National Aeronautics and Space Administration

OAI
Ohio Aerospace Institute

Multifunctional Graphene Polyimide Nanocomposites

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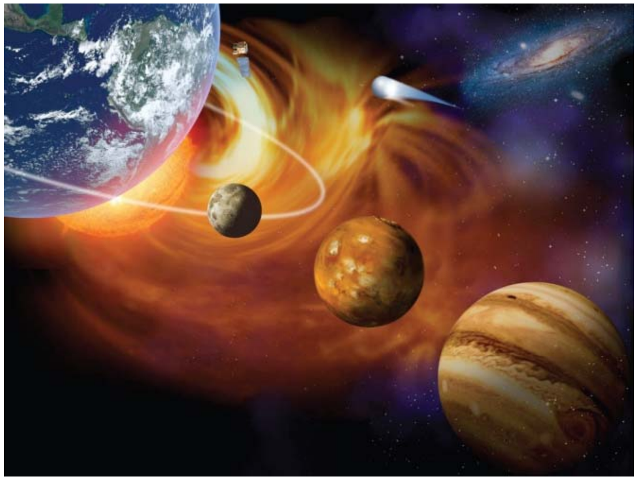
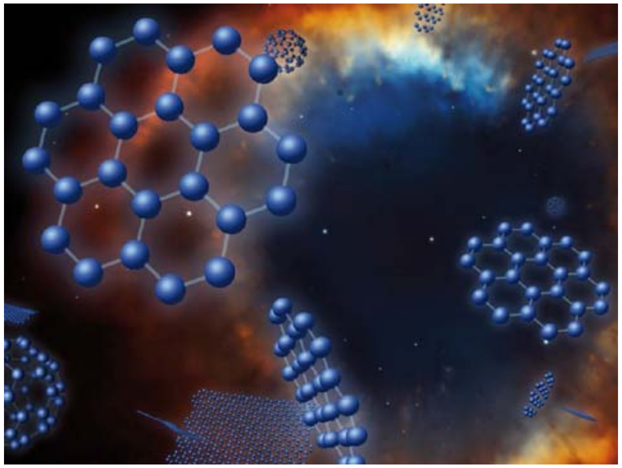
ASRC, Cleveland, OH



Nanotechnology Road map **Technology Area 10**



Graphene in Space, NASA's Spitzer Space Telescope has spotted the signature of flat carbon flakes, called graphene, in space

Nanotechnology
Engineered Materials and Structures

Light Weight Materials

- Multifunctional

-Adaptive Materials

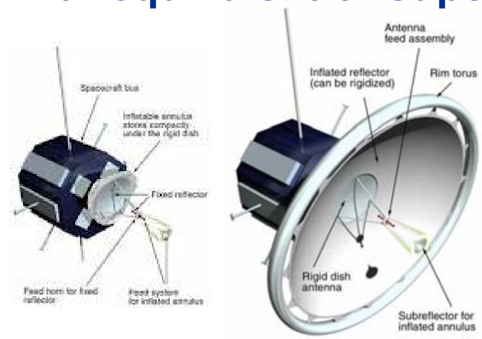
-Self Healing Materials

Development of nanostructured materials 50% lighter than conventional materials with equivalent or superior properties

Reduced Vehicle Mass

Boeing 787 composite aircraft

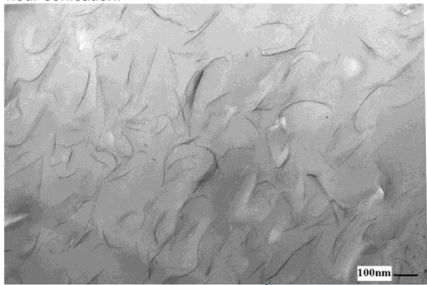
Copper mesh 4000 lb of weight



NGST ½-scale Sunshield Demonstration Model Deployment,
Cadogan, D. P.et al.

Meador, M., Files B., Li J., Manohara, H., Powell, D., Siochi, E.J. Nan otechnology Road map Technology Area 10

hour sonication.



oparticles:

s - Ijima 1991

~ 1315 m^2/g

DCNT ~ 700-800

Graphite and Graphene – Giem 2004

Graphene ~ theoretical:2600 m^2/g, 700- 1300 m^2/g

arbon nanofibers

Alumina silicates – Fukushima, Toyota 1987

ontmorillonite~ 725 m^2/g

Magadiite , Laponite, Vermiculite

agnetic Nanoparticles

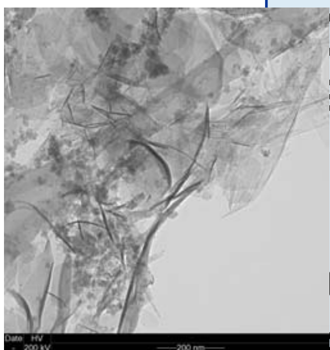
rganometallic physical crosslinkers

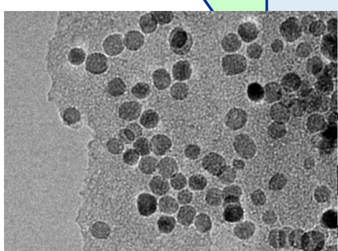
OSS

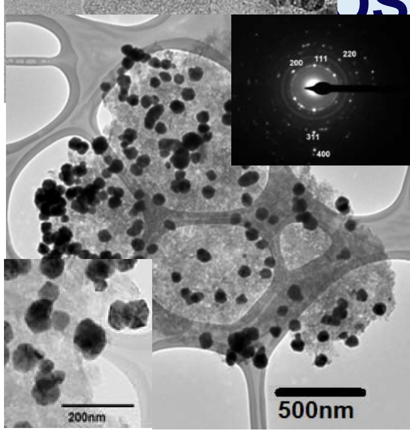
posite Nanoparticles

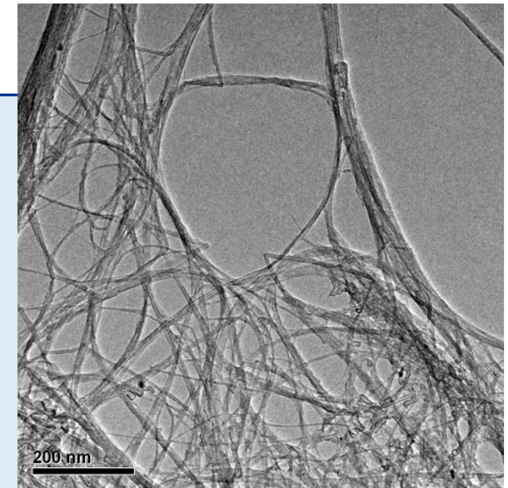
tic graphene

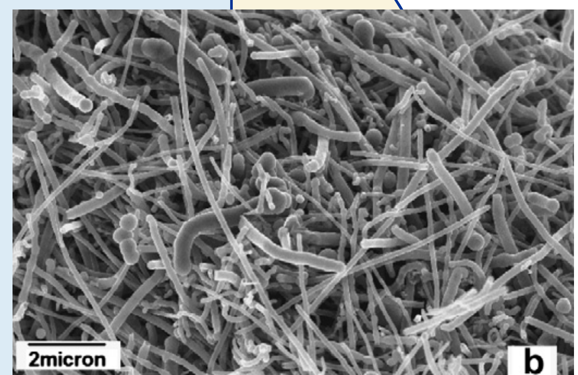
ide graphene

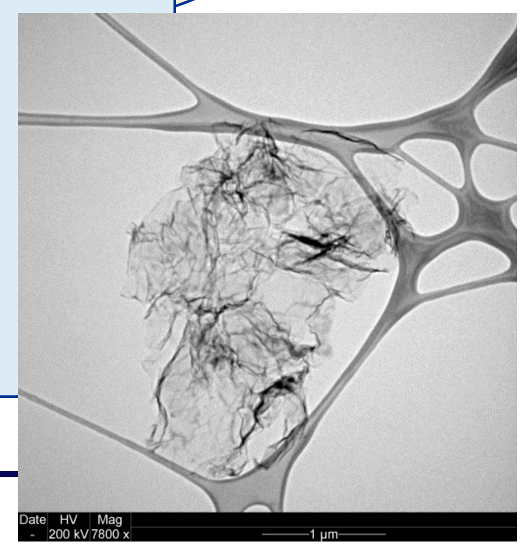


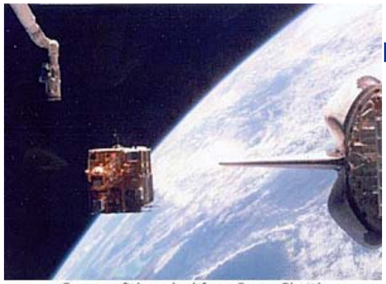











Spacecraft launched from Space Shuttle

pace Administration

Polyimide High Performance Polymer

Satellite

Aromatic polyimide:
- Low color
- Flexibility
- High thermal stability
- Dimensional stability
- Low dielectric constant
- High T_g
- Radiation resistance
- Low coefficient of thermal expansion

PMR-15 - GRC
After burner

General Ind.

Electronics and packaging

Multifunctional

- Stiffness and modulus and reinforcement
- Actuation and morphing
- Electrical performance and EMI shielding
- Thermal performance and stability

Quartz fabric–polyimide 815 °C

- •Space
- •Aero
- •Electronics

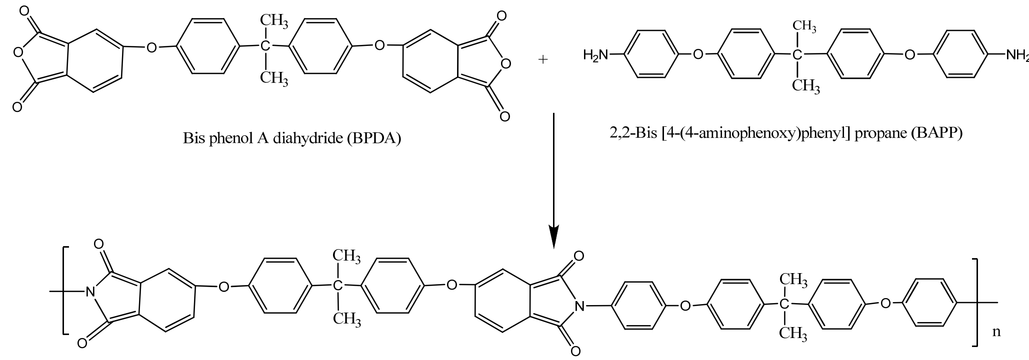
Continuous operating range between -65 °C to +357 °C

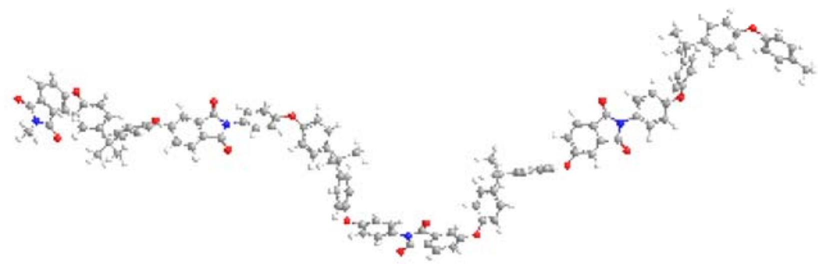
[1]Qu,L., Connell, J.W., Sun, Y.-P., Macromolecules, 2004, 37, 6055-6060.
[2]Lebron-Colon, M. Meador, M. A., Gaier, J. R., Sola, F., Scheiman, D.A., McCorkle, L.S. ACS Applied Materials and Interfaces, 2010, 2 ,3 , 669–676 .

Polyimide Graphene Nanocomposites

PMR-15
GRC
After burner

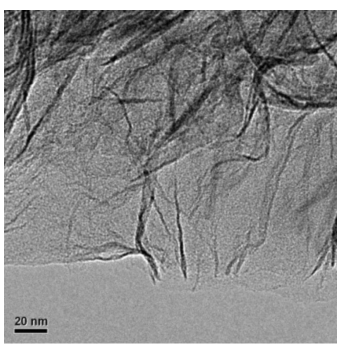
Polyimide, thermal stability >500 °C, T_g > 200 °C, flexible and semi-transparent.





Thermal imidization:

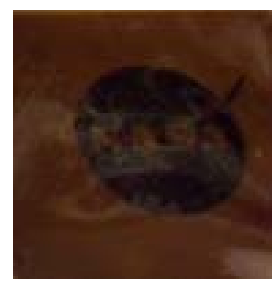
- Mixing and dissolving equi-molar ratio diamine in anhydrous-NMP under dry N_2 followed by addition of dry anhydride and stirring for 24h in flame dried vessels.
- Then, increasing the temperature ~230 °C (NMP reflux) for 3h and precipitating in methanol and drying



\+ Polyimide solution

sonication

Solvent casting →



0.025 wt% graphene/PI

[1]Qu,L., Connell, J.W., Sun, Y.-P., Macromolecules, 2004, 37, 6055-6060.
[2]Lebron-Colon, M. Meador, M. A., Gaier, J. R., Sola, F., Scheiman, D.A., McCorkle, L.S. ACS Applied Materials and Interfaces, 2010, 2 ,3 , 669–676 .

