GRAPHENE MALAYSIA 2016
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Graphene International Conference

Graphene Reinforced Polymer Nanocomposites: Recent Development and Opportunities

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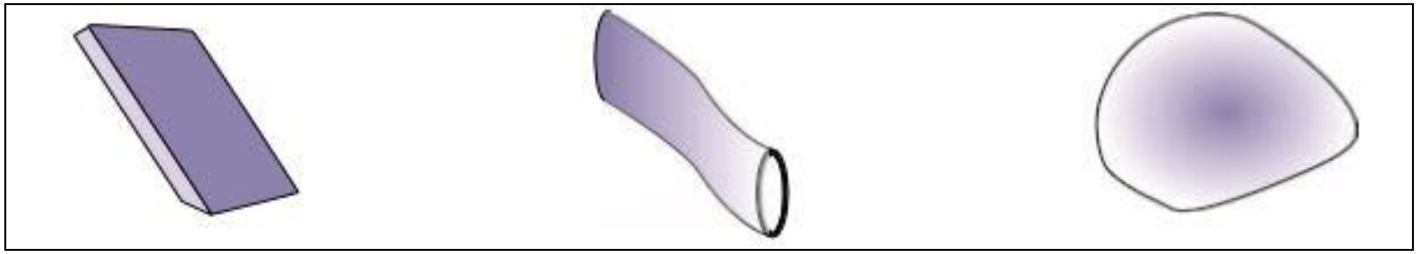
Nanocomposites

- The increased interest in nanocomposites started with the development of layered silicate polyamide 6 nanocomposites at Toyota.
- A nanocomposite is a multiphase solid material where one of the phases has one, two or three dimensions of less than **100 nm**.
- The nanoscale distribution of such high aspect ratio fillers brings improvements to the polymer matrix in terms of:
  - ✓ Mechanical properties (stiffness and strength)
  - ✓ Flame retardancy
  - ✓ Gas barrier

Types of Nanofillers

1. Carbon nanotubes
2. Metallic nanoparticles
3. Nanoclays
4. Graphene



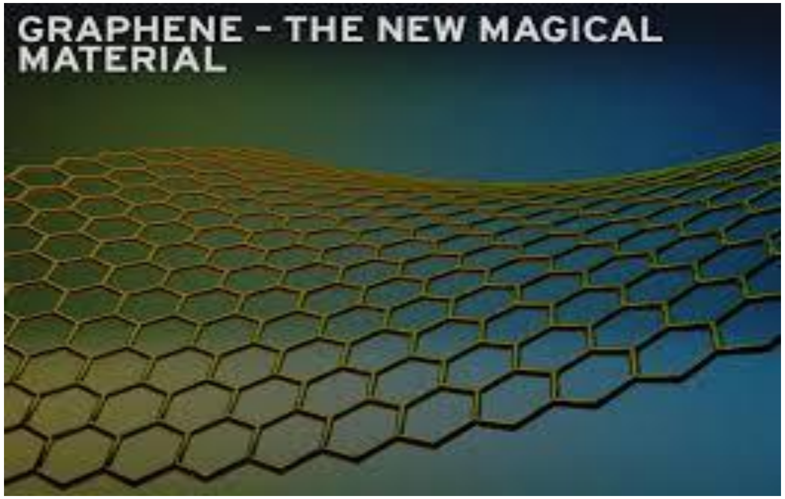
Thickness < 100nm

Diameter < 100nm

All dimensions < 100nm

Discovery of graphene

- Studies on graphite layers for past hundred years
- Discovered by Andre Geim & Kontantin Novoselov in 2004 and won Nobel Prize in 2010.
- Media refer to graphene as the miracle material of the 21st Century



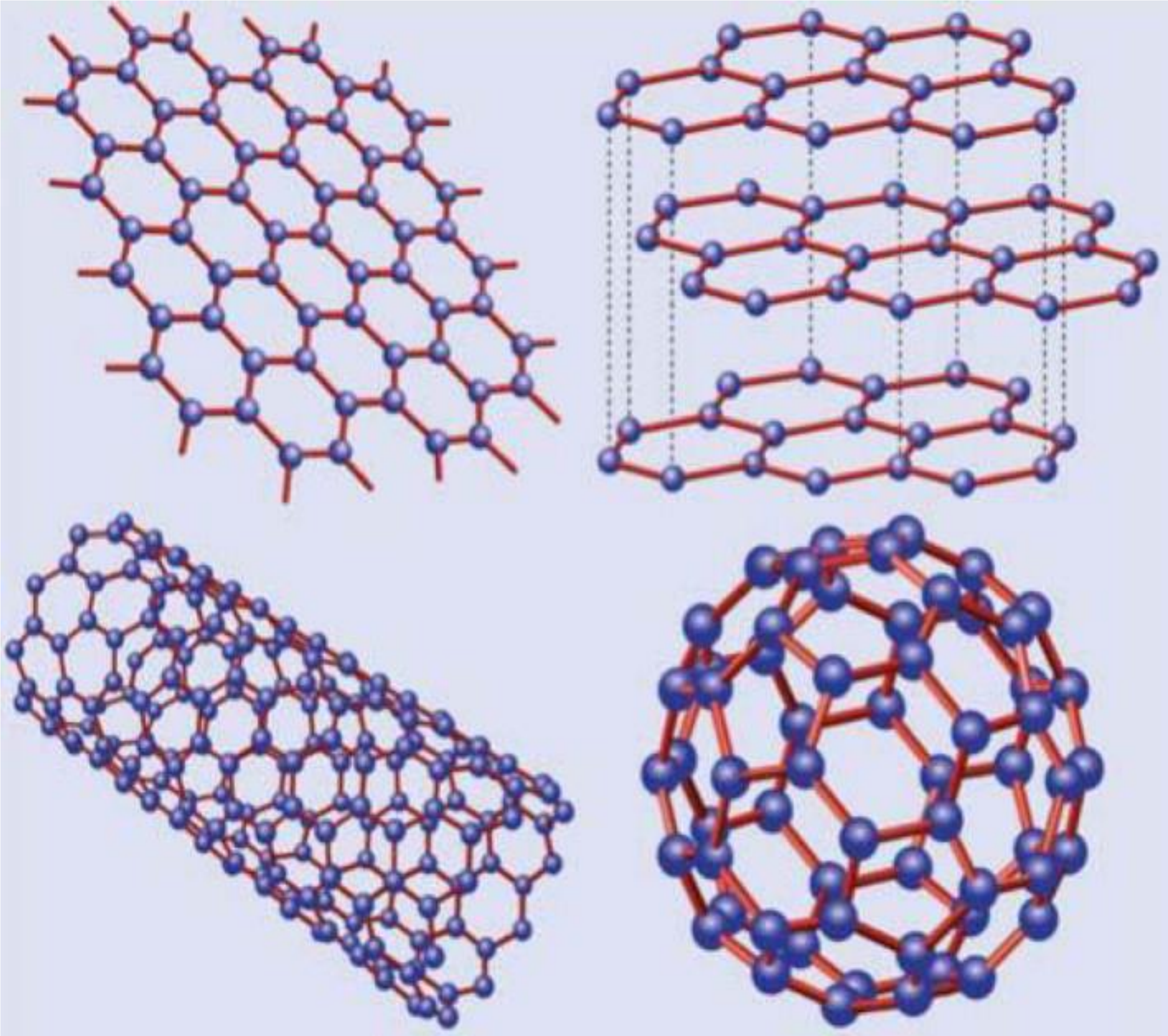
http://www.telegraph.co.uk/science/science-news/8043355/Nobel-Prize-for-Physics-won-by-Andre-Geim-and-Konstantin-Novoselov.html

Graphene (top left) is a honeycomb lattice of carbon atoms.

Graphite (top right) stack of graphene layers.

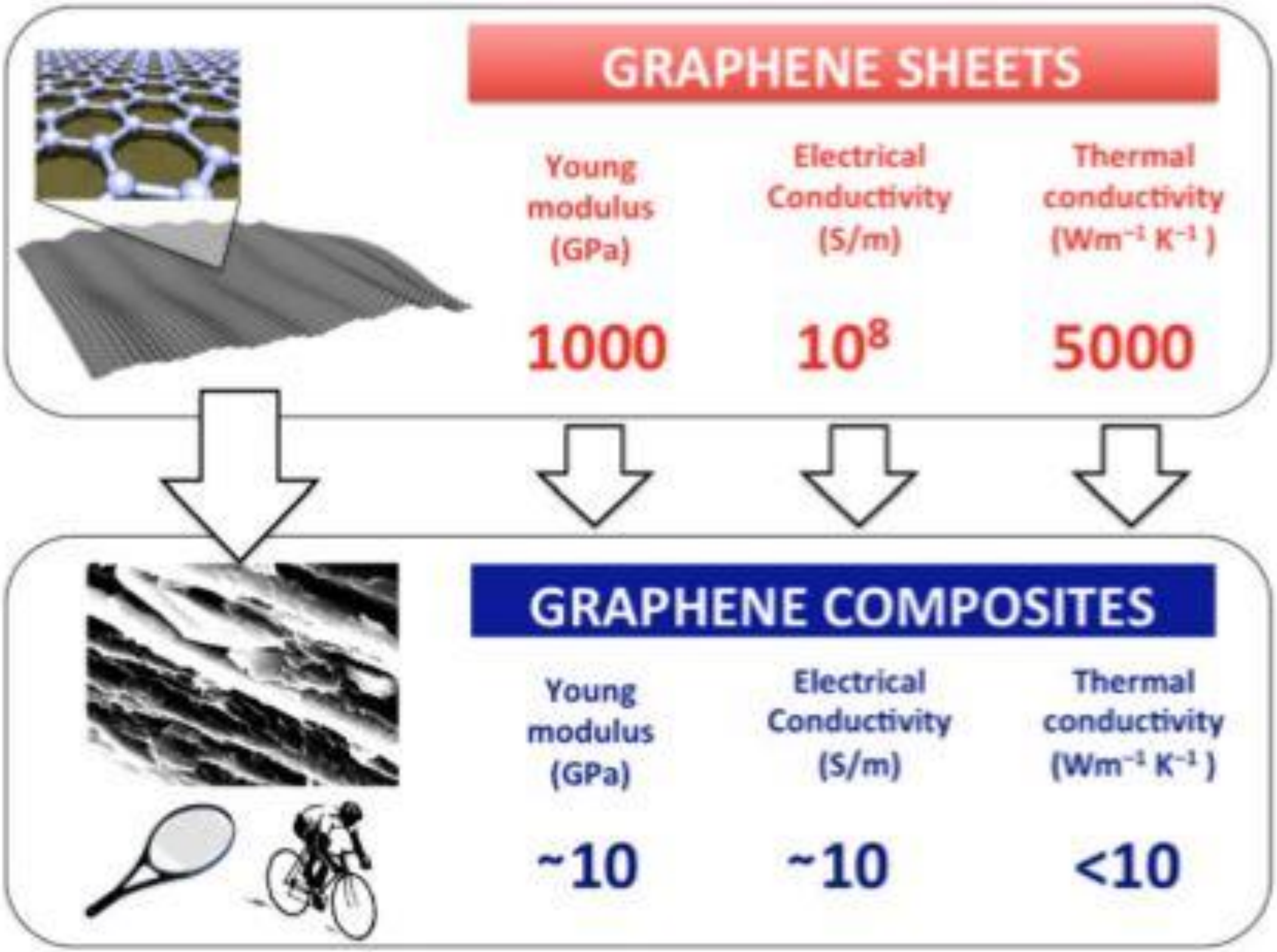
Carbon nanotubes rolled-up cylinders of graphene (bottom left).

Fullerenes (C60) molecules consisting of wrapped graphene



Comparison of Nano-reinforcement Properties

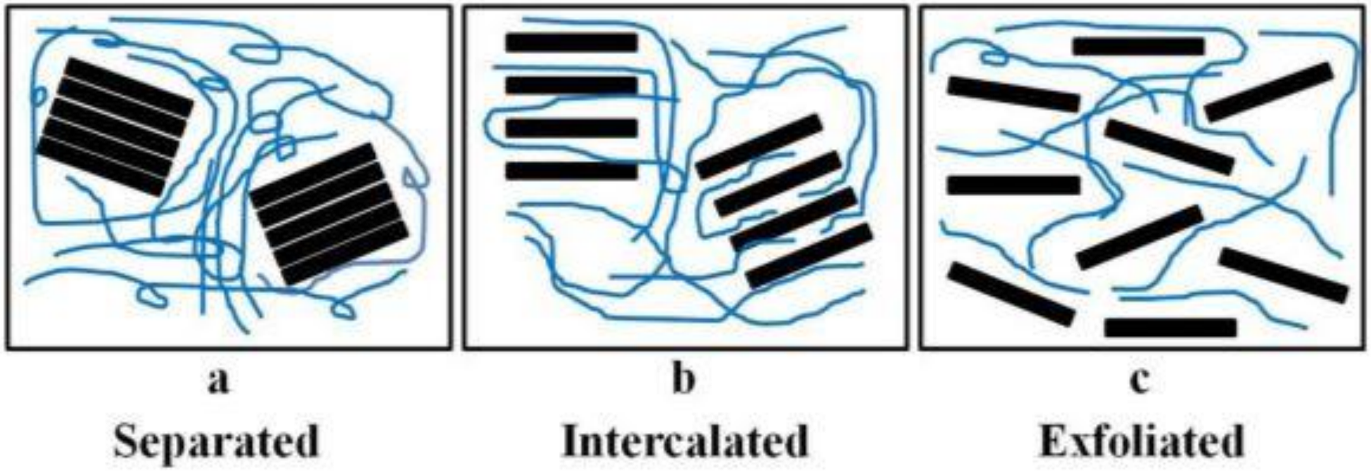
	Exfoliated Clay	Carbon Nanotube	BN Nanotubes	Cellulose Nanowhisker	xGnP-Graphite NanoPlateles
PHYSICAL STRUCTURE	Platelet ~ 1nm X 100nm	Cylinder ~1nm X 100nm	Layer	Needle-Whisker	Platelet ~ 1nm X 100nm
CHEMICAL STRUCTURE	SiO_2 , Al_2O_3 , MgO, K_2O , Fe_2O_3	Graphene (chair,zigzag, chiral)	Boron Nitride	Cellulose	Graphene
INTERACTIONS	Hydrogen bond Dipole-Dipole	Pie–pie	Hydrogen Bond	Hydrogen Bond	Pie-pie
TENSILE MODULUS	0.17 TPa	1.0-1.7 TPa	~ 1 TPa	~ 130 GPa	~ 1.0 TPa
TENSILE STRENGTH	~ 1 GPa	180 GPa	?	10 GPa	~(10 – 20 GPa)
ELECTRICAL RESISTIVITY	10^{10} -10^{16} Ω cm	50 X 10^{-6} Ω cm	Insulator	10^{10} – 10^{16} Ω cm	~ 50 X 10^{-6} Ω cm
THERMAL CONDUCTIVITY	6.7 X 10^{-1} W/m K	3000 W/m K	~3000 W/m K	Insulator	3000 W/m K
DENSITY	2.8 – 3.0 g/cm^3	1.2 – 1.4 g/cm^3	~ 2.0 g/cm^3	1.5 g/cm^3	~ 2.0 g/cm^3


GRAPHENE SHEETS
Young modulus (GPa)
Electrical Conductivity (S/m)
Thermal conductivity ($Wm^{-1} K^{-1}$)
1000
10^8
5000
GRAPHENE COMPOSITES
Young modulus (GPa)
Electrical Conductivity (S/m)
Thermal conductivity ($Wm^{-1} K^{-1}$)
~10
~10
<10

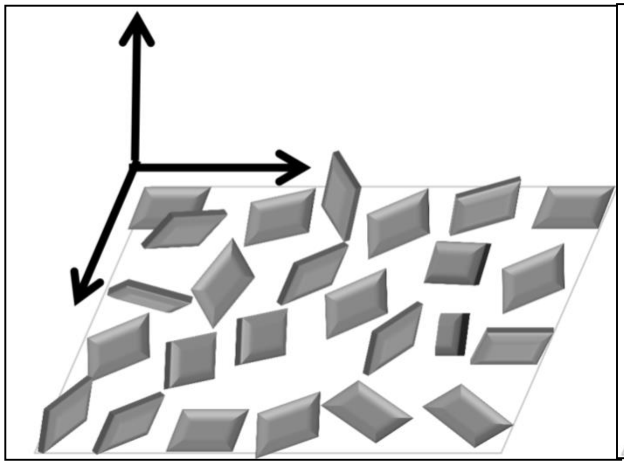
Properties of Nanomposites are dependent on:

- Constituent phases
  - Reinforcement
  - Matrix
- Relative amounts
- Geometry of reinforcement
- Interface properties
- Processing Methods
- Uniform Dispersion.
- Orientation of Reinforcement

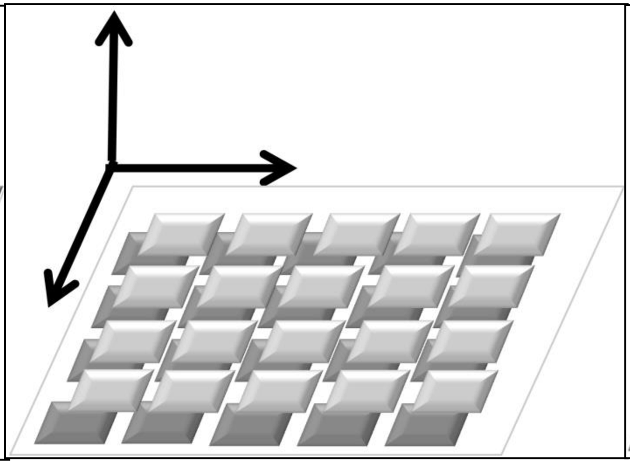
Distribution of Nanofillers



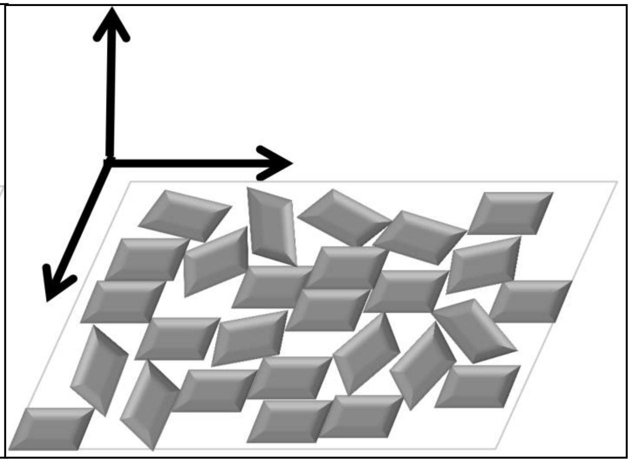
Graphene orientation



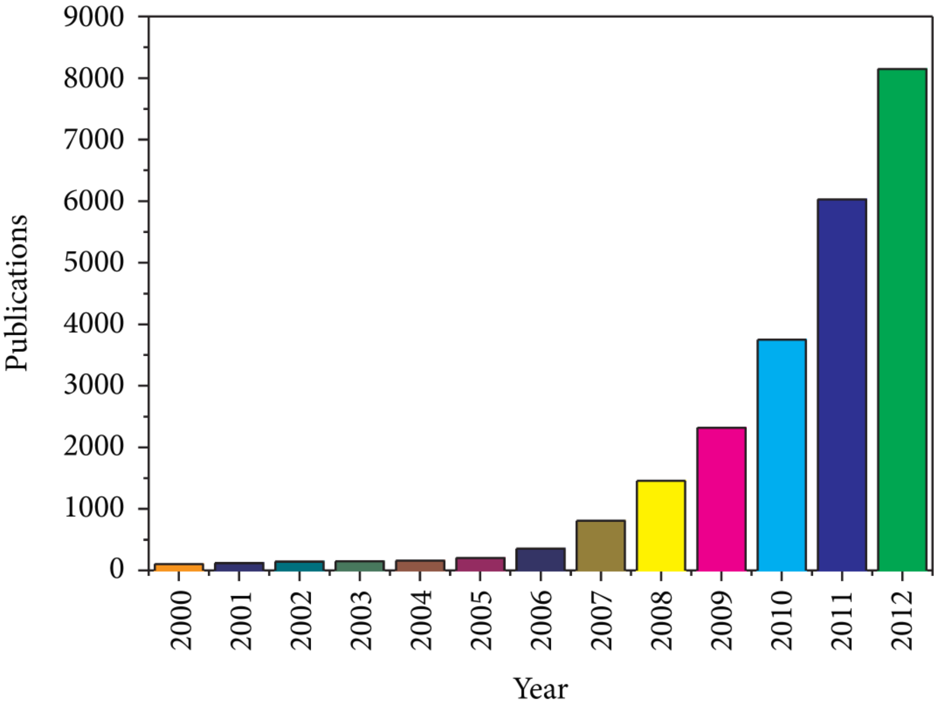
Random orientation



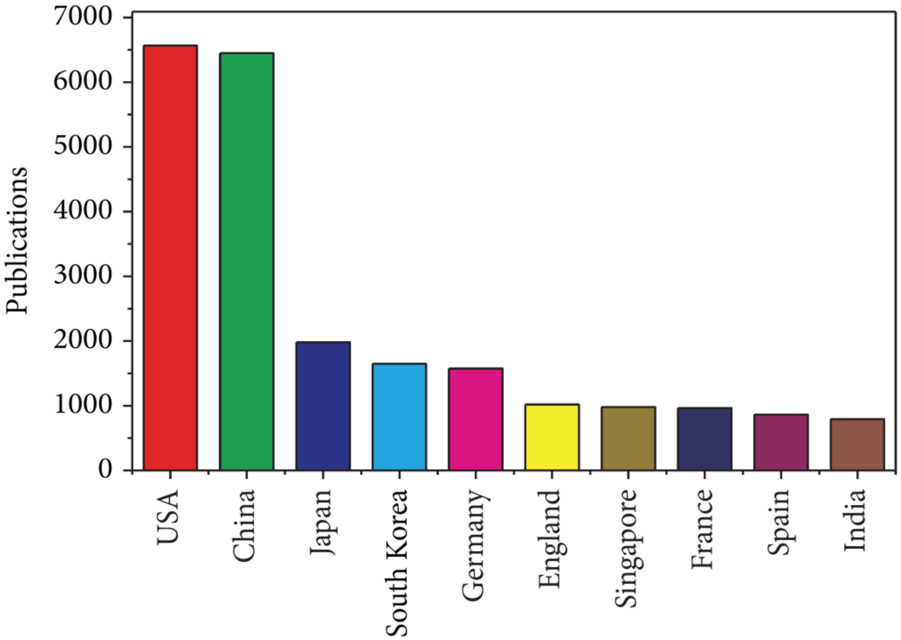
Aligned orientation



Interconnected orientation



Publication trend in graphene chronology since 2000–2012.

