National Aeronautics and Space Administration

Multifunctional Graphene Polyimide Nanocomposites

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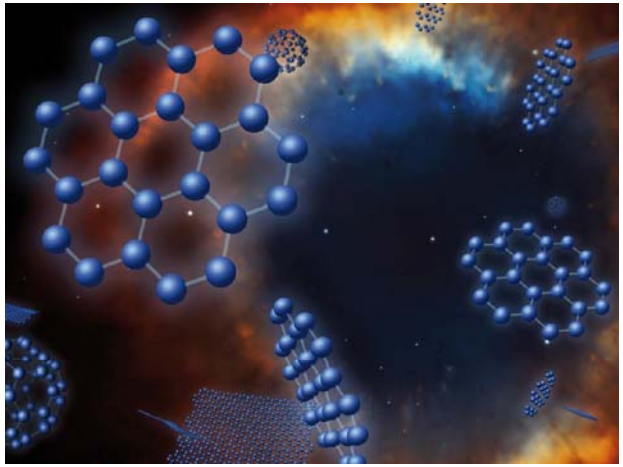
Ohio Aerospace Institute, Cleveland, OH
NASA Glenn Research Center, Cleveland, OH
ASRC, Cleveland, OH



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

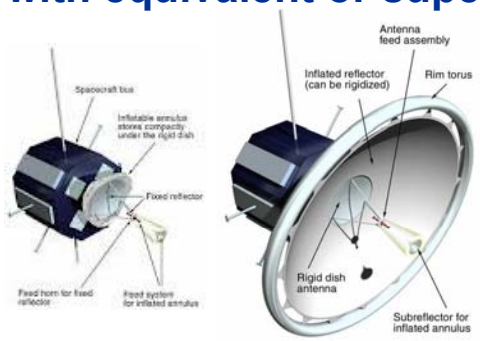
-Self Healing Materials

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

Reduced Vehicle Mass

Boeing 787 composite aircraft

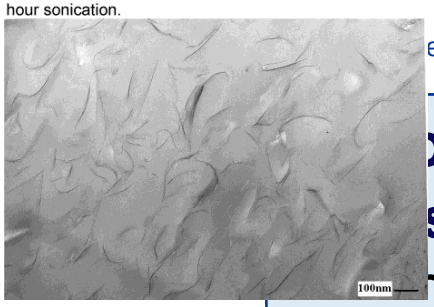
Copper mesh 4000 lb of weight



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.



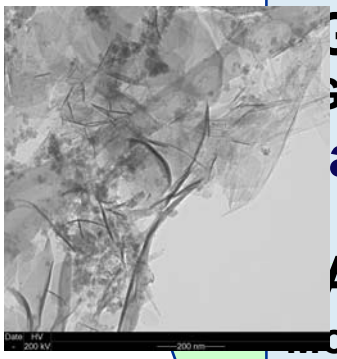
e Administration

oparticles:

s - Ijima 1991

~ 1315 m^2/g

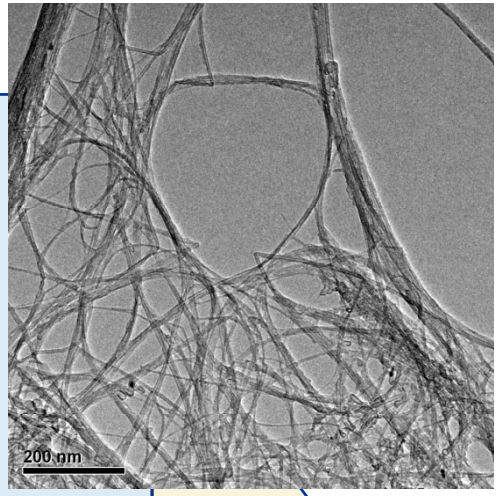
DCNT ~ 700-800

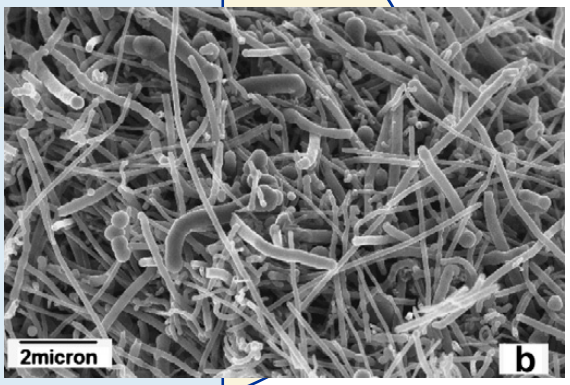


Graphite and Graphene – Giem 2004

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

arbon nanofibers

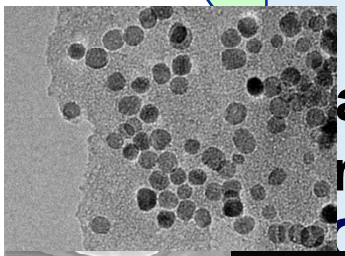




Alumina silicates – Fukushima, Toyota 1987

ontmorillonite~ 725 m^2/g

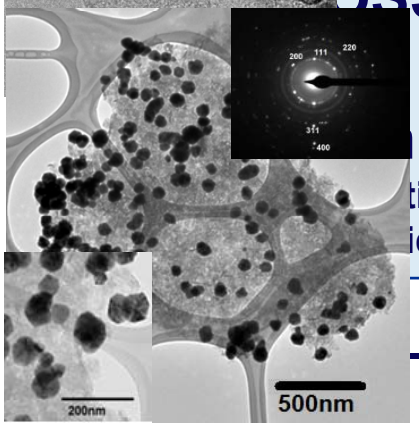
Magadiite , Laponite, Vermiculite



agnetic Nanoparticles

rganometallic physical crosslinkers

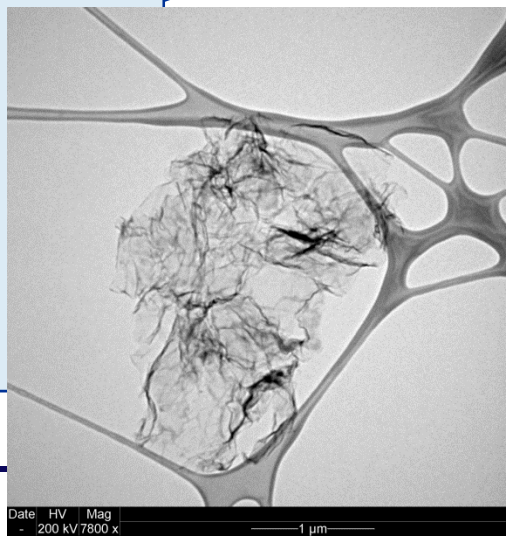
oss



posite Nanoparticles

tic graphene

ide graphene




pace Administration

Spacecraft launched from Space Shuttle

Polyimide
High Performance Polymer

PMR-15 - GRC
After burner

Satellite

Aromatic polyimide:

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

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

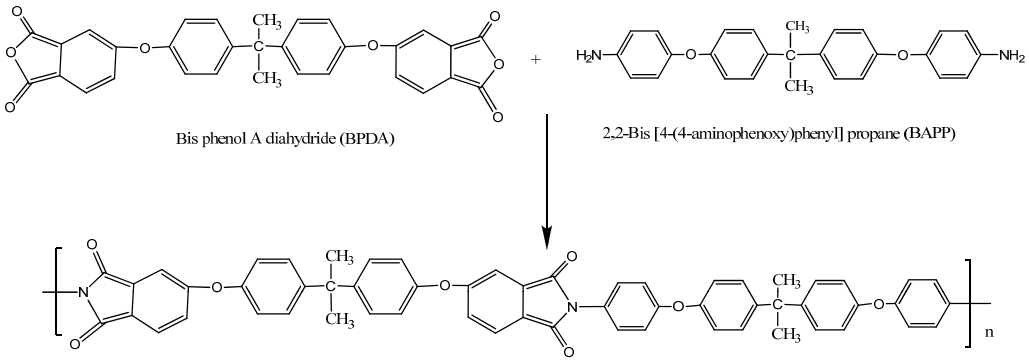
Continuous operating range between -65 °C to +357 °C

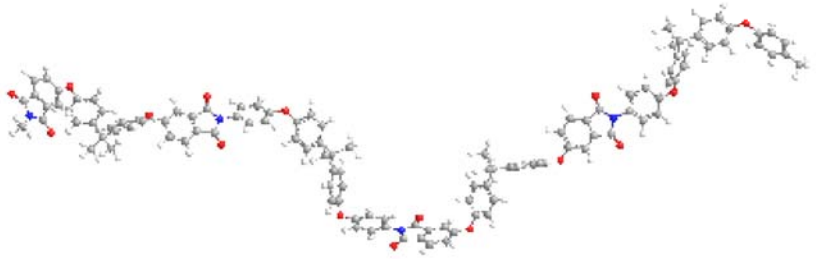
[1]Qu,L., Connell, J.W., Sun, Y.-P., Macromolecules, 2004, 37, 6055-6060.
[2]Lebron-Colon, M. Meador, M. A., Gaier, J. R., Sola, F., Scheiman, D.A., McCorkle, L.S. ACS Applied Materials and Interfaces, 2010, 2 ,3 , 669–676 .

Polyimide Graphene Nanocomposites

PMR-15
GRC
After burner

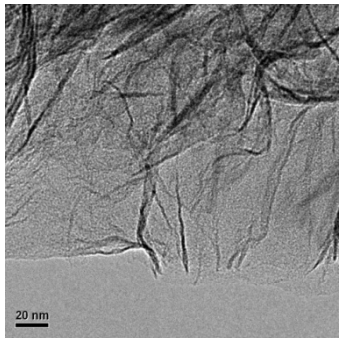
Polyimide, thermal stability >500 °C, T_g > 200 °C, flexible and semi-transparent.





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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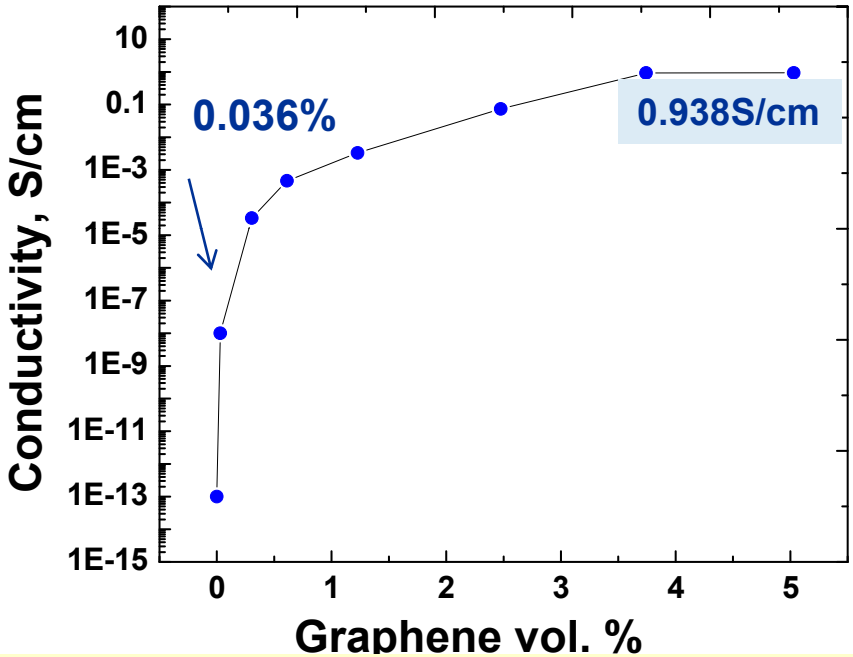
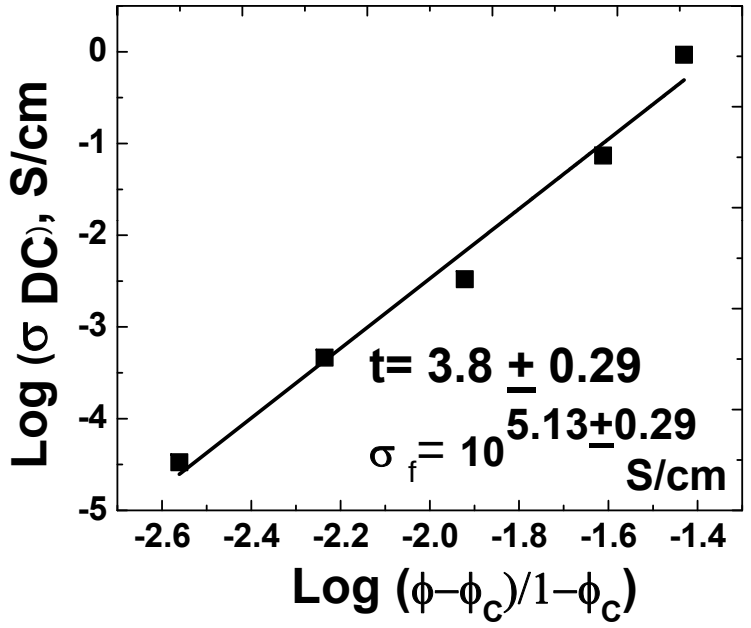
Polyimide Graphene Nanocomposites