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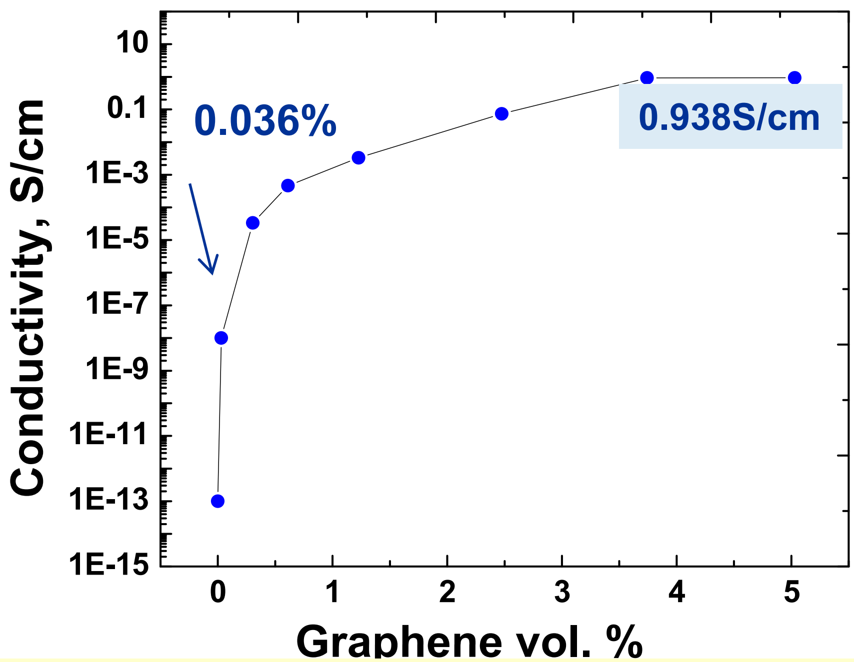
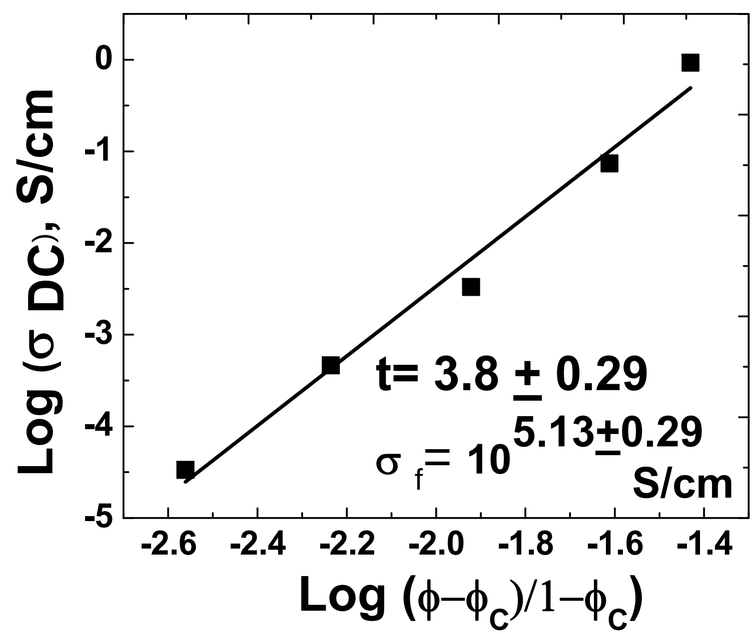
Polyimide Graphene Nanocomposites

Electrical Performance

$$\sigma_{DC} = \sigma_f[(\phi-\phi_C)/(1-\phi_C)]^t$$





	Percolation	Max. Conductivity
Chemically graphene PS nanocomposites	0.1 vol.%	0.01S/cm
PS Gr, Latex method	0.6 wt%	0.15S/cm
PET graphene	0.47 vol.%	0.021S/cm
PC graphene, emulsion	0.14 vol.%	0.512 S/cm
PC graphene, solution	0.38 vol.%	0.226 S/cm
PS CCG	0.19 vol.%	0.722S/cm

Viet Hung Pham et.al, J. Mater. Chem., 2011, 21, 11312

CNT/nanocomposites
$t = 1.2 – 2$
CNF/polyimide
$t \sim 3.1$
PET graphene
$t \sim 3.47 \pm 0.64$
PS graphene
$t \sim 2.74 \pm 0.2$

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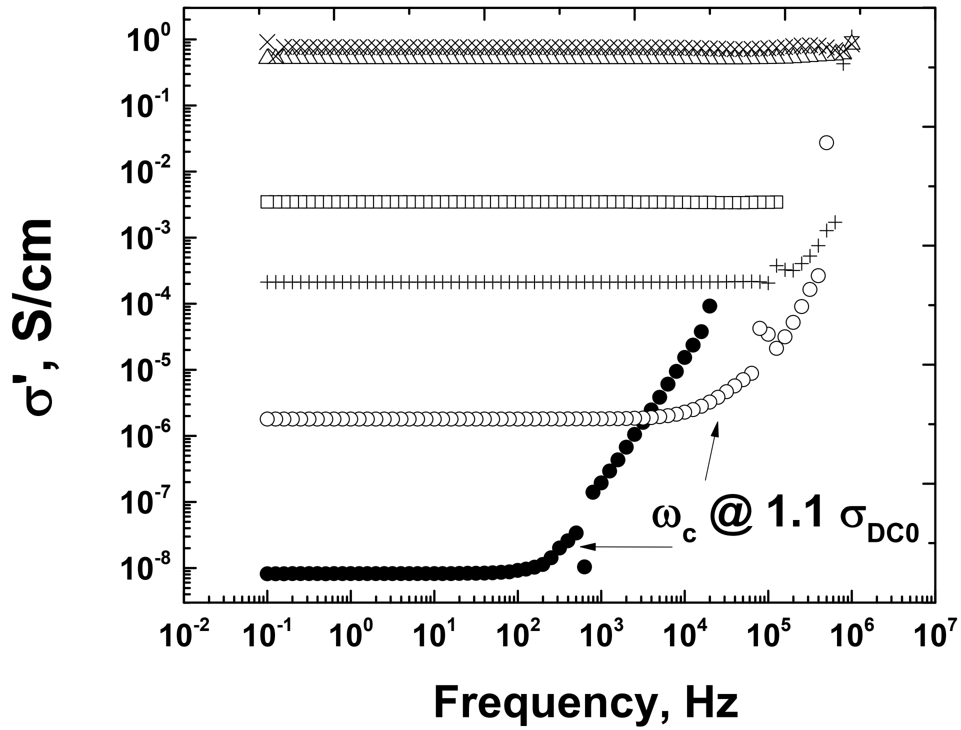
AC Electrical Performance