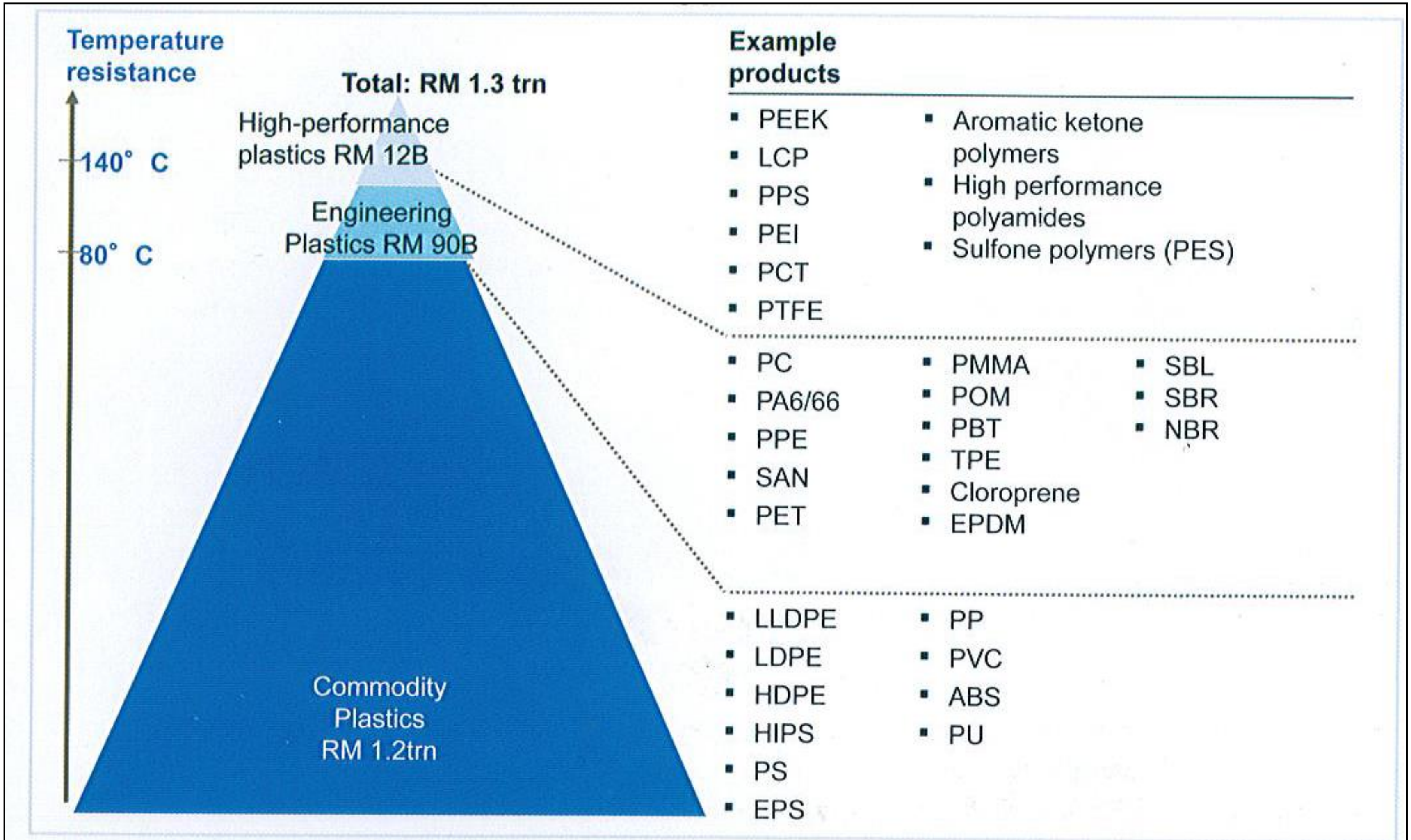


Country wise publications in graphene since 2000–2012.

Speciality of Plastics

- Low density
- Ease of processability
- **Easily modified through blends and incorporation of additives**

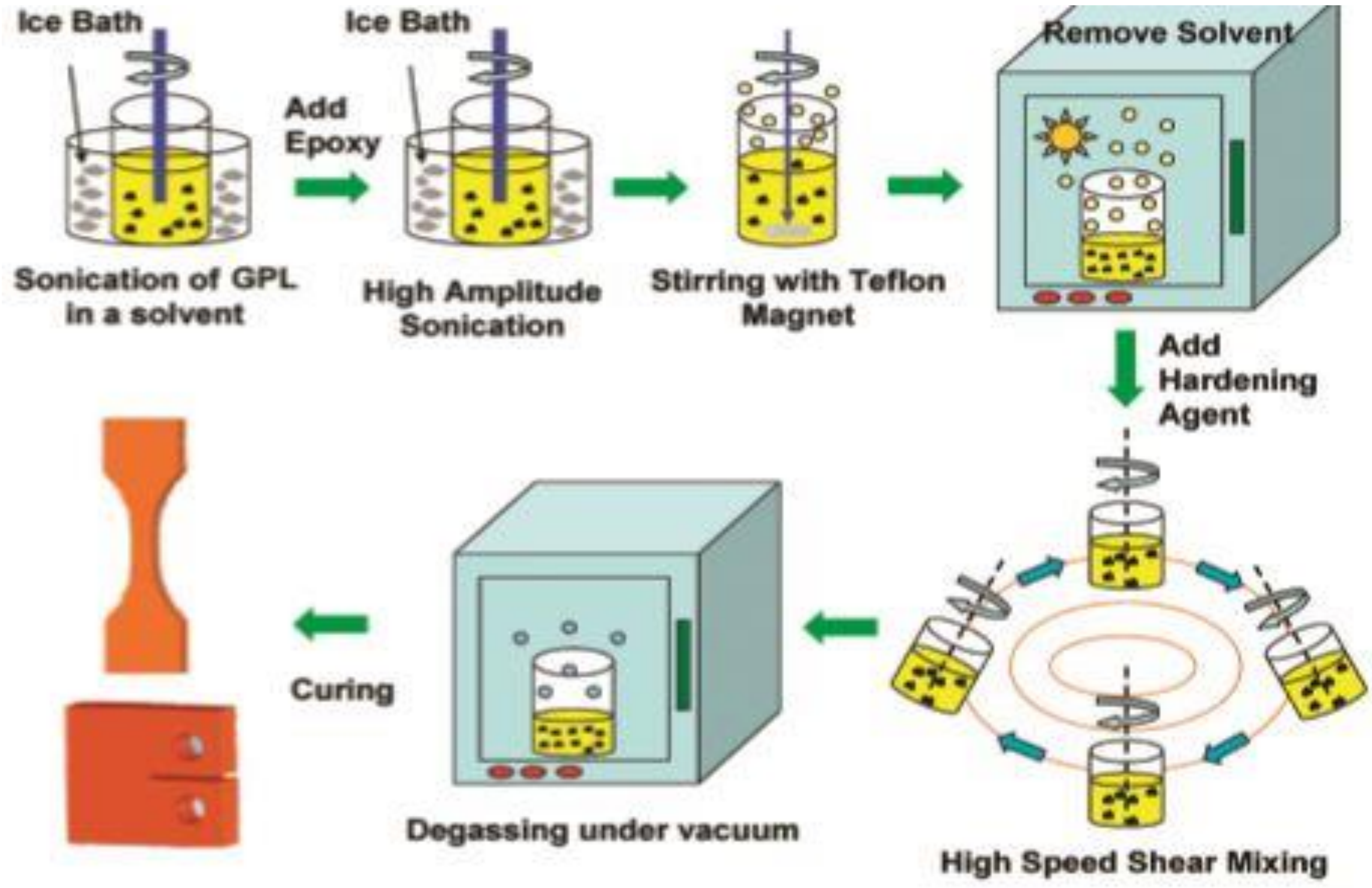
Types of Plastics: High Performance, Engineering and Commodity



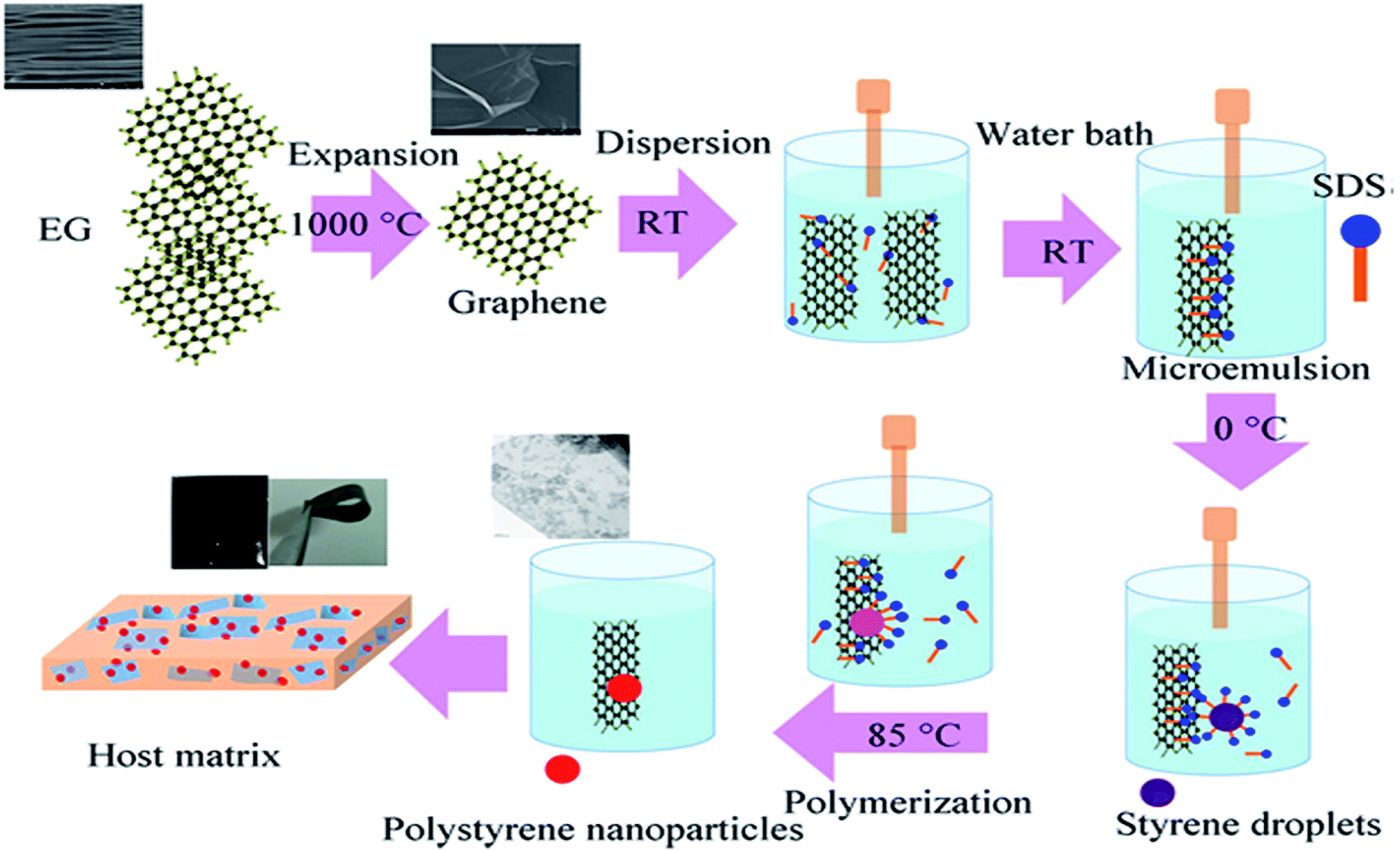
1 Marketing & Sales
SOURCE: SRI

Methods to Prepare Graphene Reinforced NanoComposites

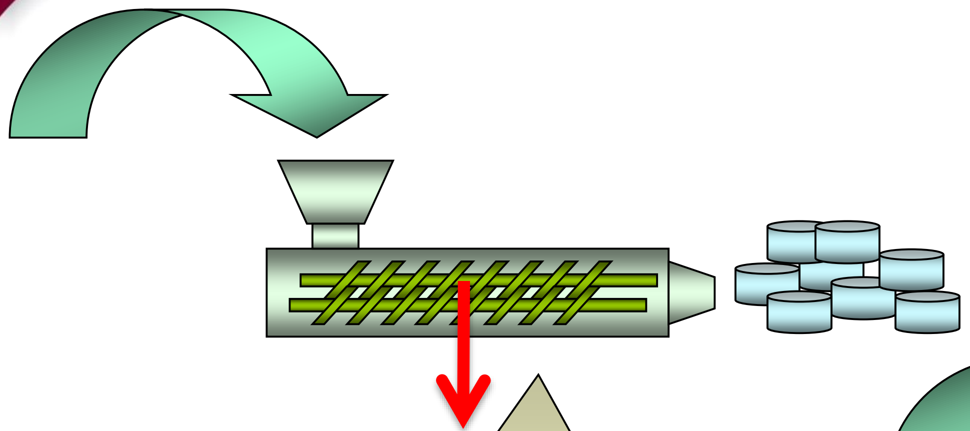
Solution blending



Insitu Polymerization

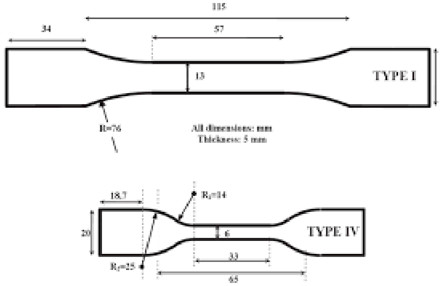


Melt Blending



Melt-blending in twin screw extruder

Injection molding of test specimens



Studies of Graphene Reinforced Polymer Nanocomposites

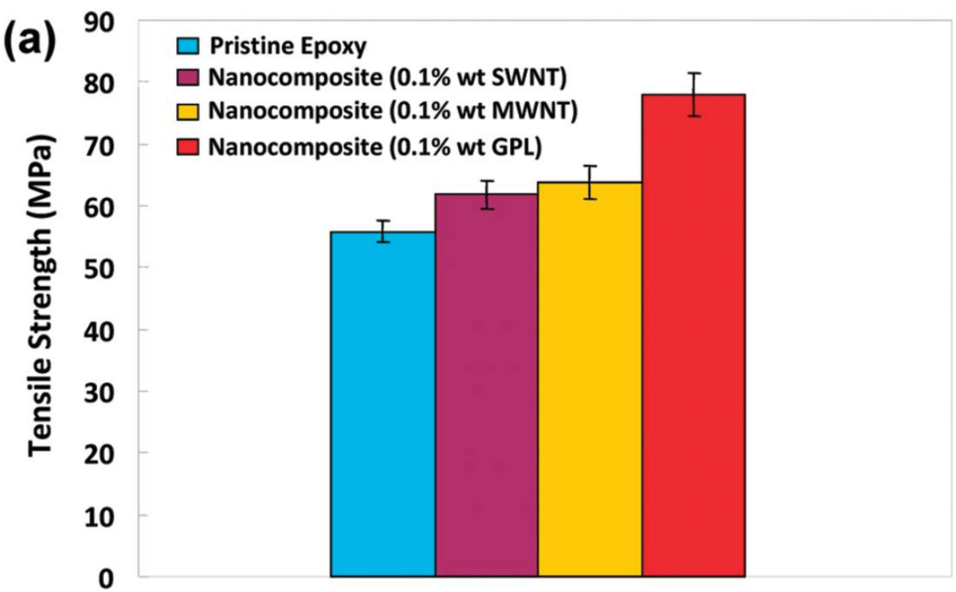
UNIVERSITI TEKNOLOGI MALAYSIA

ARTICLE

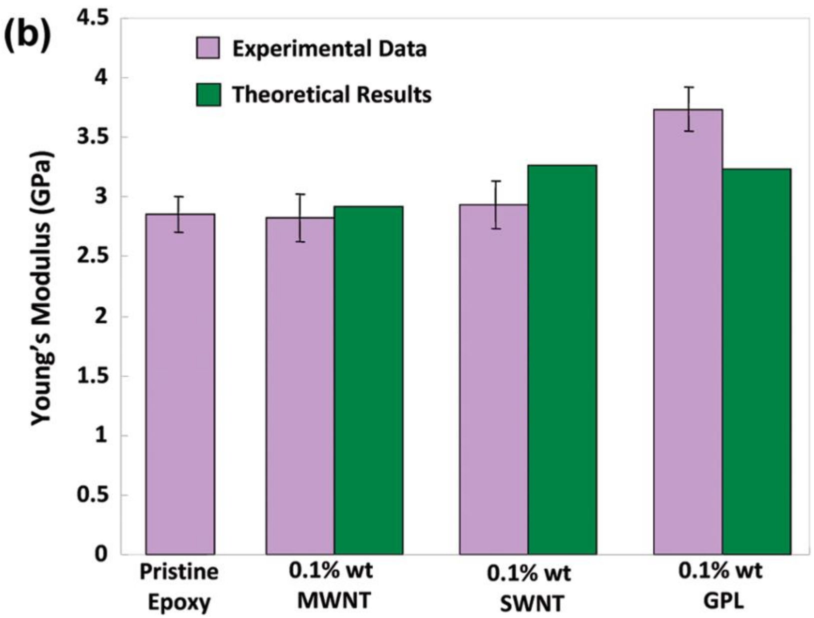
Enhanced Mechanical Properties of Nanocomposites at Low Graphene Content

Mohammad A. Rafiee,[†] Javad Rafiee,[†] Zhou Wang,[‡] Huaihe Song,[‡] Zhong-Zhen Yu,[‡] and Nikhil Koratkar[†,*]

[†]Department of Mechanical, Aerospace and Nuclear Engineering, Rensselaer Polytechnic Institute, 110 Eighth Street, Troy, New York 12180-3590, and [‡]State Key Laboratory of Chemical Resource Engineering, College of Materials Science and Engineering, Beijing University of Chemical Technology, Beijing 100029, China

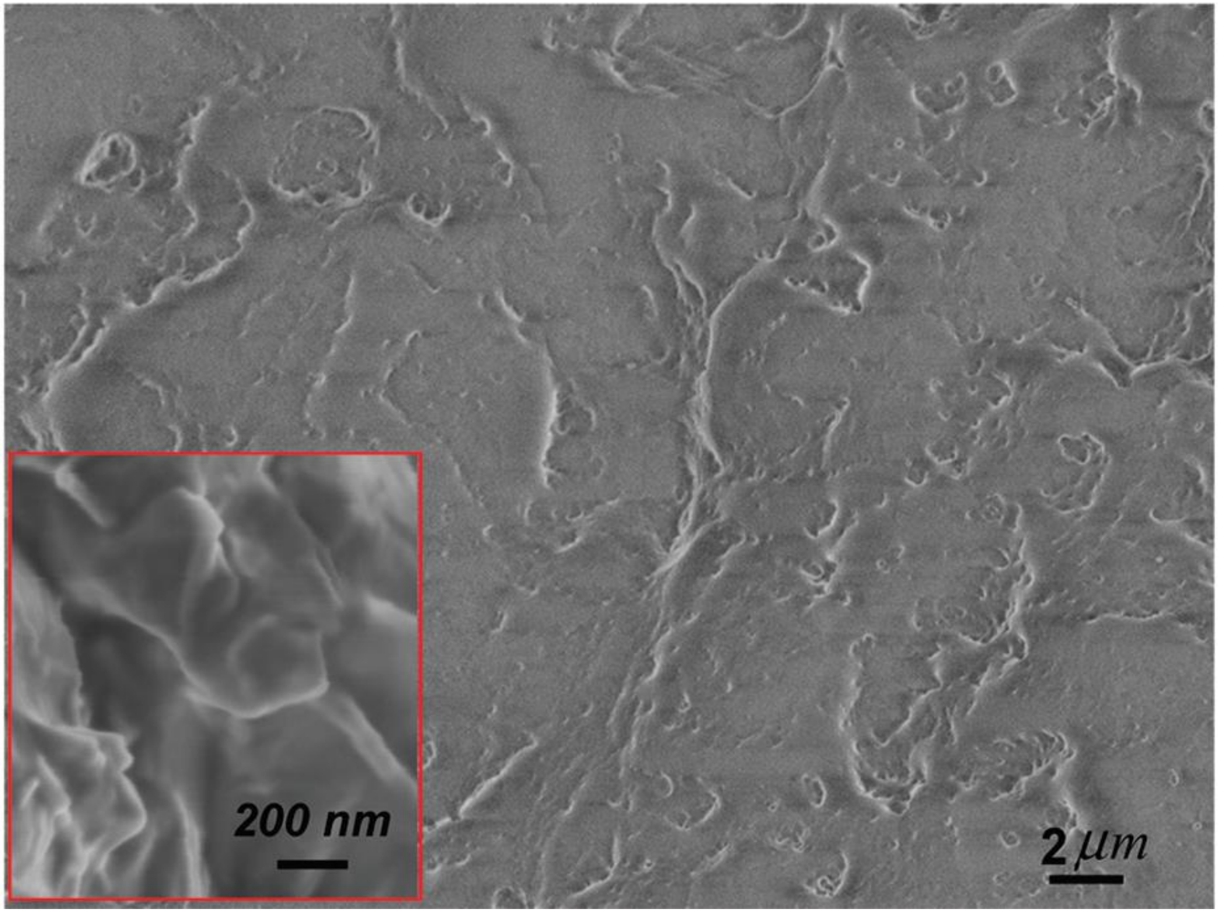


(a) Tensile strength for the baseline epoxy and GPL/epoxy, MWNT/epoxy, and SWNT/epoxy nanocomposites.