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

CNT/nanocomposites
t = 1.2 – 2
CNF/polyimide
t ~ 3.1
PET graphene
t ~ 3.47±0.64
PS graphene
t ~ 2.74 ± 0.2

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

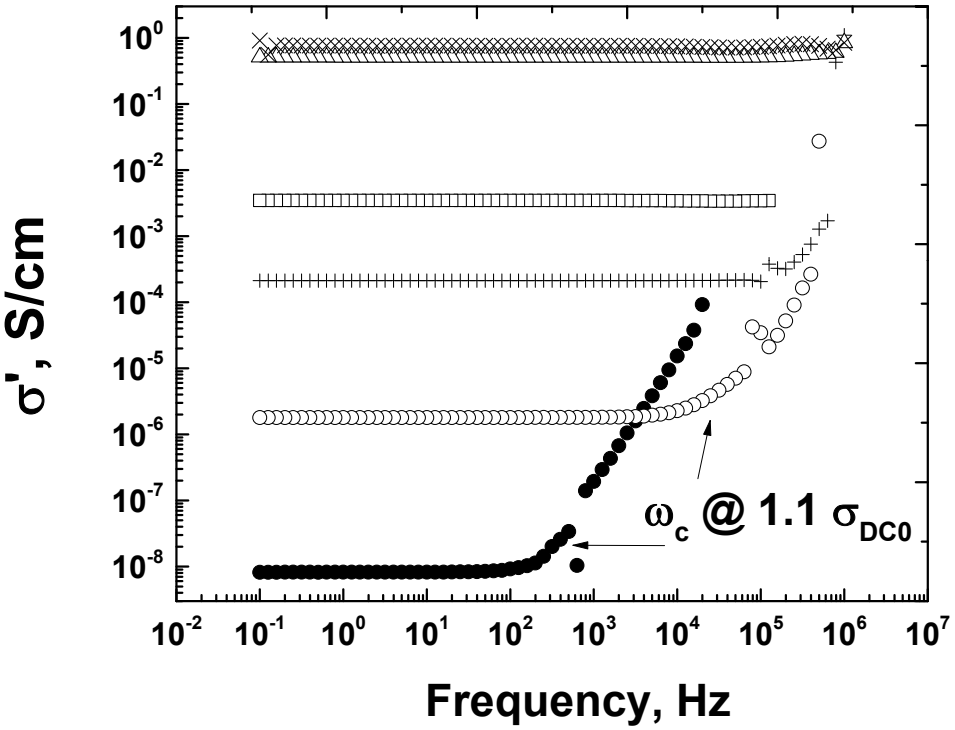
AC Electrical Performance

Broad band AC impedance spectroscopy

Extended pair approximation model

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

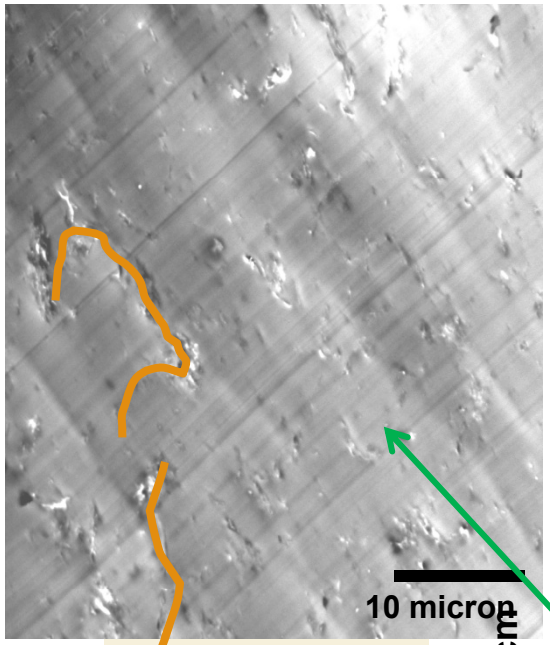
Vol.%	σ_{DC0}, S/cm	ω_c, Hz	s
0.03046	8.21e-9	150.47	0.499
0.3051	1.879e-6	7.027e3	0.647
0.6115	2.11e-4	1.241e5	0.446



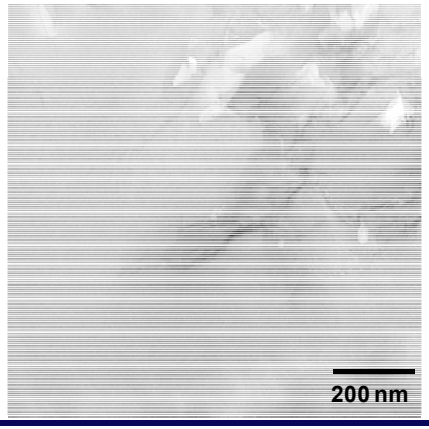
S ~ 0.99 -> hopping
S ~ 0.72 -> 3D material
S ~ 0.58 -> anomalous diffusion in fractal cluster exist

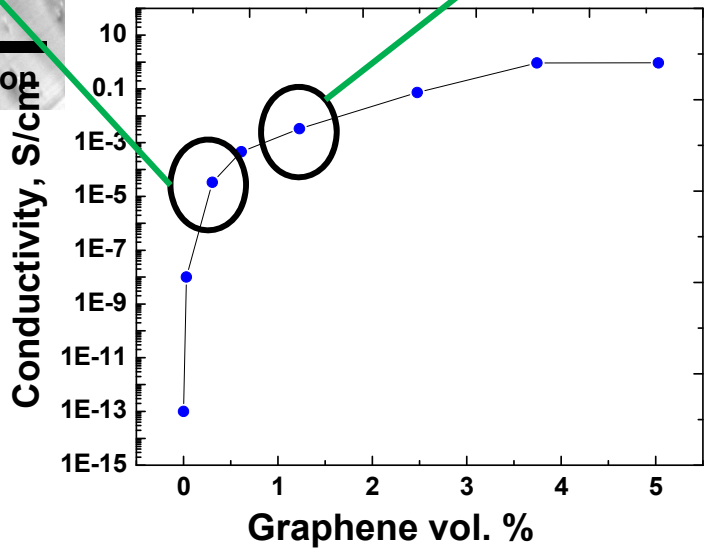
Linares, A., Ezquerra, T.A et al., Macromolecules, 2008, 41, 7090

