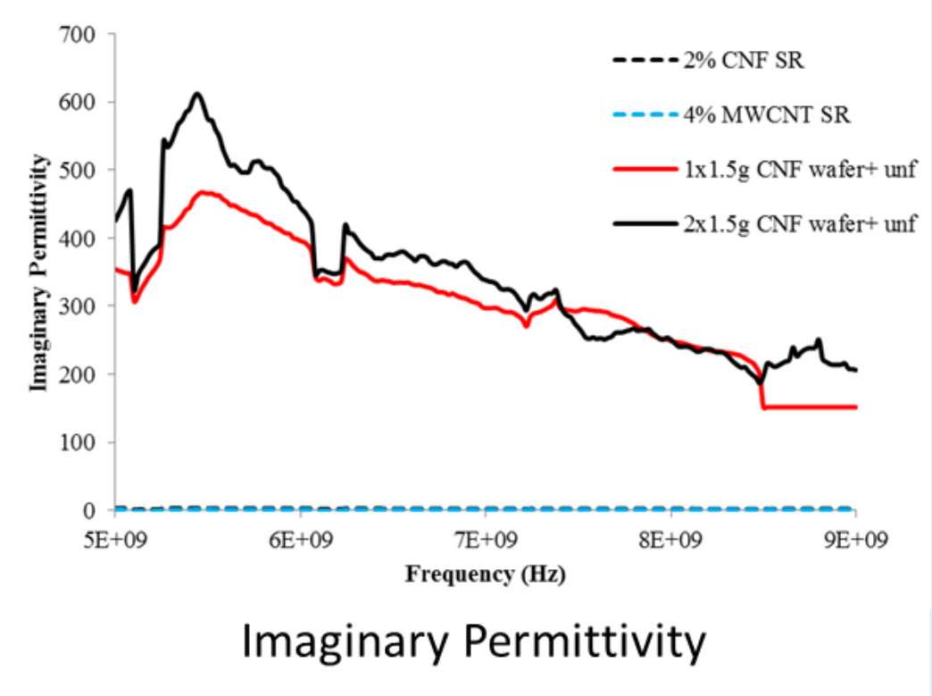
Permittivity measurements using ASTM D5568 method



Permittivity measurements as per ASTM D5568



Real Permittivity

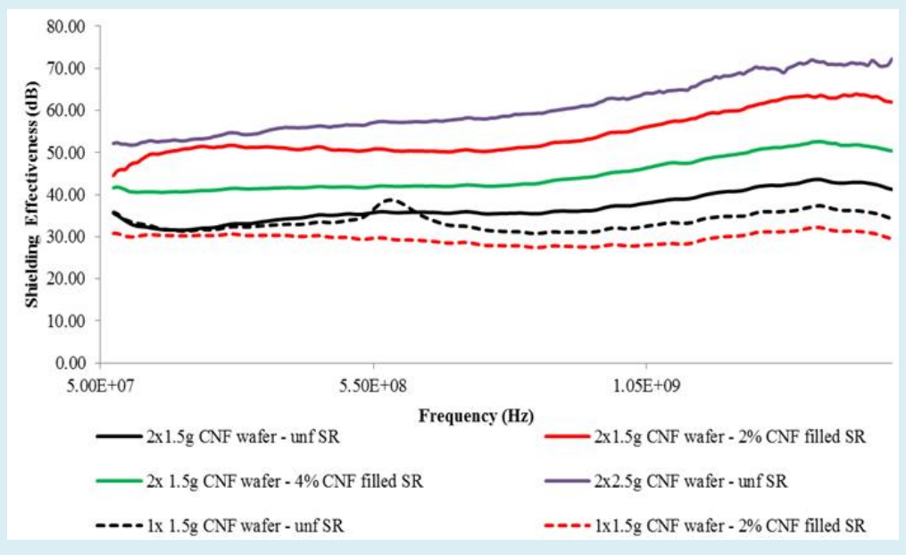


Imaginary Permittivity

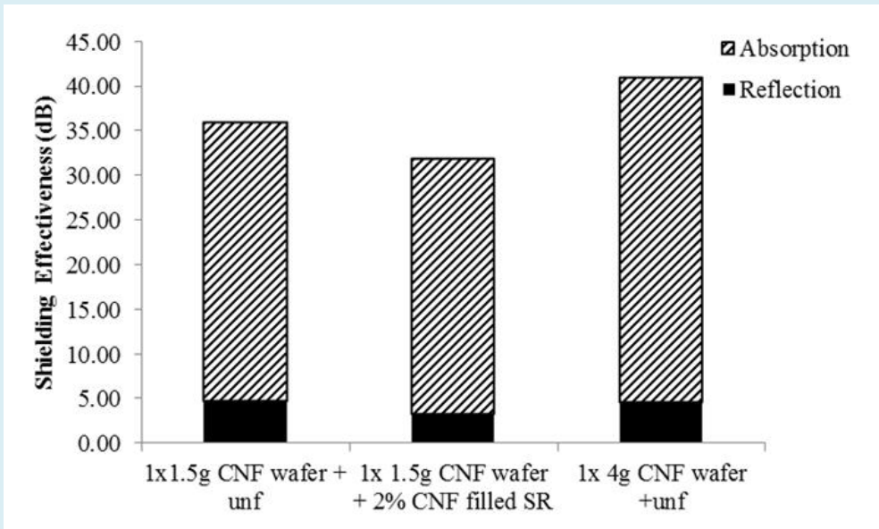
Measurement of Shielding Effectiveness

ASTM D4935 Method

- Measurements



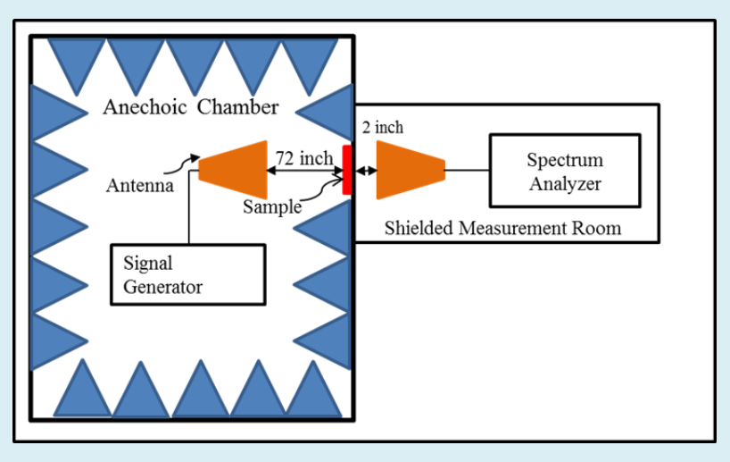
Shielding Effectiveness of different polymer samples