Broad band AC impedance spectroscopy

Extended pair approximation model

$$\sigma(\omega)/\sigma_{DC0} = 1 + k(\omega/\omega_c)^s$$

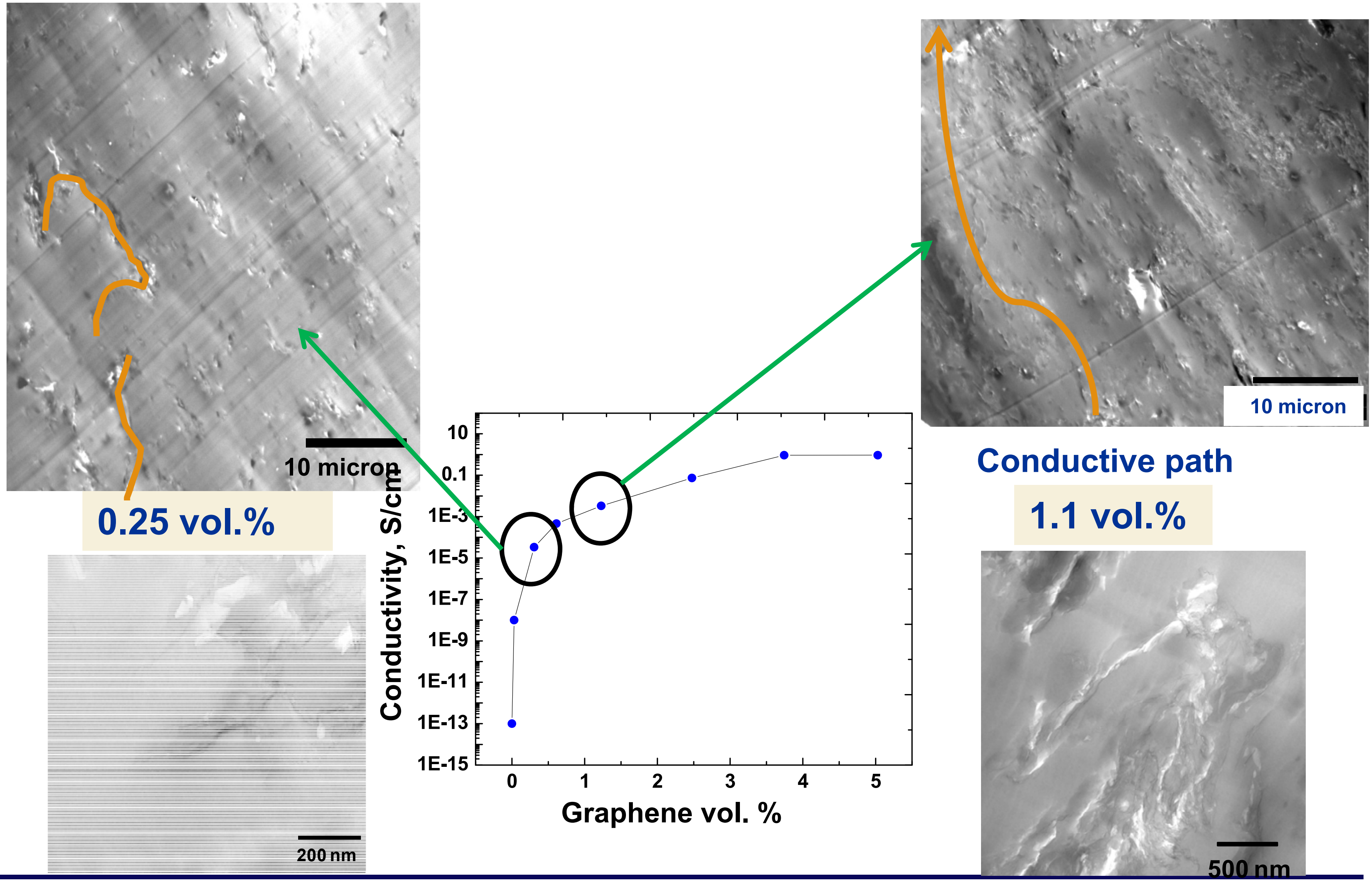
Vol.%	σ_{DC0}, S/cm	ω_c, Hz	s
0.03046	8.21e-9	150.47	0.499
0.3051	1.879e-6	7.027e3	0.647
0.6115	2.11e-4	1.241e5	0.446



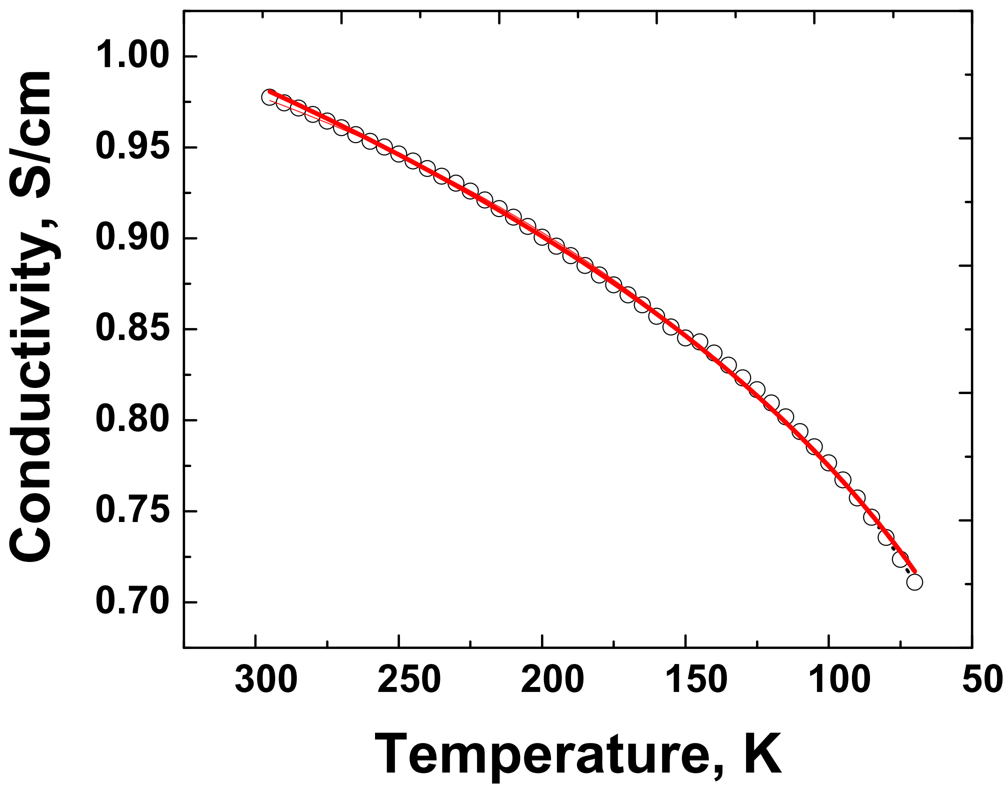
S ~ 0.99 -> hopping
S ~ 0.72 -> 3D material
S ~ 0.58 -> anomalous diffusion in fractal cluster exist