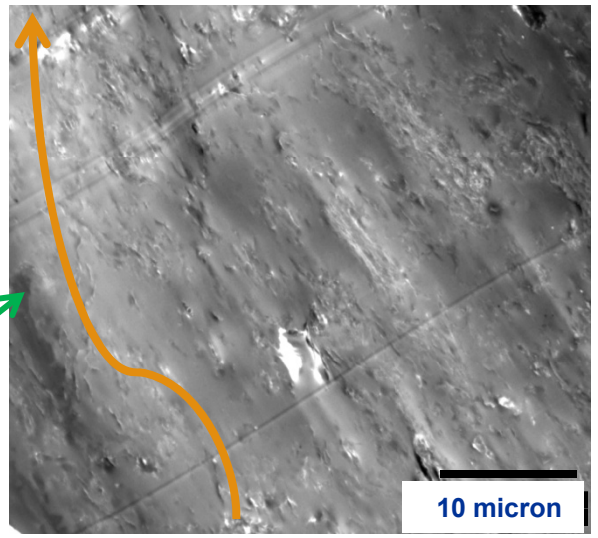
Dispersion of graphene in polyimide TEM



0.25 vol.%

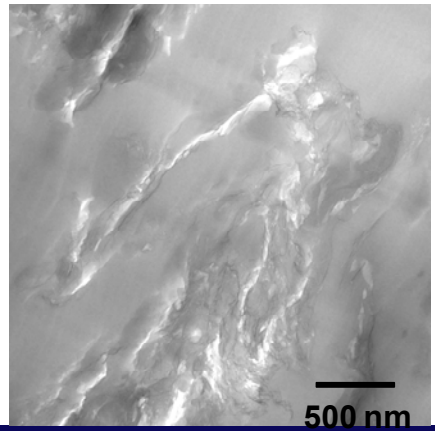




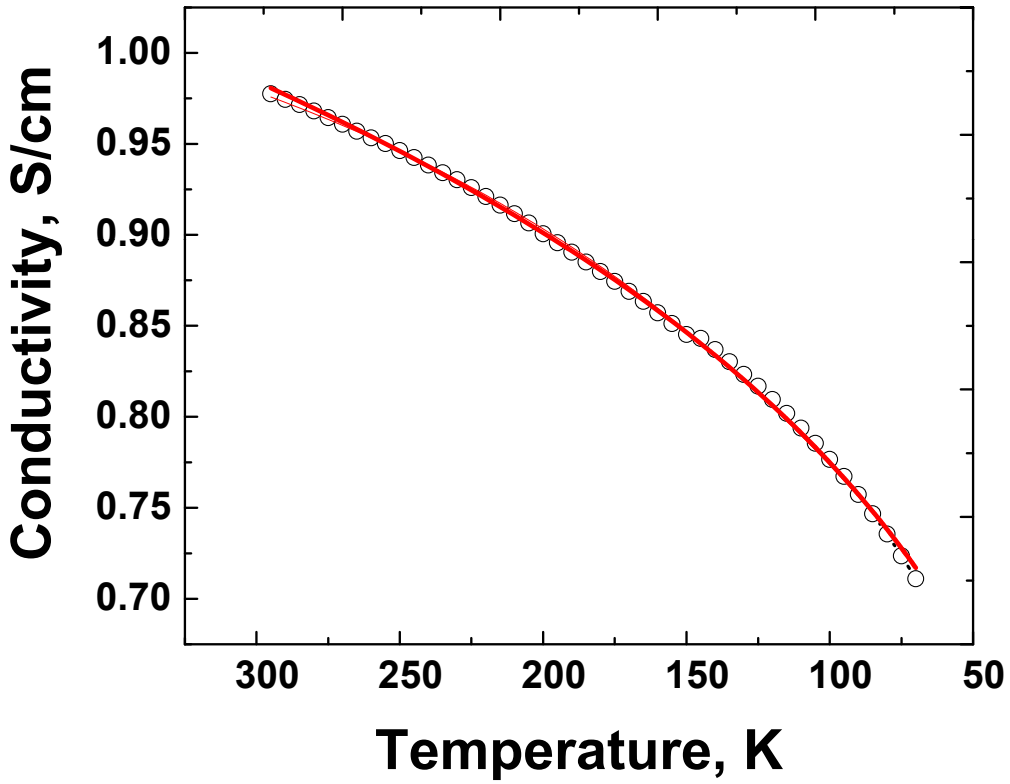


Conductive path

1.1 vol.%



Temperature Dependence Conductivity



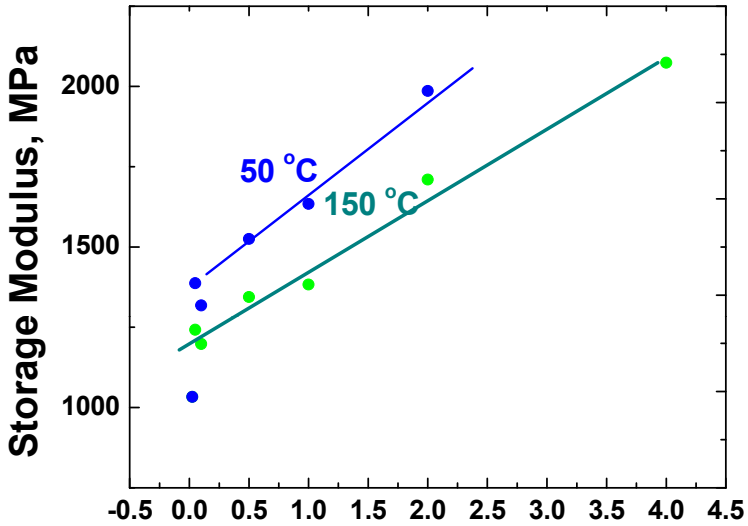
$$\sigma = 0.2844T^{0.2177}$$

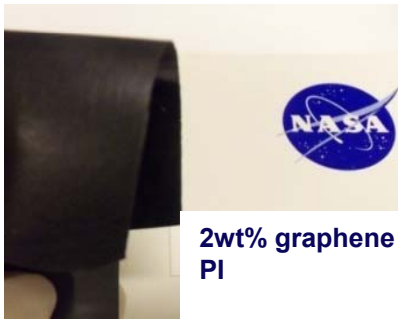
$$T = 322.404\sigma^{4.6}$$

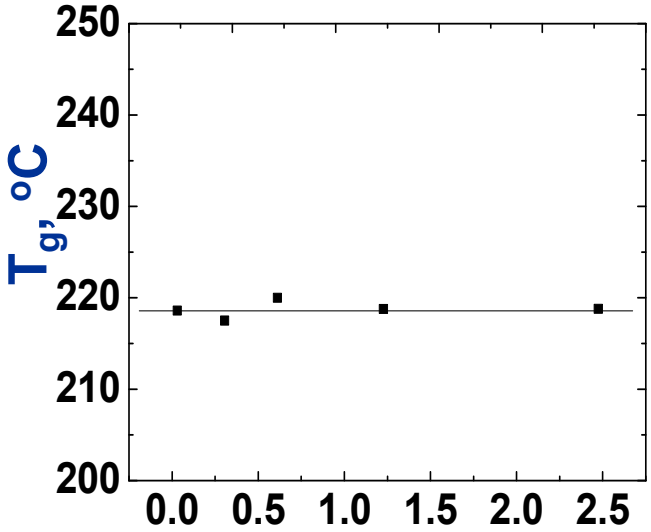
5 vol. % graphene polyimide