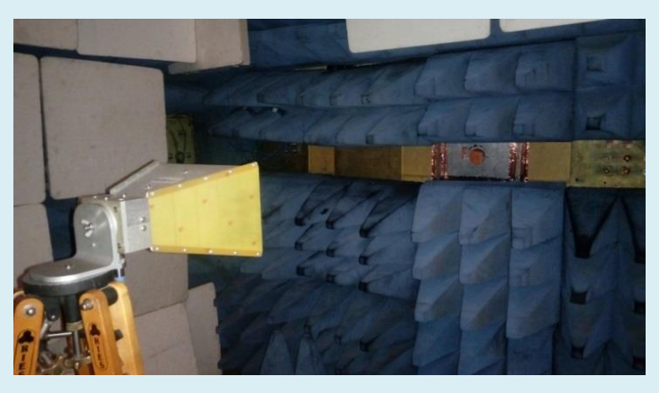
Reflection and Absorption loss

Shielding Effectiveness measurement using ASTM D4935 test fixture

Anechoic Chamber Method similar to IEEE 299

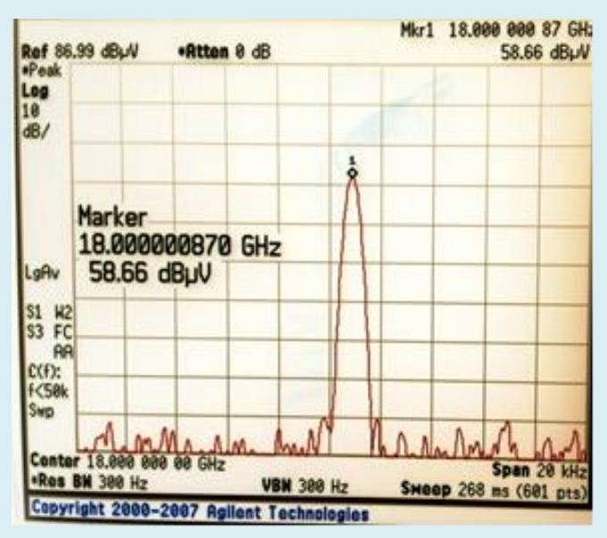


Schematic of the Measurement Set up

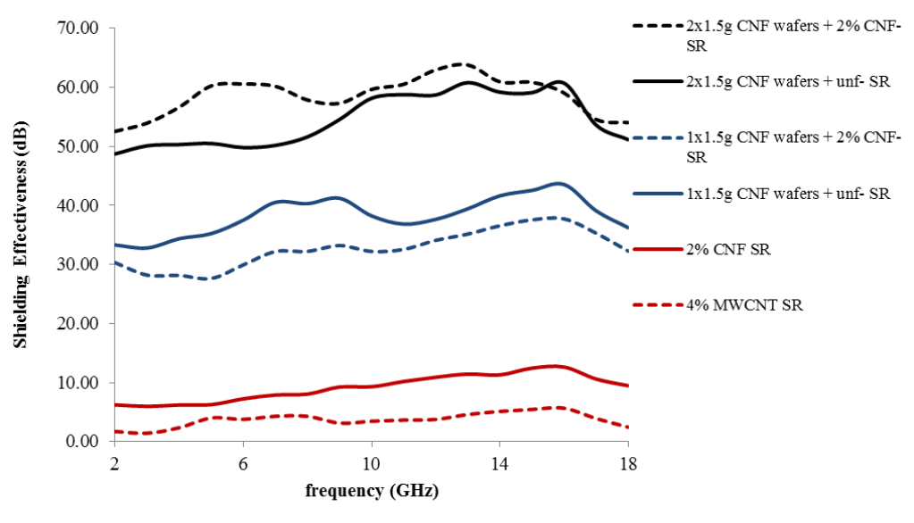


Radiated field

Horn antenna used for measurement



Measured field in the absence of the sample

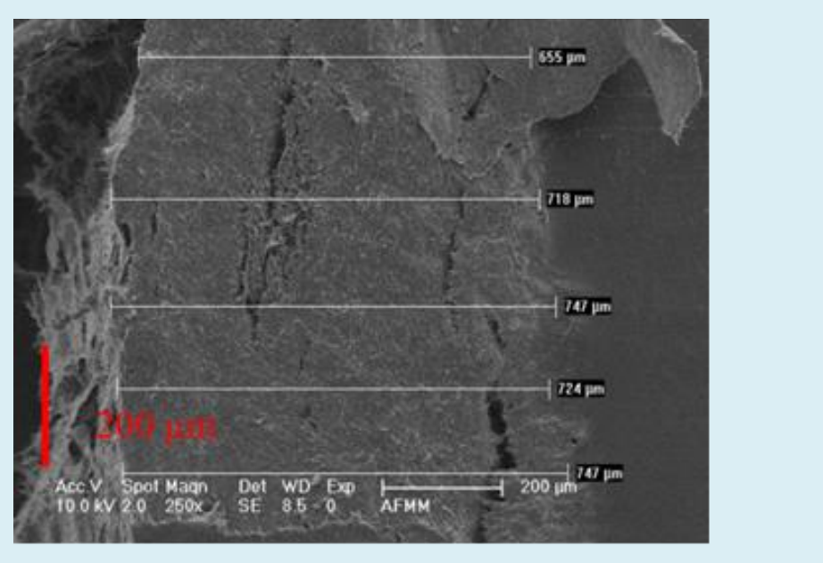


Measured Shielding Effectiveness

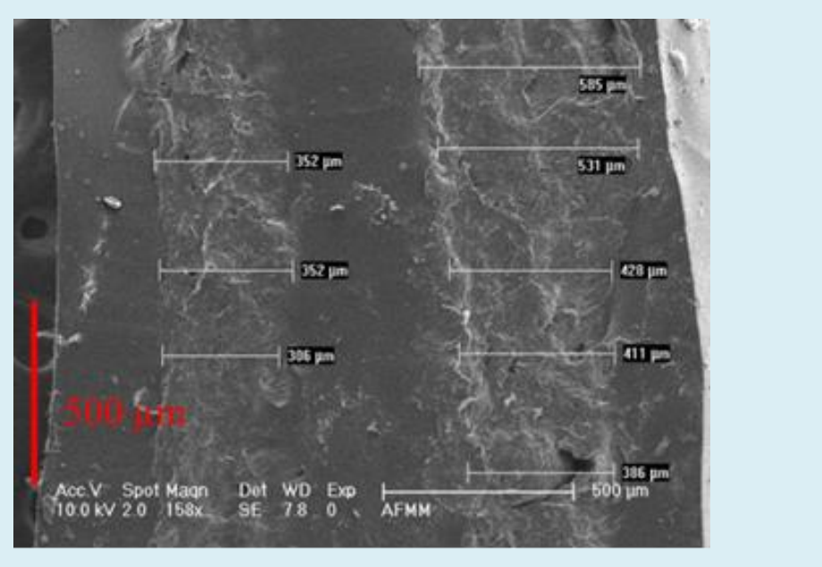
EM Modelling of Layered Composites

Absorption loss and thickness for different composites

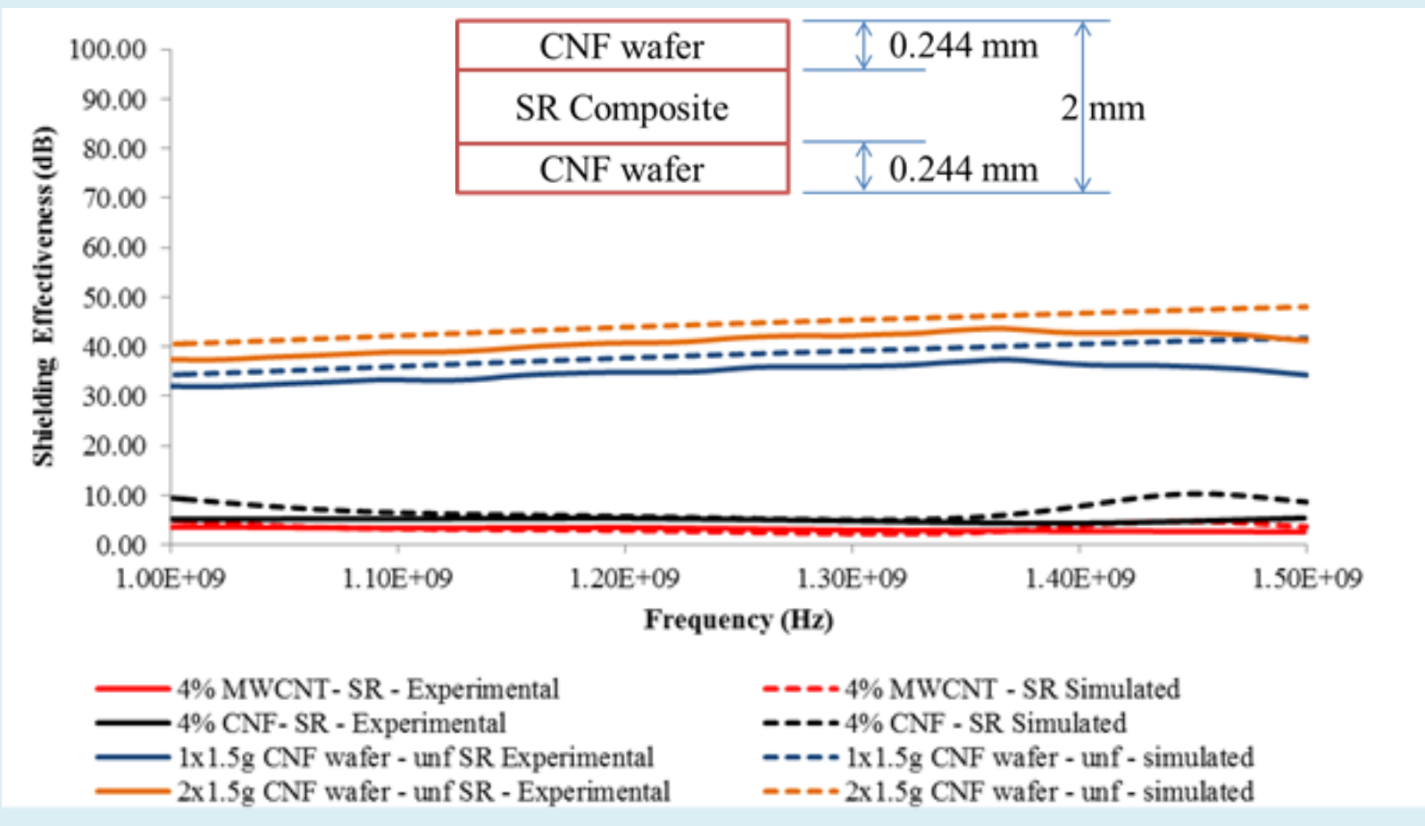
Sample Type	Absorption (dB)	Thickness (mm)
1x1.5g CNF wafer	-	0.244
1x1.5g CNF wafer - unf SR	31.32	0.729
1x1.5g CNF wafer - 2% CNF+ SR	28.61	0.697
2x1.5g CNF wafer - unf SR	34.93	0.752
2x1.5g CNF wafer - 2% CNF+ SR	52.22	1.942
2x1.5g CNF wafer - 4% CNF + SR	43.98	0.675