(b) Young's modulus of nanocomposites

SEM analysis of the freeze-fractured surface of a graphene/epoxy composite with approximately 5% weight of GPL



CARBON 49 (2011) 793–803

available at www.sciencedirect.com
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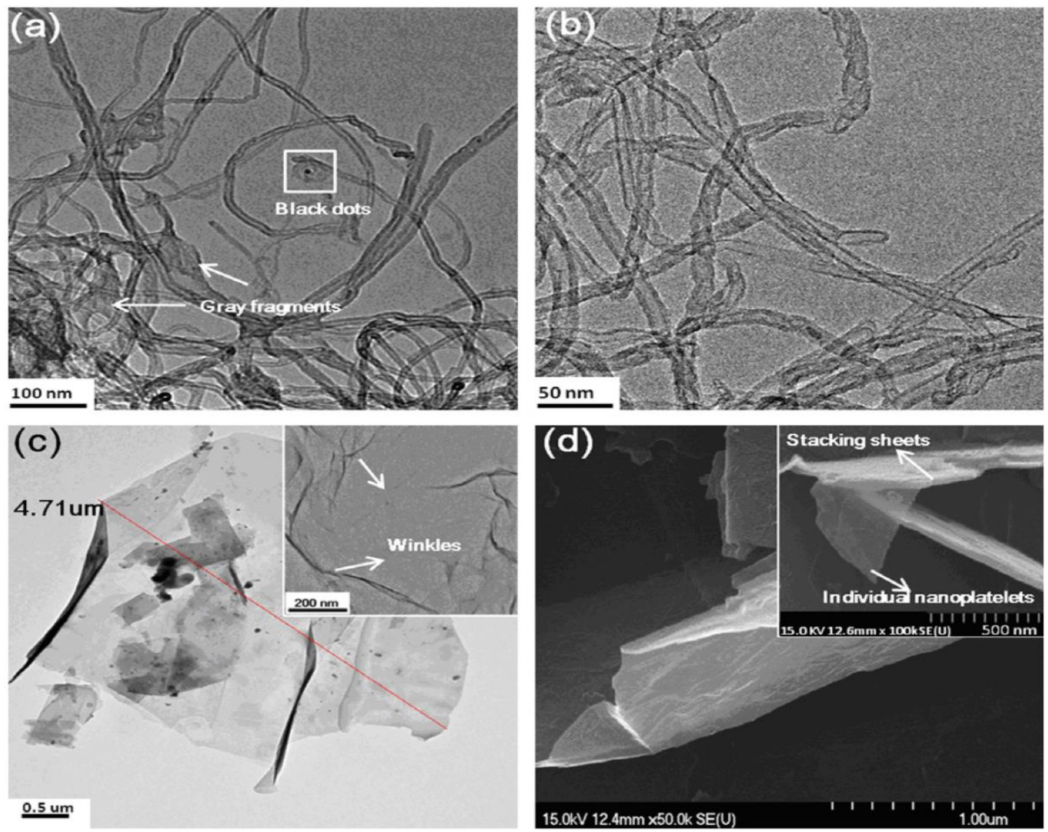
Synergetic effects of graphene platelets and carbon nanotubes on the mechanical and thermal properties of epoxy composites

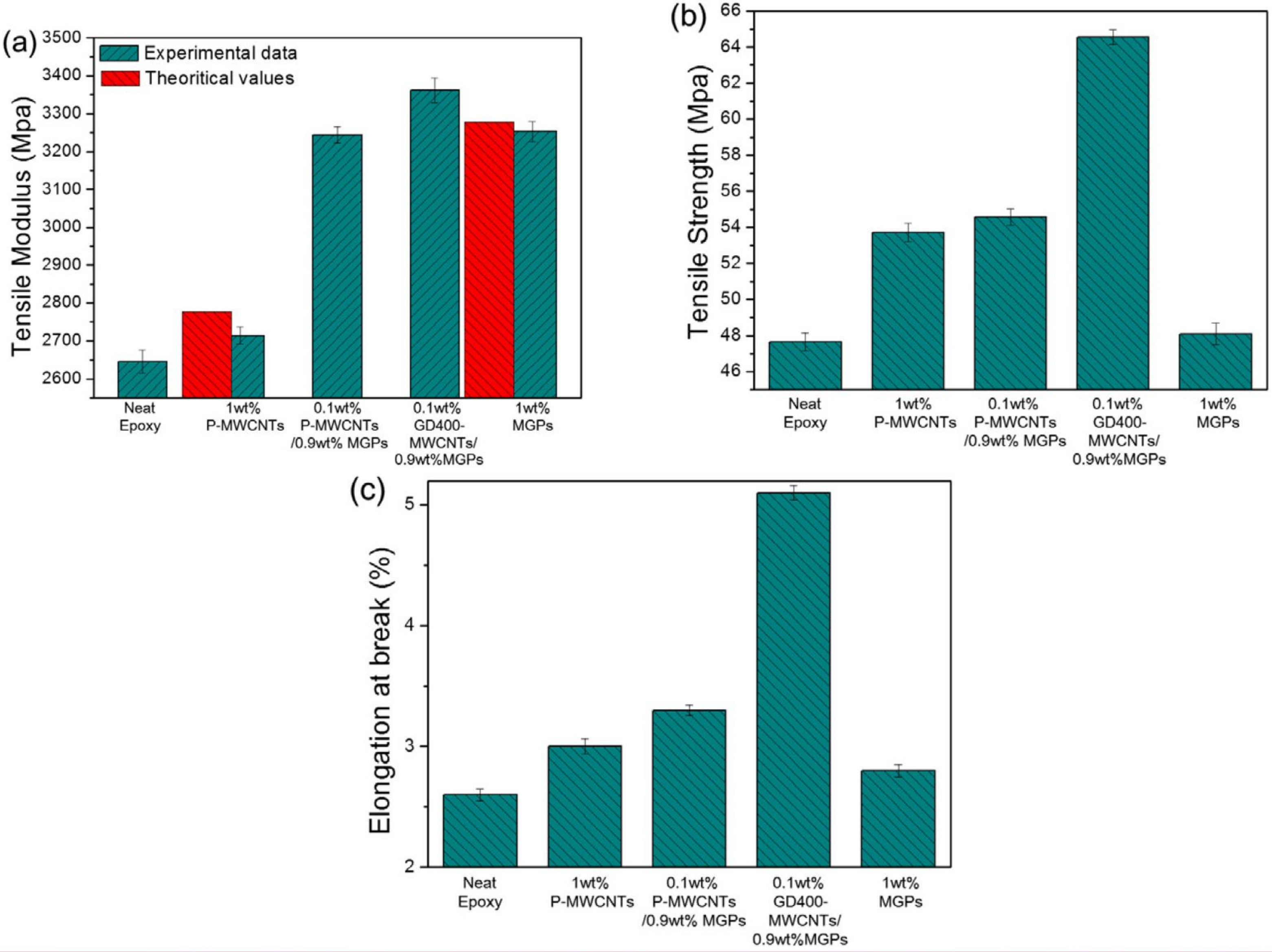
Shin-Yi Yang, Wei-Ning Lin, Yuan-Li Huang, Hsi-Wen Tien, Jeng-Yu Wang, Chen-Chi M. Ma *, Shin-Ming Li, Yu-Sheng Wang

Department of Chemical Engineering, National Tsing Hua University, Hsin-Chu 30013, Taiwan



TEM of (a) P-MWCNTs, (b) GD400-MWCNTs, (c) MGPs and FESEM of (d) MGPs

Tensile properties of neat epoxy and its composites

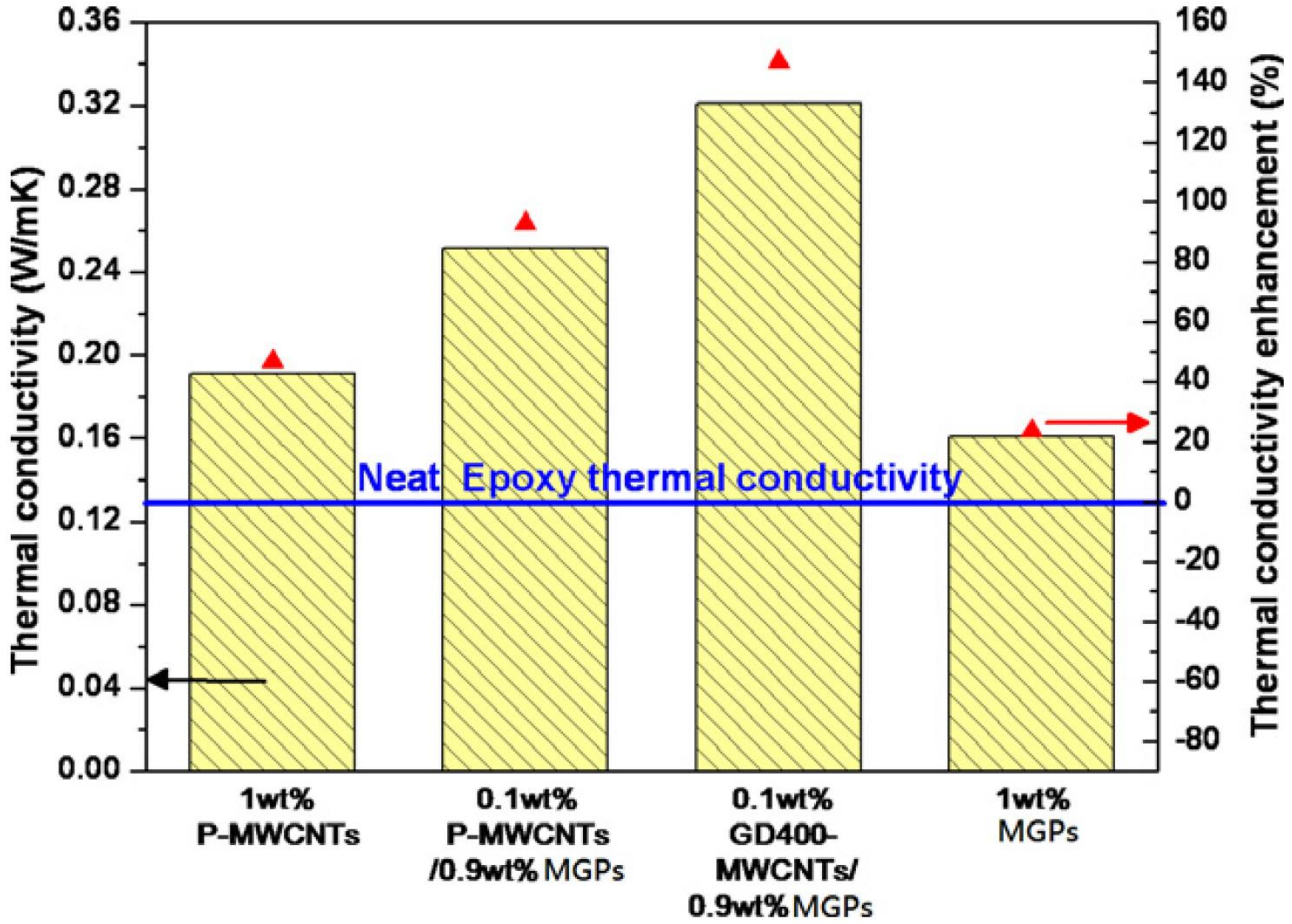


Tensile properties of neat epoxy and its composites

Sample	Tensile modulus (MPa)	(%)*	Tensile strength (MPa)	(%)*	Thermal conductivity (W/mK)	(%)*
Neat epoxy	2646	-	47.65	-	0.130	-
1 wt% P-MWCNTs	2714	2.6	53.72	12.7	0.211	62.3
1 wt% (1:9) P-MWCNTs/MGPs	3244	22.6	54.58	14.5	0.191	46.9
1 wt% (1:9) GD400-MWCNTs/MGPs	3361	27.1	64.55	35.4	0.321	146.9
1 wt% MGPs	3253	23.0	48.09	0.9	0.161	23.9

*Percentage of improvement compared to neat epoxy.

Thermal conductivity of epoxy composites



Polymer 52 (2011) 4664–4669

Contents lists available at SciVerse ScienceDirect

Polymer

journal homepage: www.elsevier.com/locate/polymer

Epoxy-Graphene UV-cured nanocomposites

M. Martin-Gallego[a], R. Verdejo[a], M.A. Lopez-Manchado[a], M. Sangermano[b,*]

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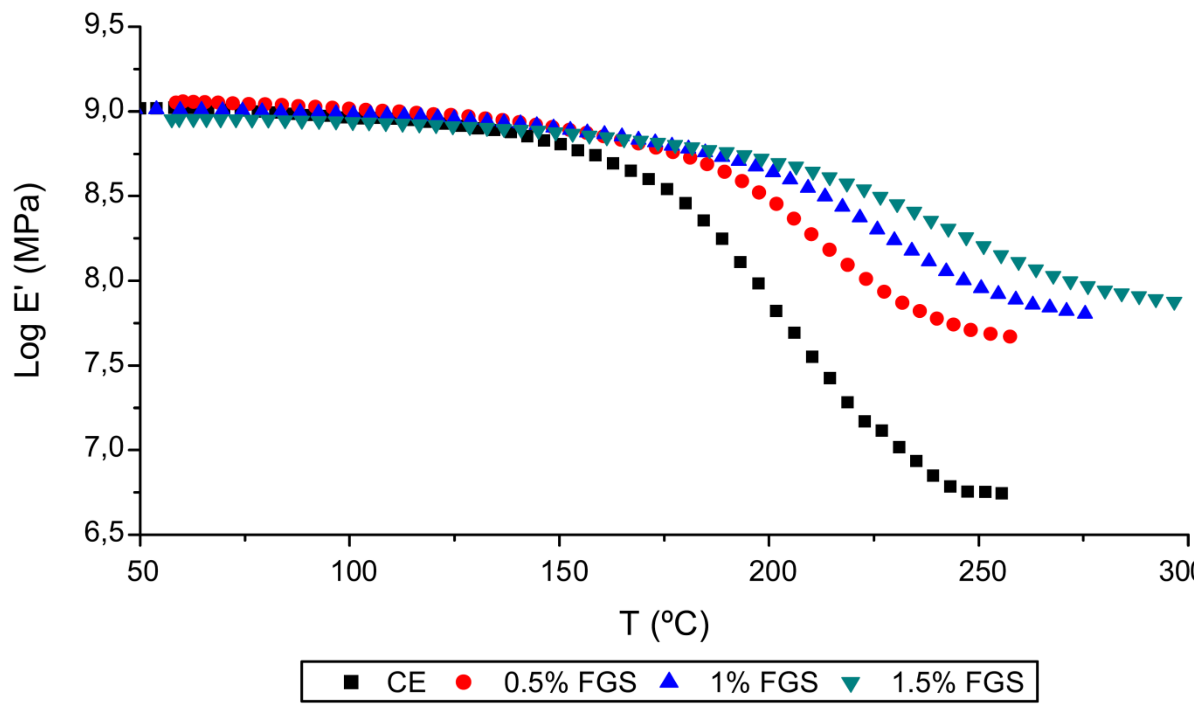
[b] *Politecnico di Torino, Dipartimento di Scienza dei Materiali e Ingegneria Chimica, C.so Duca degli Abruzzi 24, 10129 Torino, Italy*

Storage modulus curves obtained by DMTA analysis for epoxy UV-cured system
and its graphene composites.



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Polymer Composites
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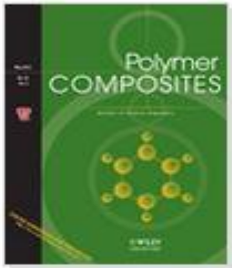
Article

Mechanical, thermal, and rheological properties of graphene-based polypropylene nanocomposites prepared by melt mixing

Mounir El Achaby, Fatima-Ezzahra Arrakhiz, Sébastien Vaudreuil, Abou el Kacem Qaiss ✉, Mostapha Bousmina, Omar Fassi-Fehri

First published: 29 March 2012 Full publication history

DOI: 10.1002/pc.22198 View/save citation

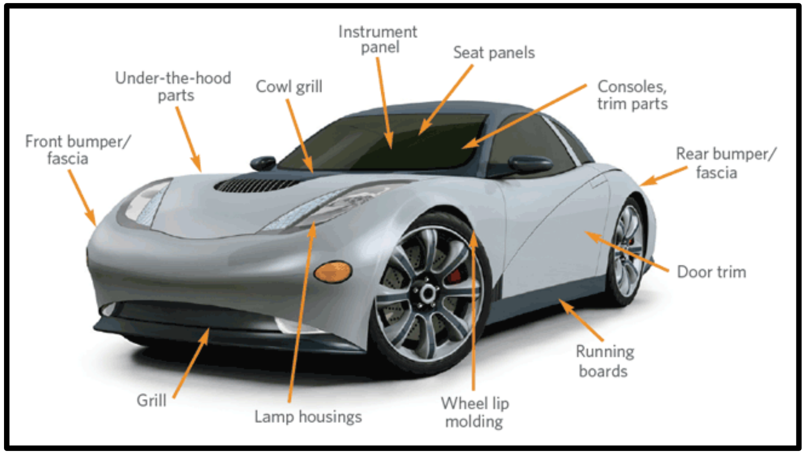


View issue TOC
Volume 33, Issue 5
May 2012
Pages 733–744

Polypropylene

Properties

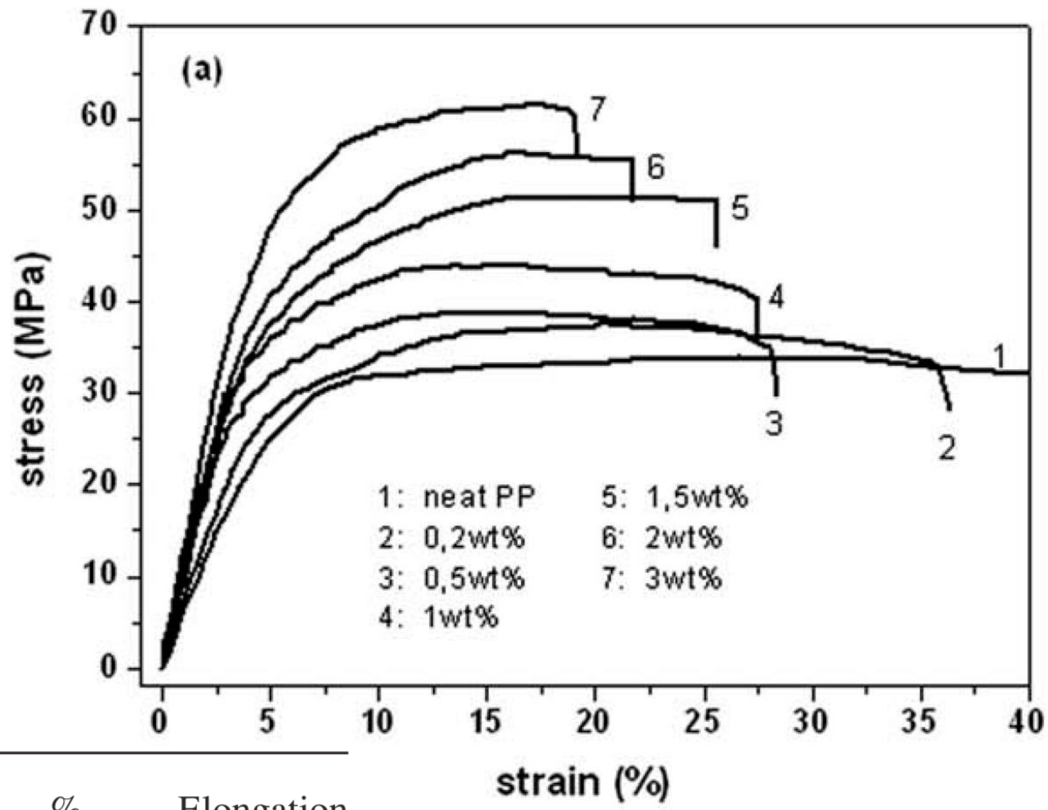
- Lightweight
- Impact resistant
- Excellent dielectric properties
- Resists most alkalis and acids
- Resists stress cracking
- Low moisture absorption
- Easily fabricated



Applications

- Automotive
- Household Goods
- Film
- Containers
- Packaging
- Electrical/Electronic

Effect of Graphene Content on Tensile Properties of Polypropylene



GNs content		Young's Modulus, E (Mpa)	% increase of E	Tensile strength, σ (Mpa)	% increase of σ	Elongation at break (%)
Wt%	Vol%					
0	—	1154.48	—	33.98	—	279.91
0.2	0.08	1252.21	14.66	37.58	10.59	36.33
0.5	0.2	1576.96	36.59	38.81	14.21	28.34
1	0.4	1785.33	54.64	43.93	29.28	27.49
1.5	0.6	1888.27	63.55	51.37	51.17	26.28
2	0.8	2040.04	76.70	56.17	65.30	21.71
3	1.2	2314.61	100.48	61.57	81.19	19.09

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GRAPHENE BASED IMPACT MODIFIED POLYPROPYLENE NANOCOMPOSITES FOR AUTOMOTIVE APPLICATIONS

Alper Kiziltas[1, 2], Alex Duguay[1], Behzad Nazari[3], Esra Erbas Kiziltas[1, 4], Douglas J. Gardner[1] & Habib J. Dagher[1]

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[3]Department of Chemical Engineering, University of Maine, Orono, ME 04469, USA