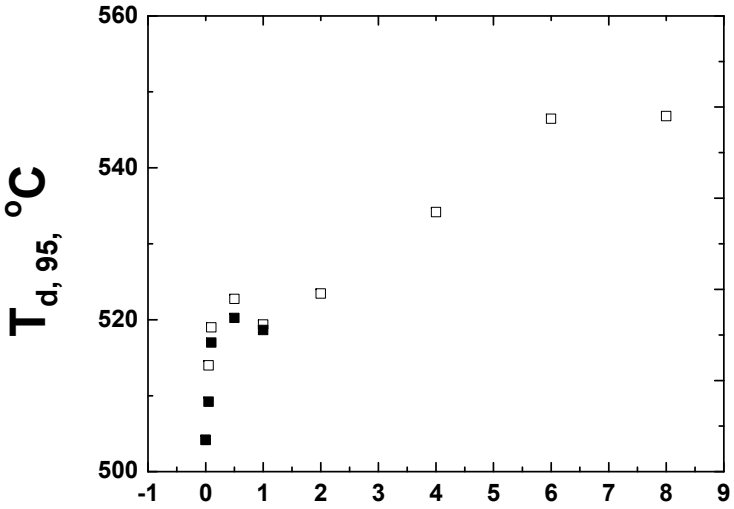
Thermal and Mechanical Properties

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







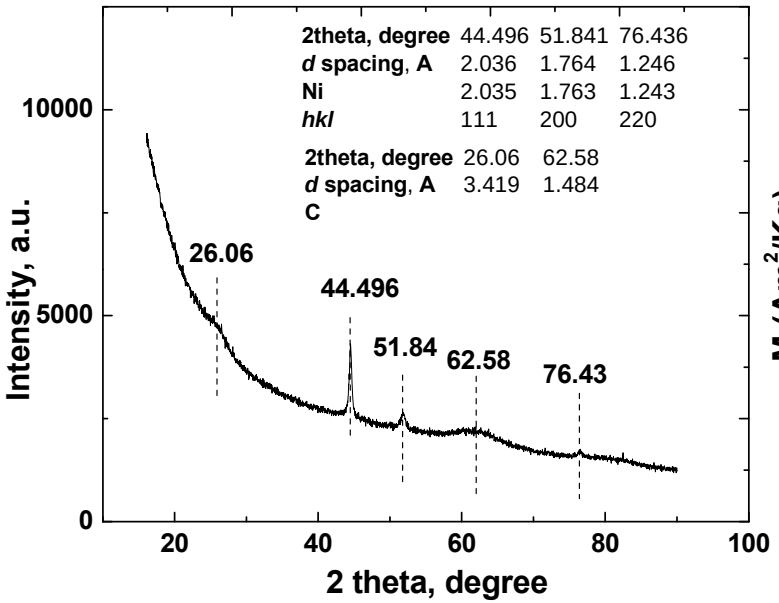


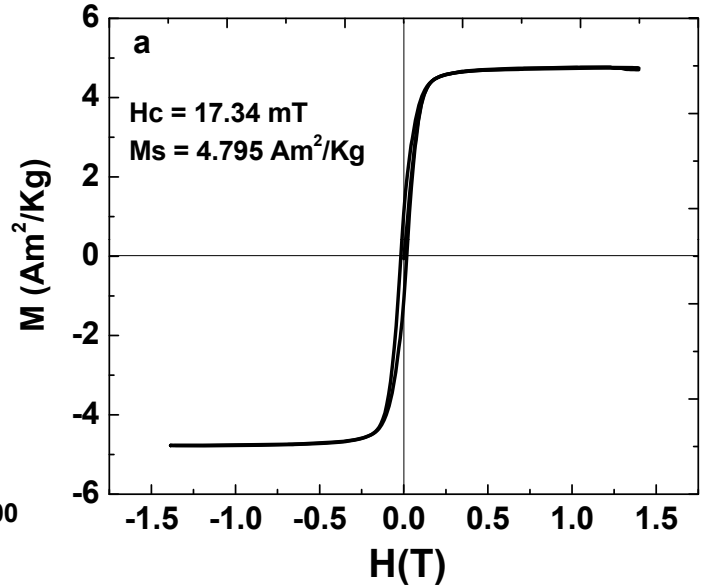
Controlled Property Direction

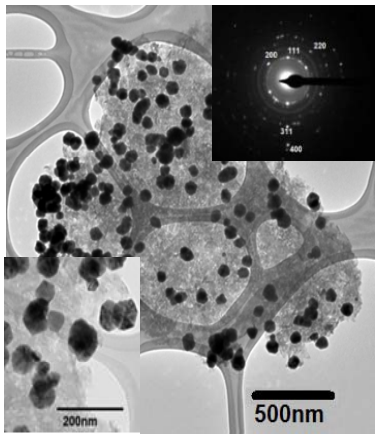
Ni-Tethered Graphene

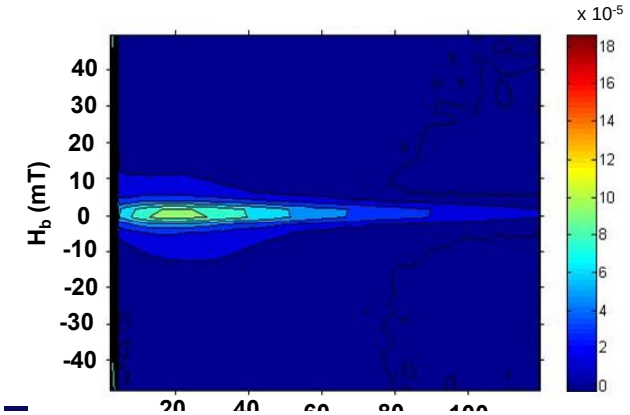
Composites Nanoparticles

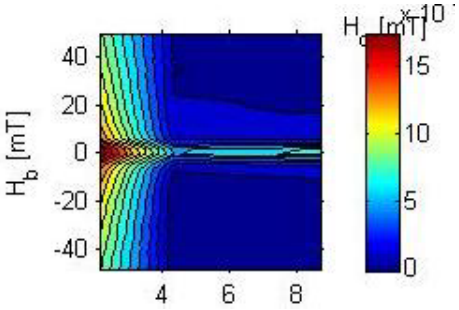
Thermal decomposition of $Ni(acac)_2$ in the presence of O-graphene