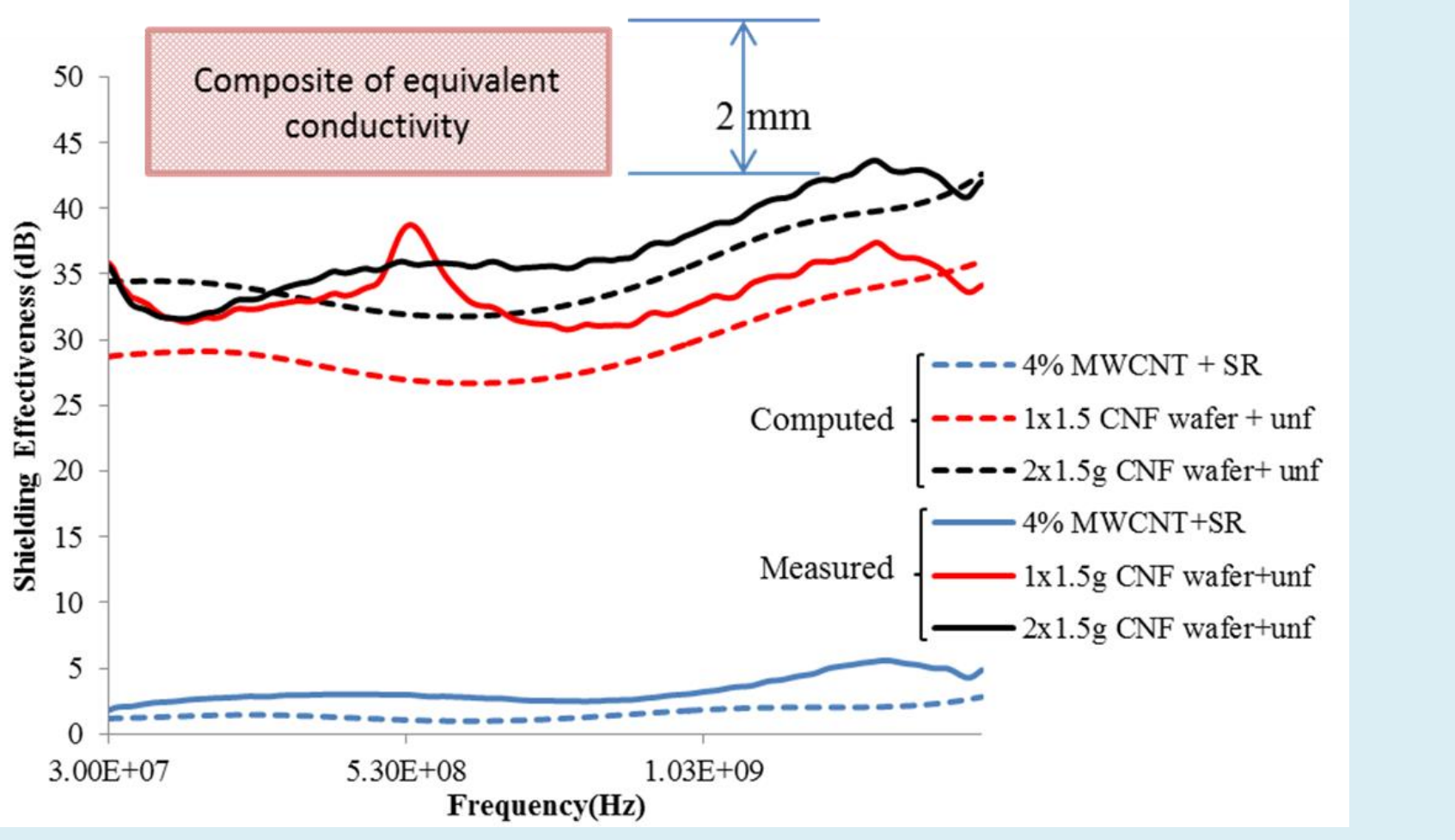
CNF wafer with 1 wafer



CNF wafer with 2 wafer



Predicted vs Measured SE using 3 layer model



Predicted vs Measured SE using permittivity values

Conclusions

- The conventional conducting polymers with MWCNT and CNF filled SR had very less conductivity and shielding effectiveness. The shielding effectiveness of the conventional conducting composites were not suitable for shielding applications.
- Even though the bulk conductivity was low, the CNF wafer composites showed good shielding behavior. This was because of the highly conducting CNF wafer layers present in the composite.
- The reflection loss of all the composites were low but increased with CNF content. This could be because of the increase in real permittivity due to increased carbon content. The large absorption loss was due to the higher imaginary permittivity of the layered composites.
- All the composites showed very low reflection loss. The shielding behavior was mainly attributed to the absorption loss. The absorption loss depends on the thickness of the CNF wafers in the SR matrix.

Publications

Intellectual Property

1. Vas J. V. and Thomas M. J., “Layered silicon rubber carbon nanocomposites for electromagnetic shielding”, IP- 201641023148, 5th July, 2016, patent pending.

Journal Article

1. Vas J. V. and Thomas M. J., “Carbon Nanofibers based Nanocomposites for Electromagnetic Shielding Applications”, IEEE Compatibility Magazine, pp. 77-79, May 2016.
2. Vas J. V. and Thomas M. J., “Electromagnetic Shielding Effectiveness of Layered Polymer Nanocomposites: Part 1”, Submitted to the IEEE Transaction on Electromagnetic Compatibility.
3. Vas J. V. and Thomas M. J, “Electromagnetic Shielding Effectiveness of Layered Polymer Nanocomposites: Part 2” Submitted to the IEEE Transactions on Electromagnetic Compatibility.
4. Vas J. V. and Thomas M. J., “Monte Carlo Modeling of Percolation and Conductivity in Carbon Filled Polymer Nanocomposites”, Submitted to the IEEE Transactions on Nanotechnology.
5. Vas J. V. and Thomas M. J., “Shielding Behavior of SR composites layered with CNF wafers in the 1-18 GHz frequency range”, under preparation.

Conferences

1. Vas J. V. and Thomas M. J., “Electromagnetic Shielding Properties of Nano Carbon Filled Silicone Rubber Composites”, Joint IEEE International Symposium on EMC, Dresden , Germany 2015.
2. Vas J. V. and Thomas M. J., “Electromagnetic Shielding Effectiveness of Multiwalled Carbon Nanotube filled Silicone Rubber”, 13th International Conference on Electromagnetic Interference and Compatibility, 22-25th July, 2015, Visakhapatnam, India.

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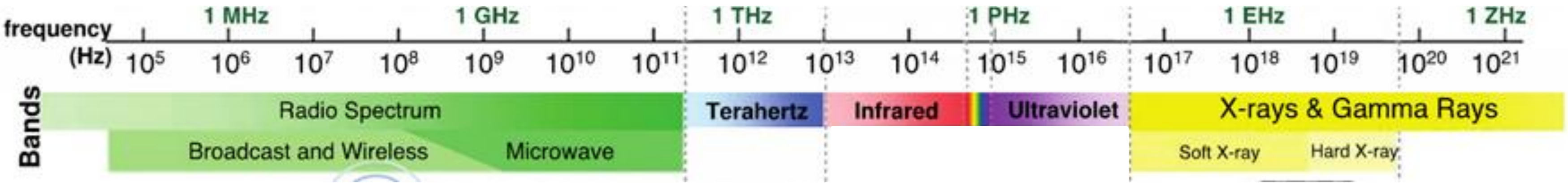


Stray EM radiation problems and how to solve it- **EMI Shielding**

- A power surge due to EMI in one of the fighter planes on ***USS Forrestal*** triggered a missile to fire on board leading to a fire and 134 lives were lost (Vietnam, 1967).

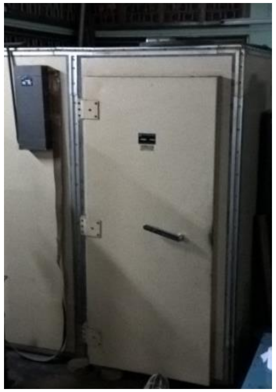
USS Forrestal (CV-59)

- ***Telesat's Anik E1 and E2***-The impulses created by this ESD permanently damaged critical components within the primary gyroscope guidance system control circuitry (Canada, 1994)

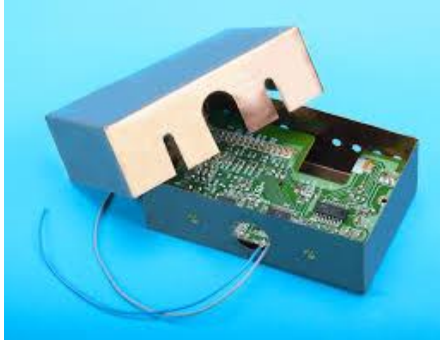
Telesat's Anik E1 satellite.