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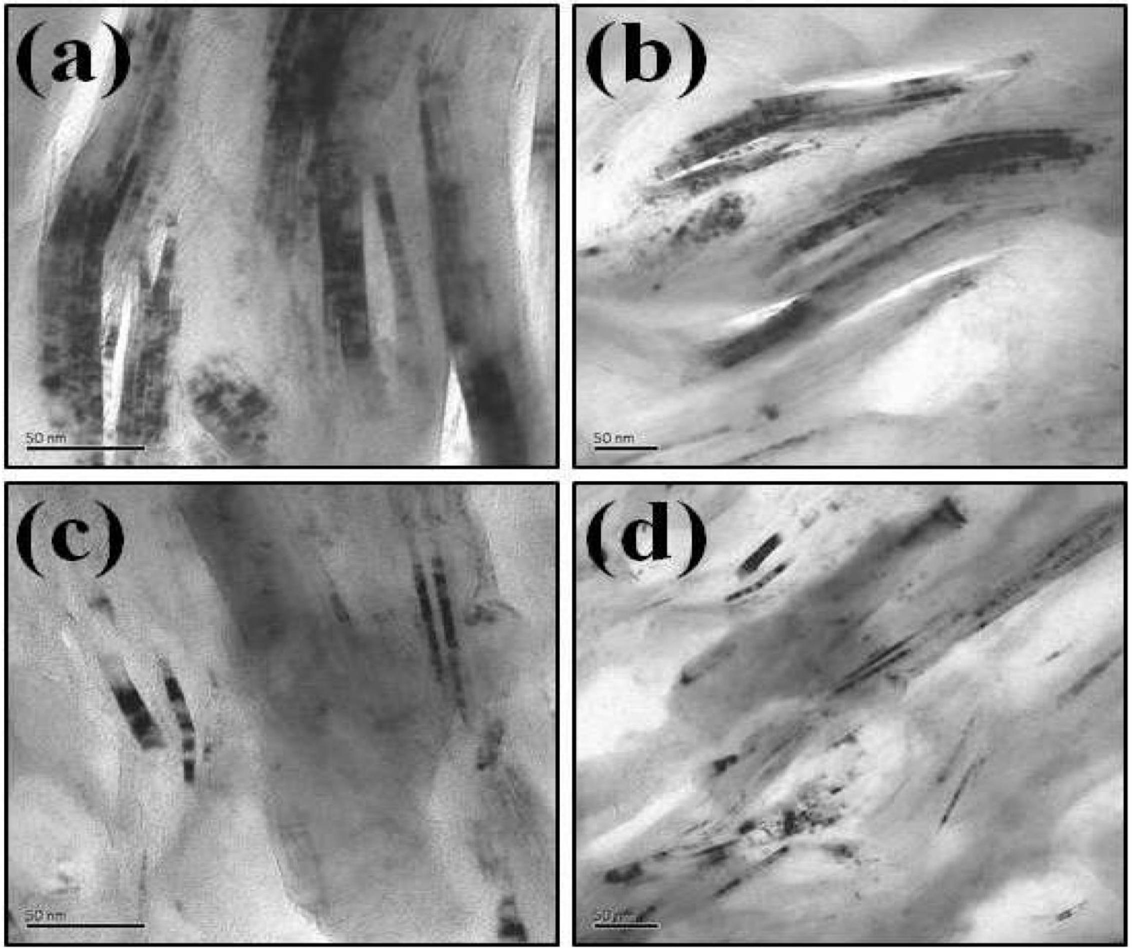
Effects of graphene and compatibilizer on flexural properties of impact modified polypropylene (IMPP)

Designation	Flexural Properties	
	Modulus (GPa)	Strength (MPa)
Neat IMPP	1.08	33.7
IMPP/xGNP2%	1.31	34.3
IMPP/xGNP4%	1.33	35.3
IMPP/xGNP6%	1.32	35.0
IMPP/xGNP8%	1.26	33.3
IMPP/PPgMAH1%/xGNP2%	1.21	35.0
IMPP/PPgMAH2%/xGNP4%	1.36	36.4
IMPP/PPgMAH3%/xGNP6%	1.52	37.9
IMPP/PPgMAH4%/xGNP8%	1.53	36.9

Thermal properties of the composites

Designation	Thermal Properties	
	T_{max} (˚C)	Residual Mass (%)
Neat IMPP	459.9	1.6
PPgMAH	453.8	4.0
IMPP/PPgMAH4%	461.1	2.0
IMPP/xGNP4%	463.7	5.5
IMPP/PPgMAH1%/xGNP2%	461.2	4.3
IMPP/PPgMAH2%/xGNP4%	462.6	6.1
IMPP/PPgMAH3%/xGNP6%	467.2	7.7
IMPP/PPgMAH4%/xGNP8%	469.1	10.0

Transmission electron micrographs of (a & b) IMPP/xGnP2% and (c & d) IMPP/PPgMAH1%/xGnP2%



Compatibilizer improved dispersion and graphene PP intercation

Tribology International 70 (2014) 165–169

Contents lists available at ScienceDirect

Tribology International

journal homepage: www.elsevier.com/locate/triboint

Short Communication

Nanotribological behavior of graphene nanoplatelet reinforced ultra high molecular weight polyethylene composites

Debrupa Lahiri [a,b], Francois Hec [a,c], Mikael Thiesse [a,c], Andriy Durygin [d], Cheng Zhang [a], Arvind Agarwal [a,*]

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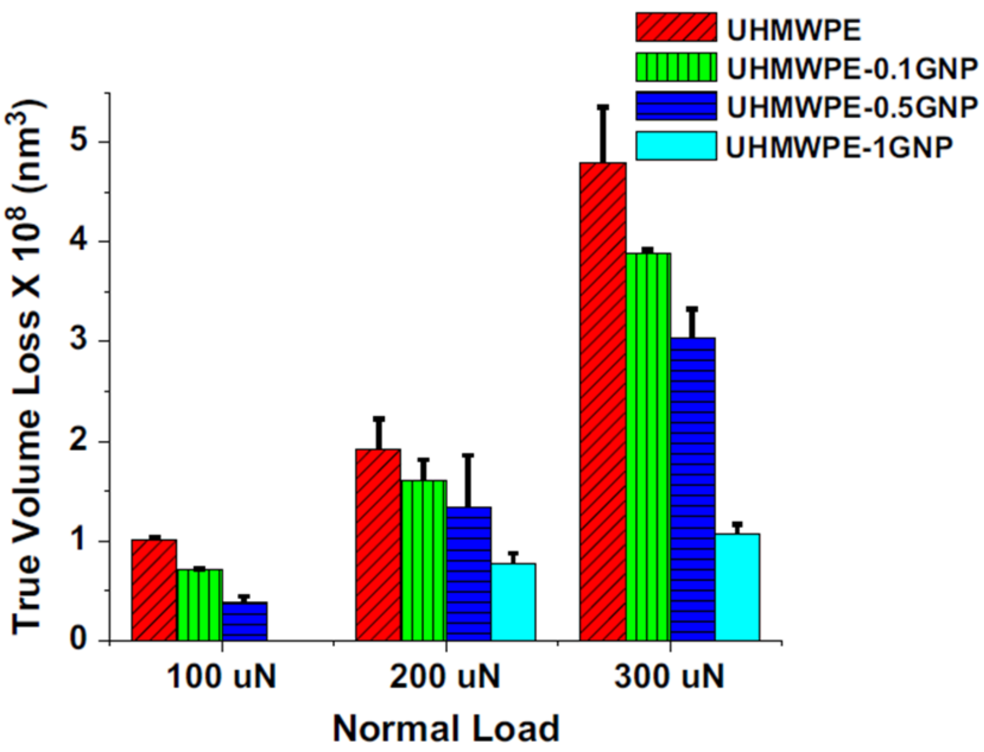
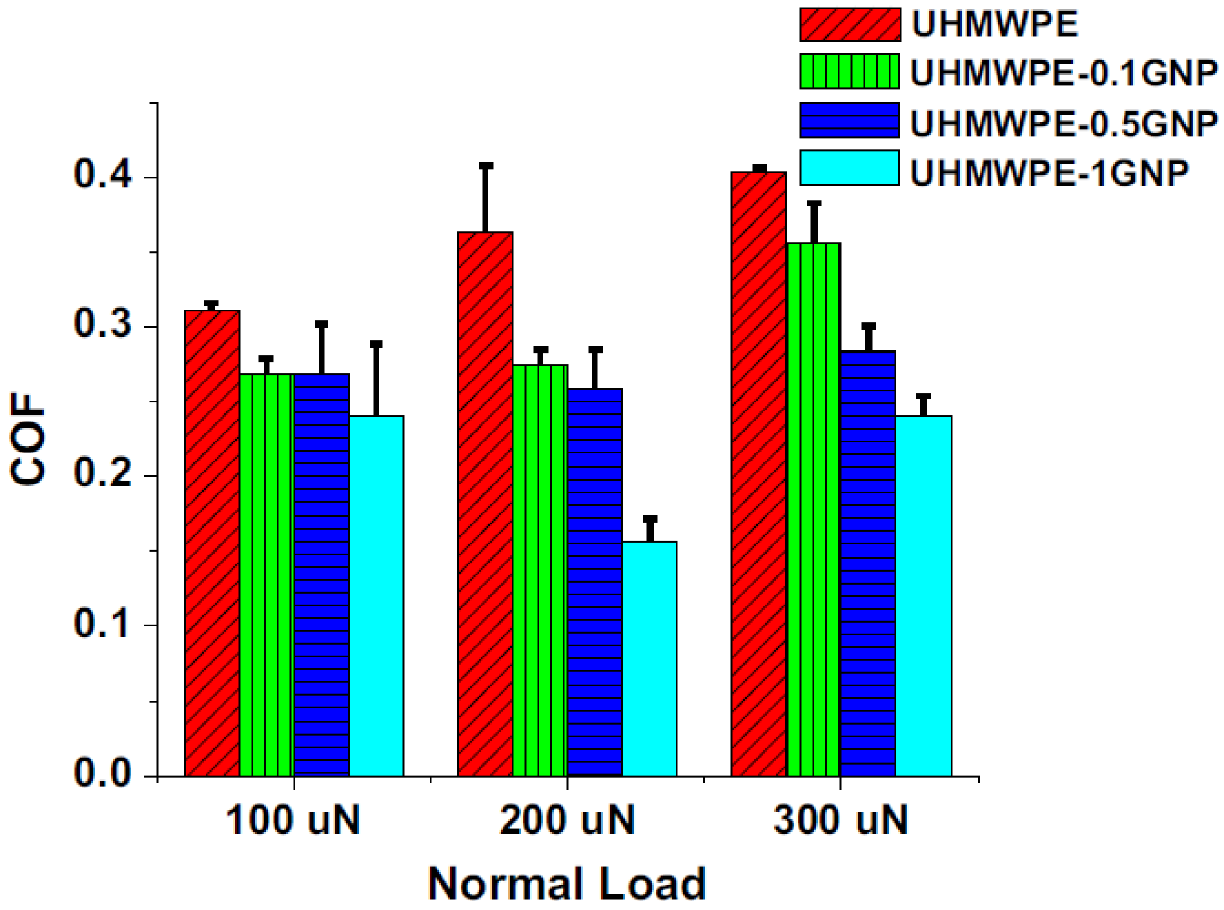
[b] Department of Metallurgical and Materials Engineering, Indian Institute of Technology Roorkee, Roorkee, Uttarakhand 247667, India
[c] Universite de Lyon-INSA de Lyon, 7, av. Jean Capelle F-69 621, Villeurbanne, France
[d] Center for Study of Extreme Materials at Extreme Conditions (CeSMEC), Department of Mechanical and Materials Engineering, Florida International University, Miami, FL 33174, USA

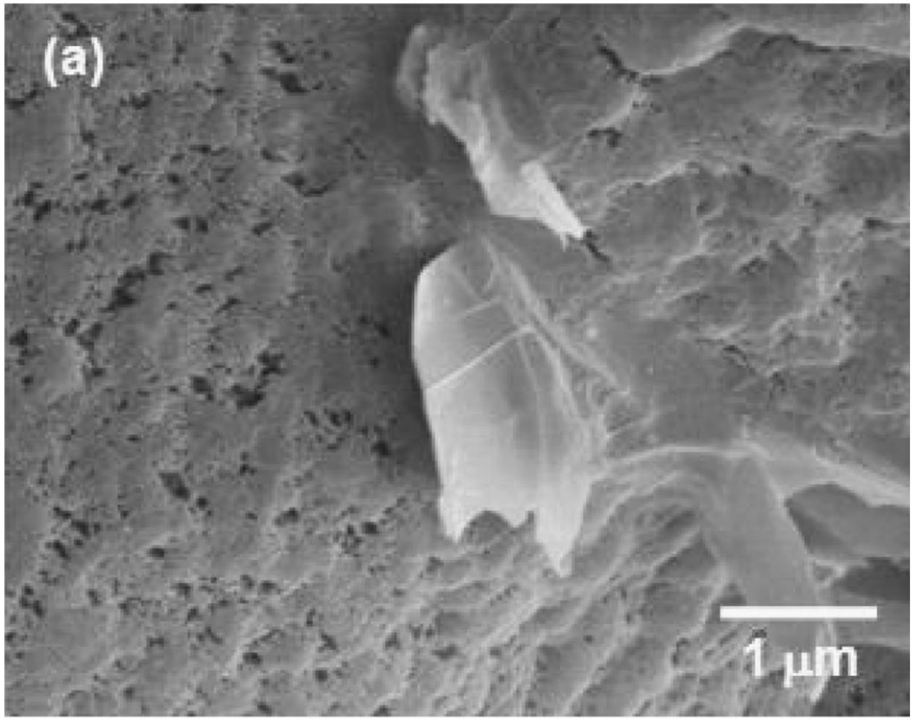
Coefficient of friction(COF) on UHMWPE-GNP composites at different loads.

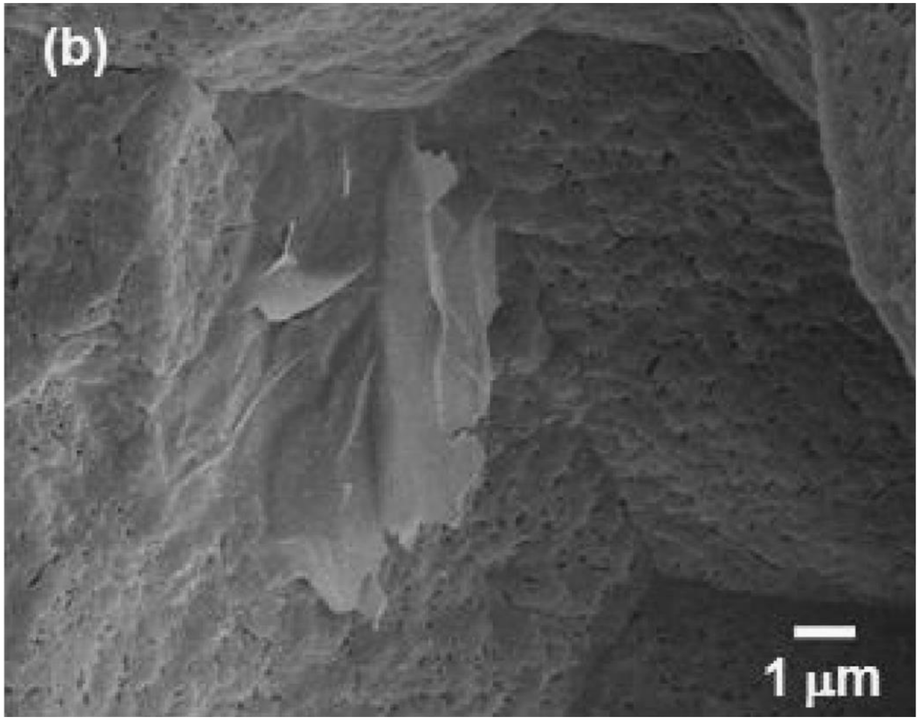


Wear volume loss during scratching on UHMWPE-GNP composites at different loads.

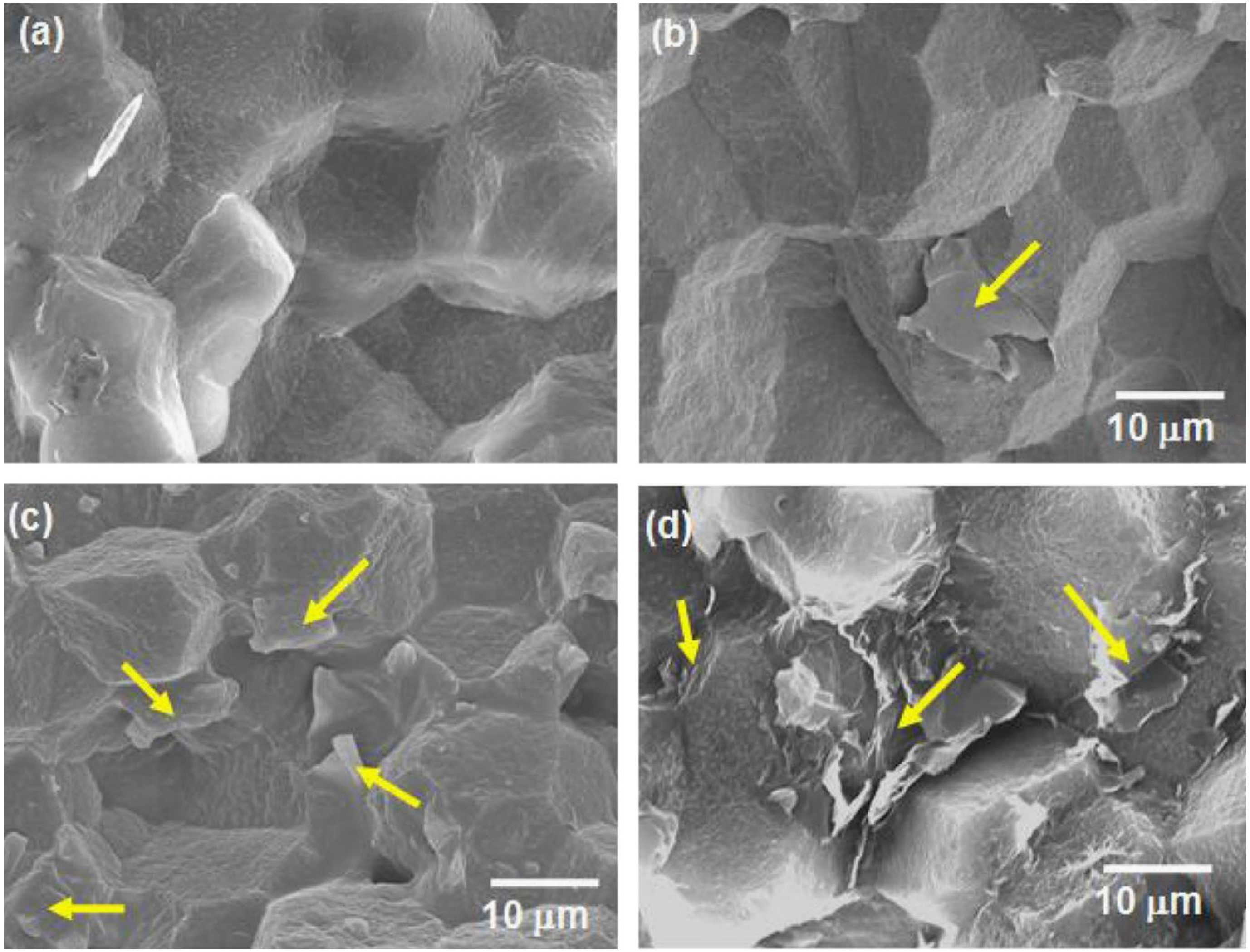


High magnification micrographs of fracture surfaces of UHMWPE 0.1GNP revealing good bonding of GNP with UHMWPE matrix





SEM micrographs of fracture surfaces of (a) UHMWPE, (b) UHMWPE-0.1GNP, (c) UHMWPE-0.5GNP and (d) UHMWPE-1GNP.



SPE Polymer Composites
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Article

Thermal stability and dynamic mechanical behavior of exfoliated graphite nanoplatelets-LLDPE nanocomposites

Sumin Kim, Inhwan Do, Lawrence T. Drzal

First published: 5 January 2009 Full publication history

DOI: 10.1002/pc.20781 View/save citation

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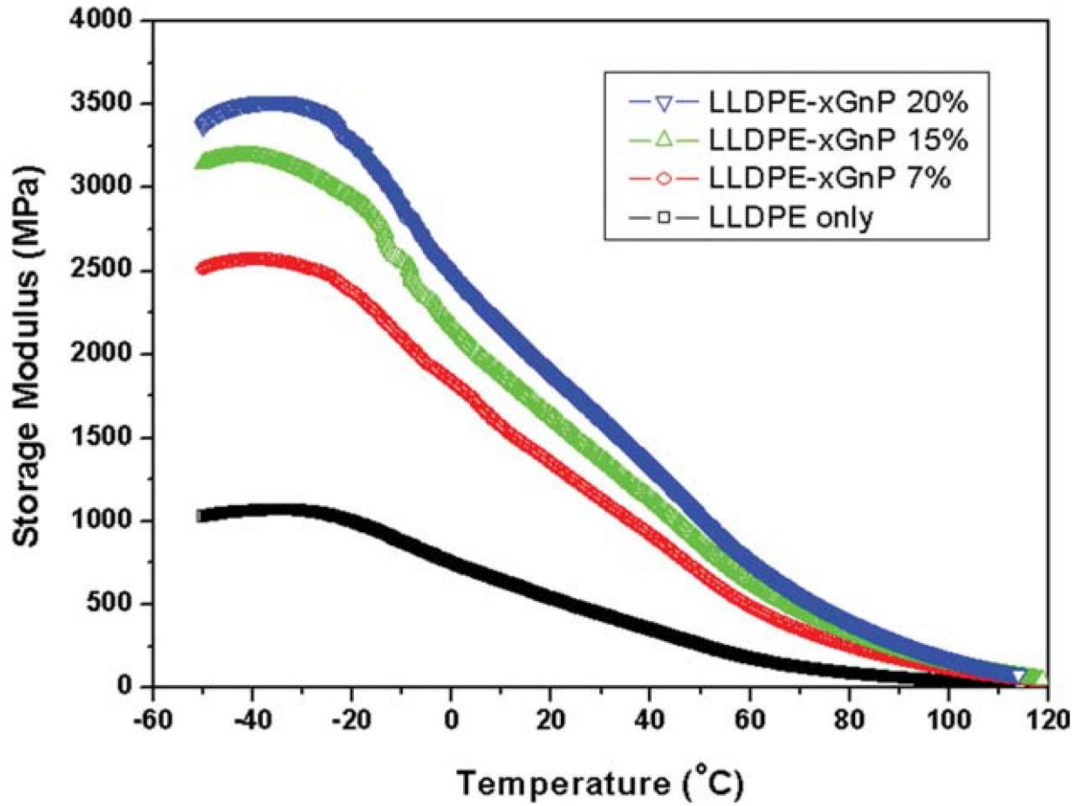
Volume 31, Issue 5
May 2010
Pages 755–761

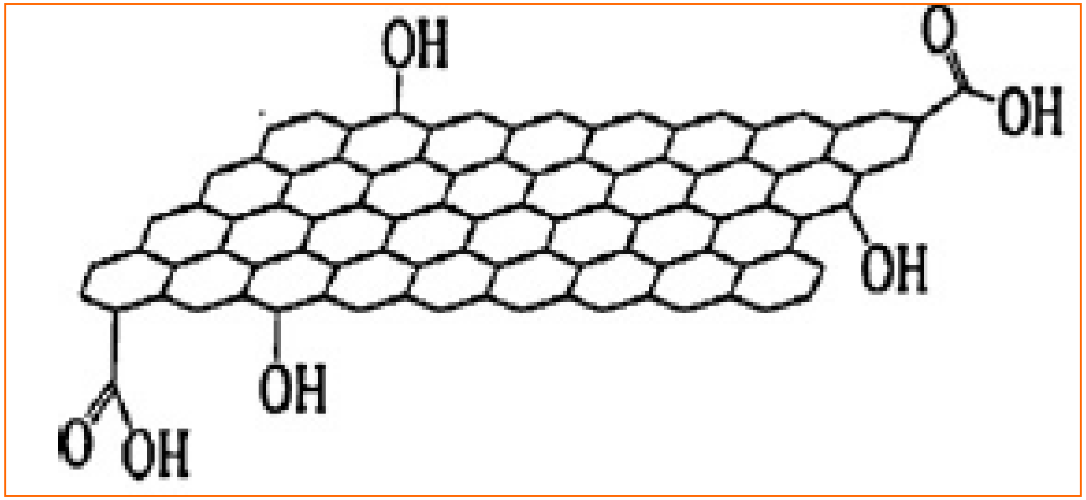
The storage modulus of xGnP-LLDPE nanocomposites by solution mixing and injection molded as xGnP loading contents.