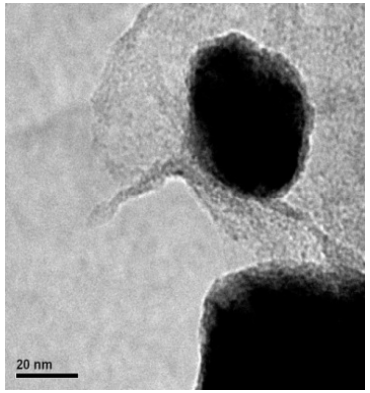






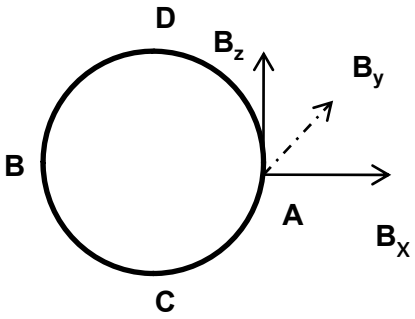




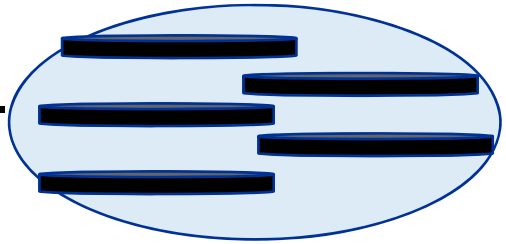


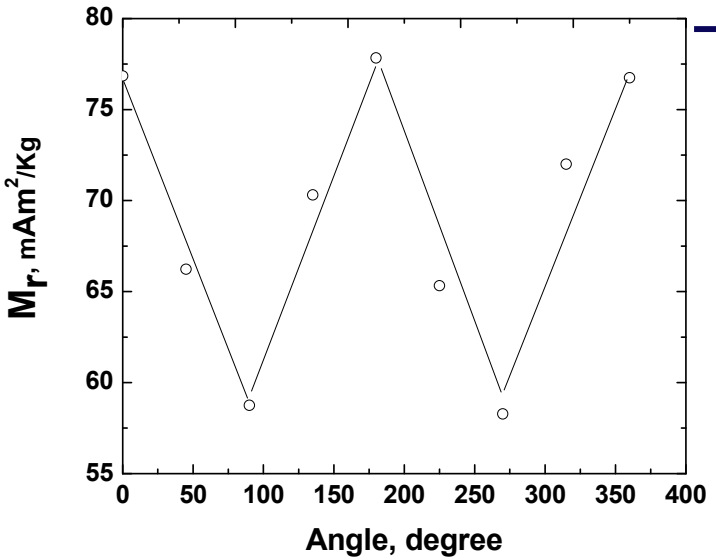
First-order reversal curve (FORC)

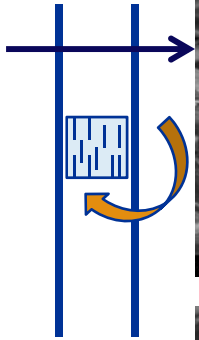
Controlled Directionality

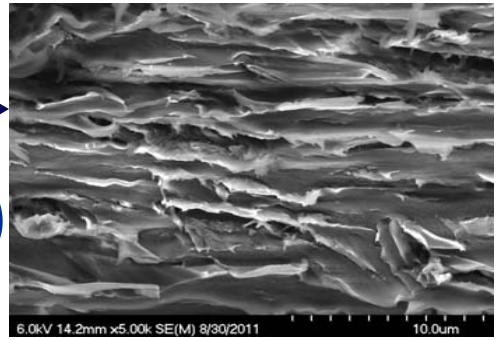


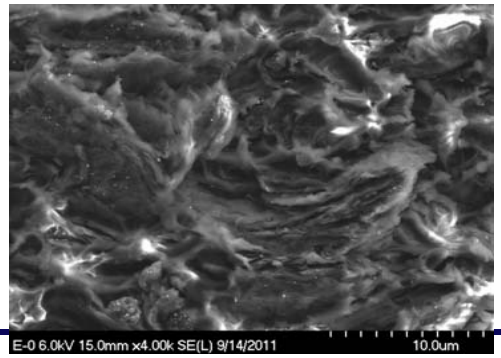
	B (Gauss)	B_x (Gauss)	B_y (Gauss)	B_z (Gauss)
A	1150	-1150	-237	-50
B	976	-948	475	50
C	440	-432	-55	-120
D	500	-520	-12	42.3