Different EM Shields

Microwave oven door



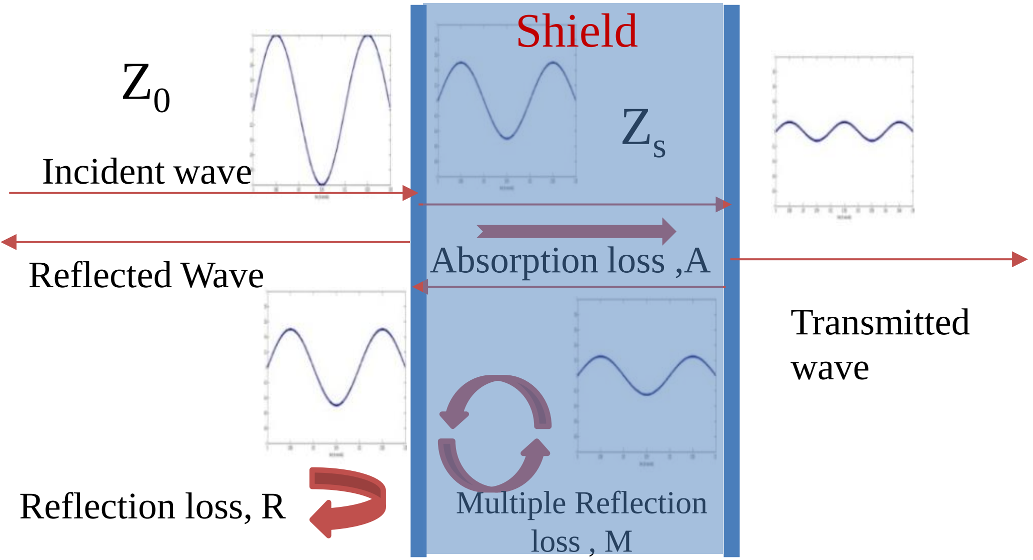
Shielding Chamber (EE, IISc)



Casing of sensitive electronics

EM Shields

Shield is any object, usually conducting, that reduces the effect of EM fields on one side from interacting with the devices or circuits on the other side.



Z_0

Shield

Z_s

Incident wave

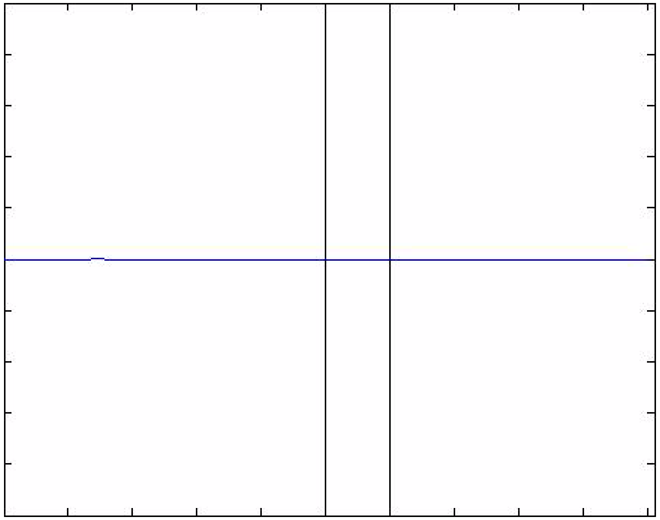
Reflected Wave

Absorption loss ,A

Transmitted wave

Reflection loss, R

Multiple Reflection loss , M



EM propagation in a thin shield

$$R = |20\log|\frac{(Z_0 + Z_s)^2}{4Z_0Z_s}$$

$$A = 8.686k_sd$$

$$M = 20\log|\frac{(Z_0 + Z_s)^2 - (Z_0 - Z_s)^2e^{-j2k_sd}}{(Z_0 + Z_s)^2}|$$

- Shielding effectiveness

$$SE = 20\log\frac{E_{tn}}{E_{ts}}\text{ dB}$$

E_{tn} and E_{ts} are the transmitted Electric fields without and with shield respectively.

$$Z_s = \sqrt{\frac{j\omega\mu_s}{\sigma_s + j\omega\varepsilon_s}} \qquad k_s = j\omega\sqrt{\mu\varepsilon}$$

ε_s – shield permittivity, σ_s– shield conductivity, μ_s– shield permeability, ω = 2πf

Literature Review - Conductivity achieved in Polymer Composites

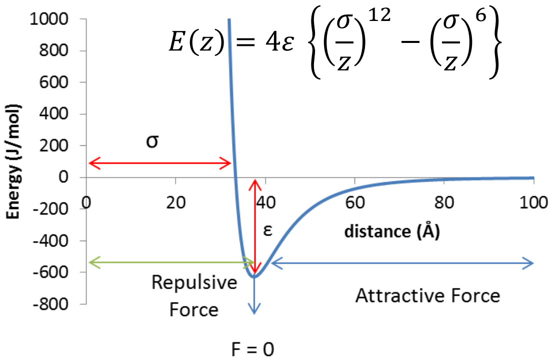
No.	Filler	Polymer	wt %	Conductivity (S/m)
1	carbon nanotubes	Epoxy	10	1.00E+05
2	Carbon fibre (.16u dia, 100u)	theroplastics	40	2.86E+01
3	Ni filament (.4u)	theroplastics	37	7.14E+03
4	Silver(0.8um)	Polyimidesiloxane	40	6.71E+07
5	CNF (50-200nm)	LCP	15	1.43E+01
6	Carbon fibre (7um)	Epoxy	47	2.22E+01
7	Ni coated Carbon fibre (16nm dia)	PES	7	2.50E+02
8	Carbon black (29nm)	EVA/NR	20	1.00E+02
9	CNT	Shape Memory Polymer	6.7	8.33E+00
10	Expandable graphite	PPS	10	1.00E+02
11	CNT	Epoxy	1	1.00E+03
12	MWCNT	Silicone	1.5	1.00E-03

Conductivity

- Carbon – 1.28 x 10^5 S/m
- Silicone rubber – 3.85 x 10^{-19} S/m
- Copper - 5.85 x 10^7 S/m

Monte Carlo Simulations for Conducting Polymer Composites

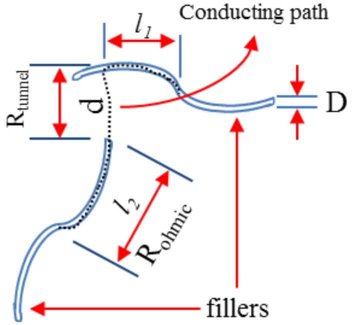
Step 1 – MC simulation performed to find the particle distribution using LJ potential.

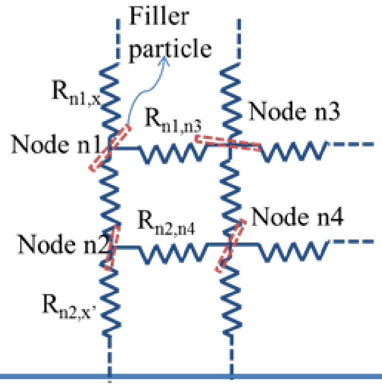


$$E(z) = 4\varepsilon\left\{\left(\frac{\sigma}{z}\right)^{12} - \left(\frac{\sigma}{z}\right)^{6}\right\}$$

The LJ potential

Step 2 – Calculation of the interparticle contact resistance using the particle orientation





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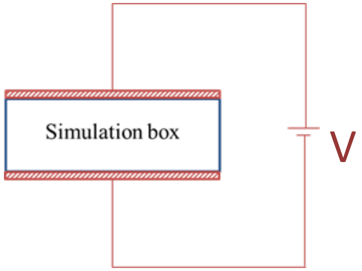
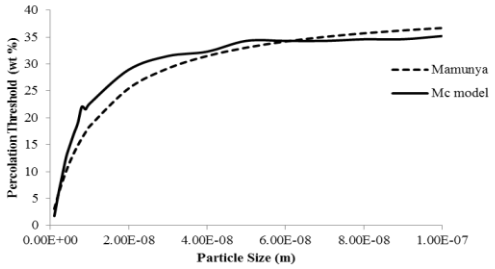
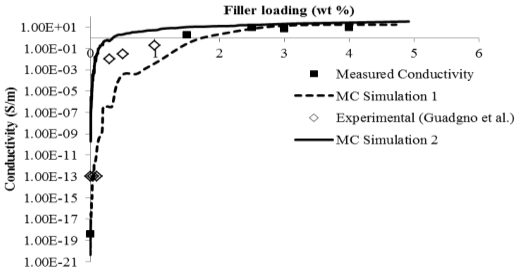


