

Advanced Materials and Technologies for Aerospace Applications

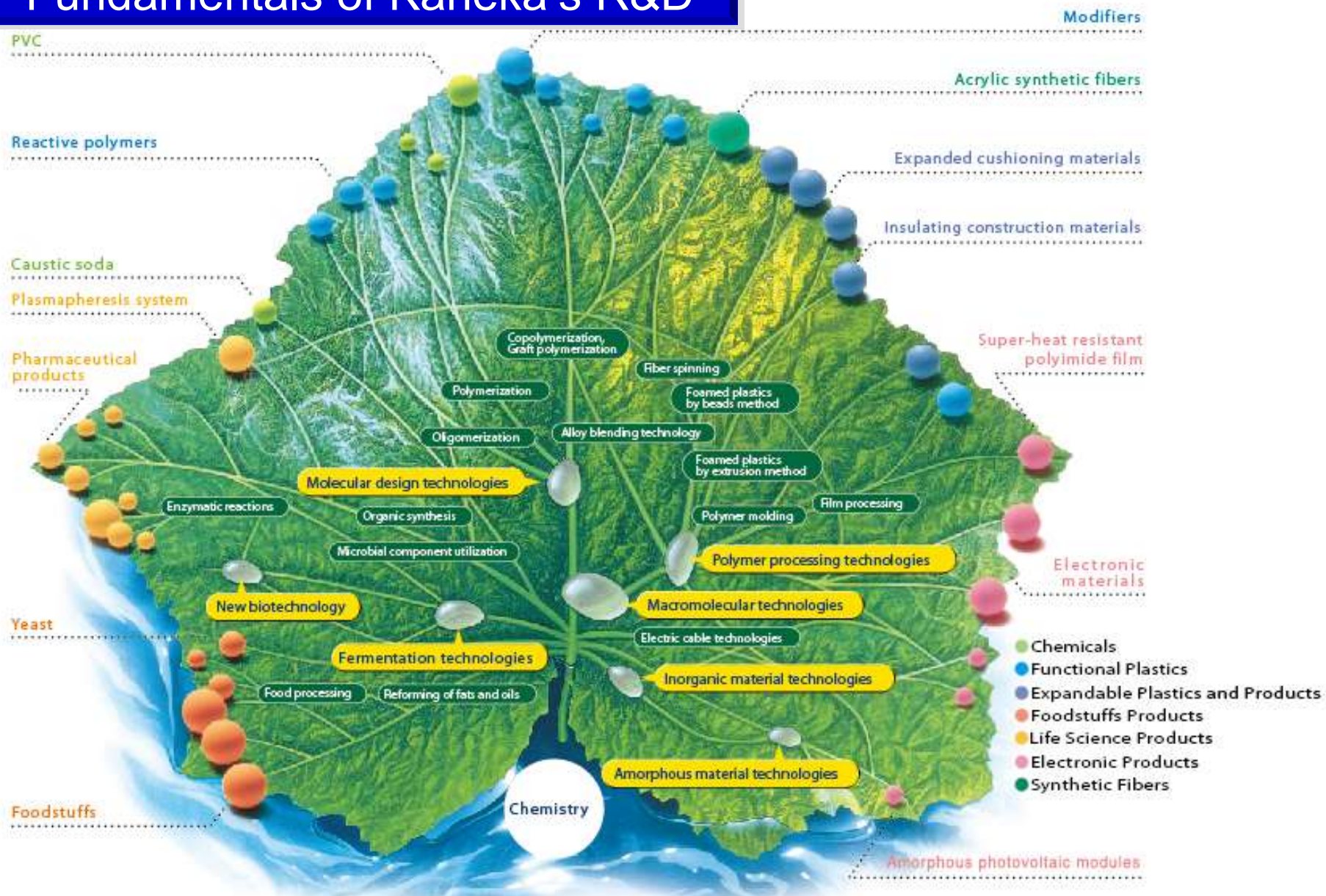
Masaya Kotaki

Kaneka US Materials Research Center
Kaneka Americas Holding, Inc.

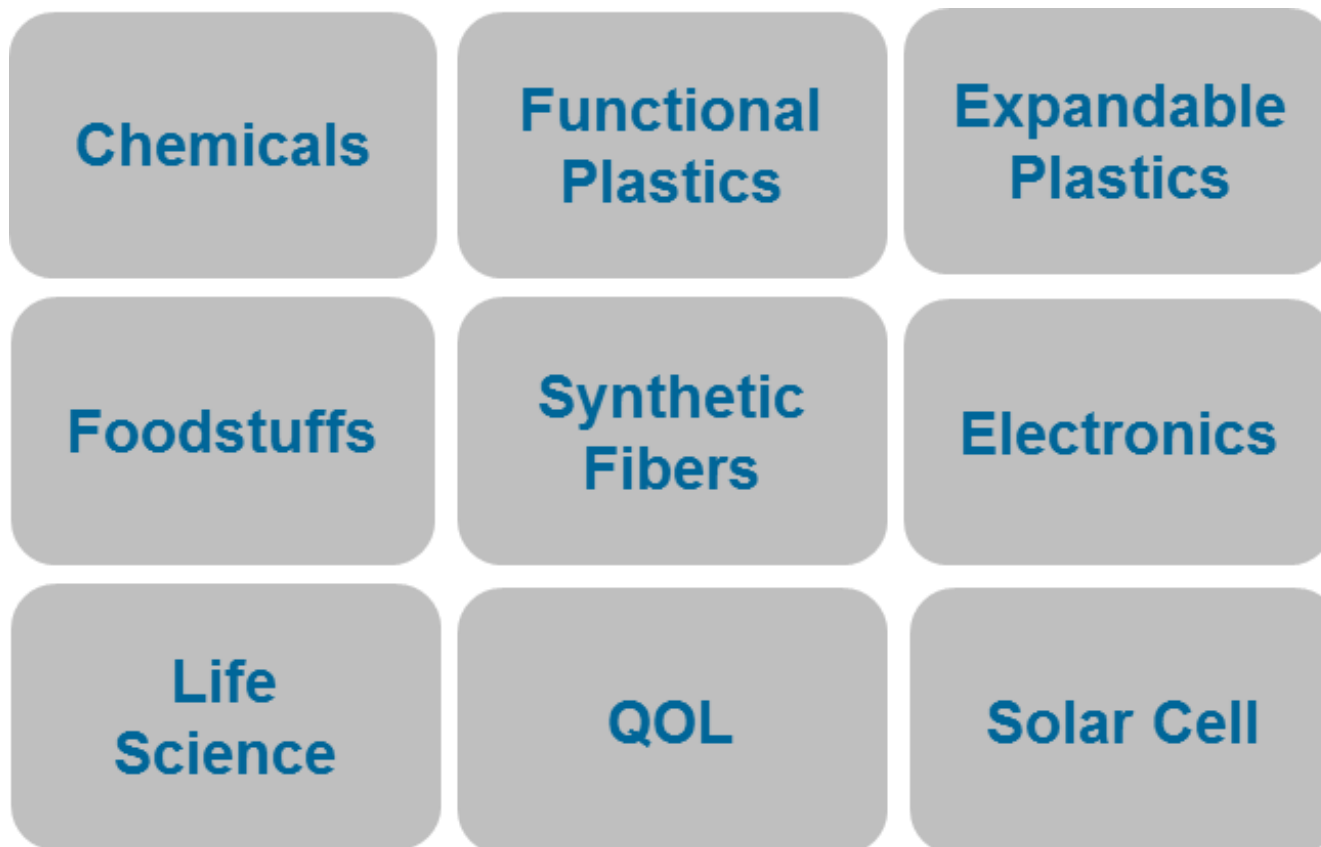


Toward an Even More Impressive and Productive Future

Fundamentals of Kaneka's R&D



Current Business Fields



Business Fields & Products ①

● Chemicals



Caustic Soda



Kanevinyl™



Kanevinyl™ Paste



Kaneka™ CPVC



Vinyl Chloride-Vinyl Acetate Copolymers



Crosslinked PVC

● Functional Plastics



Kane Ace™ B



Kaneka™ MS Polymer



Kaneka Silyl™



Kanevilack™



Kaneka Hyperite™



Sunduren™

● Expandable Plastics and Products



Eperan- PPT™



Eperan™



Kanepearl™



Kanepearl™ Soil Block



Kanelite Foam™



Kanelite™ Insert

Business Fields & Products ②

● Foodstuffs Products



Kaneka™ Margarine
Kaneka™ Shortening



PAPRÈ



Kaneka™ Yeast



LACHENTE,FRANJE



Belco



Spices

● Life Science Products



Kaneka
Plasmapheresis
System



Intervention catheters



Pharmaceutical
Intermediates



Kaneka Q10™

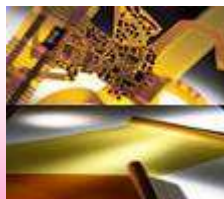


Kaneka QH™

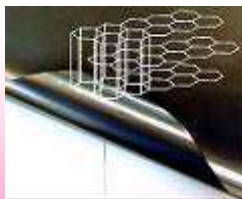


Kaneka Glavonoid™

● Electronics Products



Apical™



Graphinity™



Elmech™



Kaneka Flux™



Photovoltaic solar
power generation
system



Photovoltaic power
generation systems
for public and
industrial installation



Solar Circuit™

Business Fields & Products ③

● Synthetic Fibers



Kanekalon™, Kanecaron™



Protex™



ULTIMA™

● Business Development



“KANEKA Biopolymer AONILEX™”
(a polyester biopolymer
comprised primarily of plant oil)



Thermal solution materials
for use in electronic
device components



New heat-resistant materials
for use in optical device
components
“ILLUMIKA™”



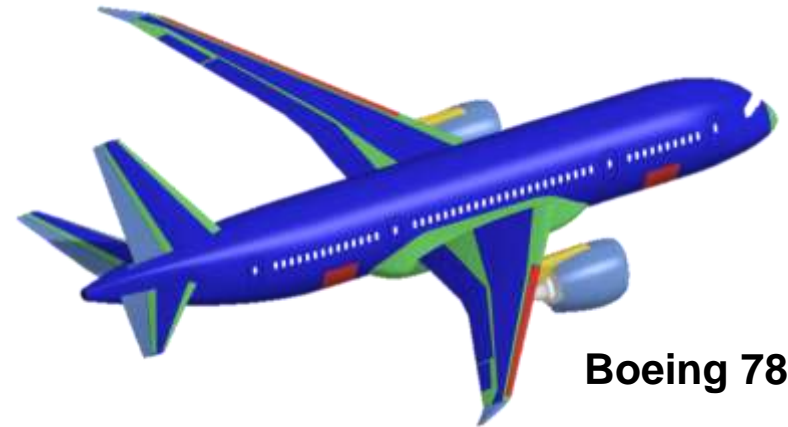
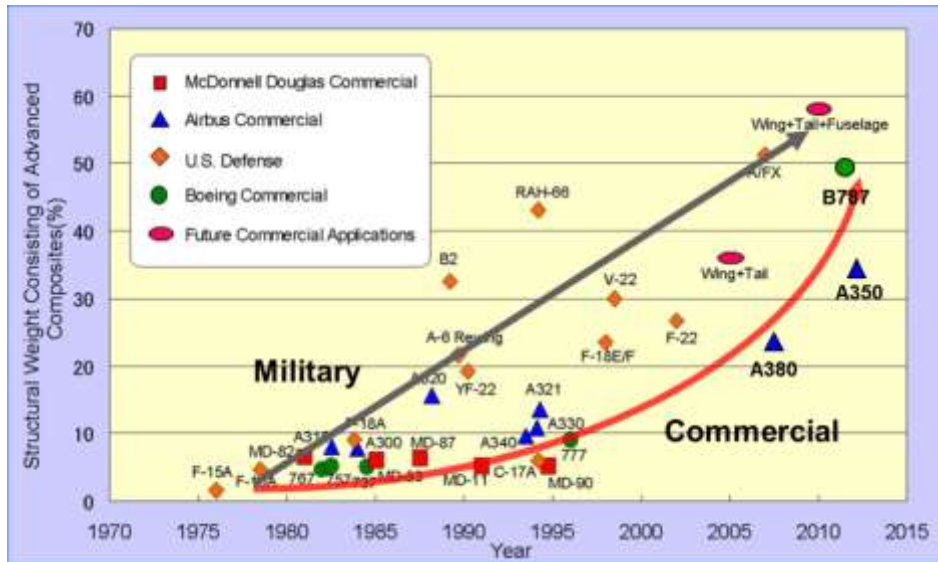
OLED Lighting Devices

Contents

- **Polyimide**
- **Toughening Agent (CSR)**
- **Graphite Sheet**
- **Nanocomposites**

CFRPs in aircrafts

- Epoxy(EP)/CFRPs and EP/GFRPs : approximately 50 % of structural weight of aircrafts
- Long-term service temp. of EP/CFRPs : **up to 120°C** due to thermal stability.
- **Polyimides** : Excellent environmental, thermal (>200°C) and mechanical properties
- **Polyimide CFRPs** : Replacement of titanium parts in jet engine