Linares, A., Ezquerra, T.A et al., Macromolecules, 2008, 41, 7090

Dispersion of graphene in polyimide TEM



Temperature Dependence Conductivity



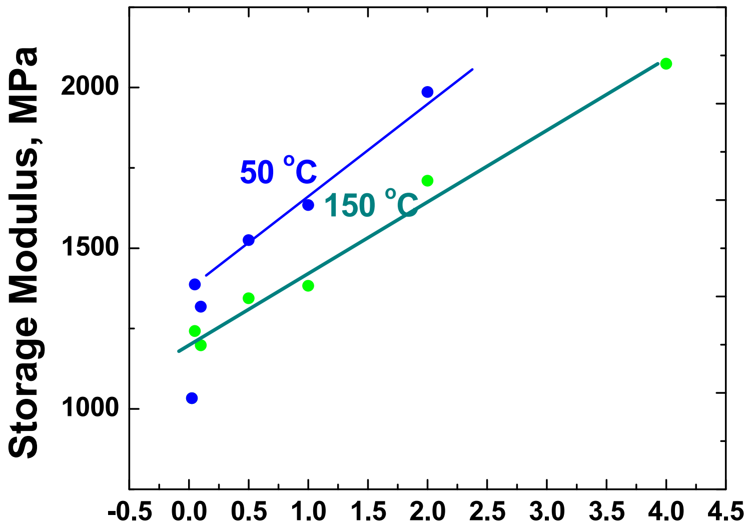
5 vol. % graphene polyimide

$$\sigma = 0.2844T^{0.2177}$$

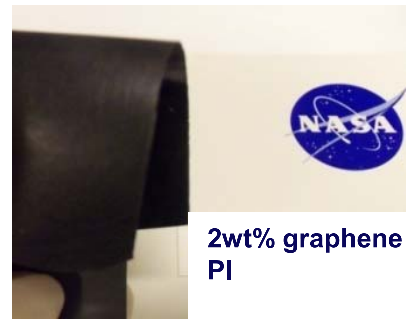
$$T = 322.404\sigma^{4.6}$$

Thermal and Mechanical Properties

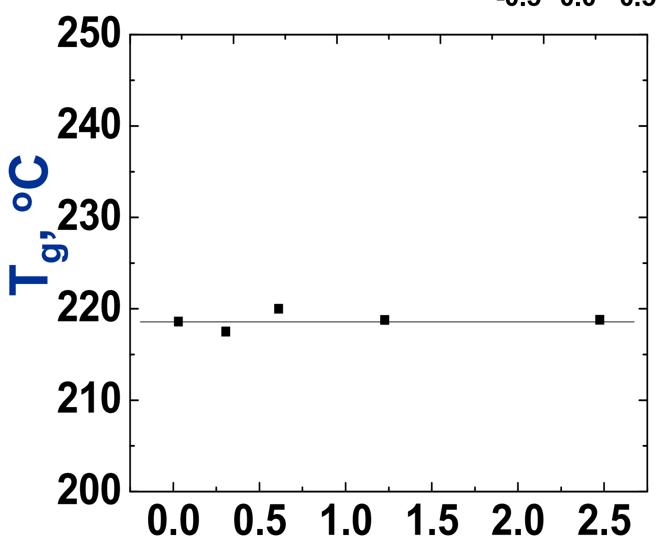
Addition of graphene resulted in composite reinforcement without adverse effect on the T_g