Validation of the model



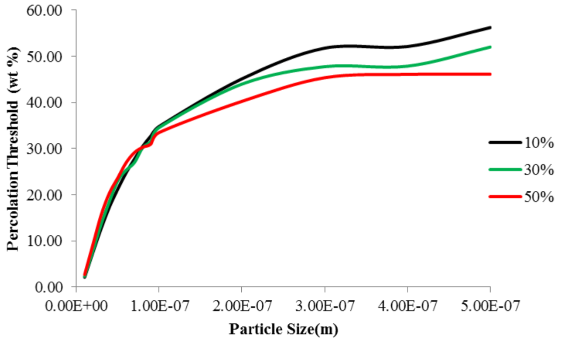
Comparison with theoretical models

Comparison with experimental data

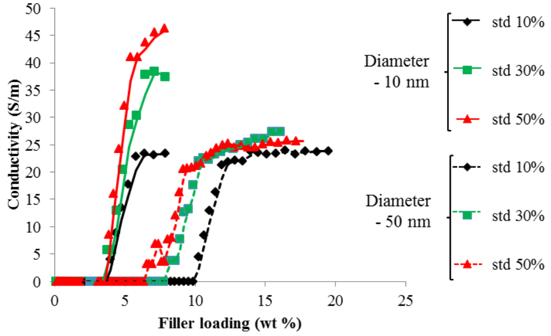


Monte Carlo Studies on Spherical and Rod like Particles

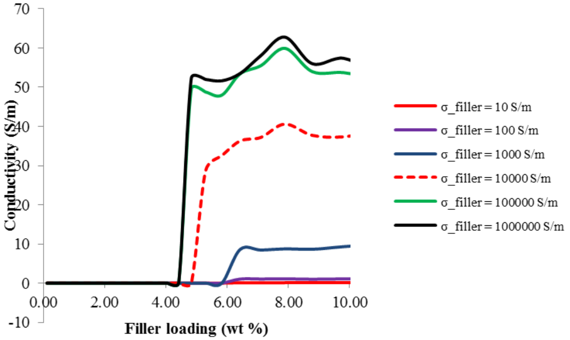
Composites with spherical particles



Percolation vs. particle size

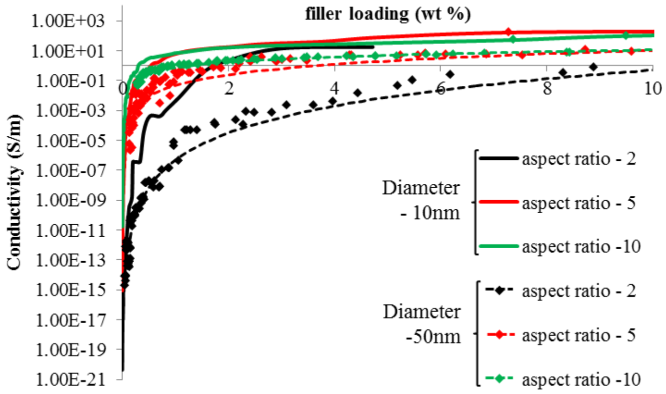


Conductivity vs. particle size



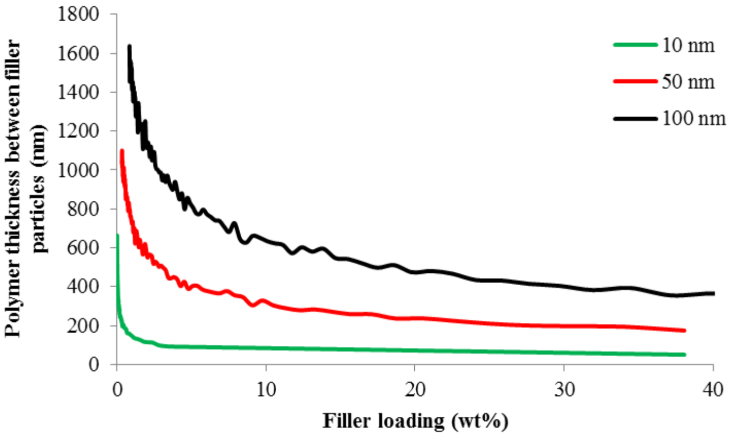
Variation of particle conductivity

Composites with rod like particles



Conductivity vs. particle size

Conductivity Limitation

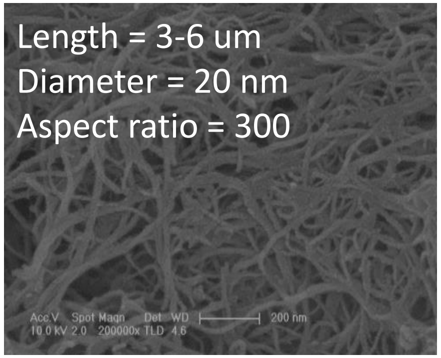


Interparticle distance vs filler loading

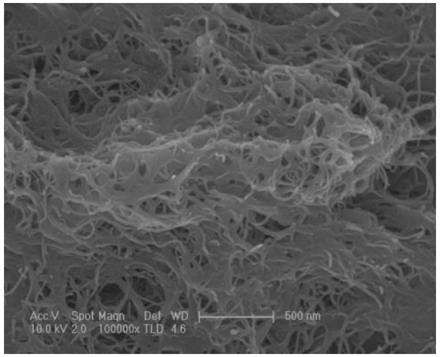
Synthesis of Conventional Composites

Nano composites synthesis

RTV SR – Polymer + Nanofillers + Solvent $\xrightarrow{\text{Ultrasonication}}$ RTV filler mixture + Pt Catalyst $\xrightarrow{\text{Curing}}$ Nano filled SR

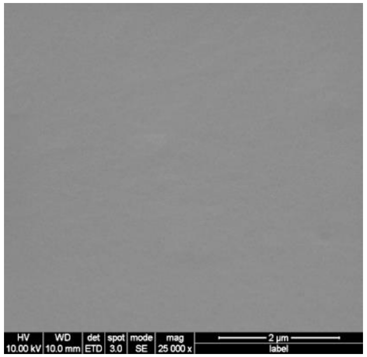

Length = 3-6 um
Diameter = 20 nm
Aspect ratio = 300

SEM of MWCNT

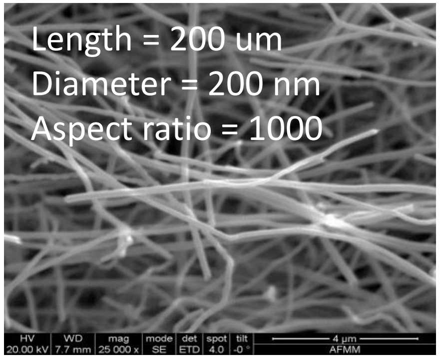


MWCNT-SR Composite

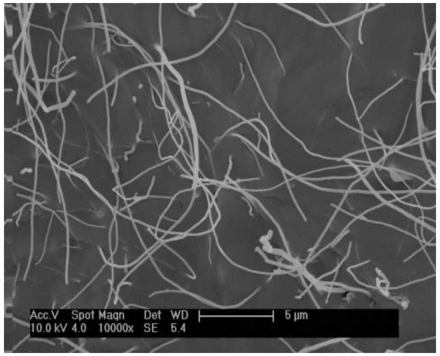
Mold



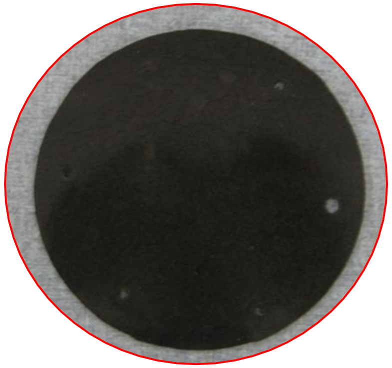
SEM of Silicone Rubber


Length = 200 um
Diameter = 200 nm
Aspect ratio = 1000

SEM of CNF



CNF-SR Composite



SR filled with nano carbon

Synthesis of SR composites layered with CNF wafers

$$\text{Silver Nitrate + Benzyl mercaptan + Solvent} \xrightarrow[\text{Ultrasonication}]{\text{Magnetic Stirrer}} \text{Nano AgS + CNF} \xrightarrow[\text{Vacuum filtration}]{\text{Ultrasonication}} \text{CNF wafer}$$