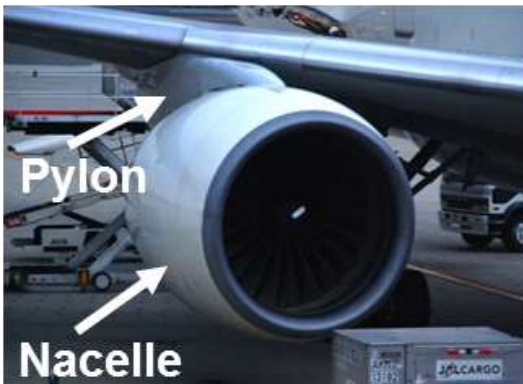


Boeing 787

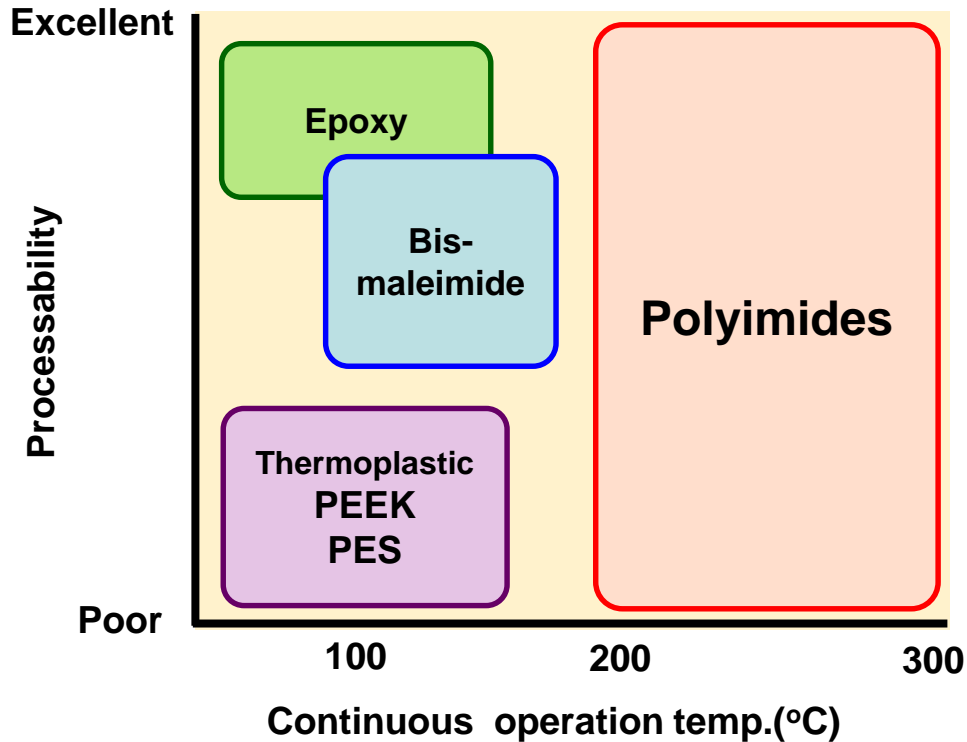
■ CFRP ■ GFRP
■ GFRP ■ Steel, Titanium(>200°C)

Source: Advanced composites engineering (2005) Baifukan

◆ Application examples of PI/CFRPs



Matrix resins for CFRPs



<Epoxy>

- Excellent processability
- Lower price

<Thermoplastic resin>

- Secondary molding
- High impact strength
- Inferior processability

<Bis-maleimide>

- Good processability
- Low toughness
- Low thermal oxidation stability

<Polyimide>

- High thermal and environmental stability
- Lower processability
- Higher price

Kaneka moldable imide resin for high temperature CFRPs

◆ Kaneka polyimide film APICAL®



APICAL® Polyimide film



Flexible Printed Circuitry



Wire and Cable coating

◆ Thermal and mechanical properties of Kaneka thermosetting “MOLDABLE” imide resin

		PMR-15	PETI-5	AFRPE	Kaneka	
Mn		1500	5000	1600	2500	4000
Uncured Imide oligomer	Min. melt viscosity (poise)	5000	60000	10	1500	5000
Cured Imide resin	T _g (°C)	343	270	370	372	340
	Modulus (GPa)	3.0	3.1	3.4	3.2	2.8
	Elongation at break(%)	1	32	2	17	19



Imide oligomer
solution



Imide oligomer
powder



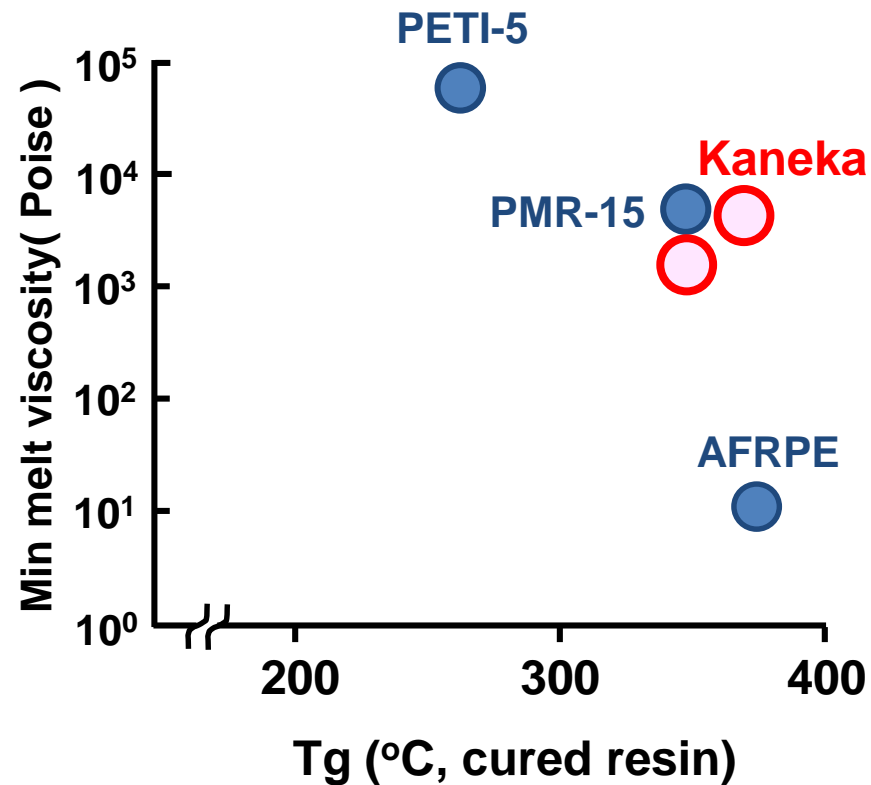
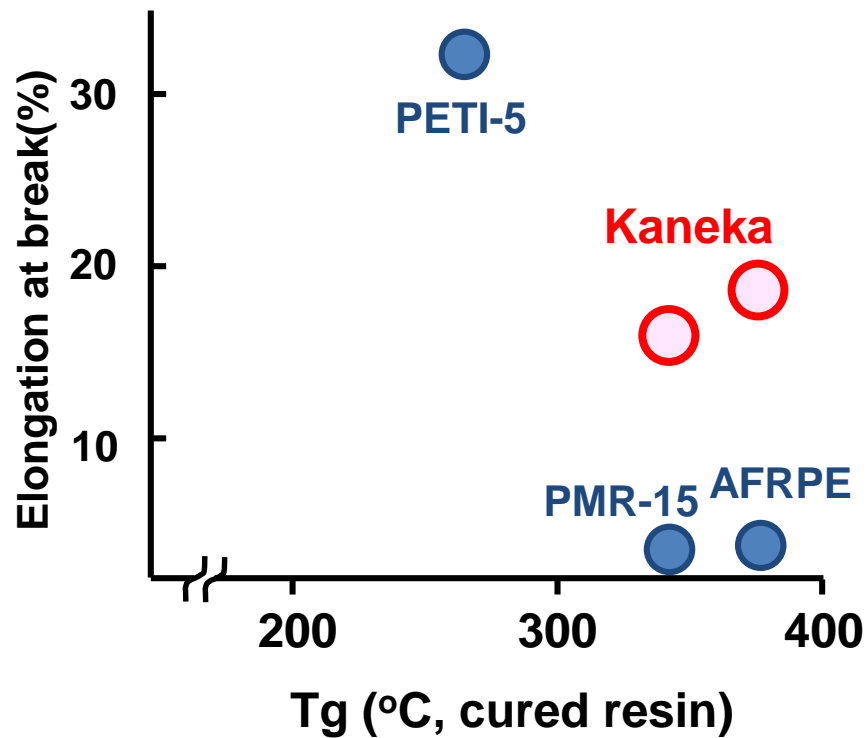
Cured resin film



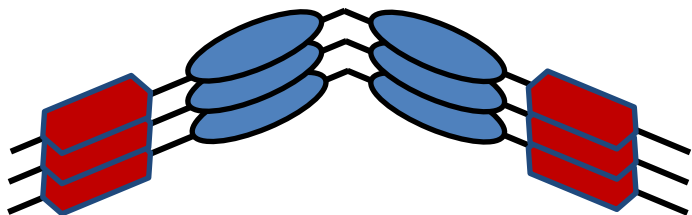
Molding

These data are not guaranteed values and may be varied by the future tests.

Thermosetting polyimides for composites



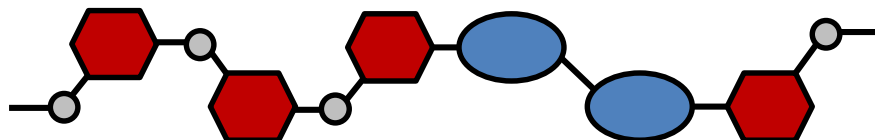
PMR-15



- Symmetric, planar rigid units
- Aggregation between rigid units
- Low molecular weight

⇒ High Tg and brittle

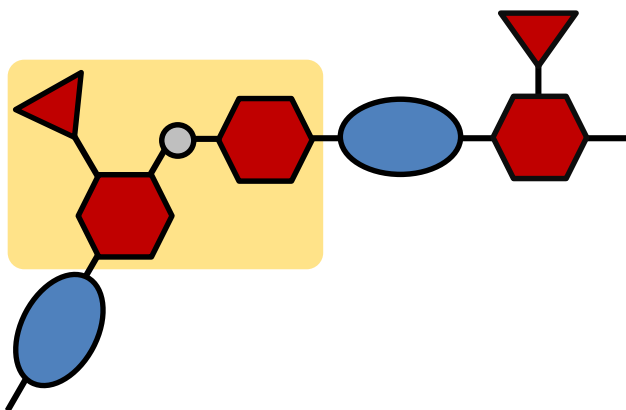
PETI-5



- *meta*-Linkage and multi-flexible ether bonds
- Disturbed regularity by copolymerization
- High molecular weight

⇒ Mid. high Tg and high elongation

Kaneka

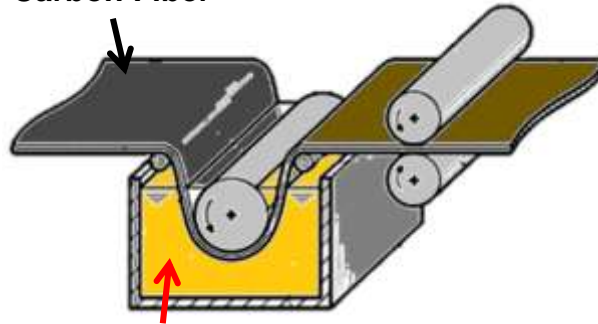


- **Asymmetric monomers with pendant group**
 - Prevention of the molecular aggregation
 - Disturbed regularity of repeated units
- Mid and high molecular weight

⇒ High Tg and excellent processability

◆ Wet prepreg (solvent wet-type)