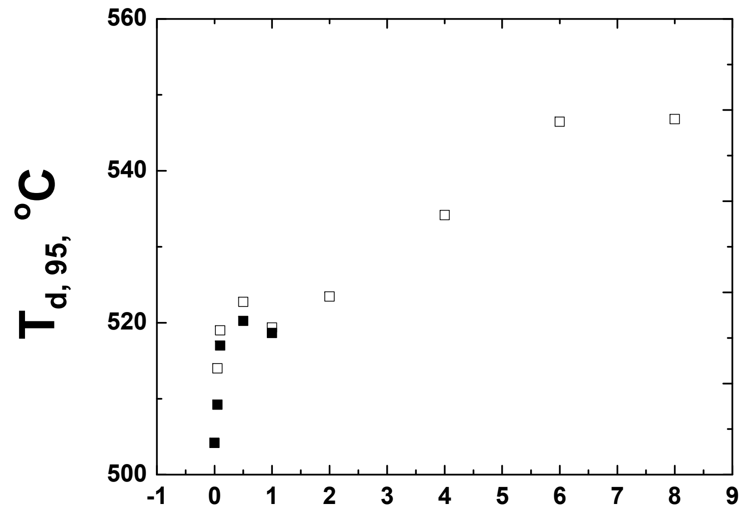
Graphene wt%



2wt% graphene PI



Graphene, vol.%



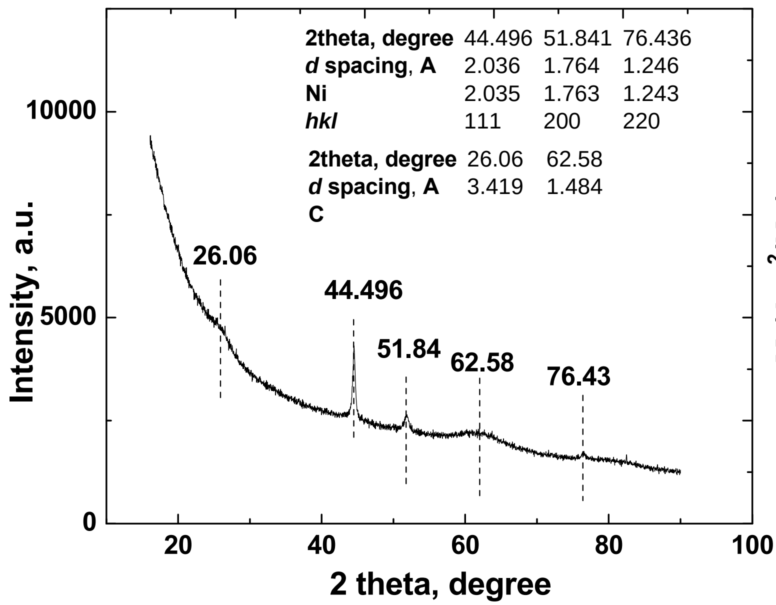
Graphene, wt%

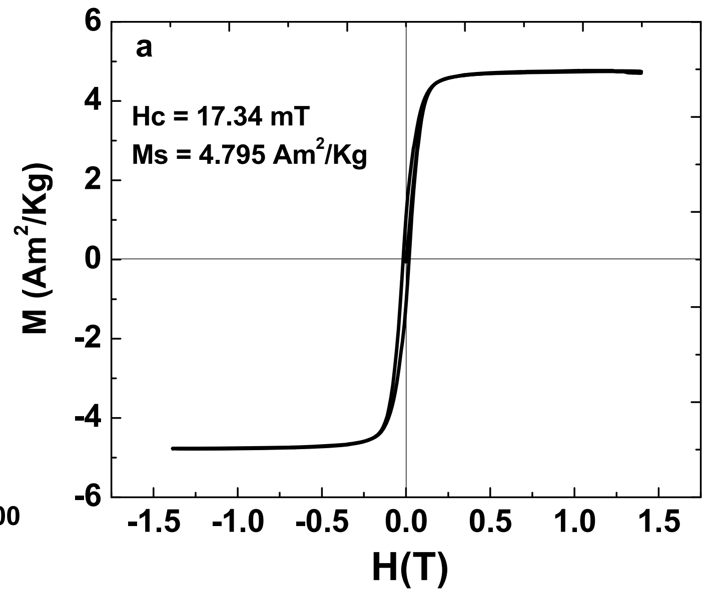
Controlled Property Direction

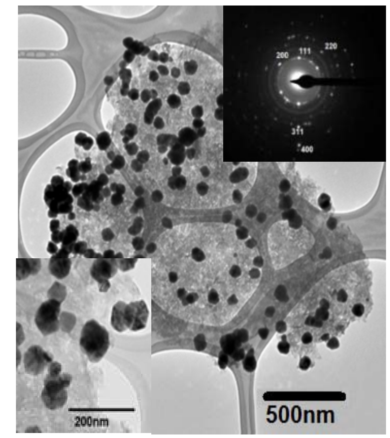
Ni-Tethered Graphene

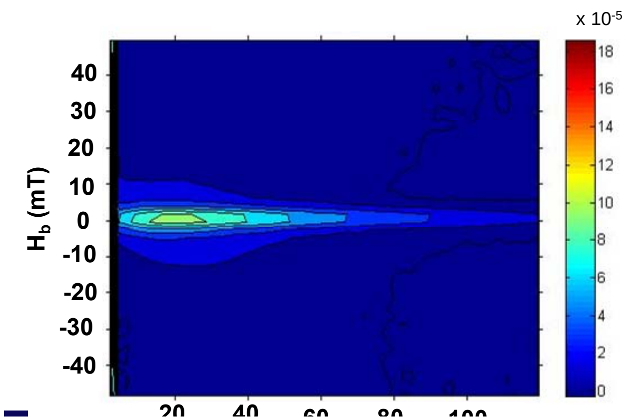
Composites Nanoparticles

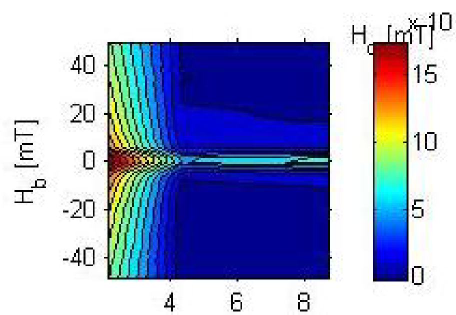
Thermal decomposition of $Ni(acac)_2$ in the presence of O-graphene

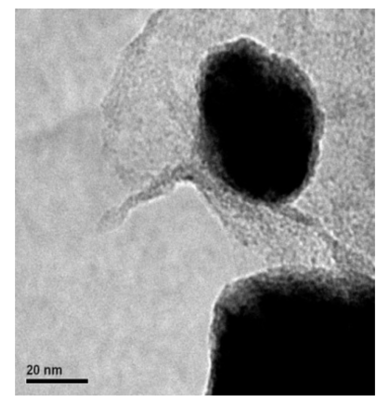






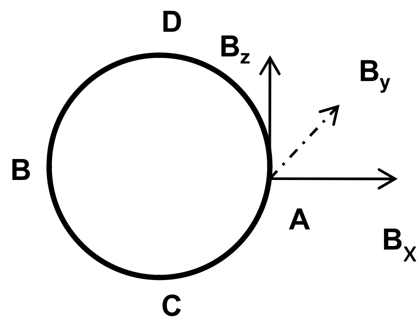




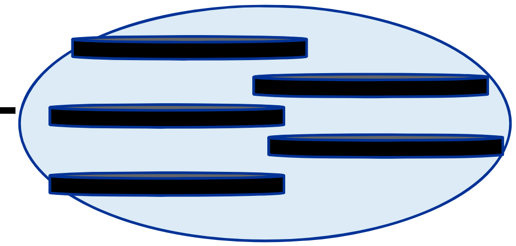


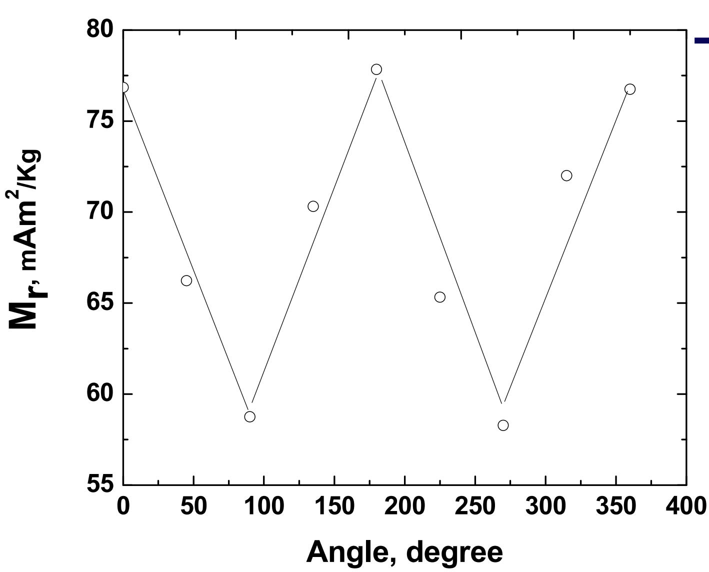
First-order reversal curve (FORC)

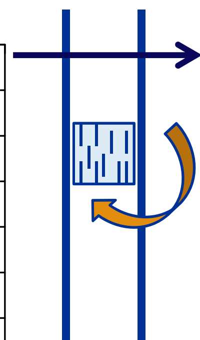
Controlled Directionality

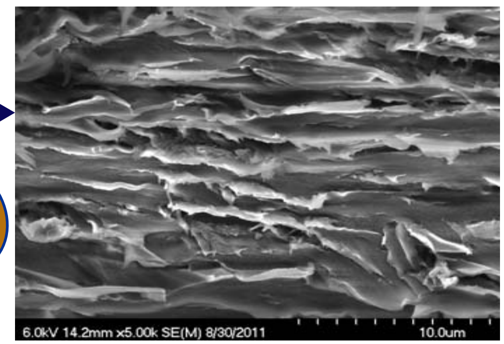


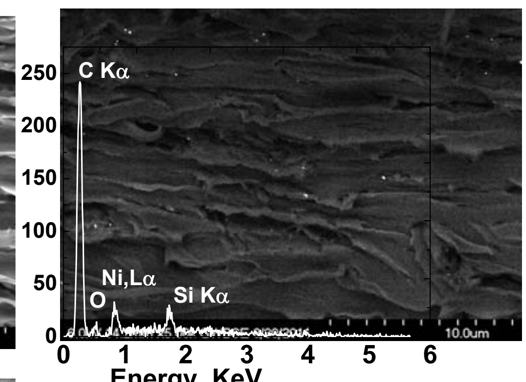
	B (Gauss)	B_x (Gauss)	B_y (Gauss)	B_z (Gauss)
A	1150	-1150	-237	-50
B	976	-948	475	50
C	440	-432	-55	-120
D	500	-520	-12	42.3