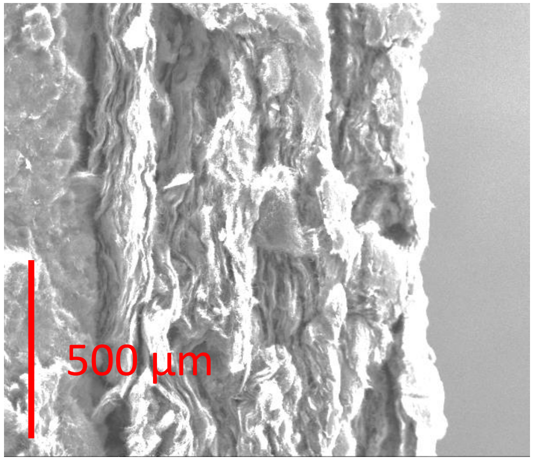
Nano AgS Synthesis

Initial mixture

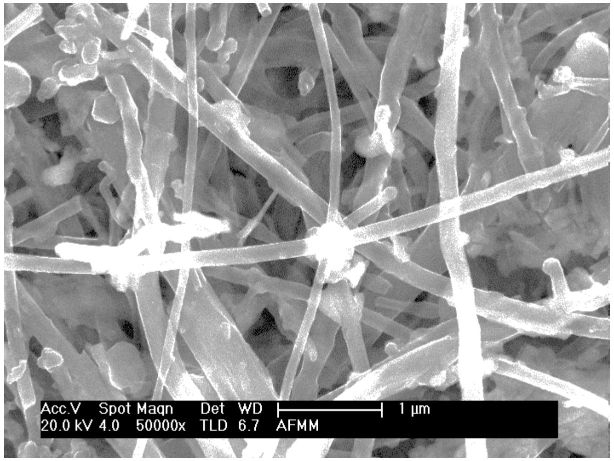
Nano AgS

CNF wafer
σ = 1320 S/m

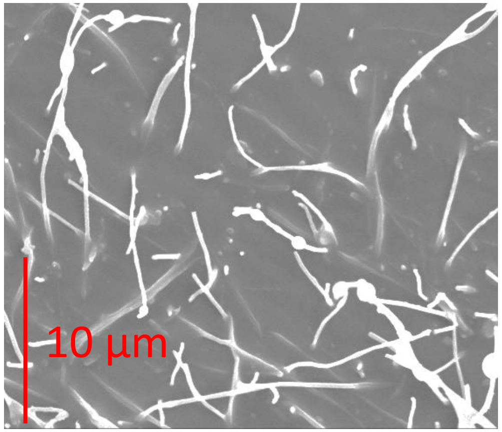
SR composites layered with CNF wafer



SEM image of cross section of CNF wafer

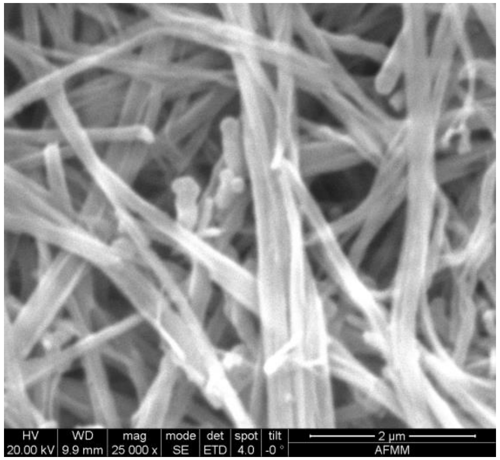


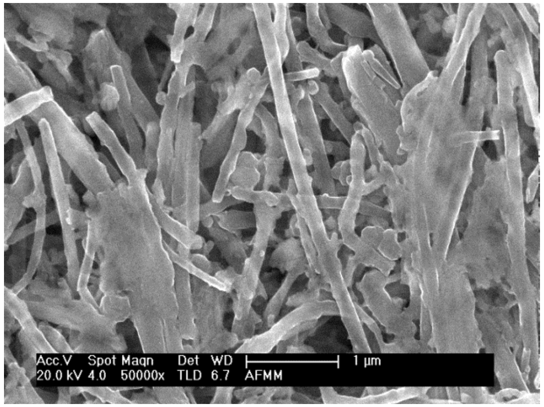
SEM image of the structure of CNF wafer

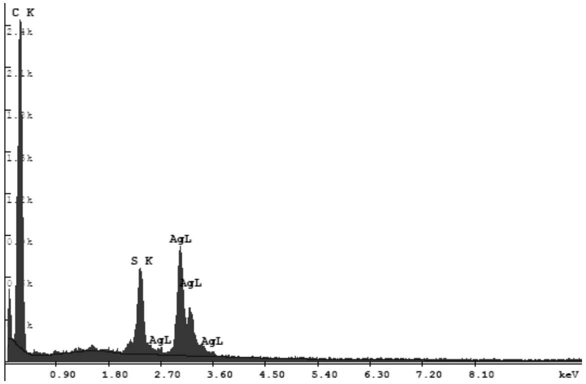


SEM image of CNF wafer- unf SR composite

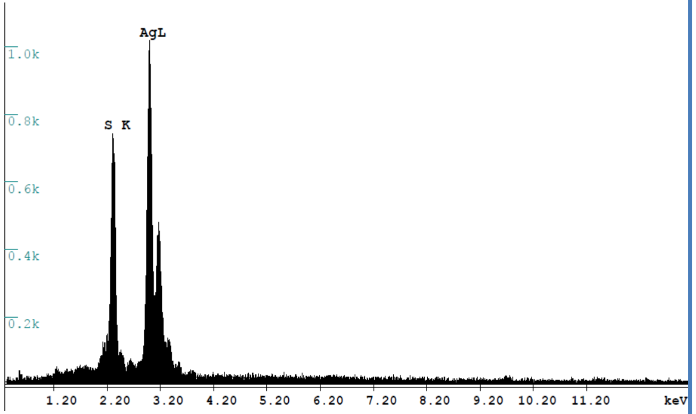
Results – SEM and EDX studies







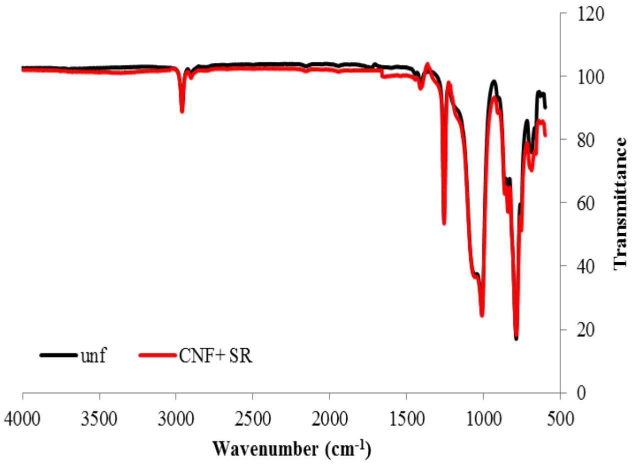
CNF -Ag- S complex



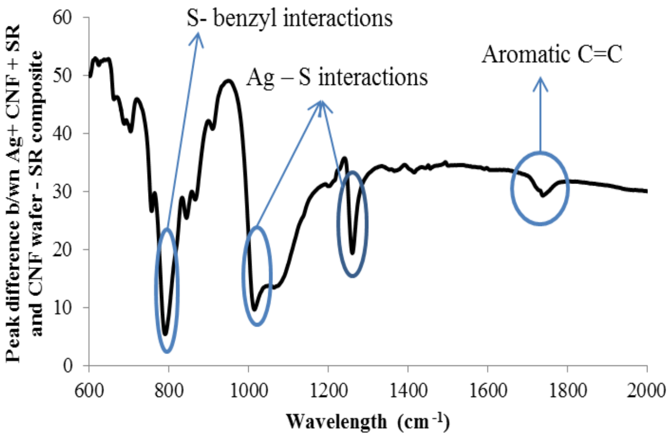
Ag- S particles

No	Sample Type	C	O	Si	Ag	S
1	unfilled	54.32	17.3	46.16		
2	CNF filler	89.47	7.4			
3	Ag-S particles				57.2	42.7
4	CNF wafer	92.84			2.66	2.36
5	CNF wafer- SR composites	70.26	12.8	15.55	0.43	0.48

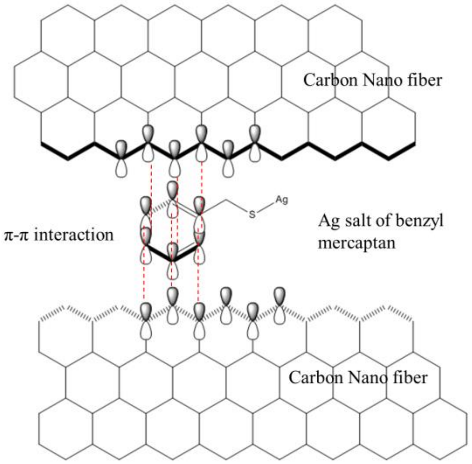
Results – FTIR



FTIR spectra of unfilled and CNF filled SR



Difference between the FTIR spectra of Ag-CNF binary composites and SR composite layered with CNF wafer



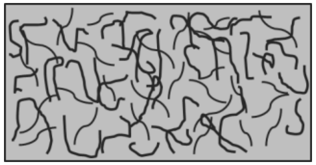
Structure of the CNF wafer

No	Group	Wave number (cm^{-1})	Material
1	-C = C- (Alkenyl group)	1680-1620	CNF interactions
2	-C = C - (Aromatic)	1700-1500	AgS nanoparticle
3	-Ag – S	1008,1355	
4	C_6H_5 –CH_2- X	690,710,730-770	

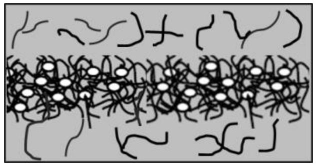
Conductivity and Permittivity Measurements

- The CNF wafer has a conductivity of 1360 S/m
- Both the conventional composites turned conducting at filler loadings less than 3%.