Carbon Fiber



Impregnation

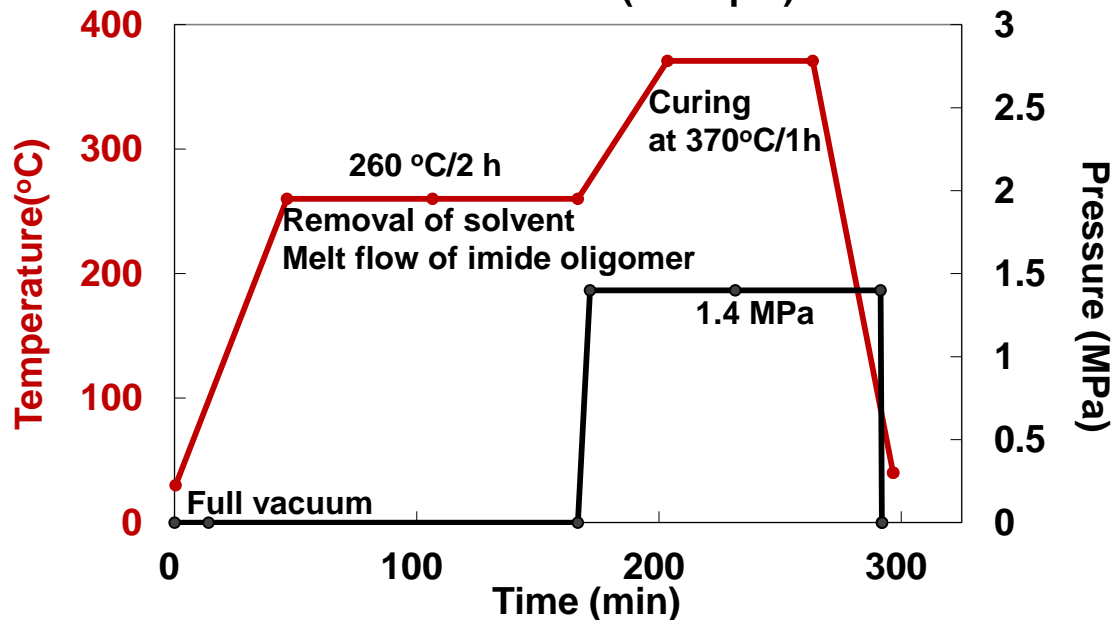
- Imide oligomer/ NMP solution
- Monomers/ alcohol solution



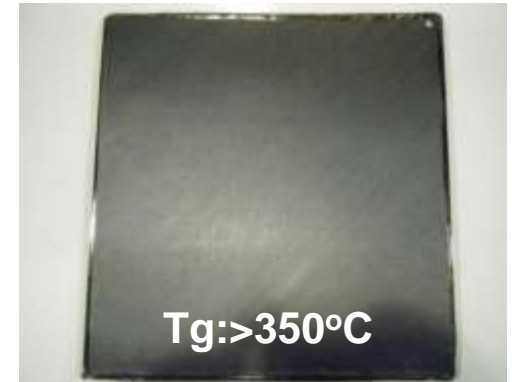
Continuous PI/CF prepreg

◆ Molding of PI CFRPs

Cure Process (example)

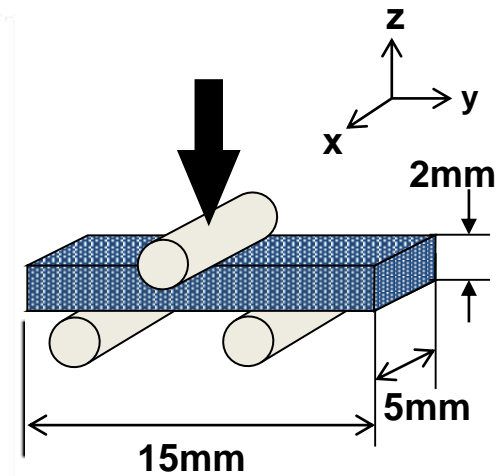
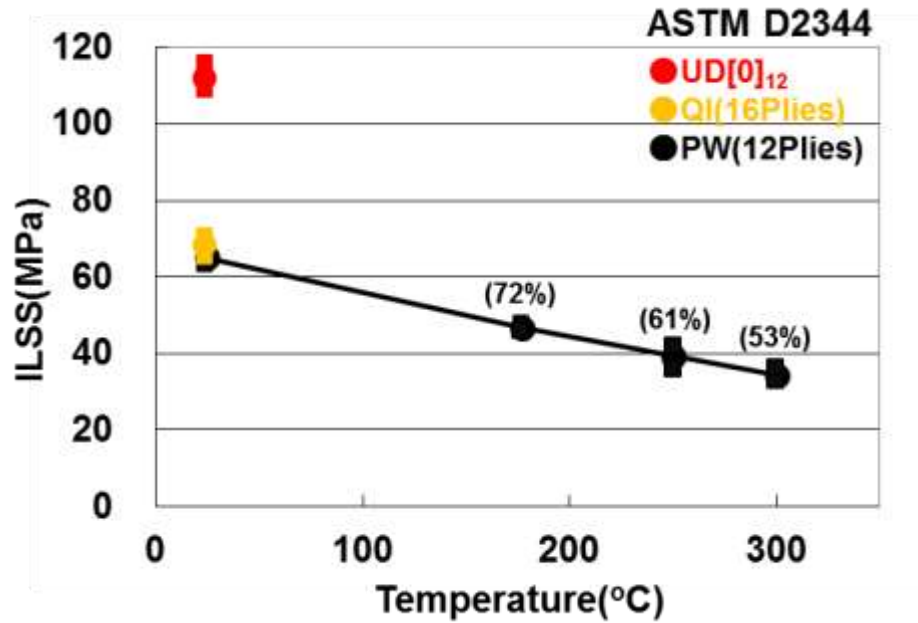


Autoclave

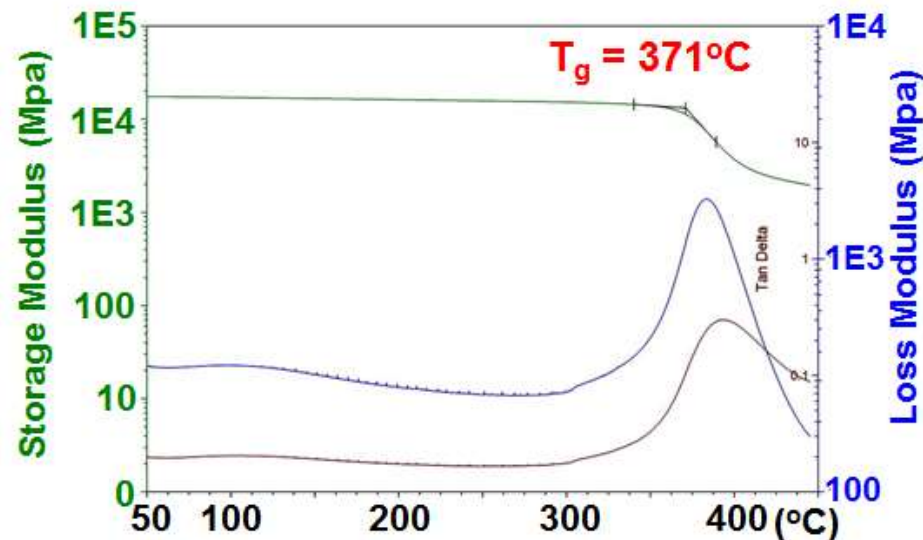


Tg:>350°C

Short beam share

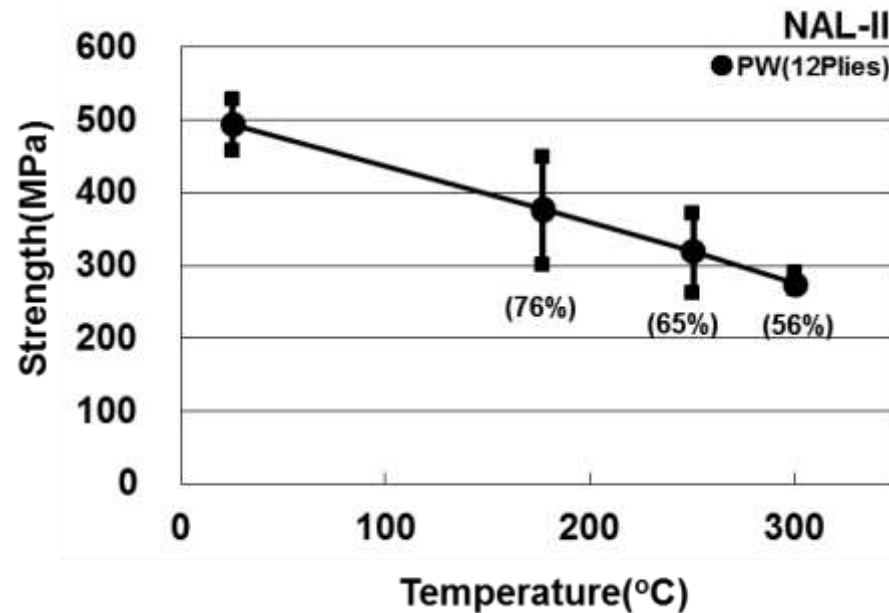
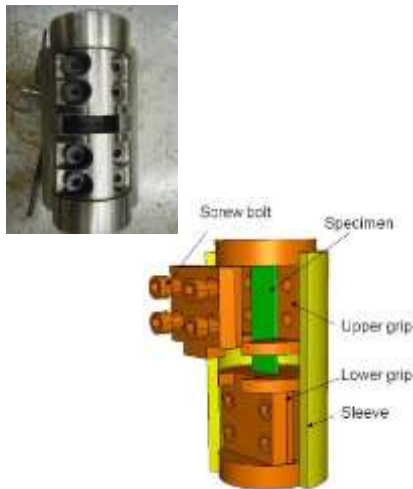


DMA data

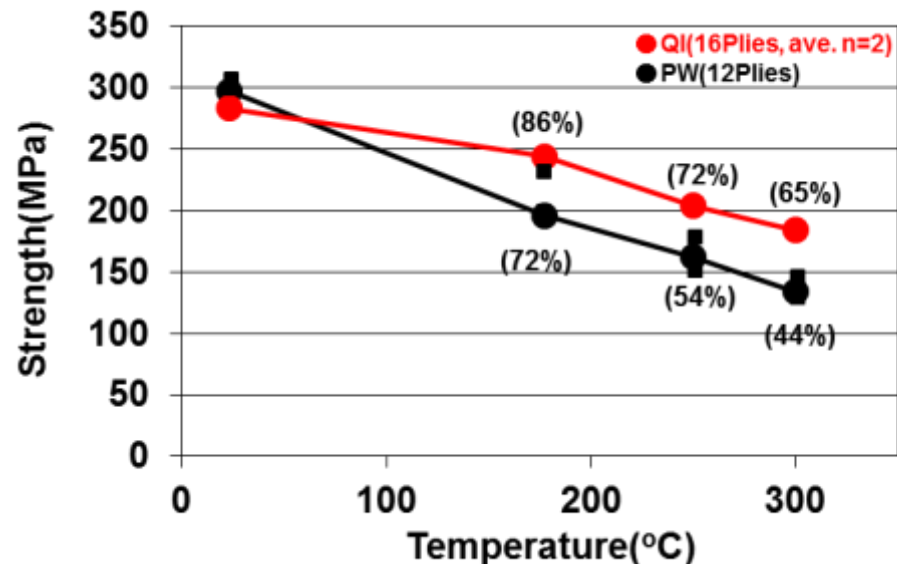


Non- and Open-hole compression tests

Non-hole compression



Open-hole compression

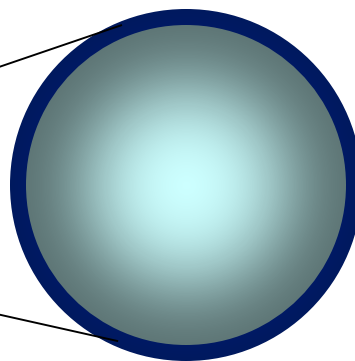
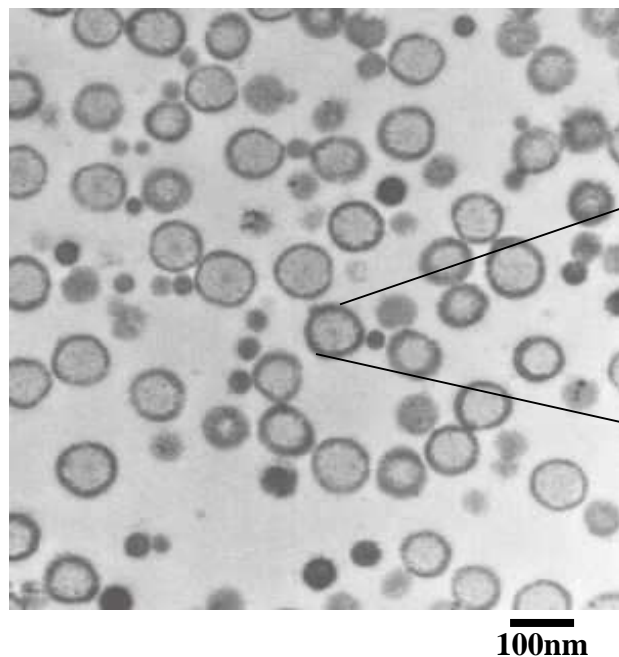