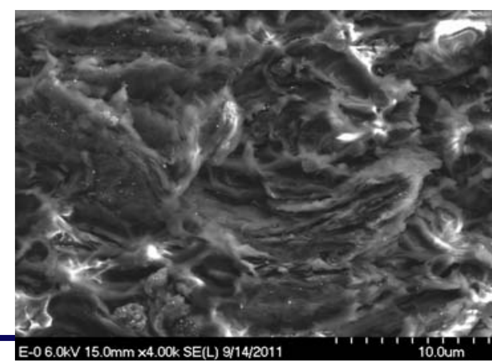


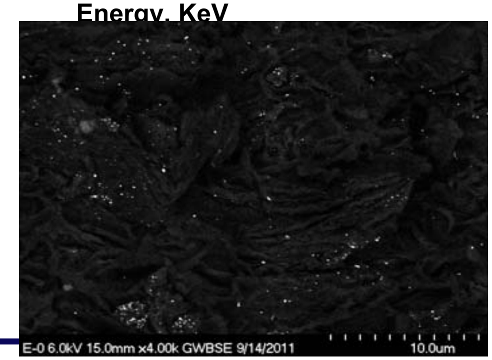








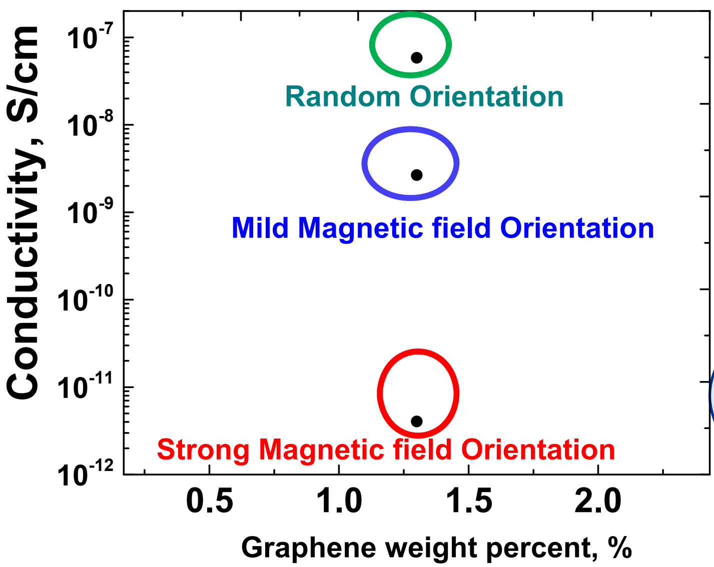


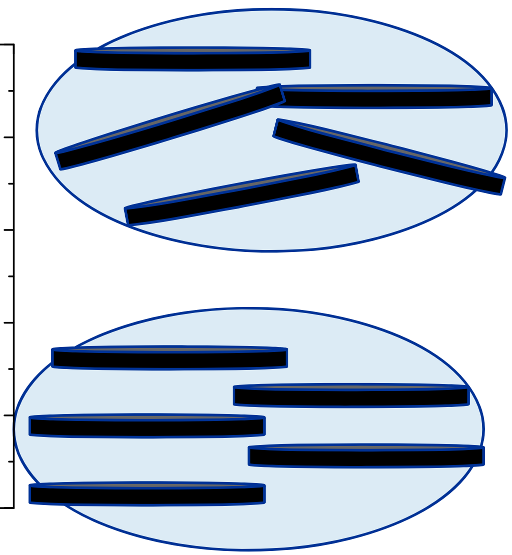


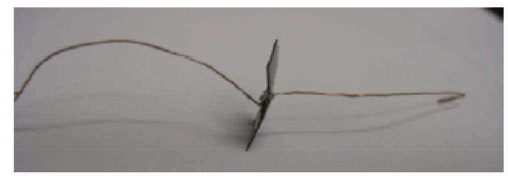
2.8 wt% Ni-Graphene polyimide nanocomposite