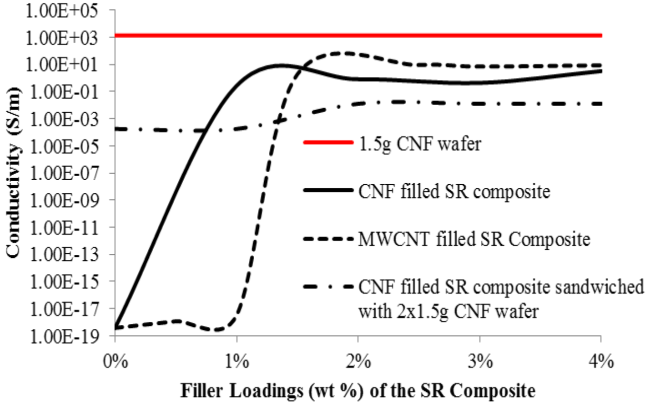
Conventional Composite

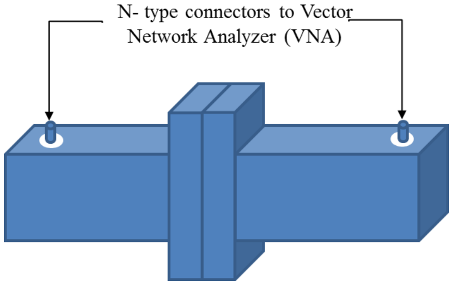


SR Composite layered with CNF wafer

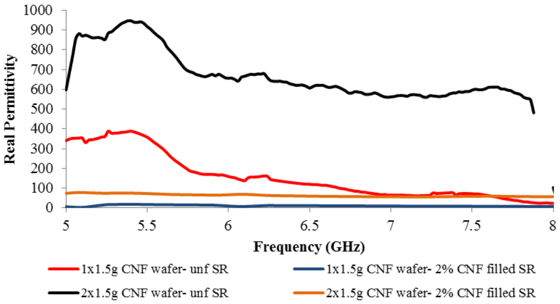


Conductivity vs filler loading for different composites

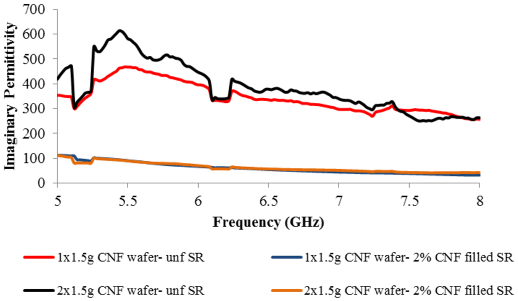




Permittivity measurement as per ASTM D5568



Real Permittivity

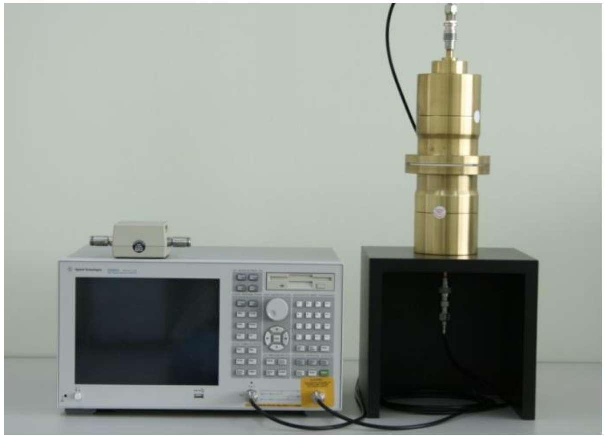


Imaginary Permittivity

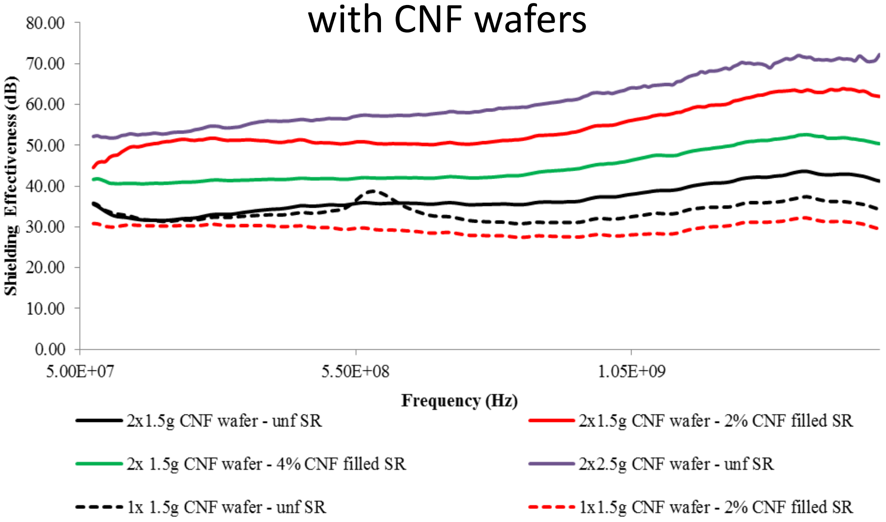
- Permittivity of conventional composites < 10
- SR composites layered with CNF wafers showed very high real and imaginary permittivities

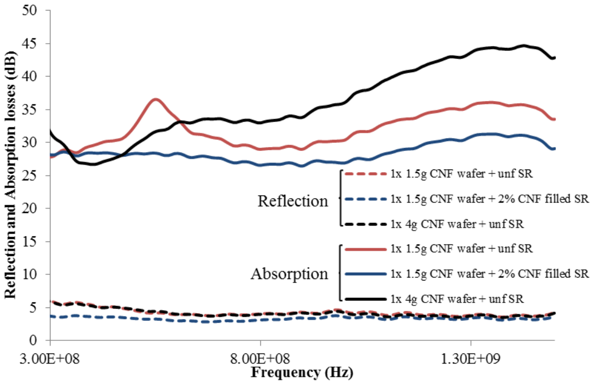
Shielding Effectiveness of different composites

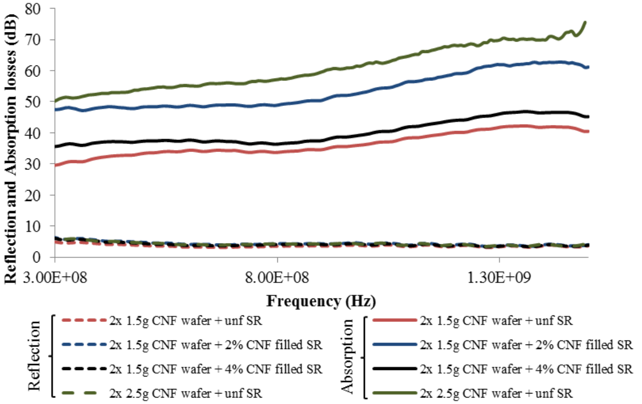
ASTM D4935 measurement set up fabricated in the lab



Shielding Effectiveness of SR composites layered with CNF wafers



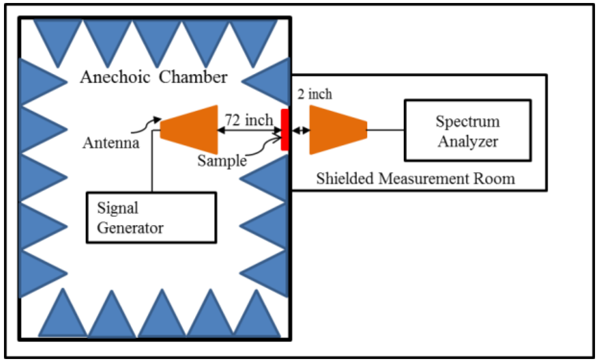


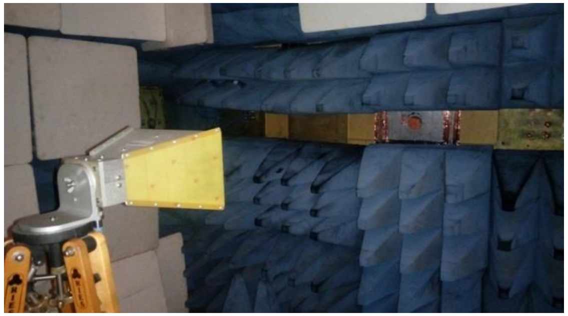


Reflection and absorption losses of SR composites with 1 CNF wafer layer

Reflection and absorption losses of SR composites with 2 CNF wafer layers

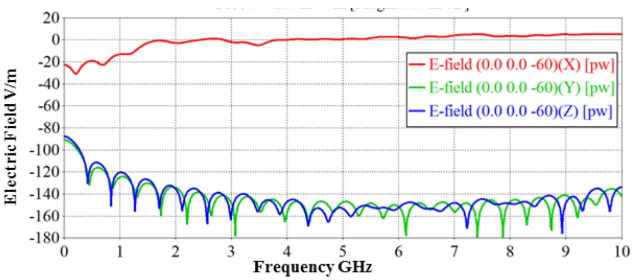
Setup used for the Anechoic Chamber measurements