Kane Ace MX®

Core-Shell Rubber (CSR) Toughener for
Thermosetting Resin Systems

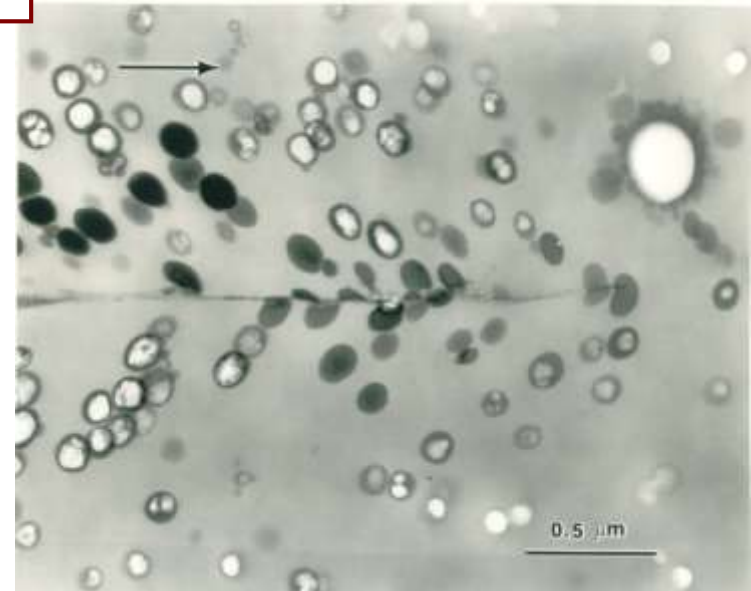
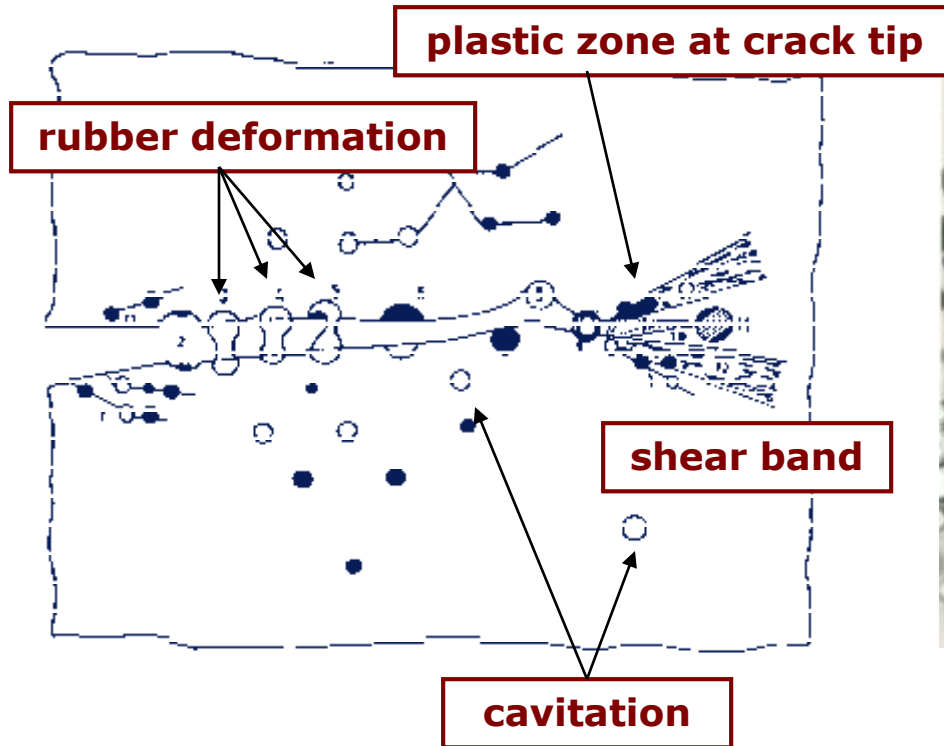
What is Core-Shell Rubber (CSR)?

The structure of a core-shell rubber particle consists of a cross-linked rubber core encased by a hard “glassy” shell.



Performance can be optimized by tailoring the structure, chemistry, particle size and distribution, as well as shell functionality.

How Does CSR Toughen?



**Rubber Cavitation and Shear Yielding
(epoxy resin/CSR)**

What is Kane Ace[®] MX?

Kane Ace[®] MX is a family of user-friendly concentrates comprised of proprietary core-shell rubber (CSR) particles pre-dispersed into thermosetting resins or other liquid media.



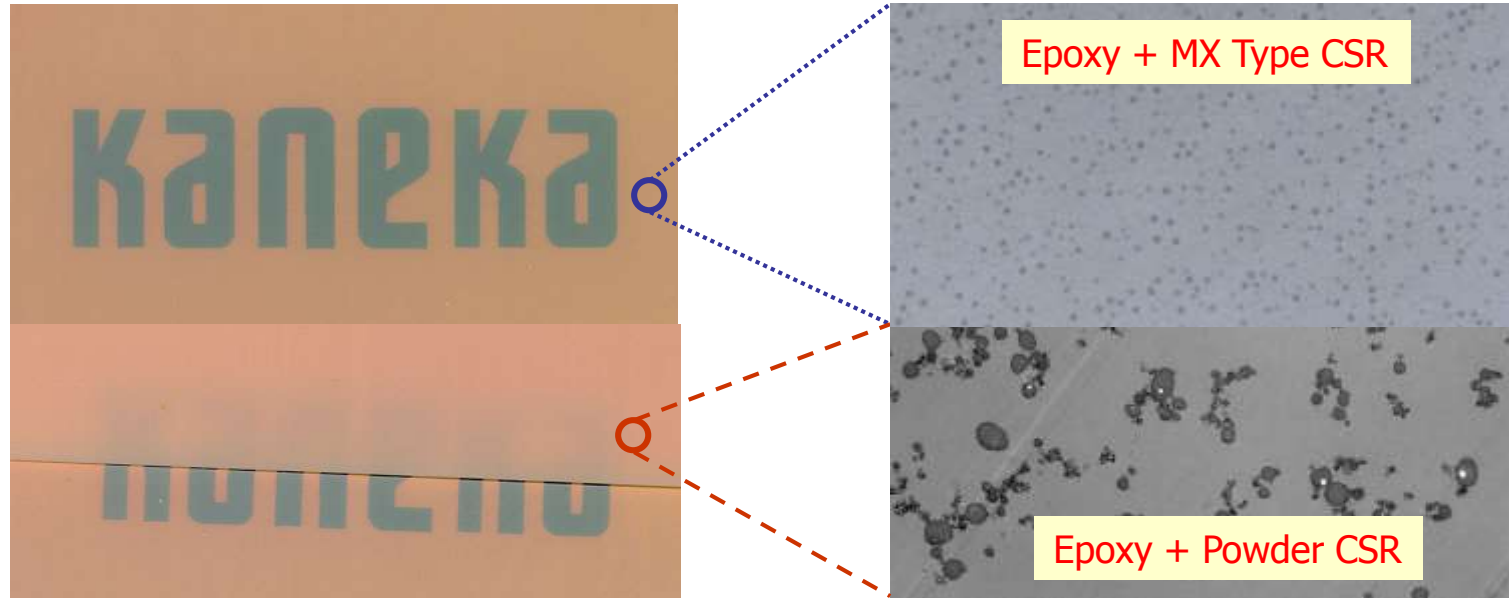
Formulating With Kane Ace™ MX

Standard Recipe	MX Modified Recipe
<p><u>Part A:</u> 70 parts liquid Bis A resin 20 parts epoxy resin Bis-F 10 parts reactive diluent</p> <p><u>Part B:</u> 30 parts Part B- Curative</p> <p><u>Mix Ratio Part A to B:</u> 100/30</p> <p><u>Core Shell Concentration:</u> 0%</p>	<p><u>Part A:</u> 52 parts liquid Bis A resin 20 parts epoxy resin B 10 parts reactive diluent 24 parts MX 125</p> <p><u>Part B:</u> 30 parts Part B-Curative</p> <p><u>Mix Ratio Part A to B:</u> 106:30</p> <p><u>Core Shell Concentration:</u> 4.4%</p>

Replacing 18 parts of epoxy with 24 parts of MX-125 is an easy way to add a modest amount of CSR without affecting the ratio of epoxy to curing agent

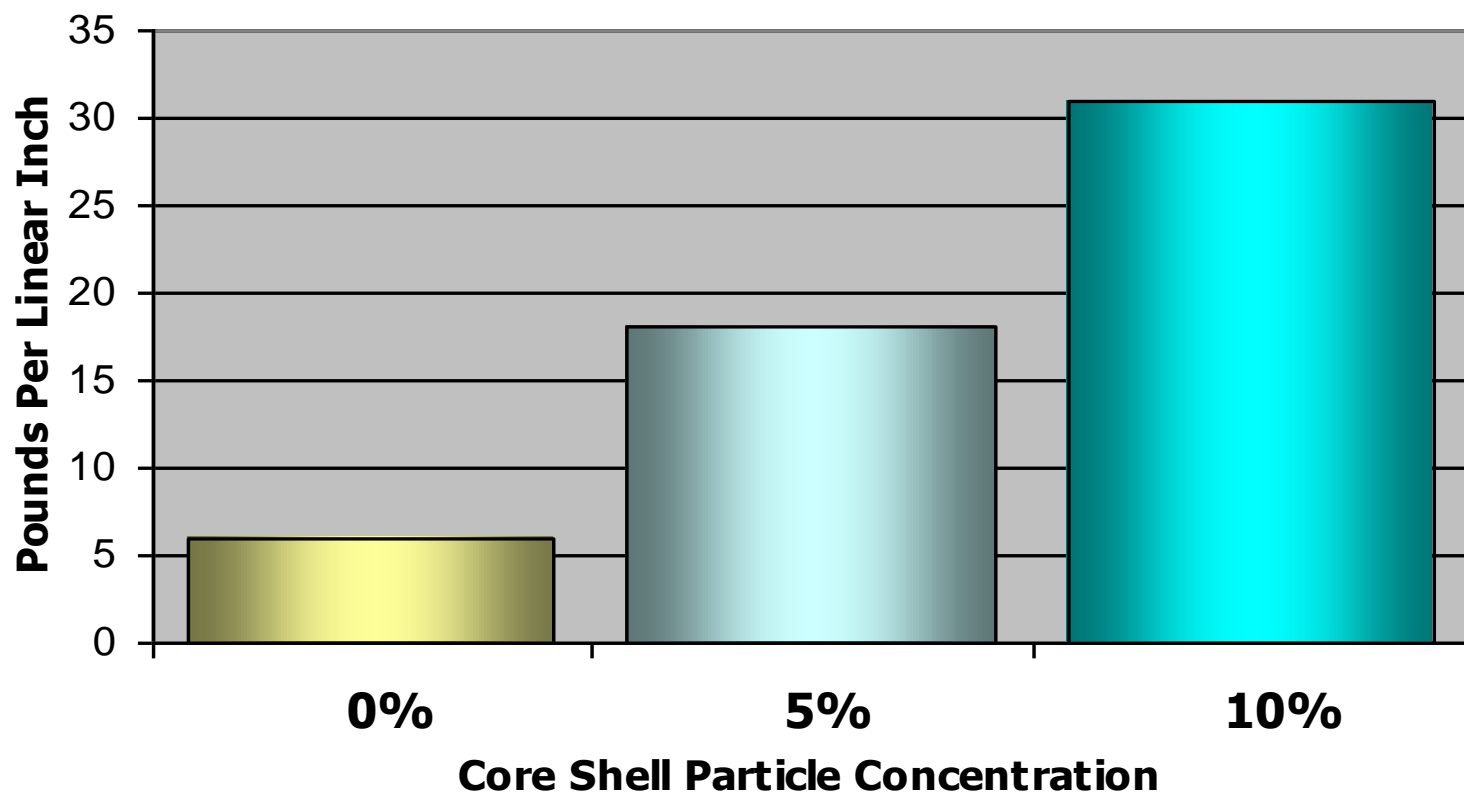
Benefits of Kane Ace® MX

DISPERSION is the key to improvement of mechanical properties!