Research on Graphene Reinforced Nanocomposites in UTM

Graphene nanoplatelets

- GNP 's are nanoparticles consisting of short stacks of one or more graphene sheets with an average thickness of 5-10nm
- Edges of the platelets are the sites for functionalization which helps facilitate for hydrogen or covalent bonding within a polymer matrix.



List of Publication

- Influence of exfoliated graphite nanoplatelets on the flammability and thermal properties of polyethylene terephthalate/polypropylene nanocomposites, *Polymer Degradation and Stability*.

- Mechanical and thermal properties of exfoliated graphite nanoplatelets reinforced polyethylene terephthalate/polypropylene composites, *Polymer Composites*.

- Characterization and mechanical properties of exfoliated graphite nanoplatelets reinforced polyethylene terephthalate/polypropylene composites, *Journal of Applied Polymer Science*.

- Mechanical, thermal, and morphological properties of graphene reinforced polycarbonate/acrylonitrile butadiene styrene nanocomposites, *Polymer Composites*.

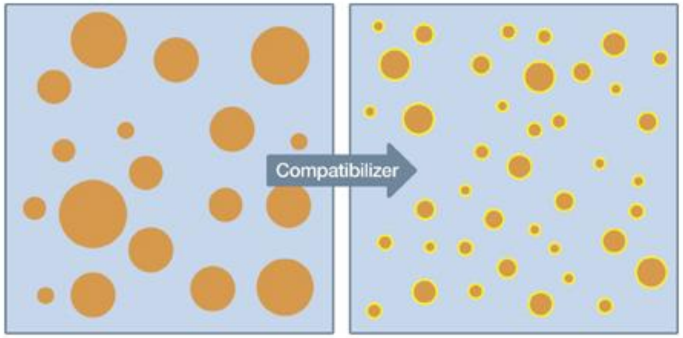
List of Publication

- Characterization and preparation of conductive exfoliated graphene nanoplatelets kenaf fibre hybrid polypropylene composites, *Synthetic Metals*.
- Flammability and thermal properties of polycarbonate/acrylonitrile-butadiene-styrene nanocomposites reinforced with multilayer graphene, *Polymer Degradation and Stability*.
- Characterization and preparation of conductive exfoliated graphene nanoplatelets kenaf fibre hybrid polypropylene composites, *Synthetic Metals*.
- Effect of exfoliated graphite nanoplatelets on thermal and heat deflection properties of kenaf polypropylene hybrid nanocomposites, *Journal of Polymer Engineering*.
- Mechanical and Thermal Properties of Hybrid Graphene/Halloysite Nanotubes Reinforced Polyethylene Terepthalate Nanocomposites, Chapter in: *Nanoclay Reinforced Polymer Composites*.

Why polymer blends?

- The ever-increasing demand for high performance polymers has shifted the focus from the development of new homopolymers to the development of new blends.

- Although polymer blending is combining two or more different advantageous properties in a single system, it is not always successful due to the inherent incompatibility between polymers to be blended



Why PET/PP Blends

- The increasing cost of engineering thermoplastics leads to finding alternative materials with good properties at cheaper price.
- Commodity thermoplastics such as PET and PP are inexpensive, easy to process and well understood.
- PP and PET constitute more than half of all plastic waste. Blending the two polymers is very convenient in terms of recycling and environmental conservation efforts.

Research Objectives

- To develop a multifunctional nanocomposites with equal or higher performance properties than engineering thermoplastics at a lower cost.

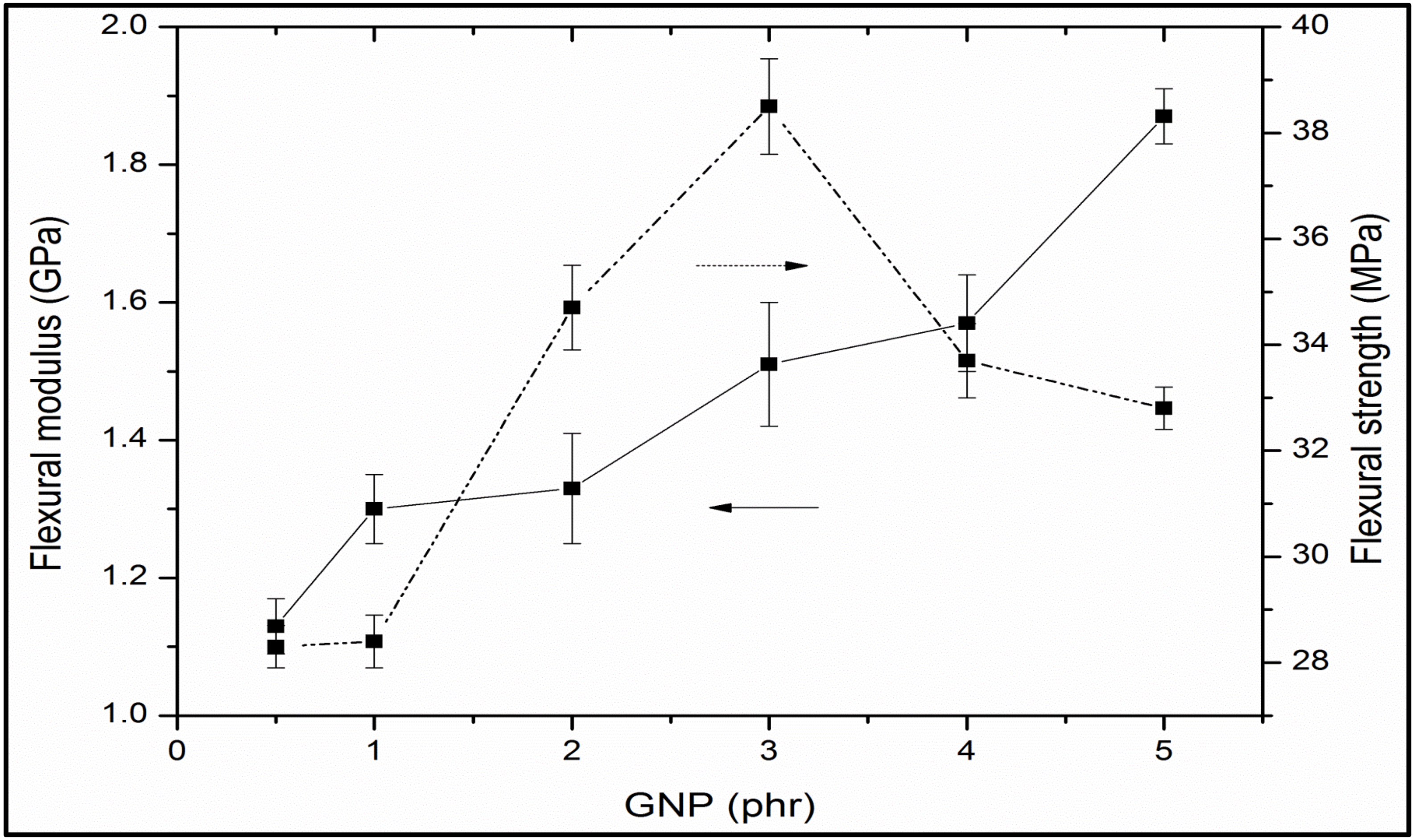
- To characterize the influence of graphene nanoplatelets, filler loading and the addition of compatibilizing agents on the mechanical, electrical, flammability and thermal properties of graphene filled PET/PP nanocomposites.

Testing and characterization

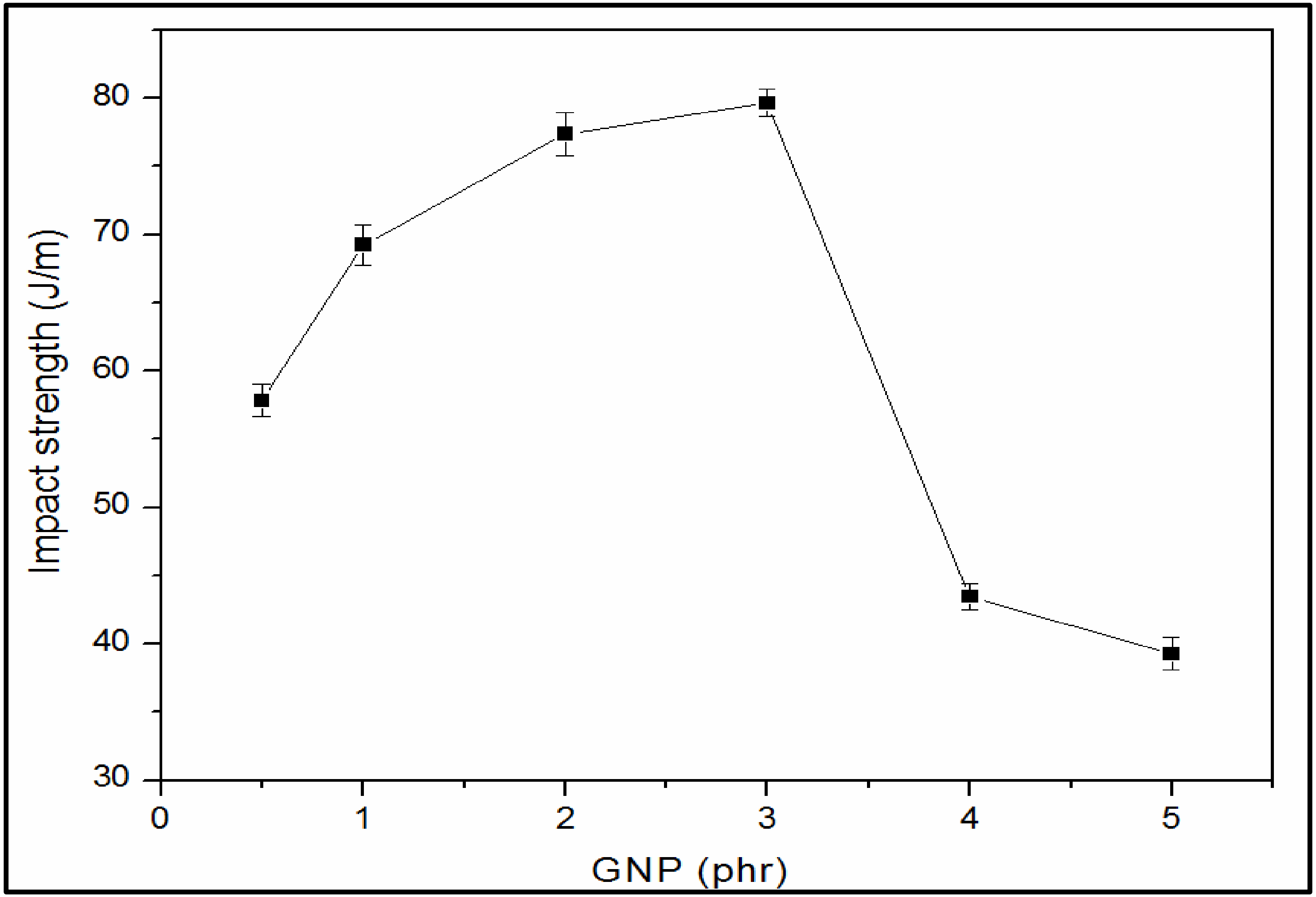
- Tensile test
- Impact test
- Cone calorimeter
- Limiting oxygen index
- UL-94
- Heat distortion Temperature

- Thermal conductivity
- TEM
- FESEM
- XRD
- Thermogravimatric analysis (TGA)

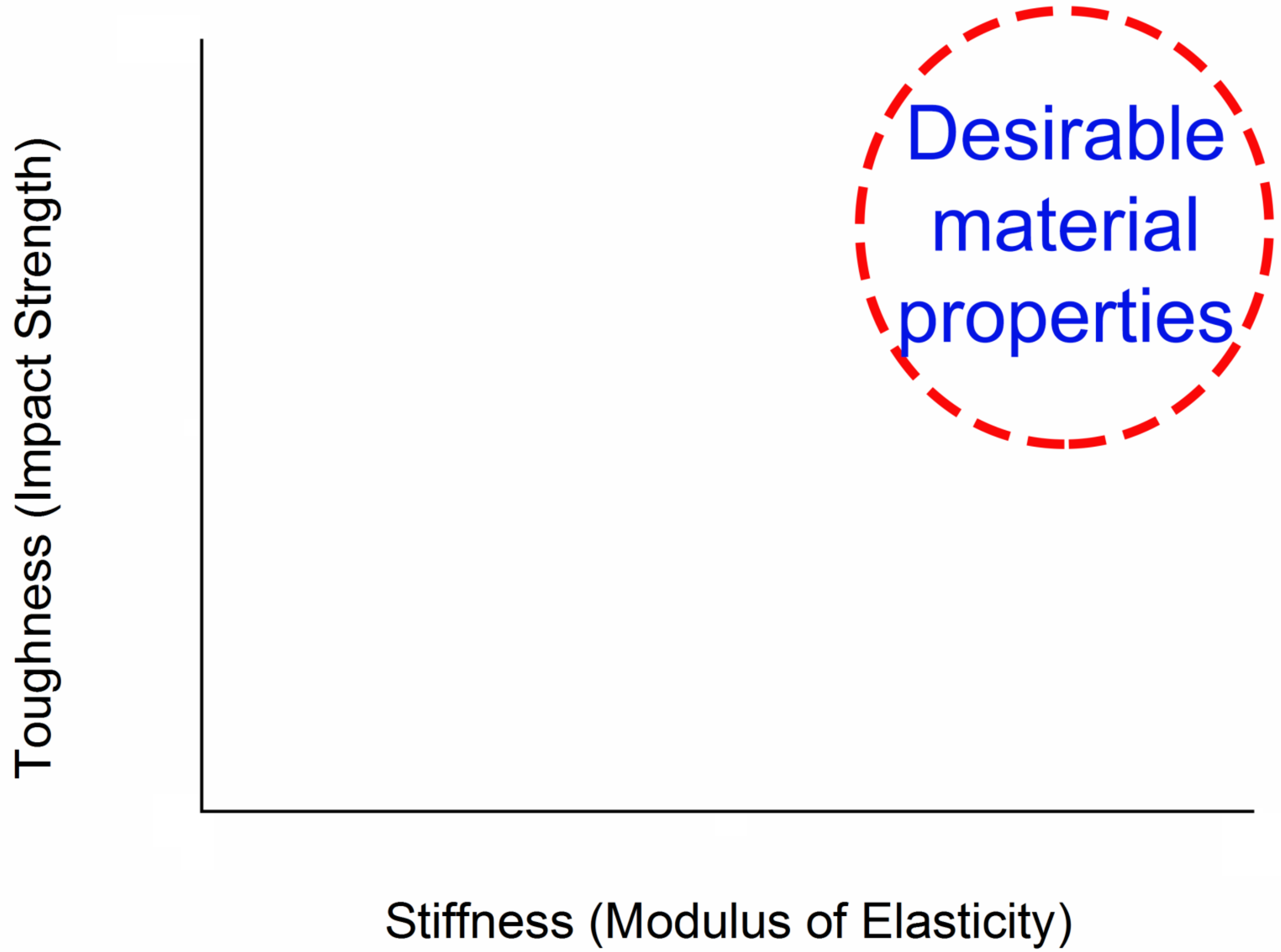
Effect of GNP content on flexural properties



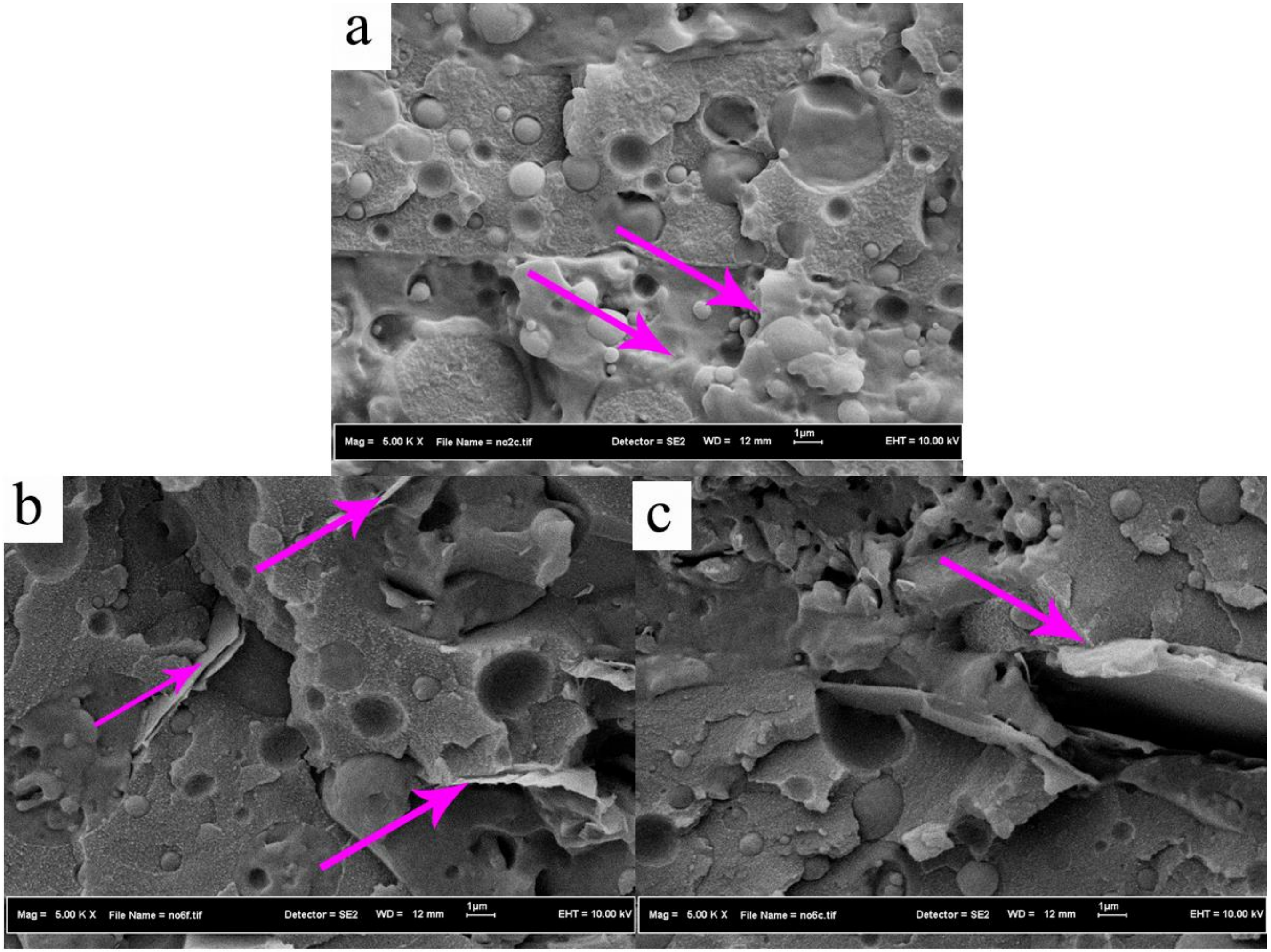
Effect of GNP content on impact strength



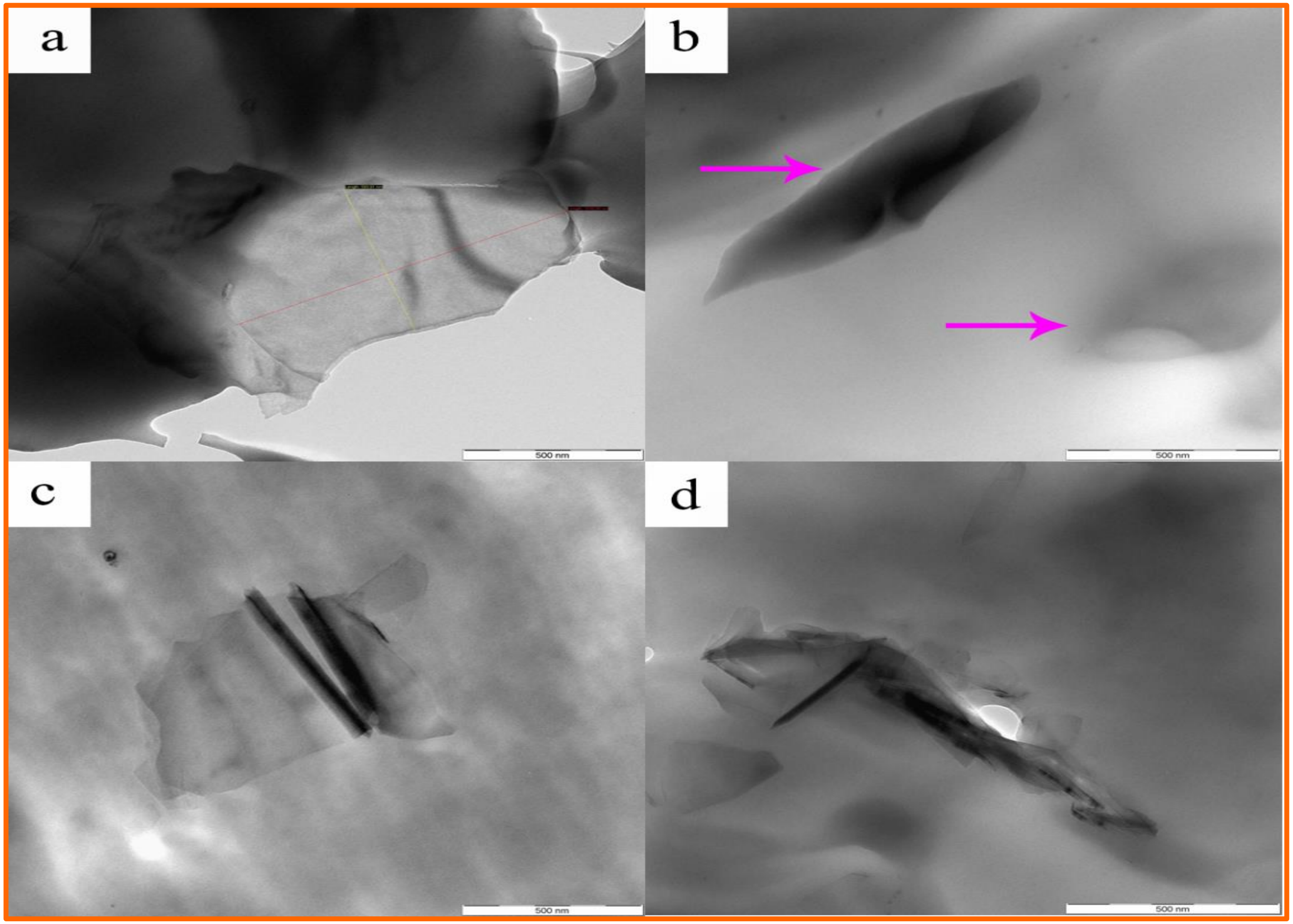
In terms of mechanical properties, the aim is to obtain materials with balance properties in terms of stiffness and toughness.