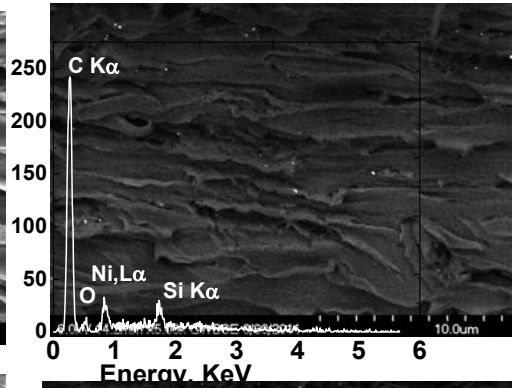


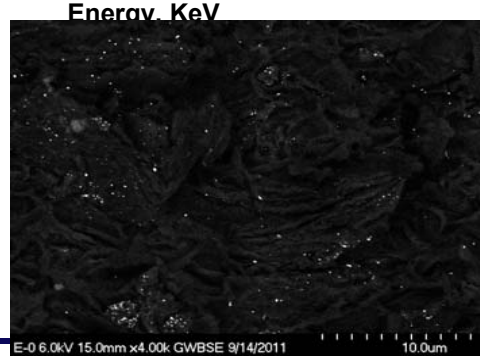








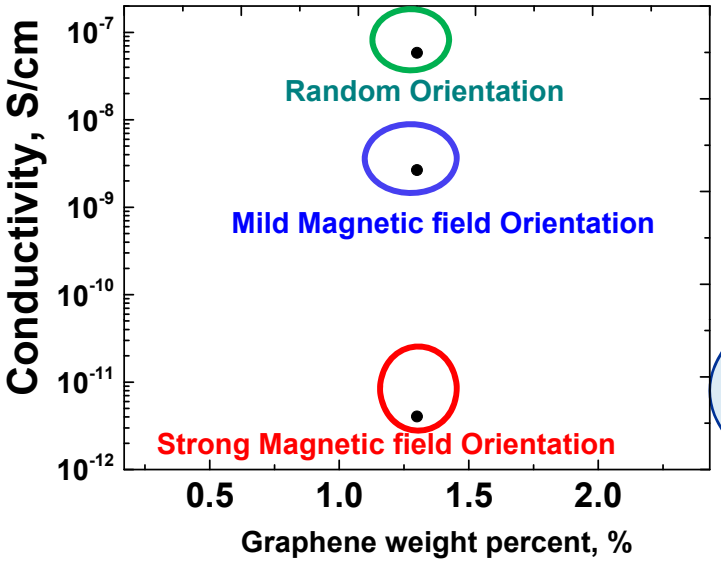


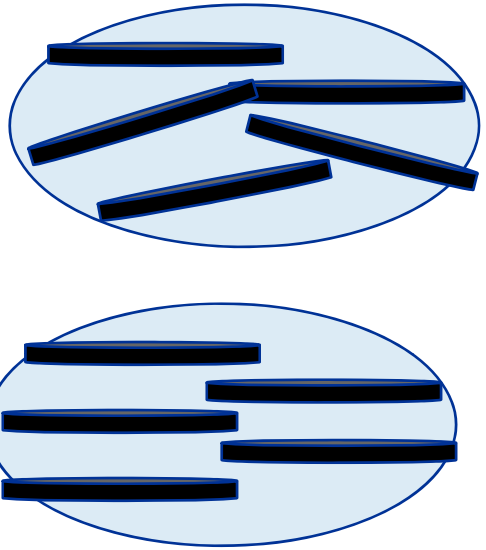


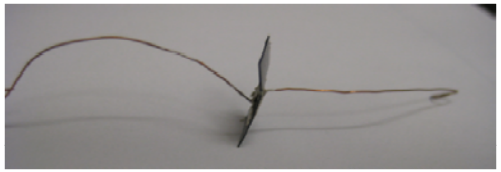
2.8 wt% Ni-Graphene polyimide nanocomposite