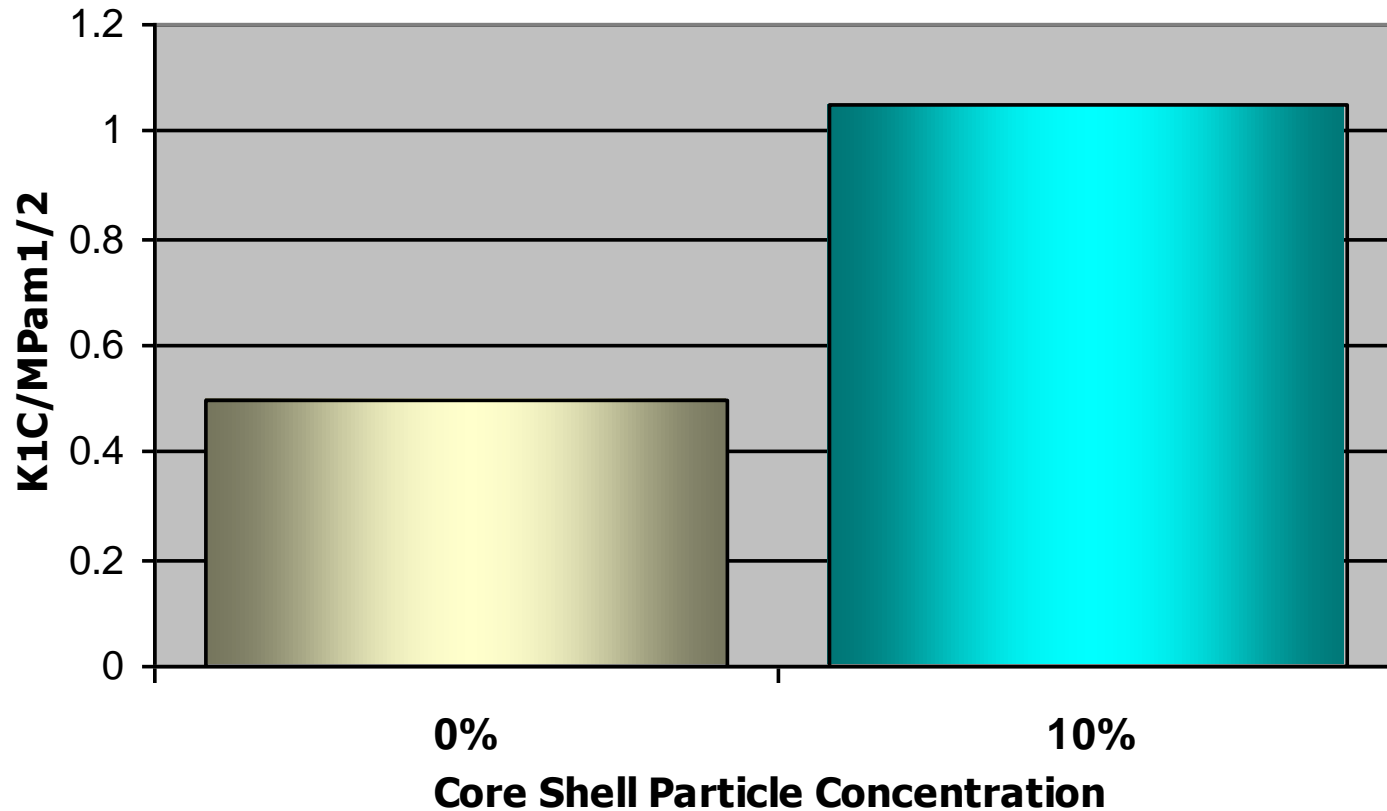
TEM analysis shows complete dispersion of CSR particles via MX while conventional tougheners suffer from agglomeration issues.

Benefits: Increased Peel Strength



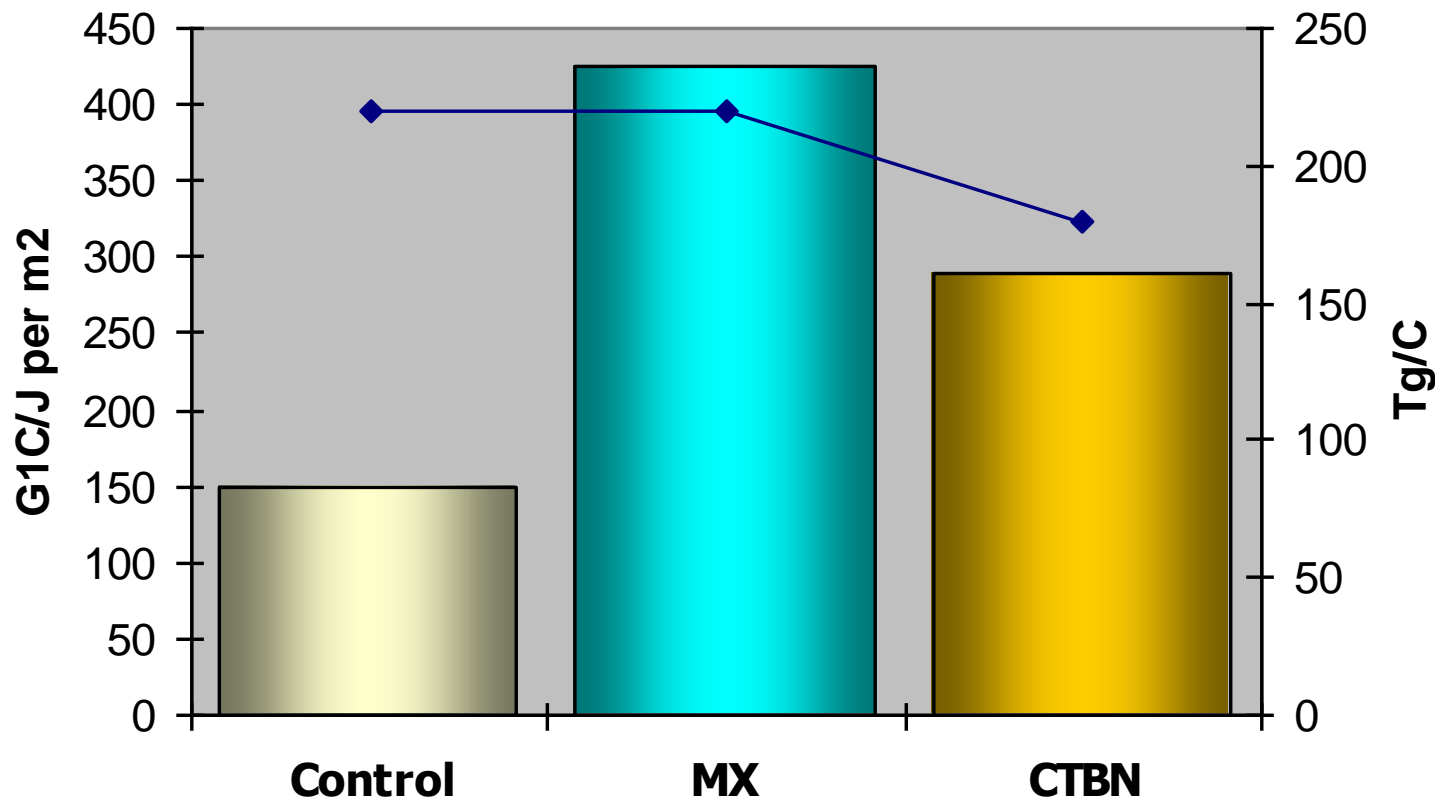
(Recipe: Bis A Epoxy + Curing Agent + MX)

Benefits: Fracture Toughness (K_{1C})



(Recipe: Bis A Epoxy + Curing Agent + MX)

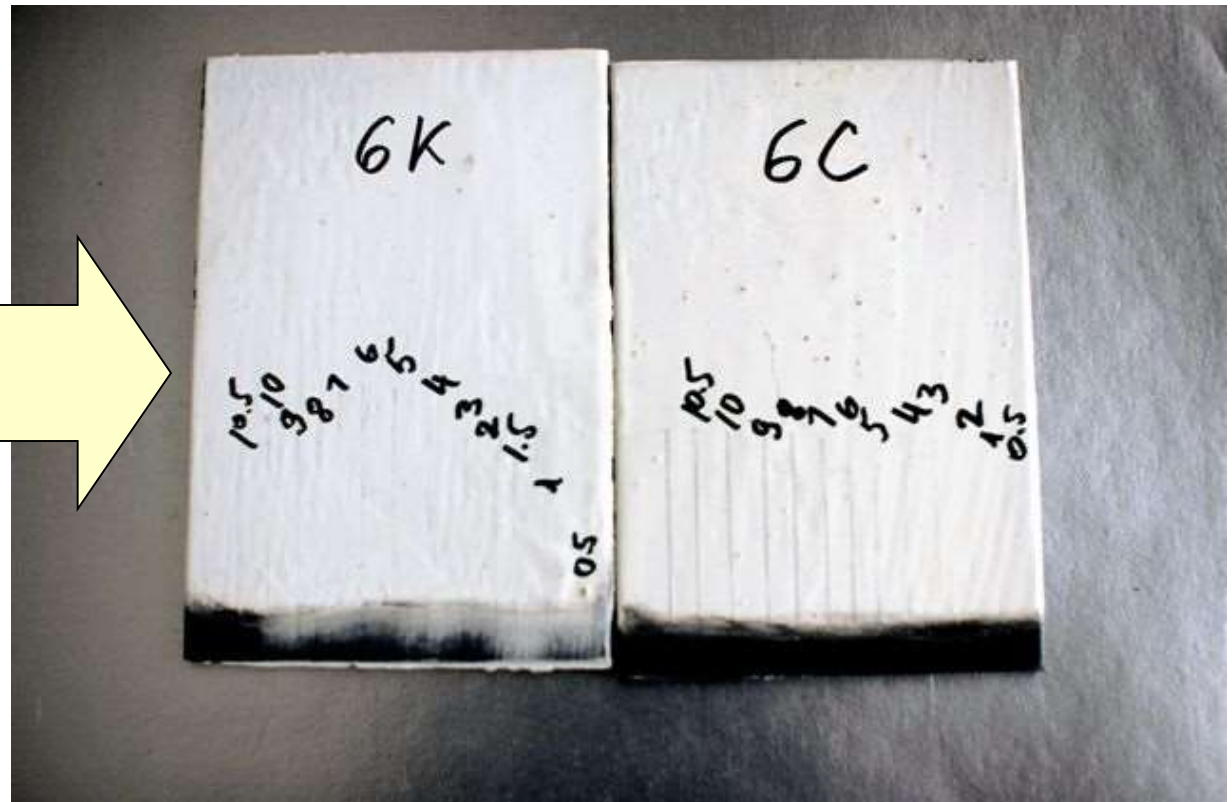
Benefits: Toughness versus T_g



(Recipe: Bis A Epoxy + Curing Agent + MX)

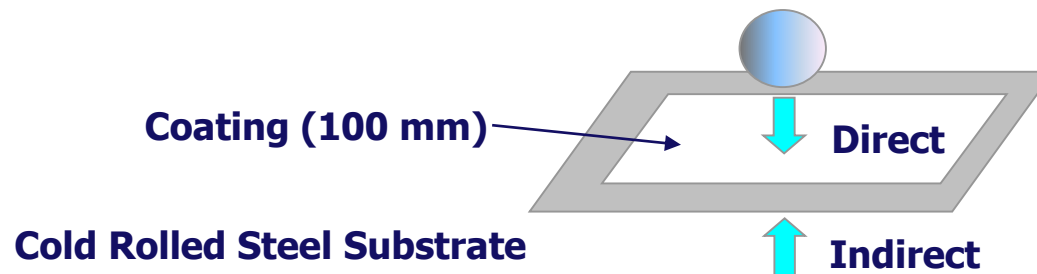
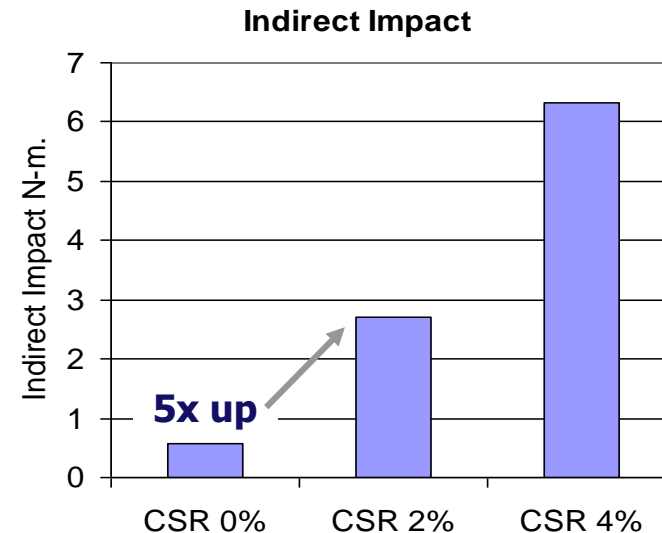
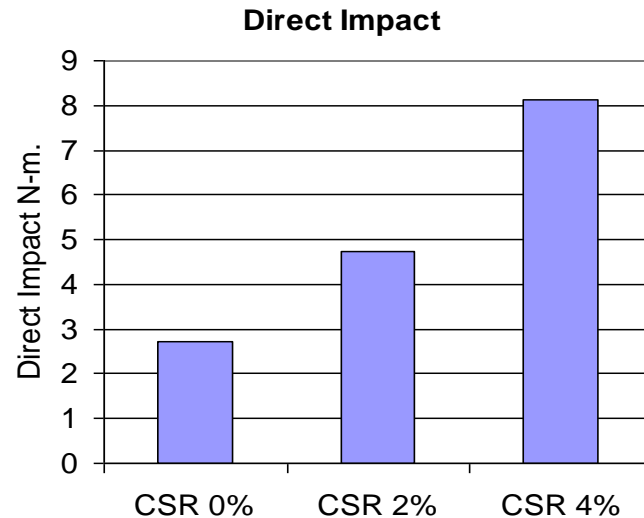
Benefits: Mar & Scratch Resistance

Kane Ace® MX can provide improvements to mar/scratch and abrasion resistance of thermosetting materials.