Morphology: FESEM



Morphology: TEM



Flammability - drawback of many plastics

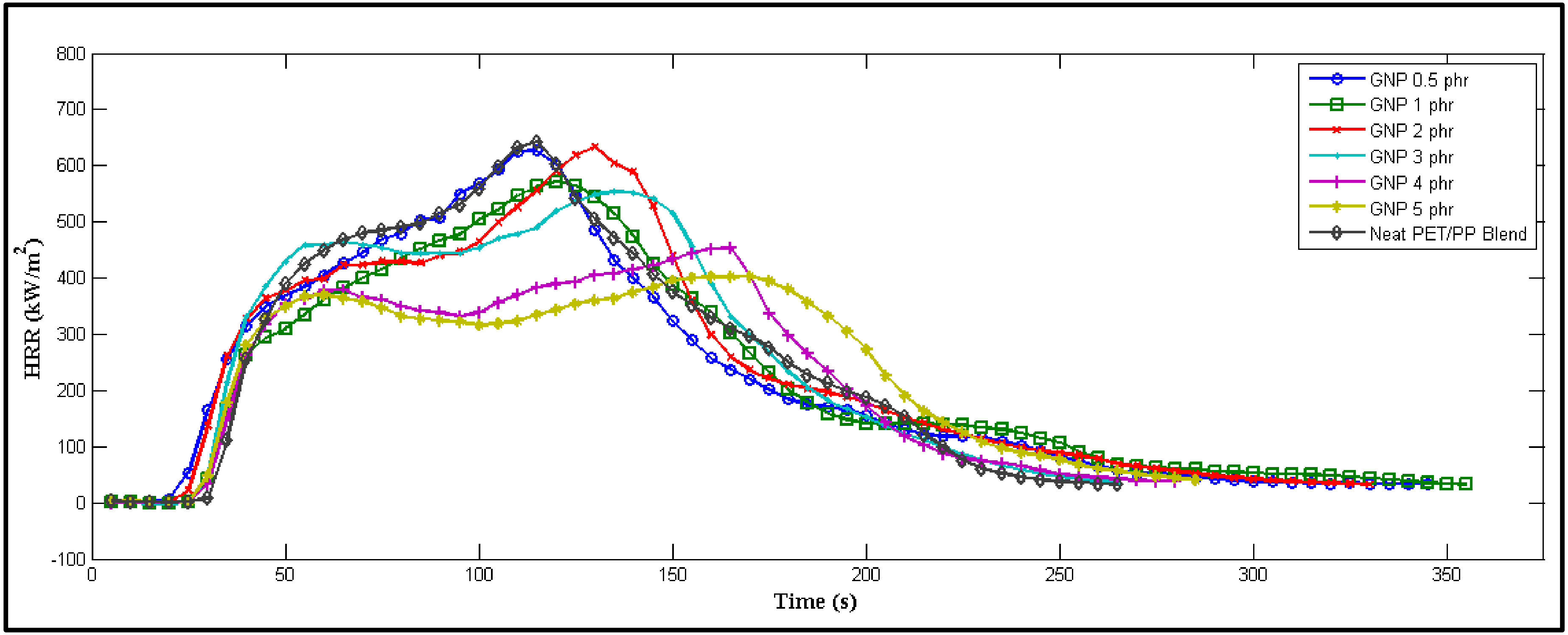
Plastic type	LOI
Acrylic	17
Polypropylene	17
Polyethylene	17
Polystyrene	18
ABS	19
Polycarbonate	26
Polyvinyl chloride	45
Polytetrafluoroethylene	>95

In 2014, there were **1.3 million** fires reported in the United States causing **$11.6 billion** in property damage

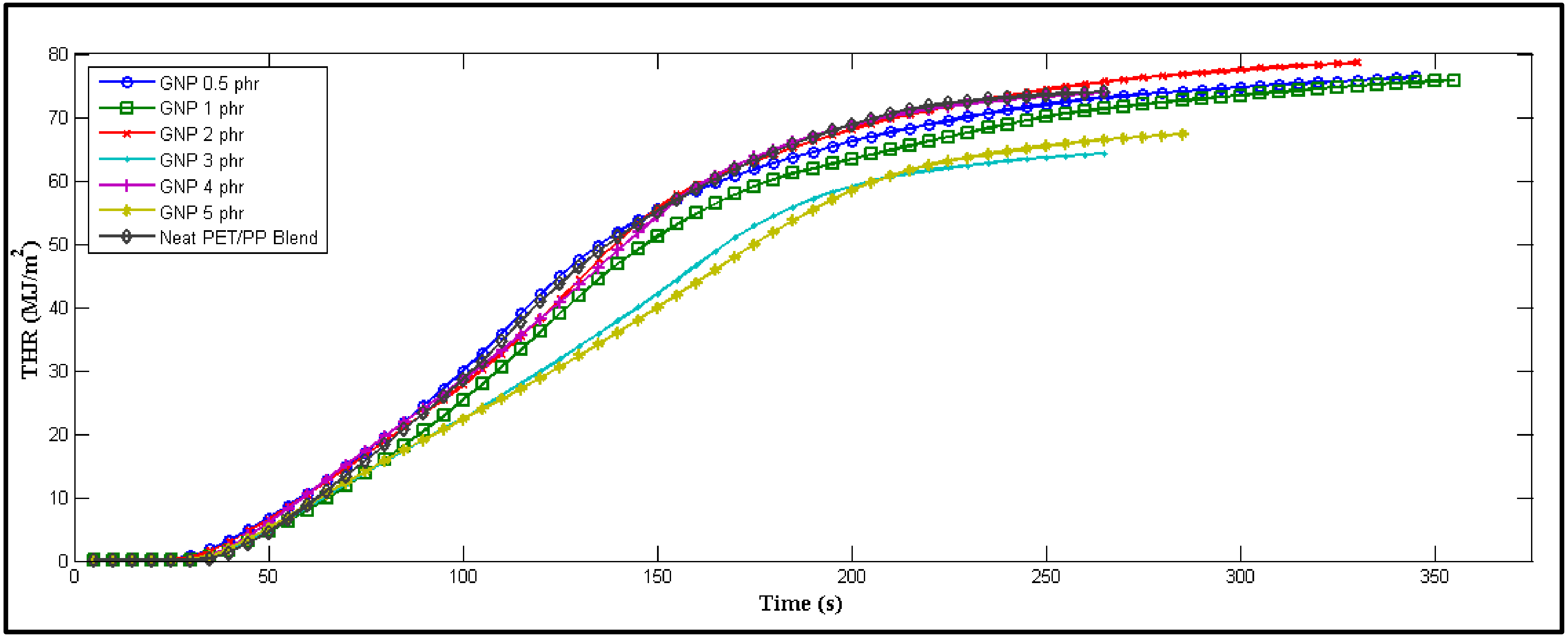
Cone Calorimeter

Measurement of the **heat release rate** is based on the principle that the gross **heat of combustion of** any organic material.

Heat Release Rate



Total Heat Release



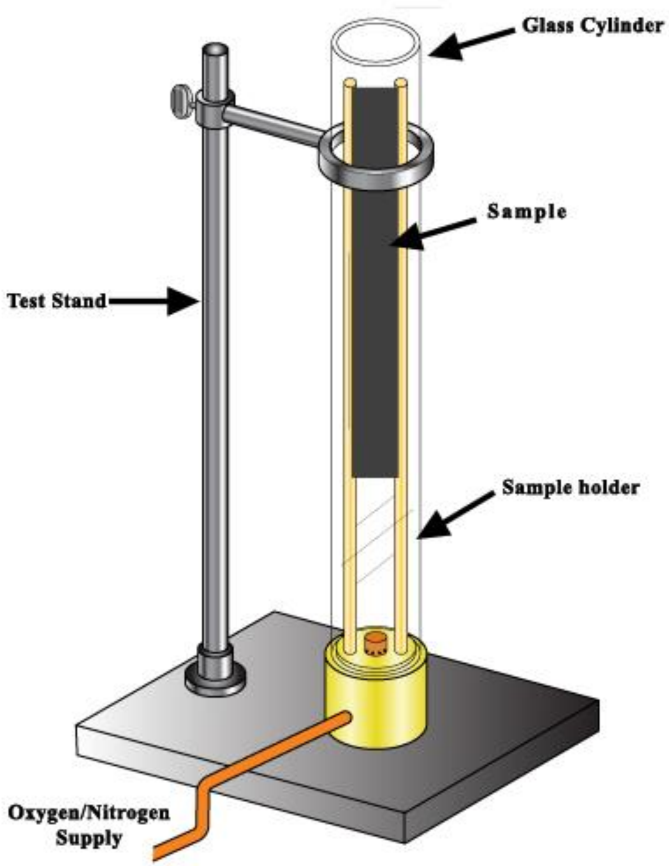
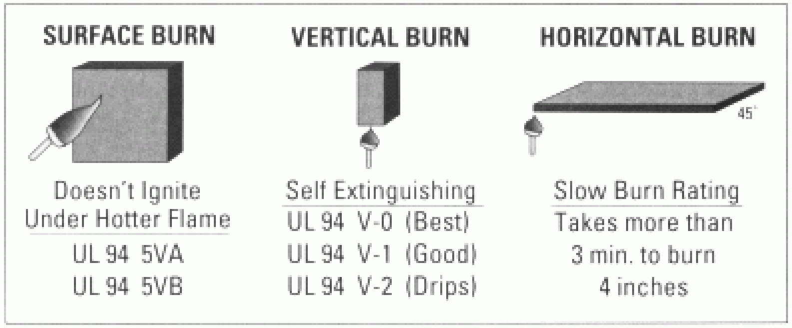
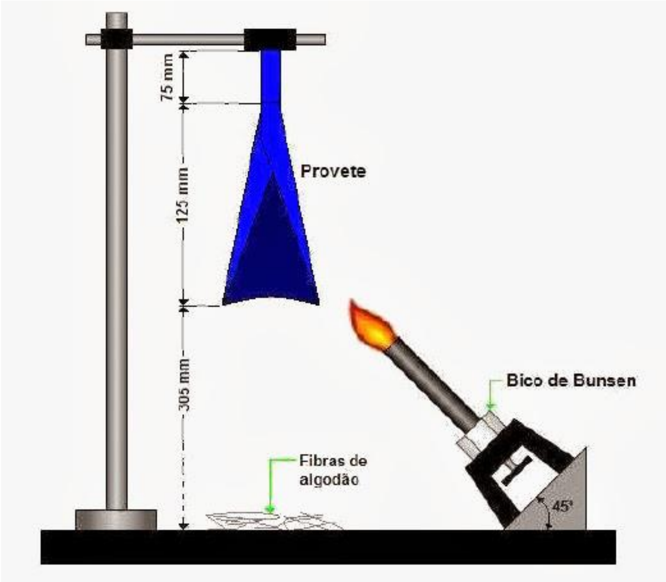


Figure 1 – Schematic of oxygen index test system

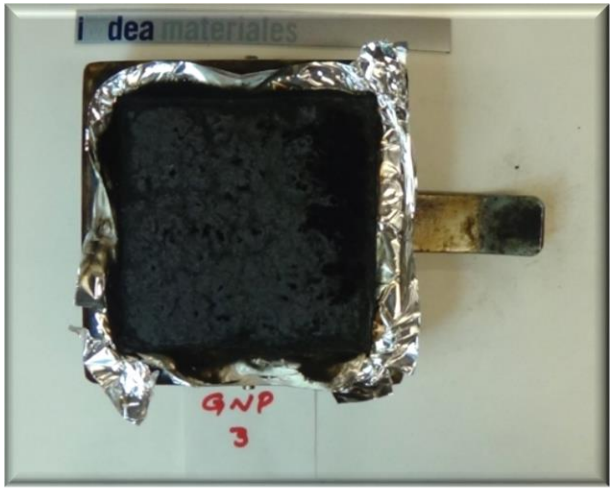




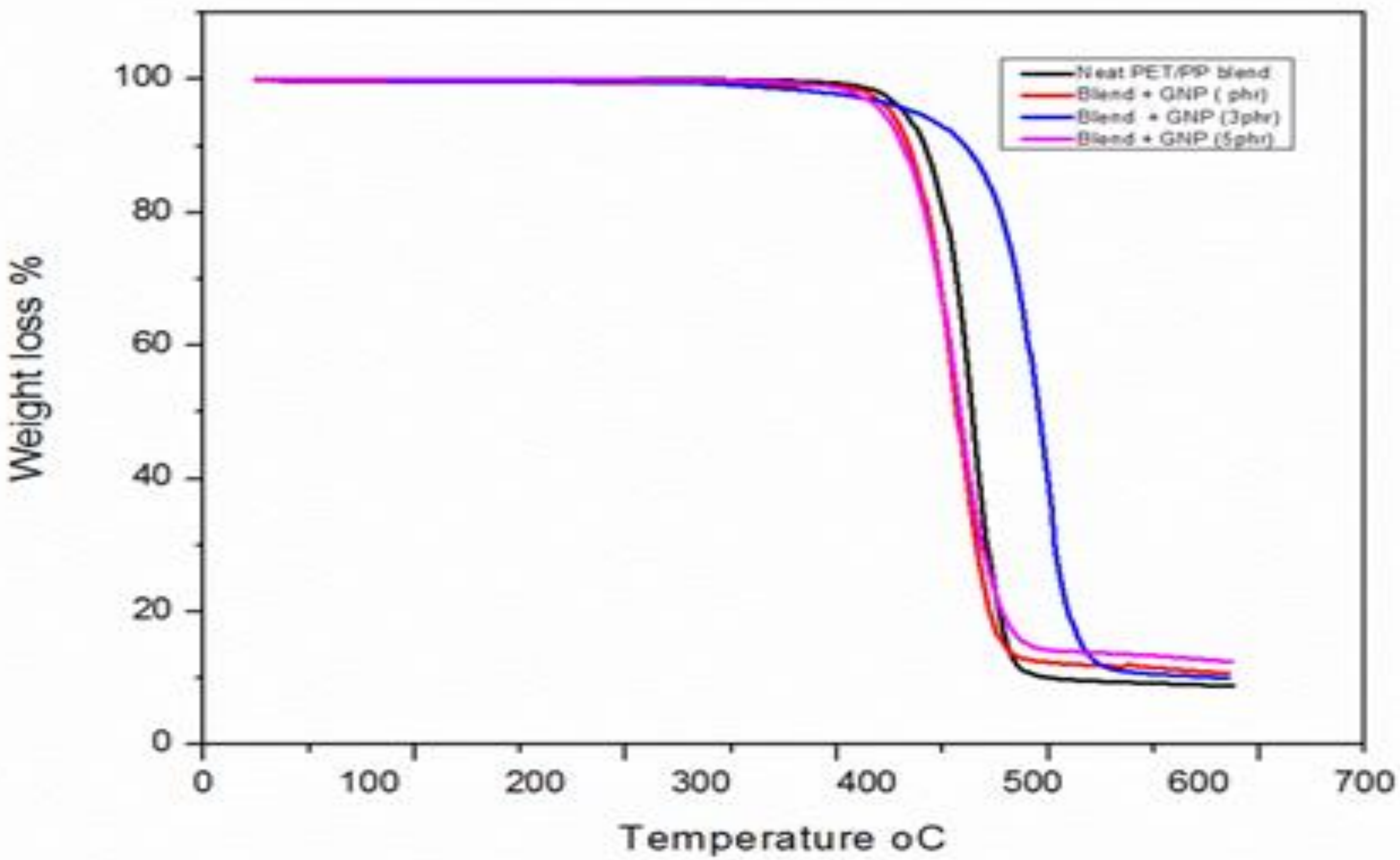
Effect of Graphene Content on Flammability Proprieties: Cone Calorimetry, LOI and UL-94

Sample Design	PHRR (kW/m^2)	TPHRR (s)	TTI (s)	CO (g/s)	FPI (TTI/PHRR)	LOI (%)	UL-94 ratings	
GNP0	643	115	20	0.01182	0.0310	21	Burning	Dripping
GNP0.5	627	115	22	0.00970	0.0351	24	V2	Moderate dripping
GNP1	572	120	25	0.00944	0.0437	26	V2	Low dripping
GNP2	553	130	27	0.00928	0.0488	27	V2	Less dripping
GNP3	534	140	30	0.00732	0.0561	28	V0	No dripping
GNP4	453	165	33	0.00678	0.0728	29	V0	No dripping
GNP5	403	170	35	0.00591	0.0868	31	V0	Not ignited