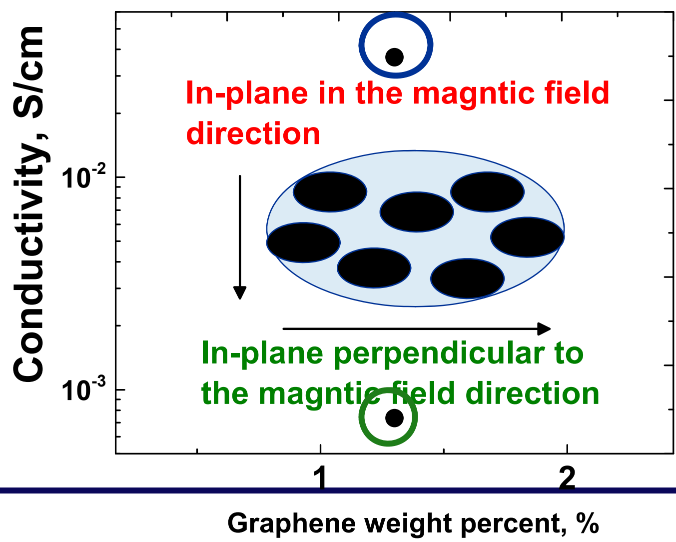
Anisotropic Properties

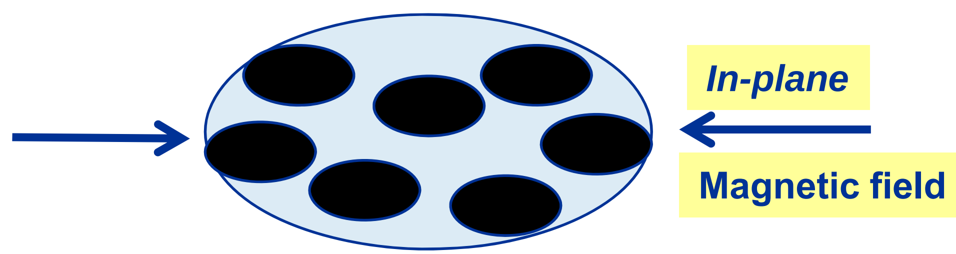
Electrical properties





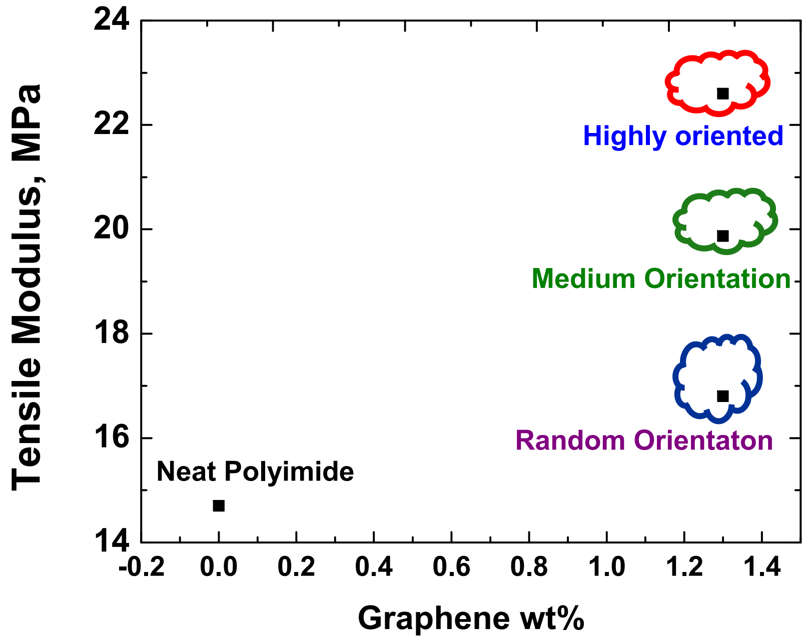


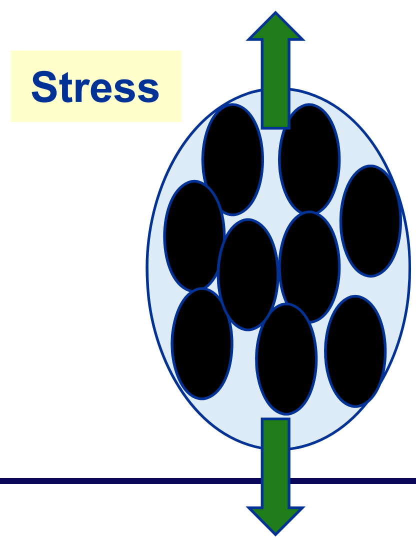


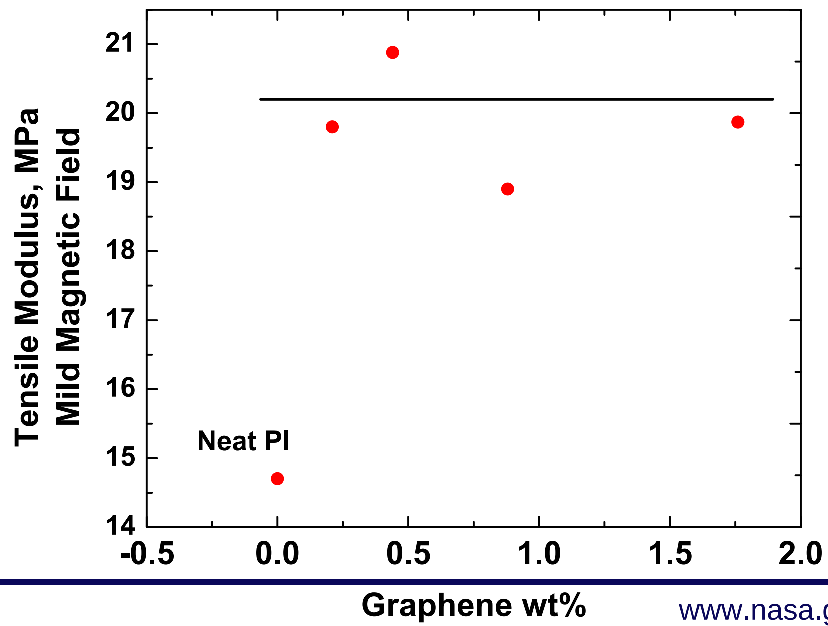


Anisotropic Properties

Mechanical properties

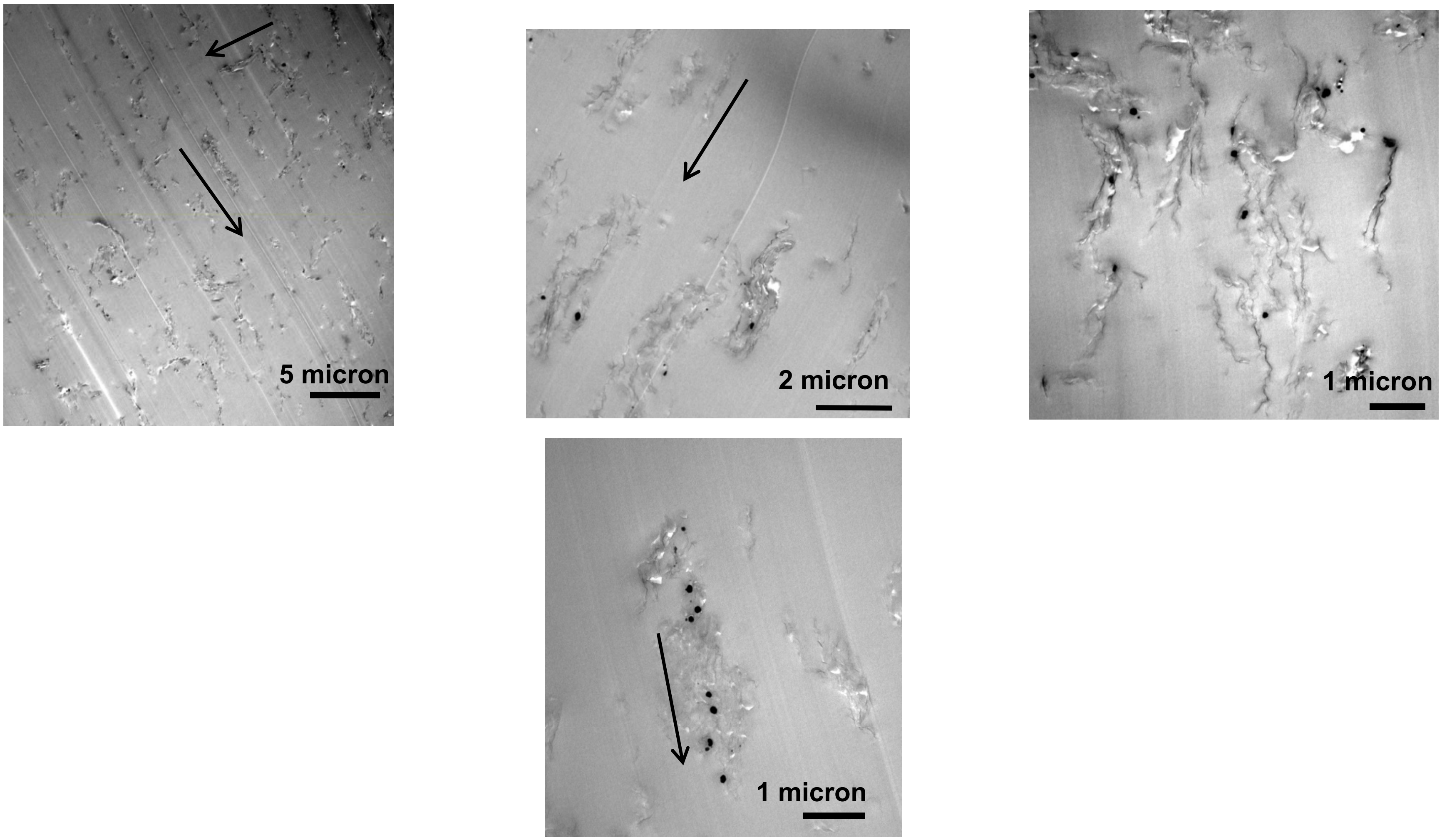






Transmission Electron Microscopy

1.77 wt% Ni-graphene polyimide
90% parallel and 5% perpendicular



Conclusions

-**Addition of graphene resulted in nanocomposites with high conductivity with a percolation as low as 0.036 vol.% and a maximum conductivity of 0.94 S/cm**

-**Dynamic moduli of the nanocompsoties increased with addition of graphene with no adverse effect on T_g or flexibility.**

-Magnetic graphene were synthesized enabled controlled orientation of graphene in magnetic fields.

-**Ni-graphene/PI nanocomposites were obtained which has e-2 S/cm *in-plane* conductivity and insulating in the *through-plane* direction.**

-**Ni-graphene/PI nanocomposites exhibited increased modulus with increasing orientation.**

-**The orientation was verified by magnetic characterization and TEM studies.**

Acknowledgements

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