Huntsman "Zero VOC" White Epoxy High-Gloss topcoat (WEHGT)

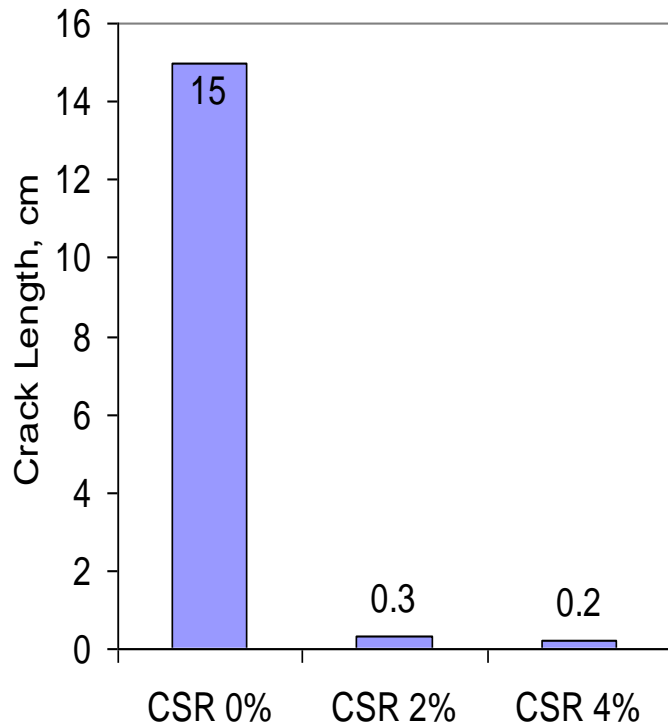
Gardner Impact



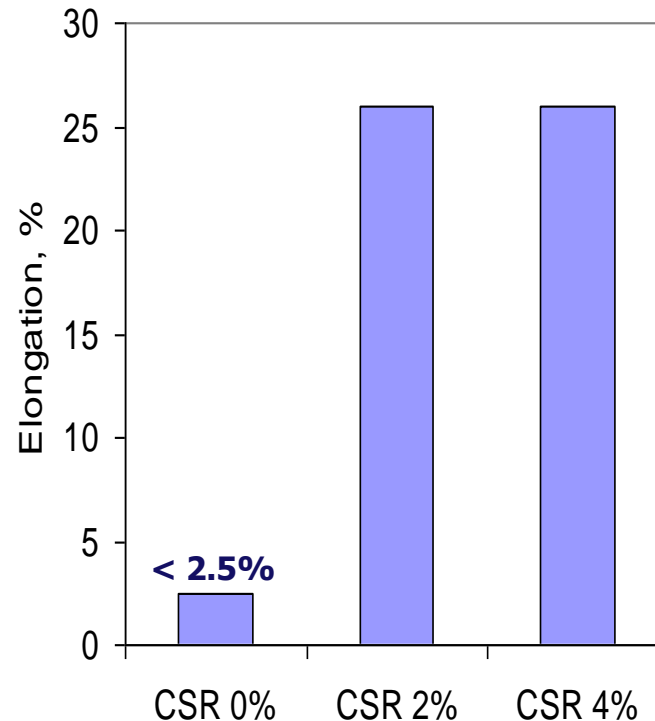
Test temperature 23°C
ASTM D-2794

Mandrel Bend Testing

Crack Length



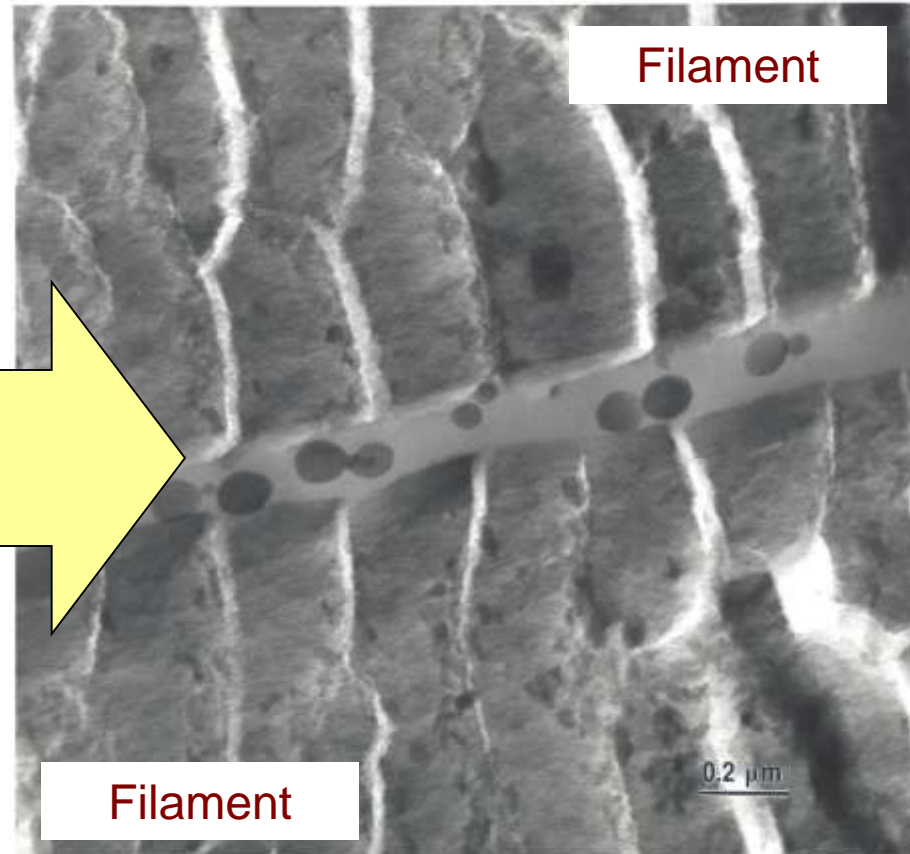
Estimated Elongation



ASTM D-522

Benefits: Fiber Reinforced Systems

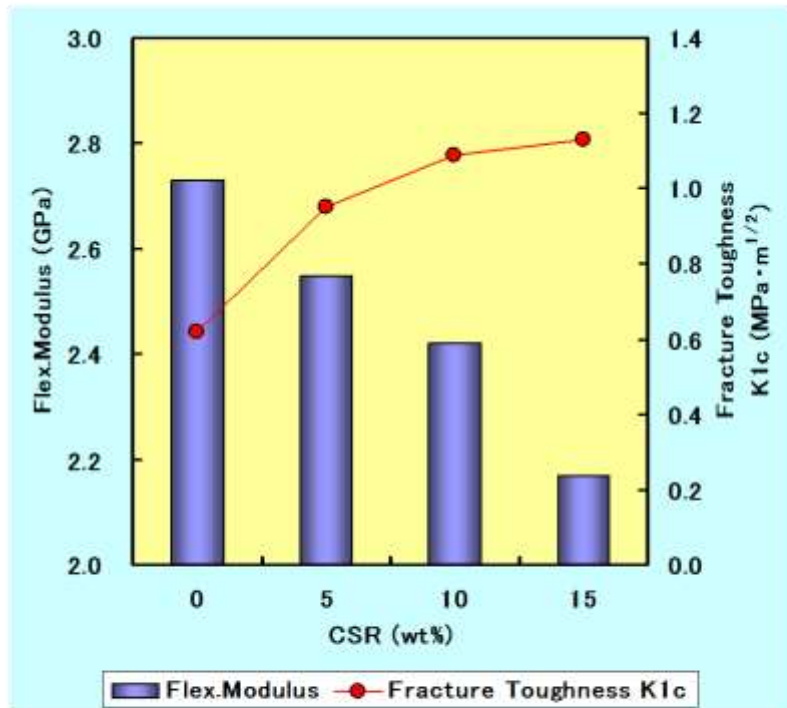
The technology associated with the production of Kane Ace™ MX avoids agglomeration of CSR particles. As a result fibers (such as carbon or glass) do not filter out the CSR particles, allowing them to toughen even the resin-rich area between single filaments.



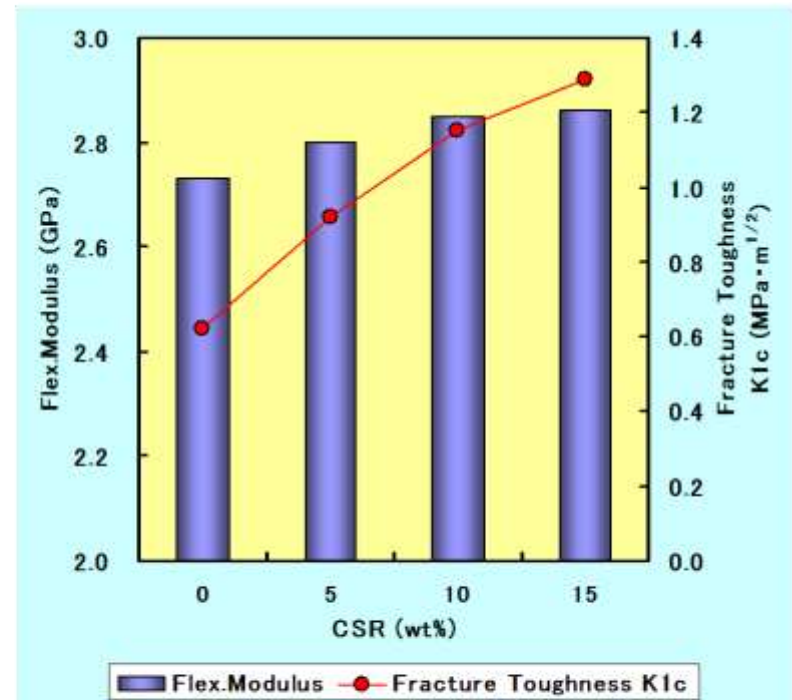
MX-170 for Composite Applications

Improves fracture toughness without sacrificing modulus

Relationship between Fracture toughness (K1c) and Flex. Modulus



Kane Ace™ MX-153 (Conventional)

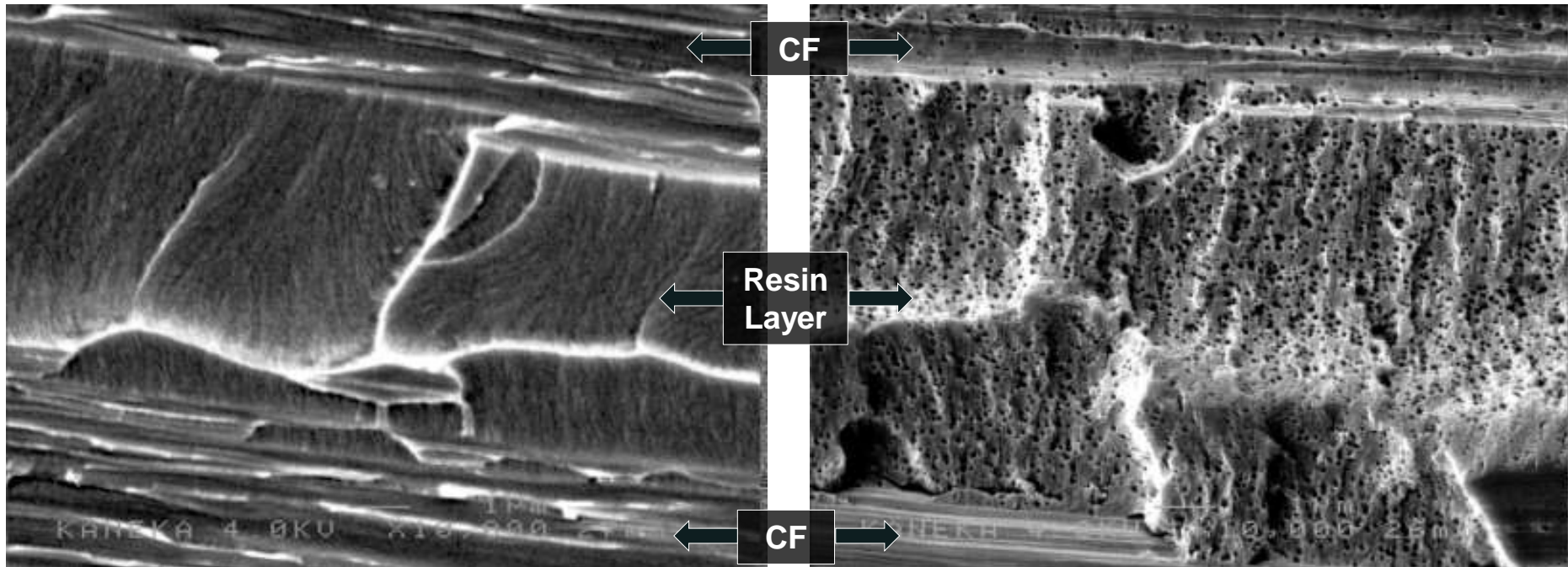


Kane Ace™ MX-170

本表の数値は測定値であり保証値ではありません。 These data are not guaranteed values but measurement values.

MX-170 for Composite Applications

Submicron-sized particles (CSR) are uniformly dispersed as primary particles in the resin layer between fibers, that means the particles are not filtered out by fibers through resin transfer molding process.