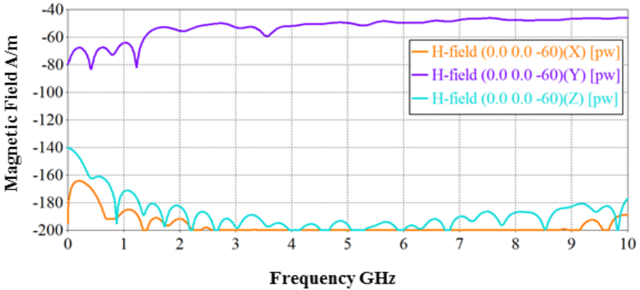




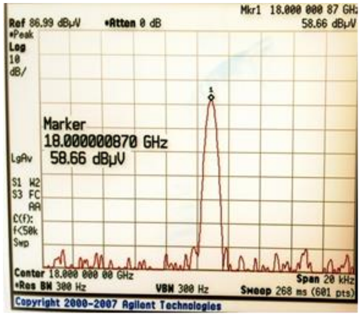
Radiated field

Horn antenna used for measurement



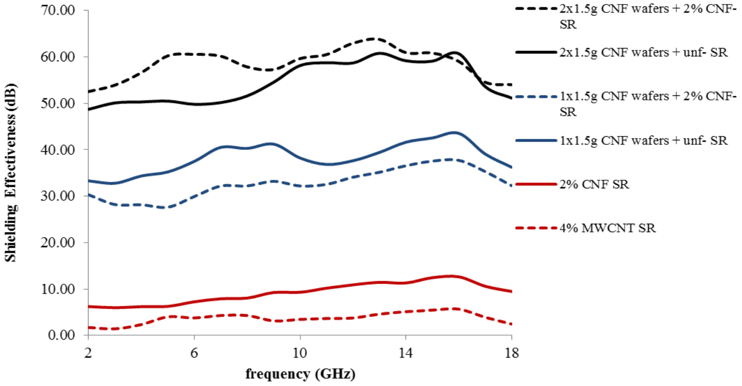


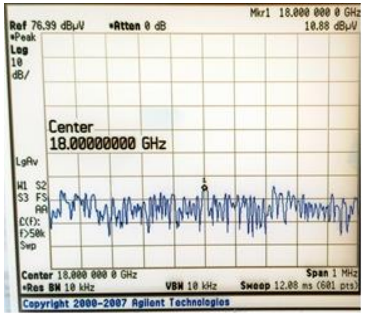
Electric and Magnetic Fields Experienced by the sample



Measured field in the absence of the sample

Shielding Effectiveness Measured in the 2-18 GHz frequency range

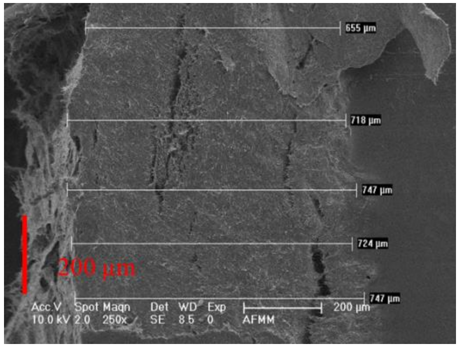




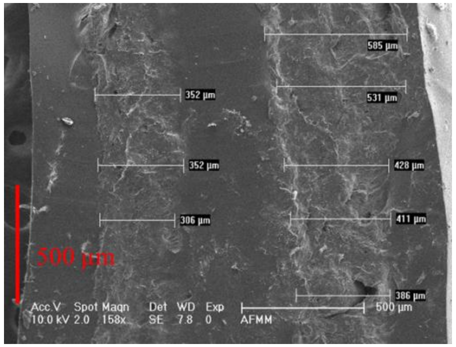
Measured field with the sample

EM Modelling of Layered Composites

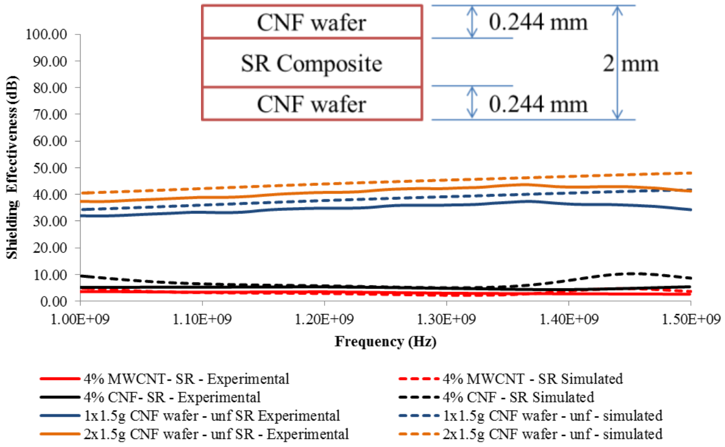
Sample Type	Absorption (dB)	Thickness (mm)
1x1.5g CNF wafer	-	0.244
1x1.5g CNF wafer - unf SR	31.32	0.729
1x1.5g CNF wafer - 2% CNF+ SR	28.61	0.697
2x1.5g CNF wafer - unf SR	34.93	0.752
2x1.5g CNF wafer - 2% CNF+ SR	52.22	1.942
2x1.5g CNF wafer - 4% CNF + SR	43.98	0.675



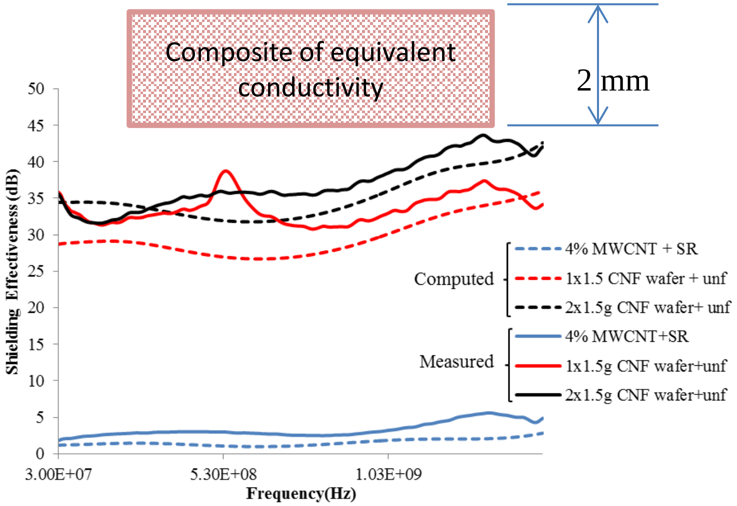
CNF wafer with 1 wafer



CNF wafer with 2 wafer



Prediction of SE based on 3 layer model



Prediction of SE based on permittivity

Conclusions

Studies on Conventional Composites

- The conventional conducting polymers with MWCNT and CNF filled SR had very less conductivity and shielding effectiveness.
- The shielding effectiveness of the conventional conducting composites were not suitable for shielding applications.

Studies on SR composites with CNF wafers

- Composites were synthesized with highly conducting CNF wafers.
- Even though the bulk conductivity of the composites were low, the CNF wafer composites showed good shielding behavior.
- This was because of the highly conducting CNF wafer layers present in the composite.

Waveguide measurements

- The shielding behavior, reflection loss, absorption loss was measured in the frequency range 5-18 GHz.
- Samples showed trends similar to the Anechoic Chamber method and the coaxial fixture method for low frequency.
- The reflection loss of all the composites were low but increased with CNF content.
- This could be because of the increase in real permittivity due to increased carbon content.
- The large absorption loss was due to the higher imaginary permittivity of the layered composites.

Shielding, Reflection and Absorption Measurements

- The set up used was designed such that the samples experienced a TEM wave.
- The conventional composites showed very low shielding effectiveness.
- The SR composites with different layers of CNF wafers had very high shielding effectiveness
- All the composites showed very low reflection loss
- The shielding behavior was mainly attributed to the absorption loss
- The absorption loss depends on the thickness of the CNF wafers in the SR matrix
- The reflection loss marginally increased with CNF content.