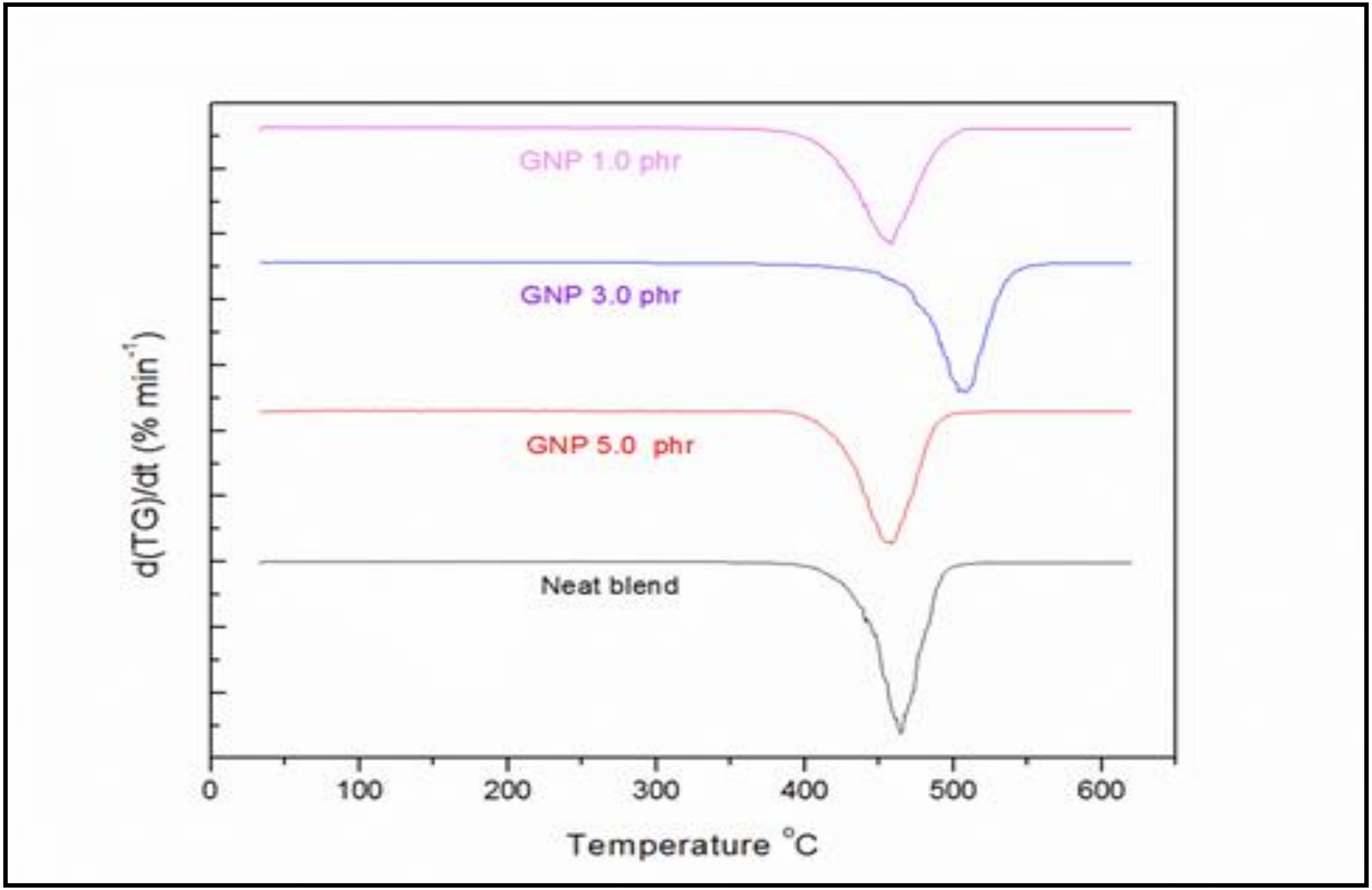
Cone calorimeter analysis: char residue



Thermogravimetry Analysis: Decomposition Temperature



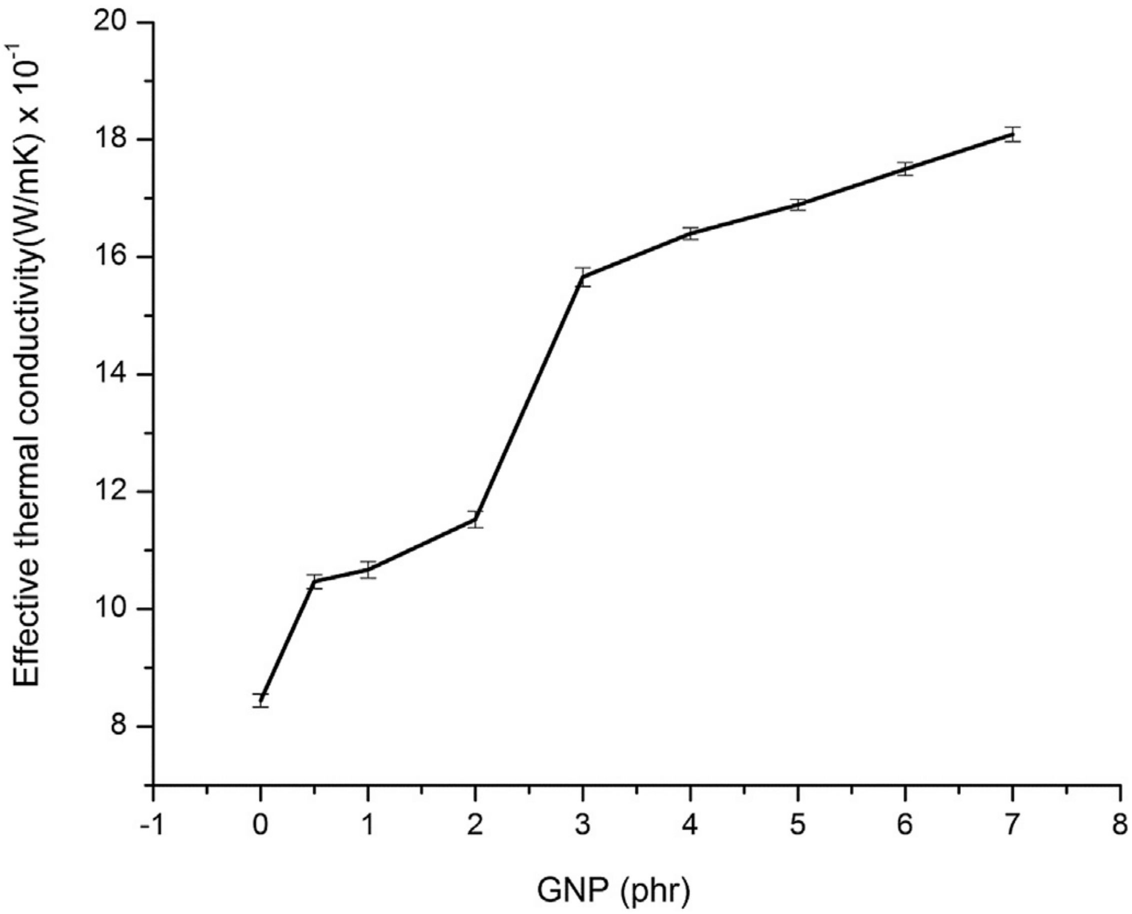
Thermogravimetry Analysis: Maximum Decomposition temperature



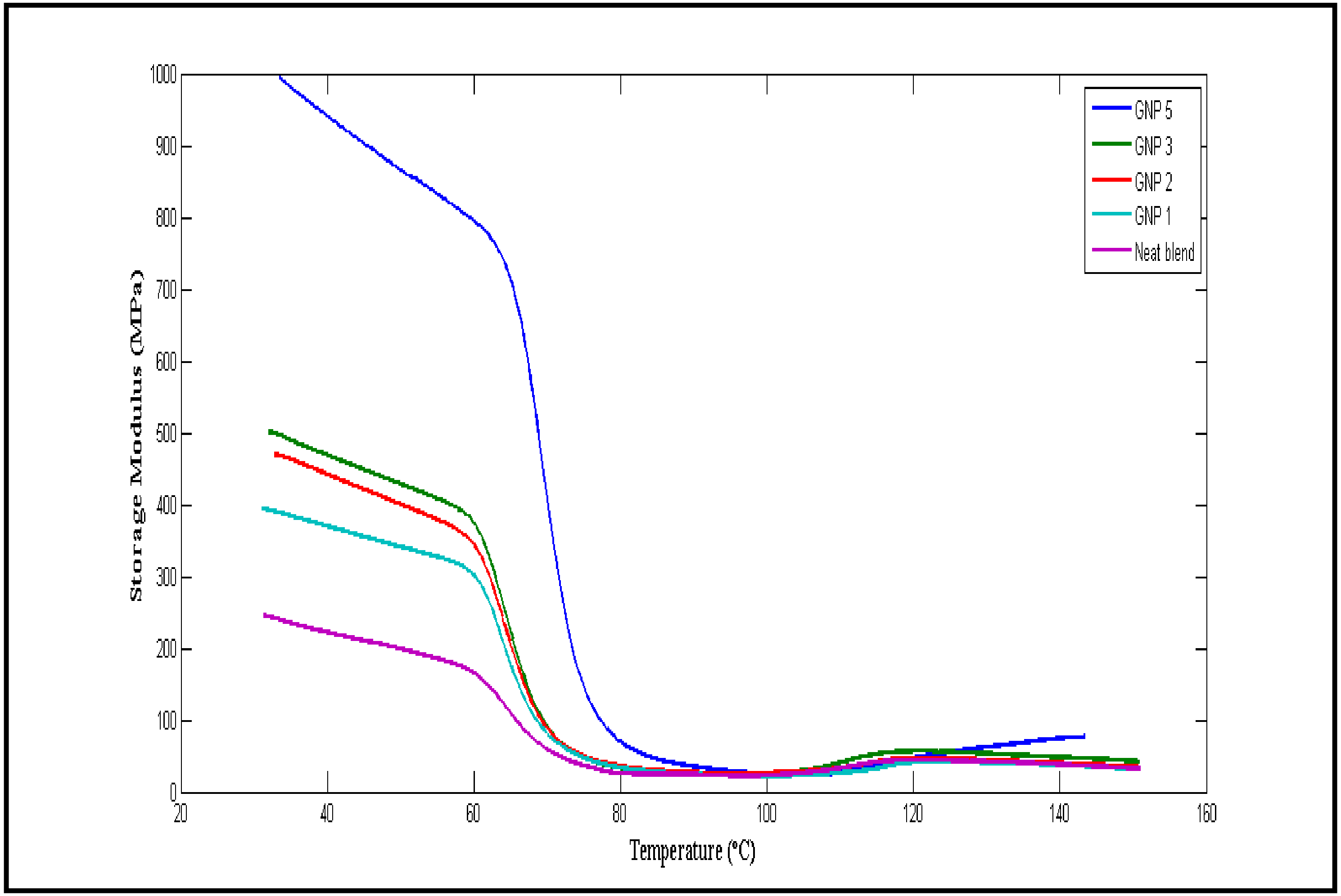
TGA data of PET/PP/GNP nanocomposites

Sample	Degradation Temp. (°C)		DTG Peak Temp. (°C)	Residual Weight (%) at 600 °C
	T_{10}	**T_{50}**	**T_{max}**	
Neat Blend	242	454	466	8.7
GNP1	426	457	457	10.1
GNP3	458	502	505	11.8
GNP5	421	456	455	12.

Thermal Conductivity



Dynamic Mechanical Analysis: Effect of GNP Content on Storage Modulus.



Conclusion

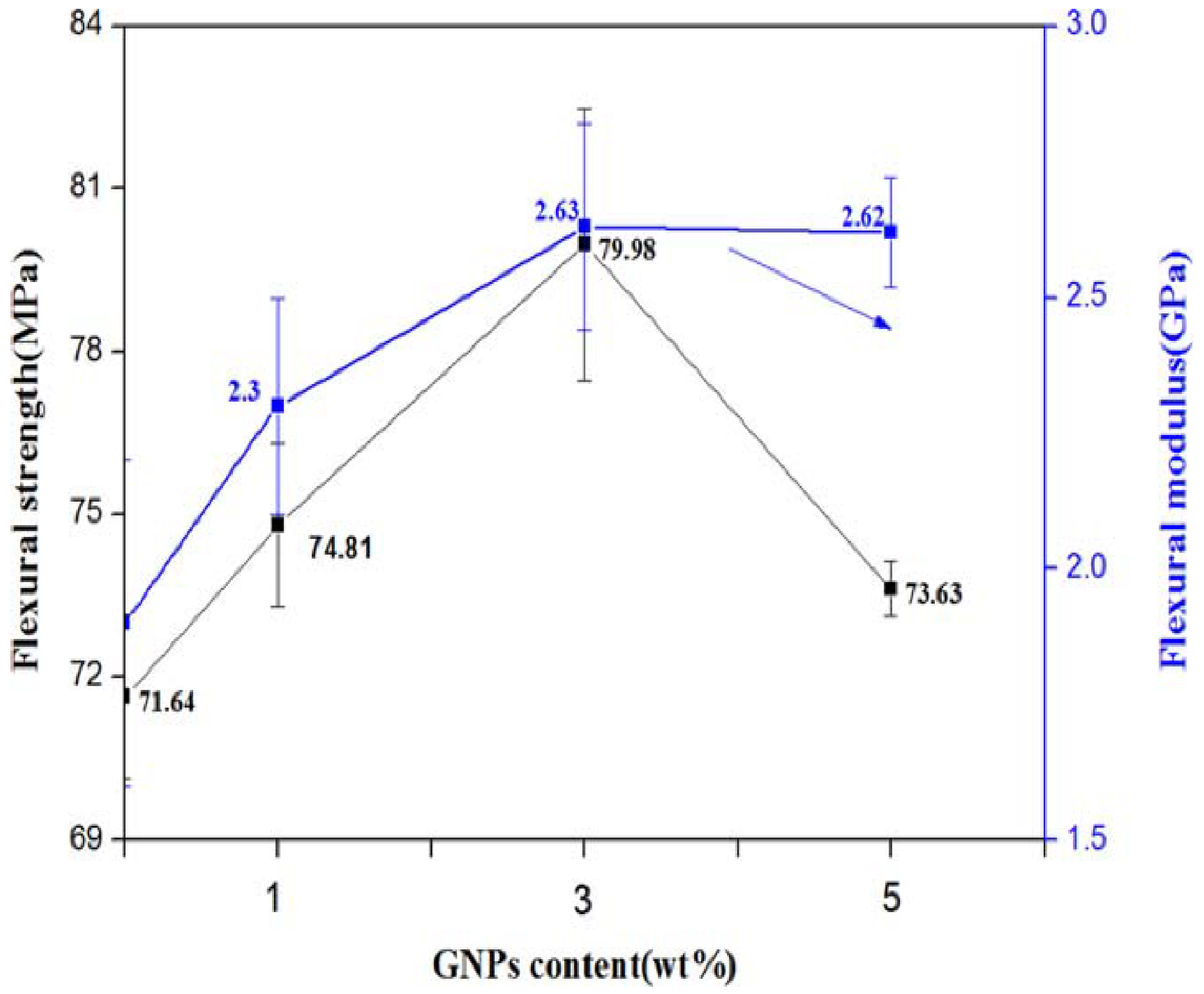
- 3 phr GNP loading was established as the optimum filler loading for mechanical and thermal properties.
- Conductivity and flammability varies directly as filler content
- TEM, FESEM, and XRD studies have shown that GNPs have uniformly and homogenously dispersed in the matrix at 3 phr.
- No significant changes in the peak positions of FTIR spectra were noticed. This indicated no substantial chemical interaction between GNP and the blends.

PC/ABS graphene reinforced Nanocomposites

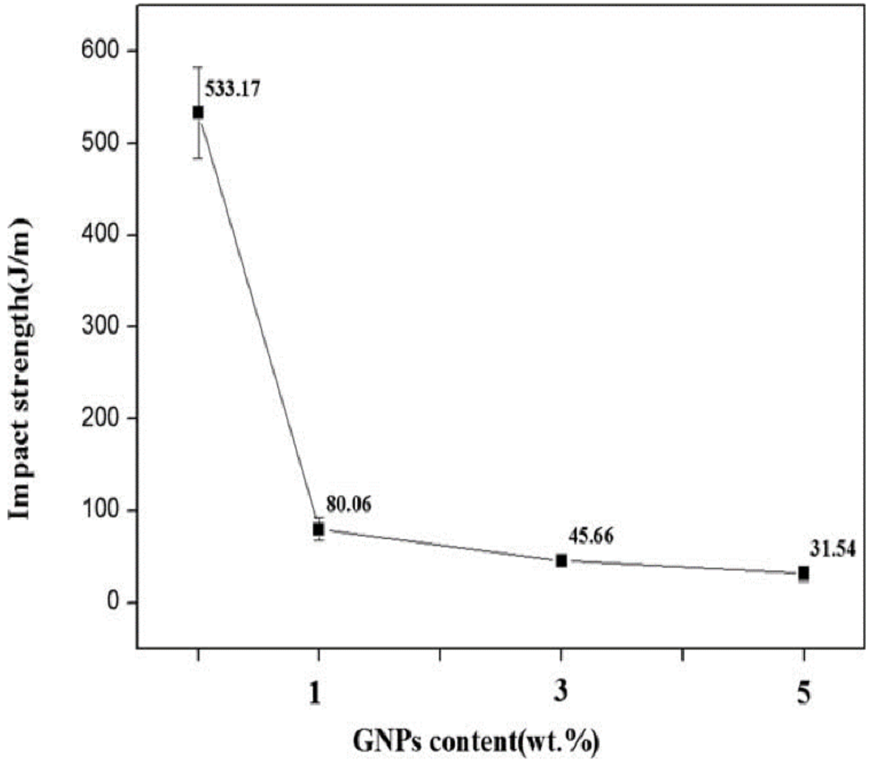
Why PC/ABS Blends?

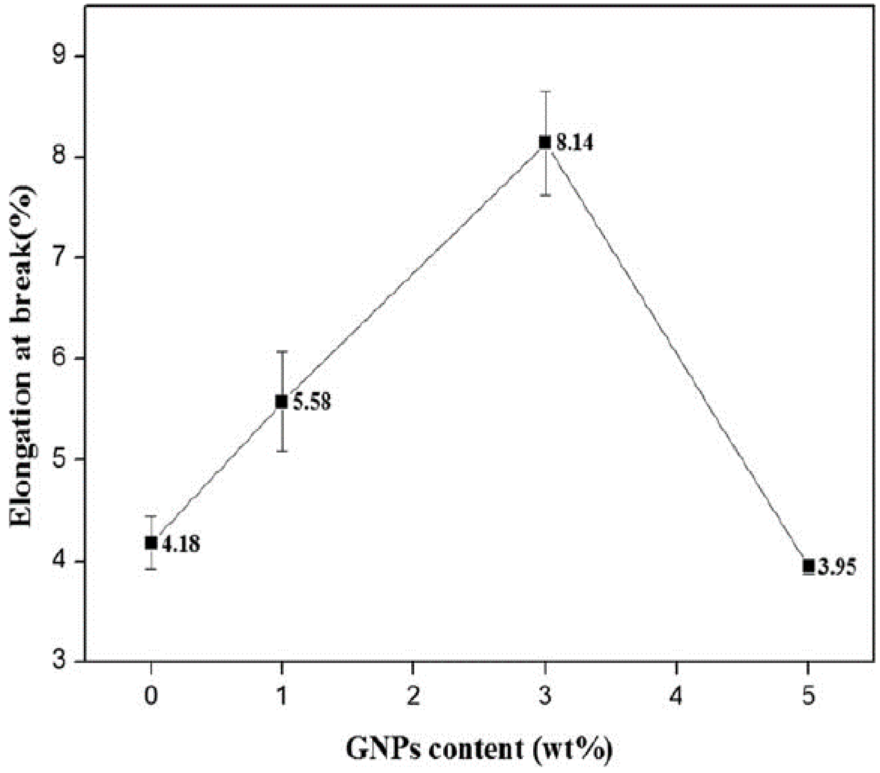
- ABS provides the benefits of economics, processability and more reliable impact resistance.
- PC contributes the improvements of tensile, flexural, thermal properties and flammability resistance to the blends.
- For PC major blends, the weak properties of PC can be overcome by ABS while maintaining the inherent good properties.
- PC/ABS blends are widely used in automotive, electronics and telecommunications applications.

Flexural strength and flexural modulus of PC/ABS/GNP nanocomposites

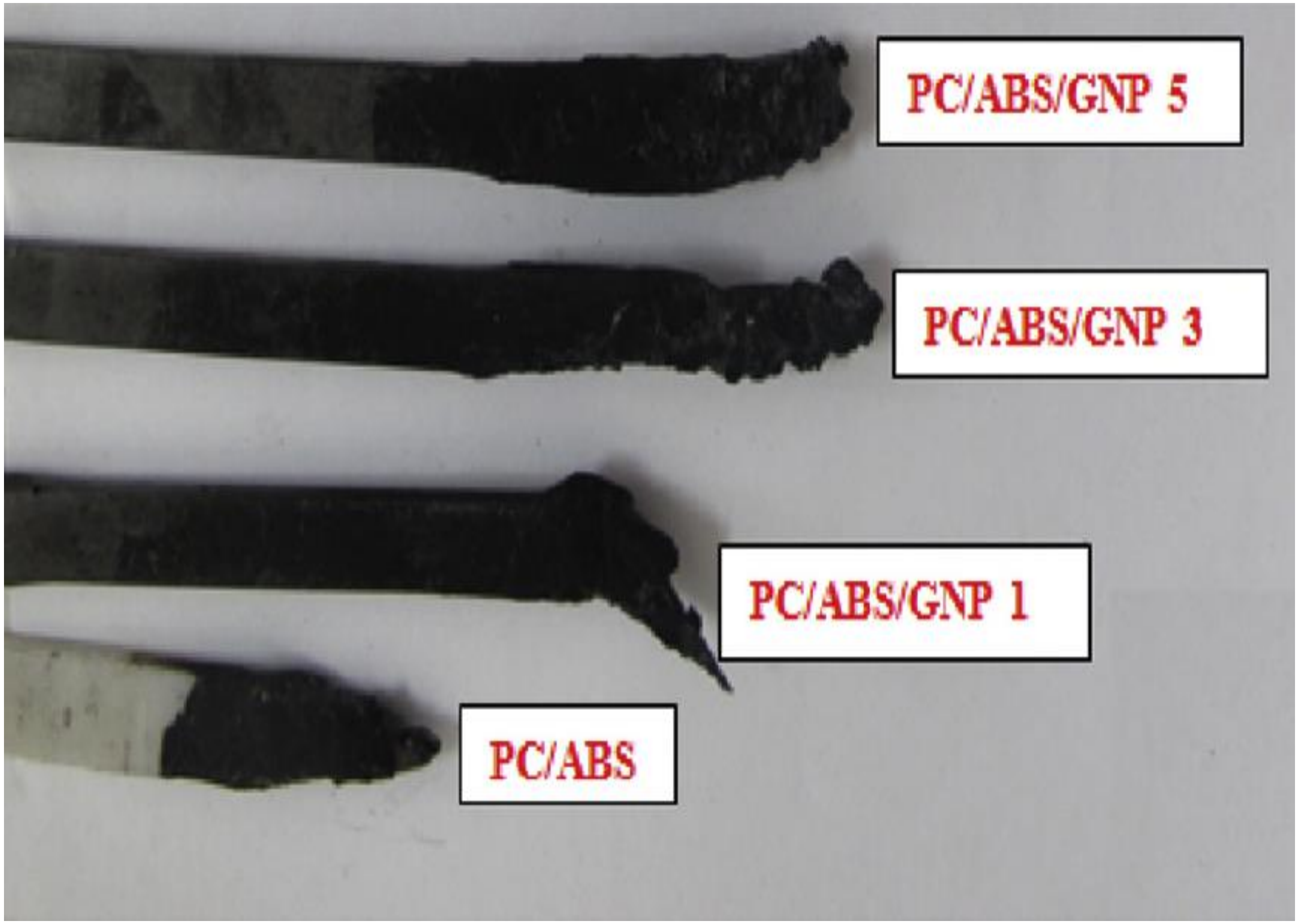


Impact strength and elongation at break of PC/ABS/GNP nanocomposites





LOI test specimens of PC/ABS and its nanocomposites after test



Interpretation of cone calorimeter data, LOI and UL-94 ratings

Compound	LOI (%)	UL-94 ratings		PHRR (kW/m^2)	THR (MJ/m^2)	Residue (wt%)
PC/ABS	23	HB	Dripping	177.5	62.1	8.7
PC/ABS/GNP1	24	HB	Moderate dripping	169.4	59.1	14.3
PC/ABS/GNP3	26	V-2	Low dripping	136.1	49.9	16.9
PC/ABS/GNP5	26	V-2	Low dripping	150.5	52.4	18.1

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Enhanced Mechanical and Thermal Properties of Hybrid Graphene Nanoplatelets/Multiwall Carbon Nanotubes Reinforced Polyethylene Terephthalate Nanocomposites