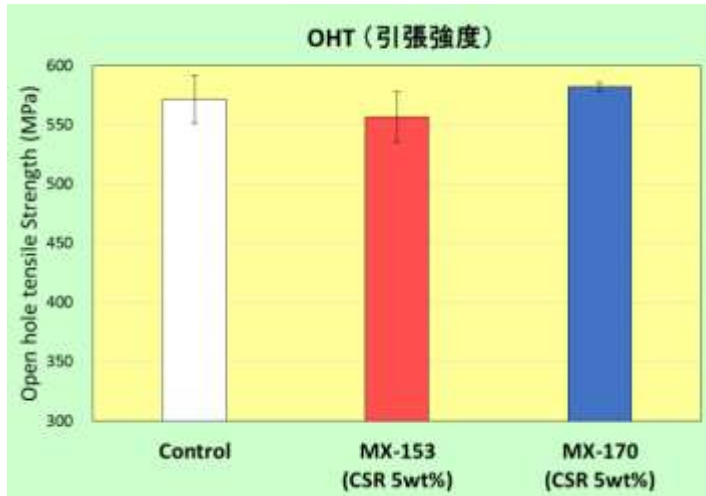
CFRP - No MX

CFRP with MX (VaRTM) CSR 10wt%

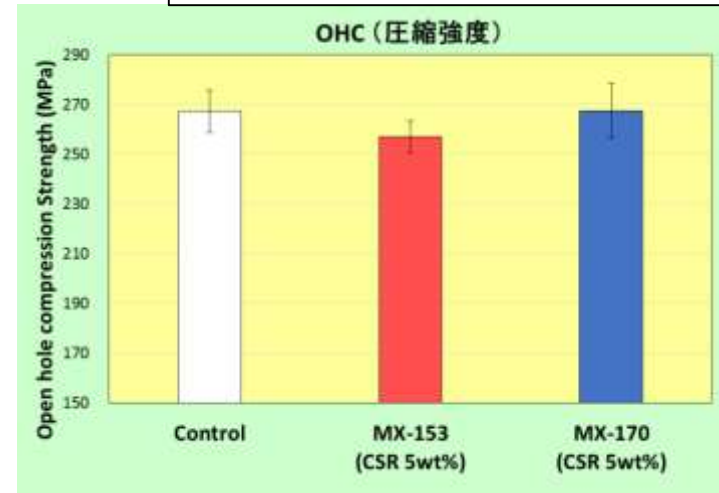
MX-170 for Composite Applications

Properties as CFRP (VaRTM)

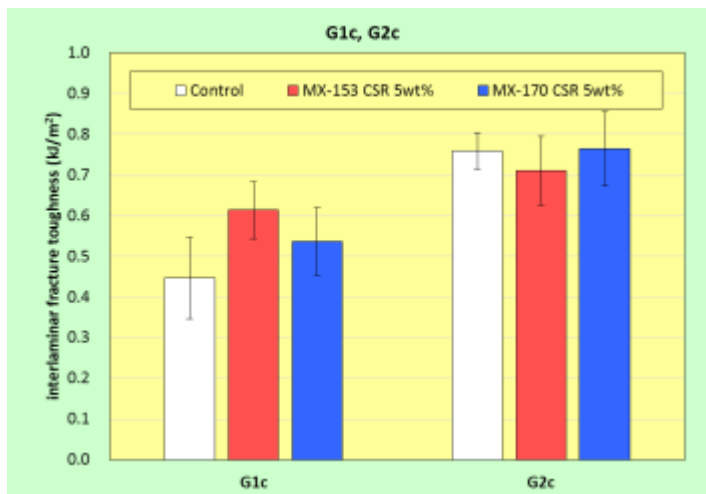
Resin: DENATool XNR6809/XNH6809 = 100/95
CF: T800UD
OHT, OHC: [45/0/-45/90]2s, t=3.35mm
CAI: [45/0/-45/90]3s, t=4.8mm
Toughener: 5 wt% vs. Resin



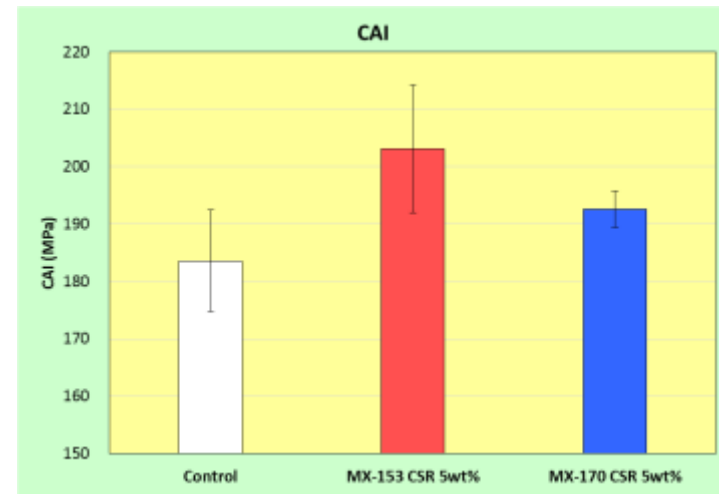
JIS K 7094



JIS K 7093



JIS K 7086



JIS K 7089

Products

Kane Ace MX products are available in a variety of carrier resins including:

Bis-A epoxy

Bis-F epoxy

Phenol Novolac epoxy


Multi-functional epoxy

Polyol for Urethane systems

Polyaspartic

Vinyl ester and UPE compatible

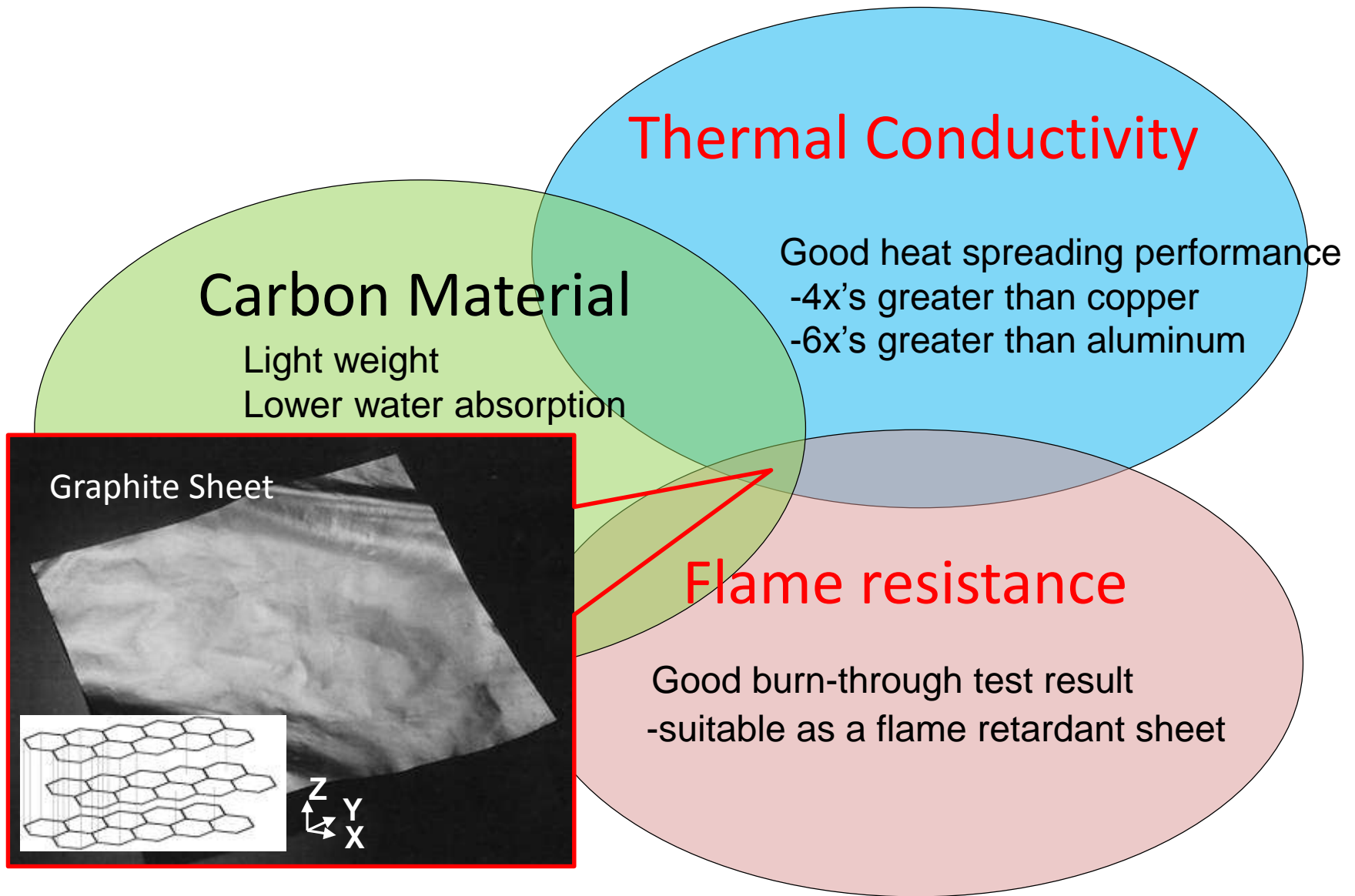
Acrylic system for UV curable applications



Kaneka's Graphite Materials For Air Craft

**Kaneka Corporation
Electrical & Electronic Materials Division**

General Characteristic of Graphite Sheet



General Characteristics of our Graphite material

1) High thermal conductivity in the in-plane direction

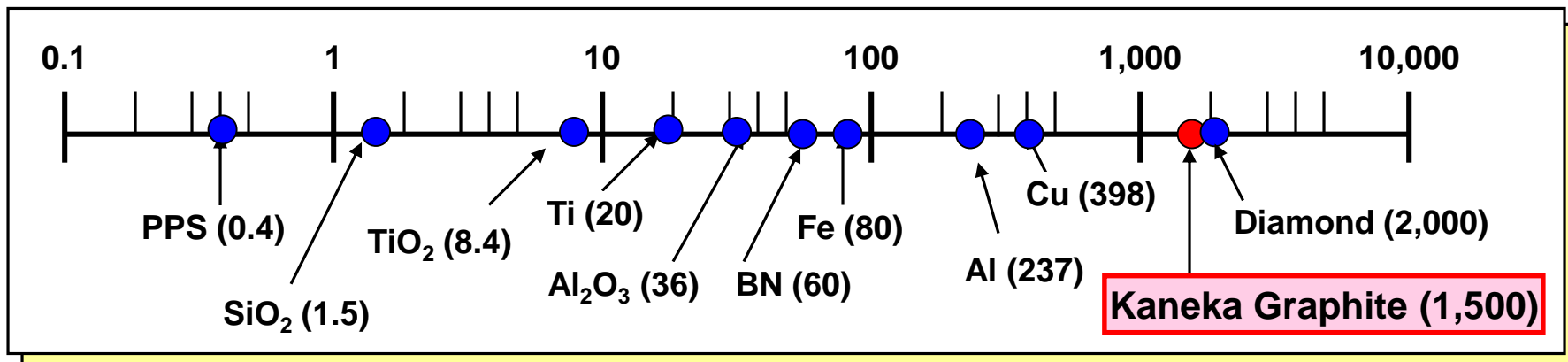
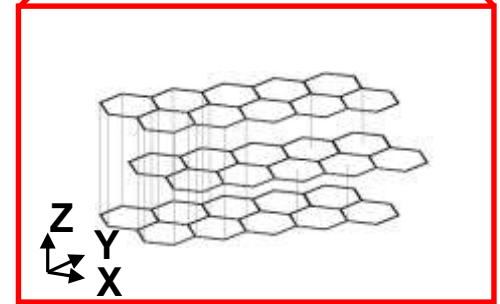
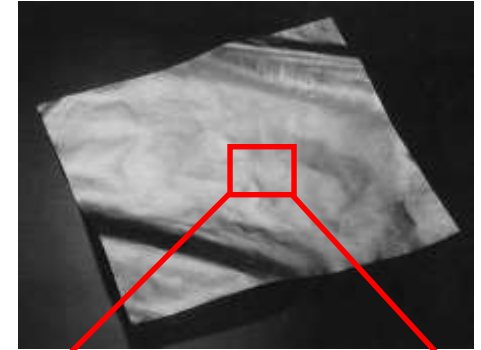
- Graphite: 1,500 W/mK (in-plane direction)
- about 4x's greater than Copper (398 W/mK)
- about 6x's greater than Aluminum (237 W/mK)

2) Anisotropy of thermal conductivity

- Graphite: 5 W/mK (thickness direction)
- Ratio: in-plane / thickness = 300

3) Low density

- Graphite : 2.0 g/cm³,
- Copper: 8.9 g/cm³, Aluminum: 2.7 g/cm³



➤ Basic Properties of our Graphite Sheet

- 10um to 40um thick Graphite Sheets are commercially available.