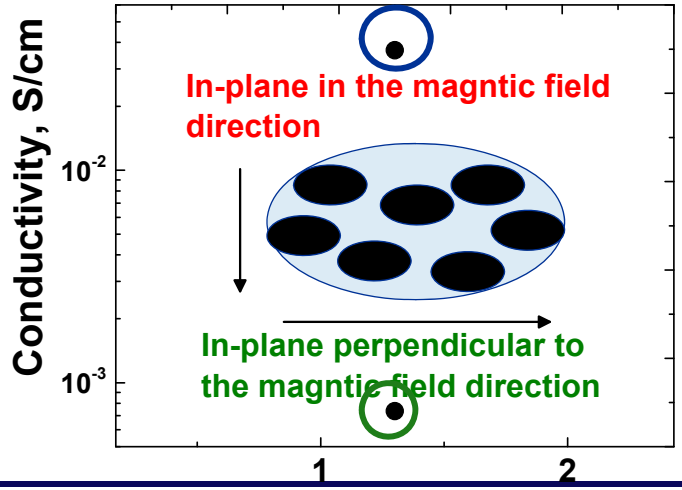
Anisotropic Properties

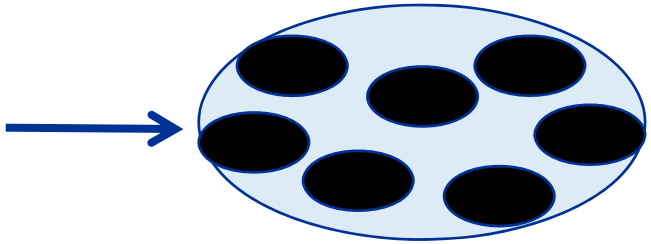
Electrical properties









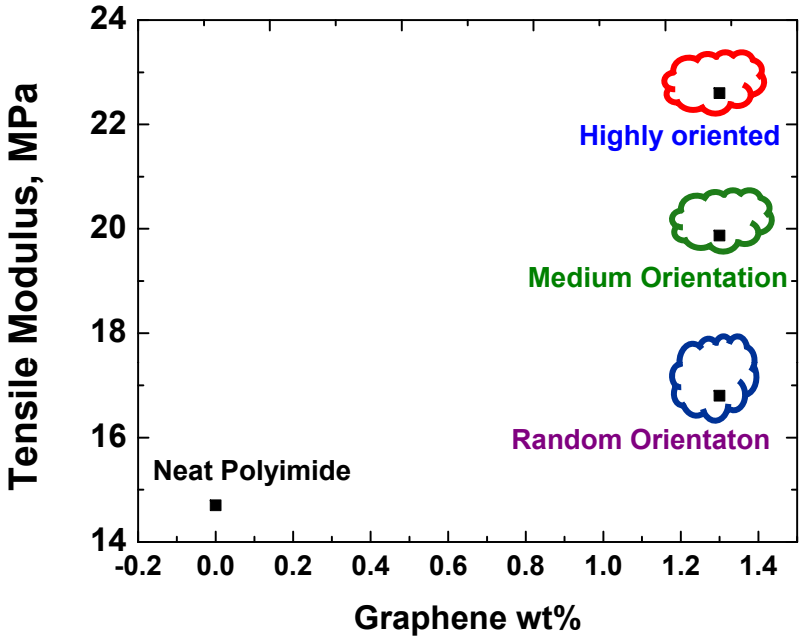


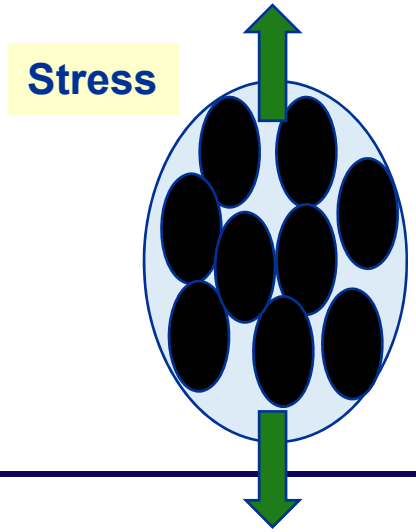
In-plane

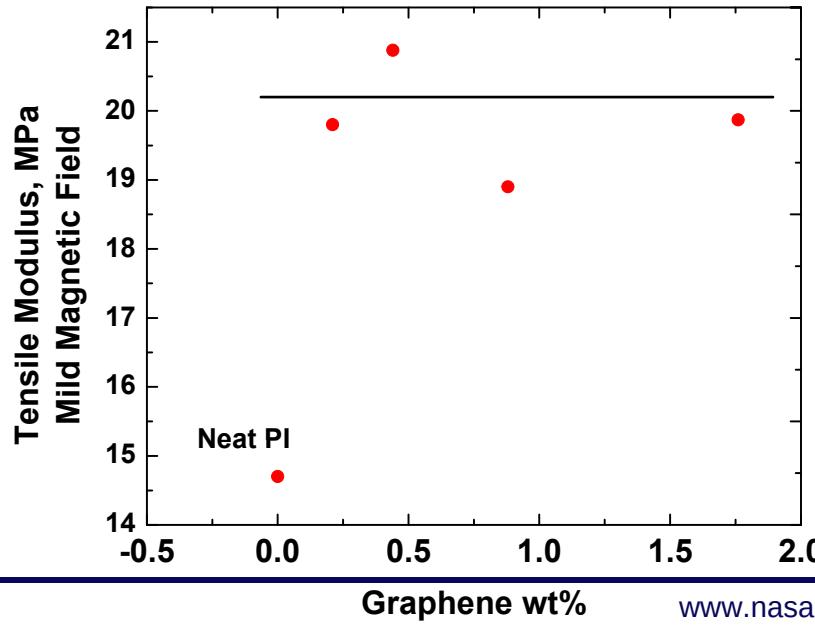
Magnetic field

Anisotropic Properties

Mechanical properties

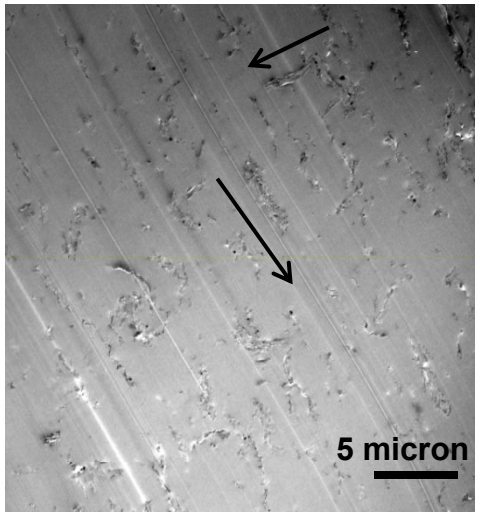


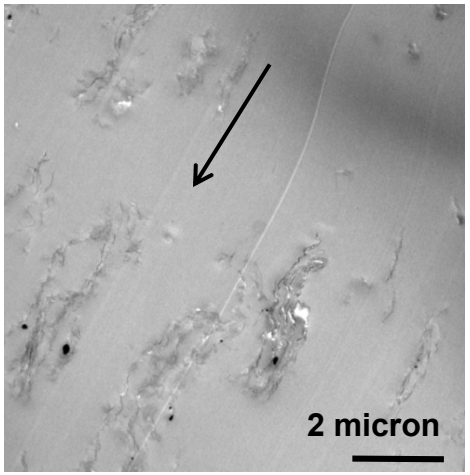


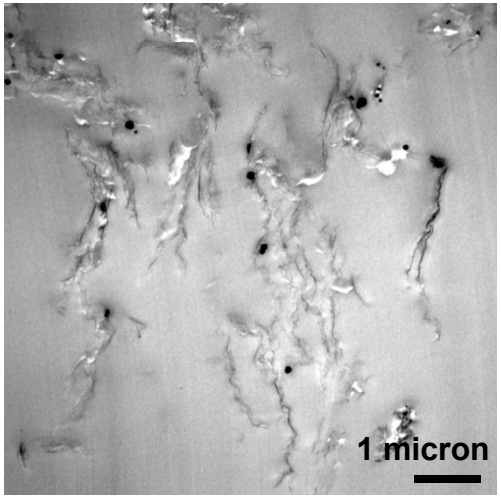


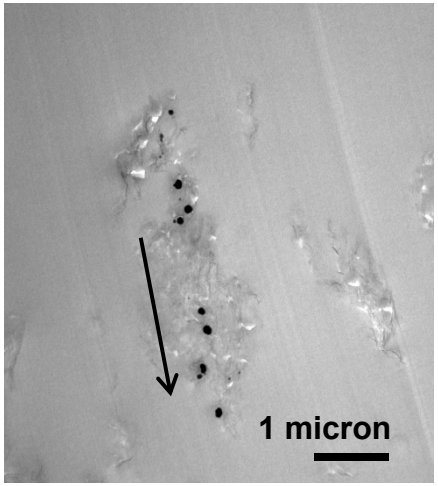
Transmission Electron Microscopy

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.

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