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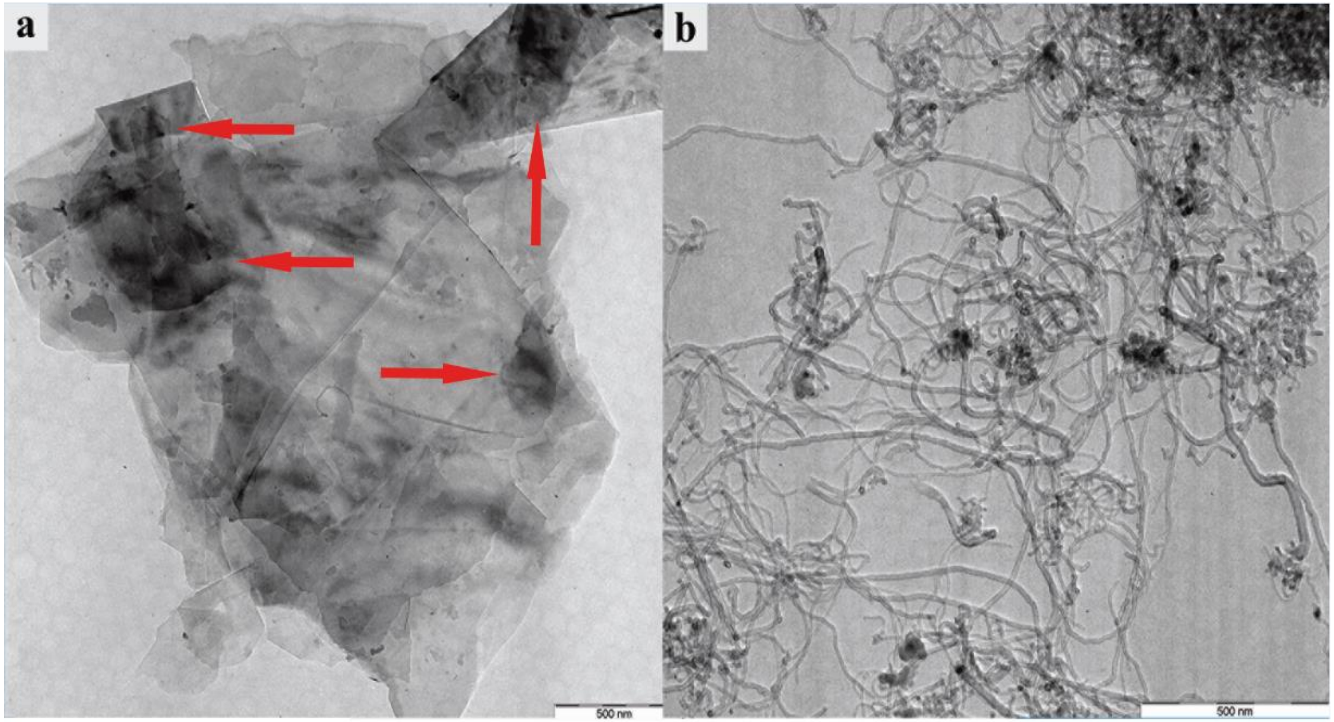
Flexural and tensile properties of neat PET and PET nanocomposites

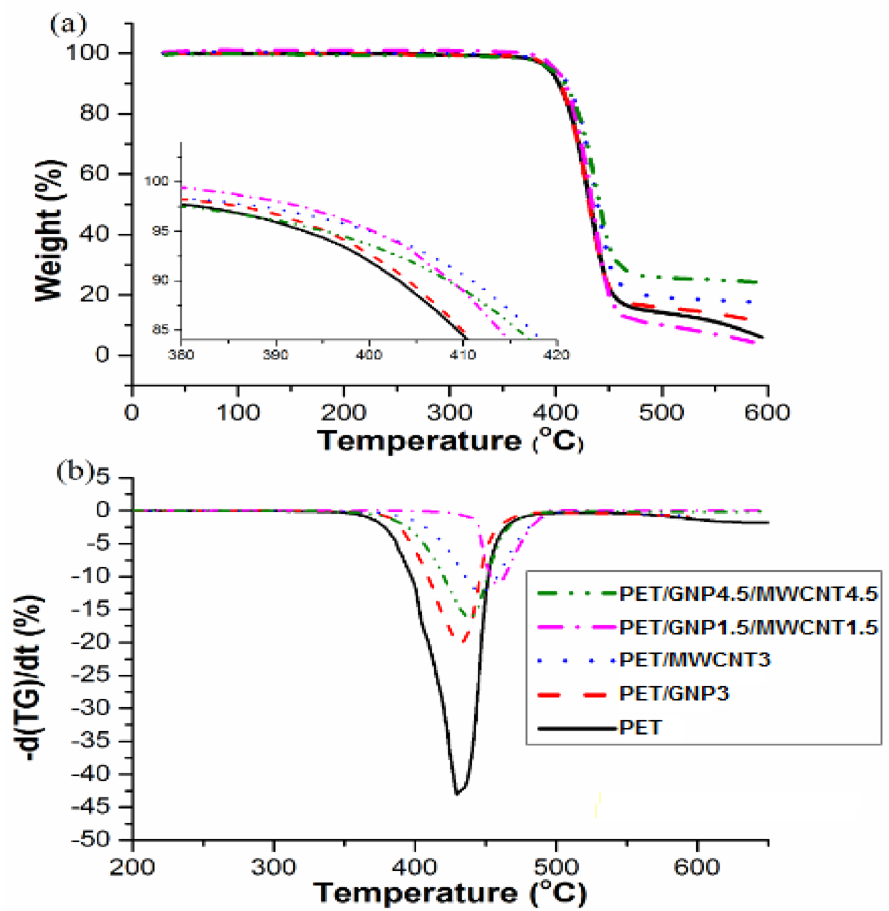
Sample name	Flexural strength (MPa)	Flexural modulus (GPa)	Tensile strength (MPa)	Young's modulus (GPa)	Elongation at break (%)
PET	75.9 ± 21.5	2.7 ± 0.2	32.1 ± 11.4	1.1 ± 0.1	3.9 ± 1.0
PET/GNP3	78.3 ± 6.9	3.3 ± 0.2	51.6 ± 3.9	2.8 ± 0.1	2.6 ± 0.3
PET/MWCNT3	77.5 ± 7.5	3.0 ± 0.1	44.4 ± 7.9	2.5 ± 0.4	2.5 ± 0.5
PET/GNP1.5/MWCNT1.5	84.5 ± 13.1	3.4 ± 0.2	57.4 ± 9.4	2.3 ± 0.6	3.0 ± 1.2
PET/GNP4.5/MWCNT4.5	75.7 ± 16.0	3.5 ± 0.3	44.0 ± 7.8	2.7 ± 0.7	2.5 ± 0.8

TEM micrographs of (a) GNP and (b) MWCNT powders

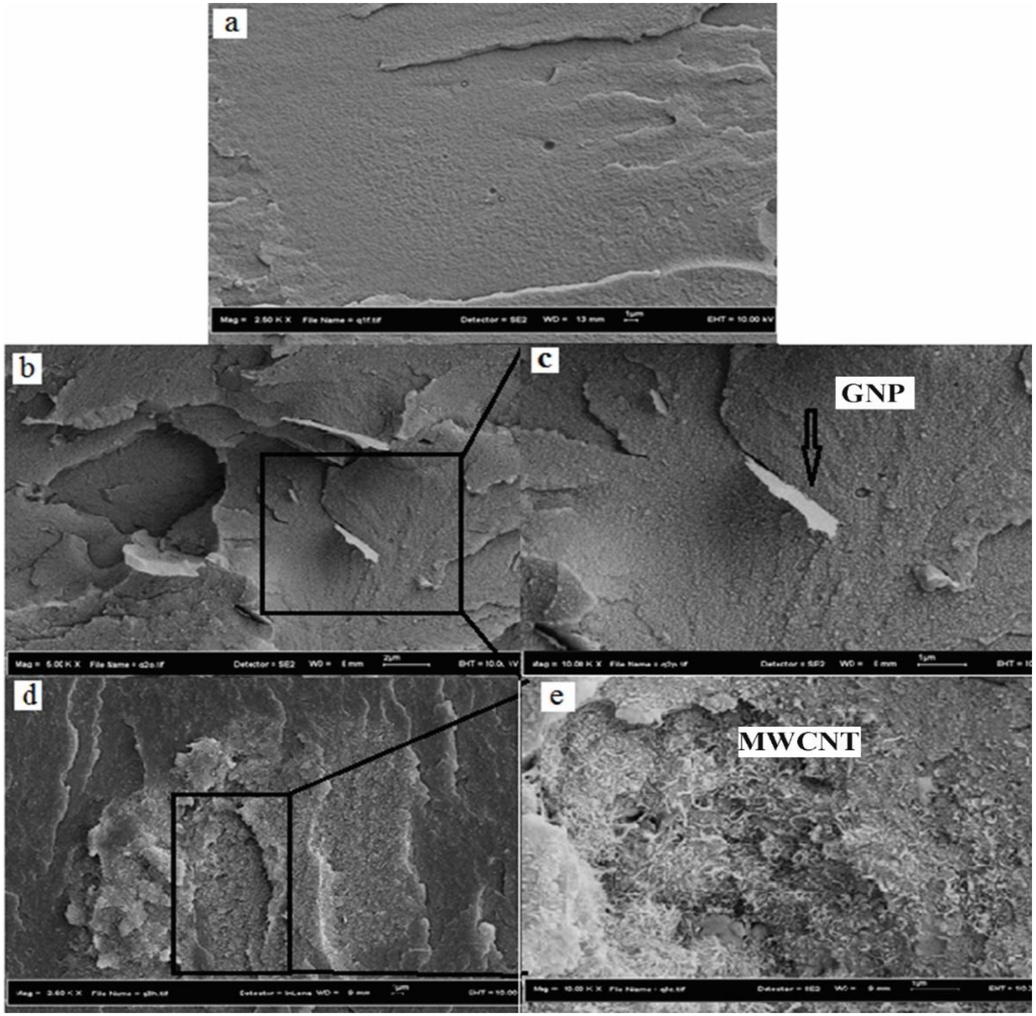


TGA and DTG thermograms of PET/GNP/MWCNT hybrid nanocomposites

Designation	T_{onset} (˚C)	T_{max}	Char Residue(%)
PET	380.0	425.9	6.1
PET/GNP3	388.7	432.6	11.1
PET/MWCNT3	391.7	452.3	17.5
PET/GNP1.5/MWCNT1.5	393.4	468.4	23.8
PET/GNP4.5/MWCNT4.5	391.0	439.6	24.1

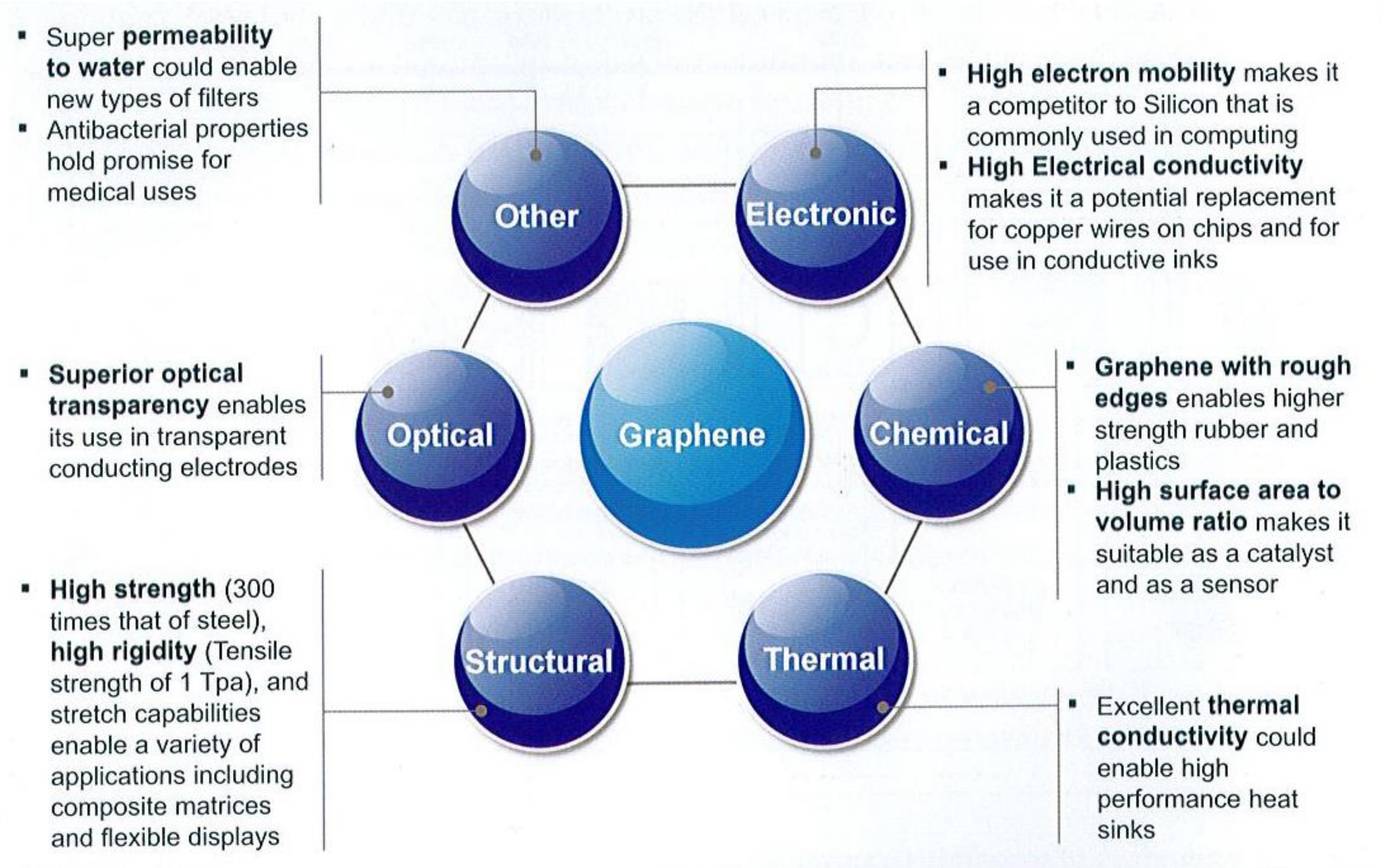


FESEM images showing the morphology of fractured surface of the PET nanocomposites; (a) neat PET, (b) PET/GNP3 nanocomposite, (c) magnified section of (b), (d) PET/MWCNT3 nanocomposite and (e) magnified section of (d).

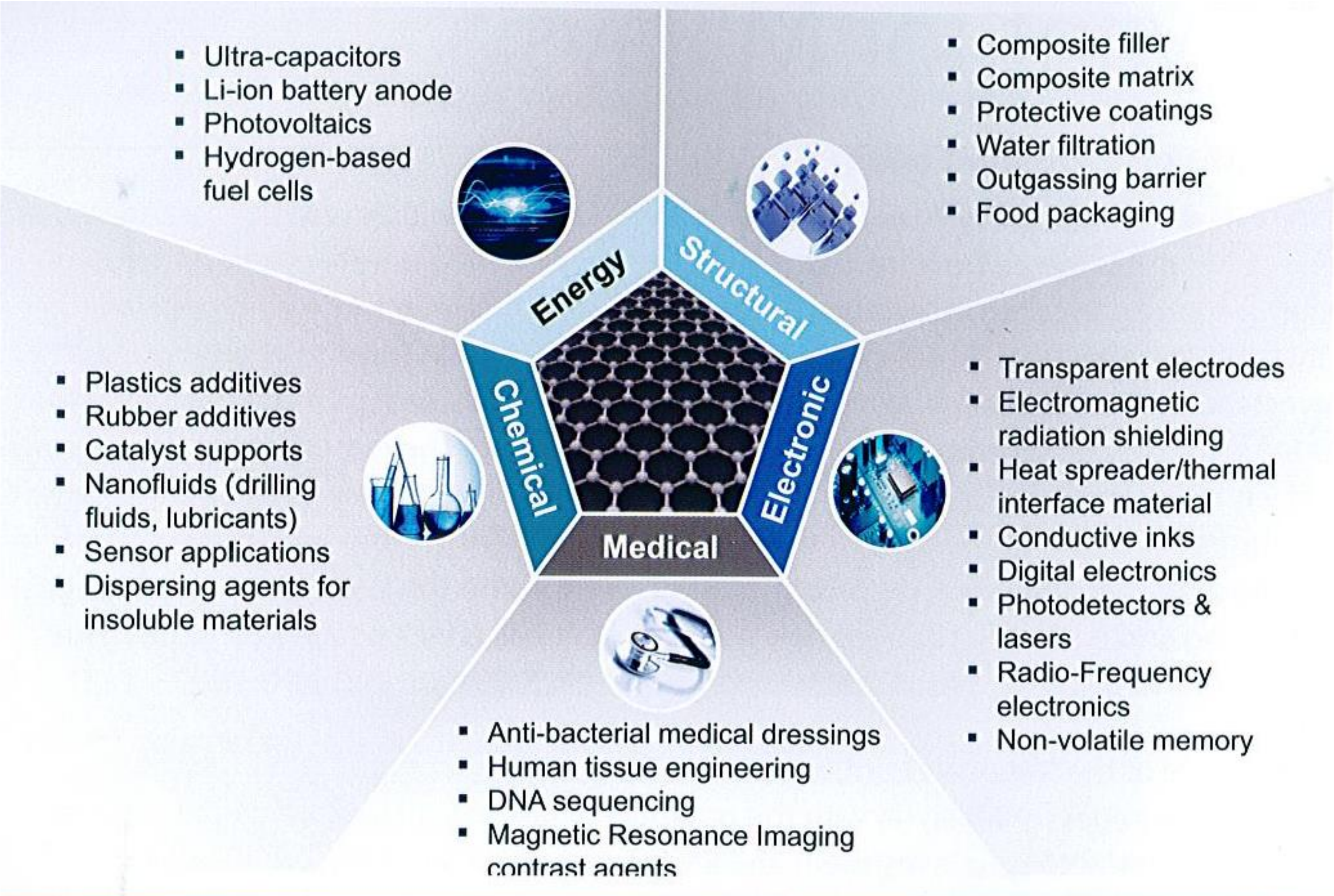


Summary and Future Outlook

Graphene: unique combination of properties



Graphene Applications Evaluated for Technical Feasibility



- Many of the current and potential applications of carbon nanotubes may be replaced by graphene at much lower cost.

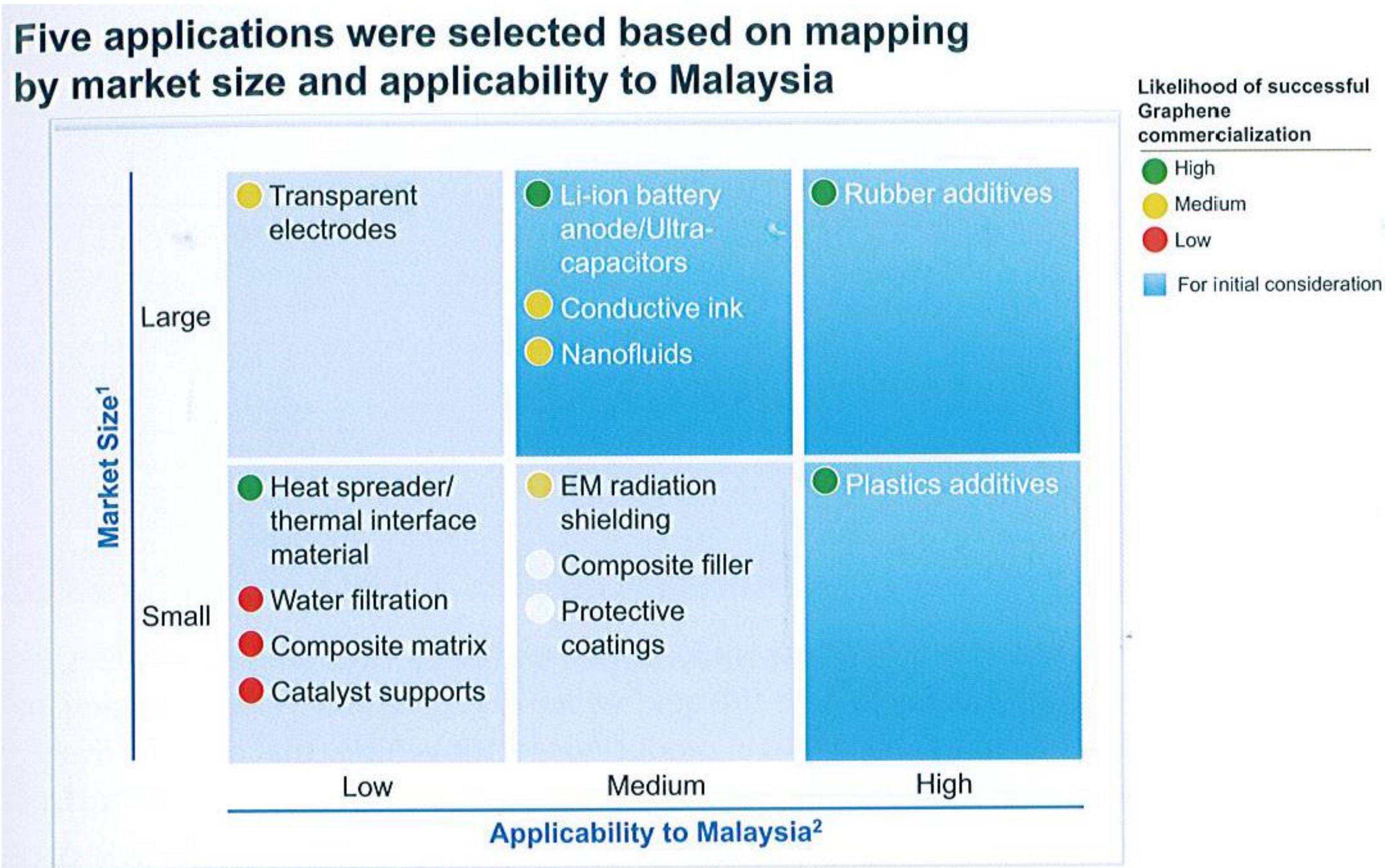
- Compatibility of graphene with polymers need to be further improved to form homogeneous composites with superior properties. Surface modification of graphene can be used to enhance graphene–polymer interaction.

- Based on the huge interest, enhanced properties as well as ease of production and handling, various countries are allocating huge funds:

✓ European Union: 10 year $1.73 billion coordination action on graphene.
✓ South Korea: $350 million on commercialization initiatives
✓ United Kingdom: $76 million in a commercialization hub.

▪ Studies on hybridization with other nanofillers and using polymer blends as matrices are also worth pursuing

Potential applications in Malaysia based on market size.



Thank You