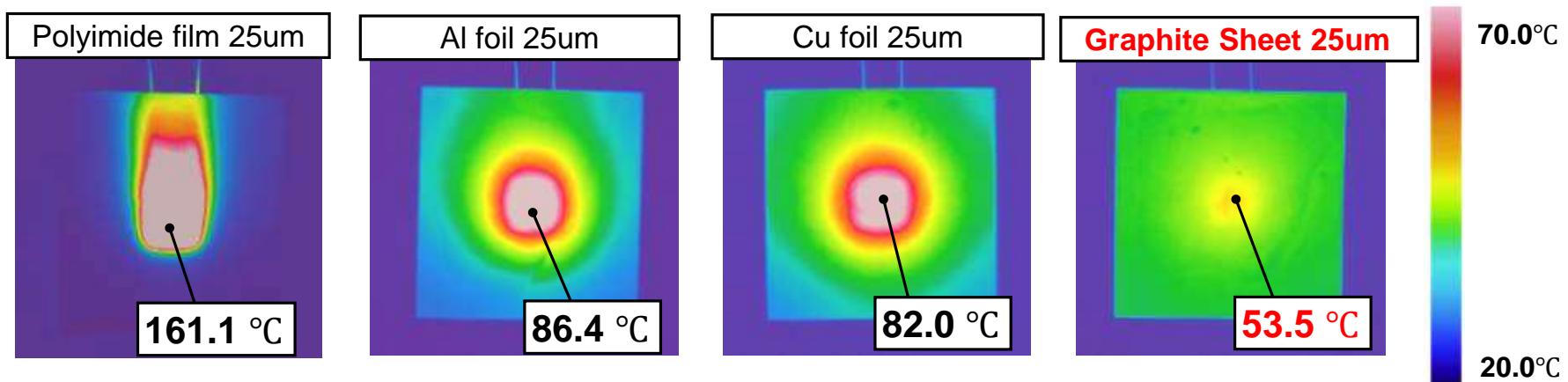
		Current grade				
Thickness (um)		10	18	25	32	40
Thermal conductivity (W/m·K)	XY axis	1600	1600	1500	1500	1500
	Z axis	5	5	5	5	5
Thermal diffusivity (cm ² /s)		9.0	9.0	9.0	9.0	9.0
Specific Density (g/cm ³)		2.1	2.1	2.0	2.0	2.0

Other properties:

No Water absorption, No outgas, Electric conductivity, EMI shield, Good corrosion resistant

Heat spreading performance

- Kaneka's Graphite Sheet has a much larger heat spreading ability than copper and aluminum with the same thickness.



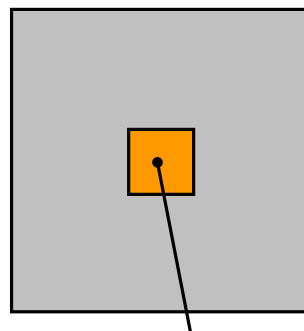
□ Test material:
50x50x0.025mm

Thermography

□ TIM

6.5W/mK, 10x10x0.3mm

Thermal Interface Material



□ Heater

2.0W, 10x10x1.8mm

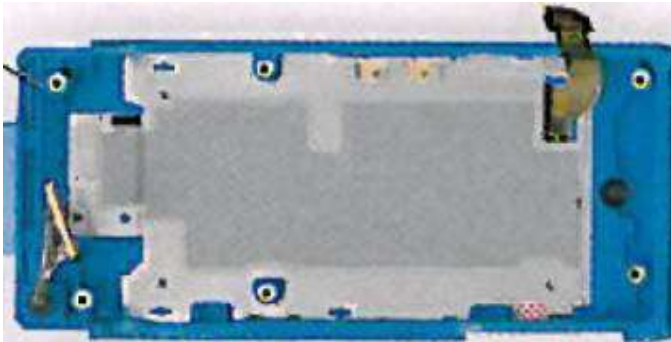
Measurement Conditions

- Room Temp. : 24°C
- Heater Wattage : 2.0 W
- The emissivity was adjusted as 0.94 by black carbon spray

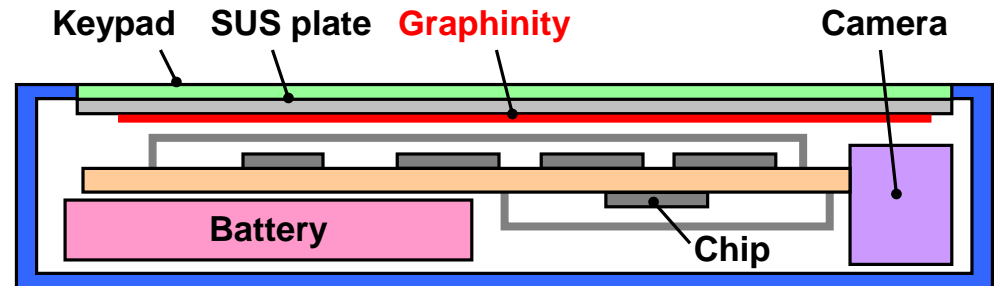
Application example of our Graphite Sheet

➤ Graphite Sticker

- Kaneka's Graphite Sticker is suitable as a heat spreader because of its high thermal conductivity and thinness.



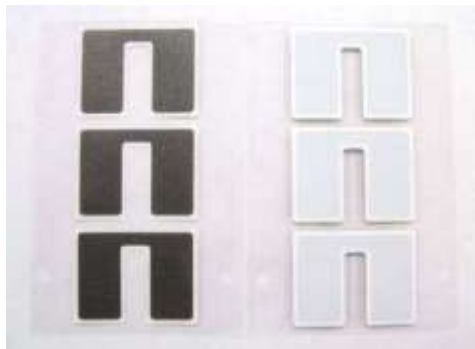
10umPET/**25umGS**/10umPSA,
30×80mm



Graphite Sheet transfers the heat from the Chips, Camera and Battery.

PSA side

PET tape side



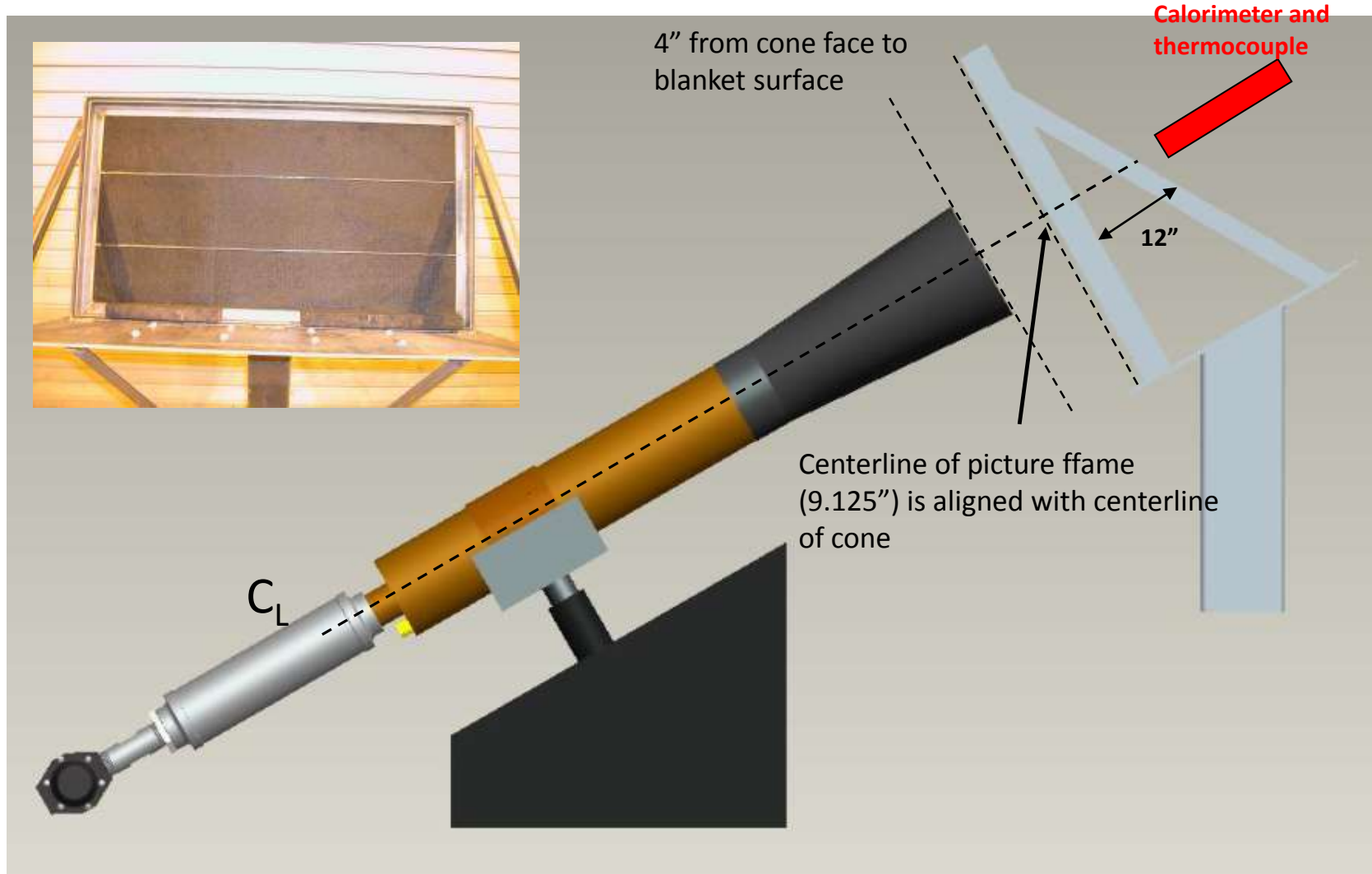
Protect layer/ Graphite/ Adhesive



← PET tape
← Graphite Sheet
← Adhesive(PSA)
← Release film

Burn-through Test Results

➤ Picture Fframe Test (to compare with ceramic paper)



Burn-through Test Results

➤ Flame penetration

25um



40um



Graphite sheet (25um,40um) can meet FAA requirement

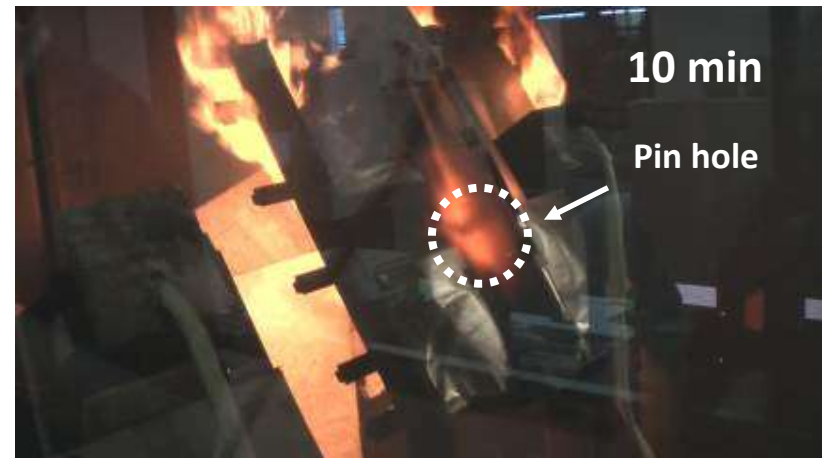
Burn-through Test Results

➤ Flame penetration

25um



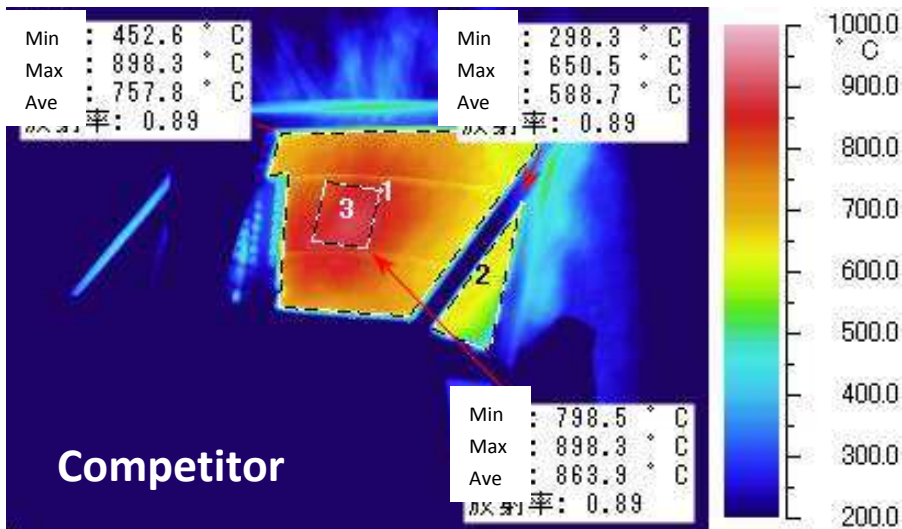
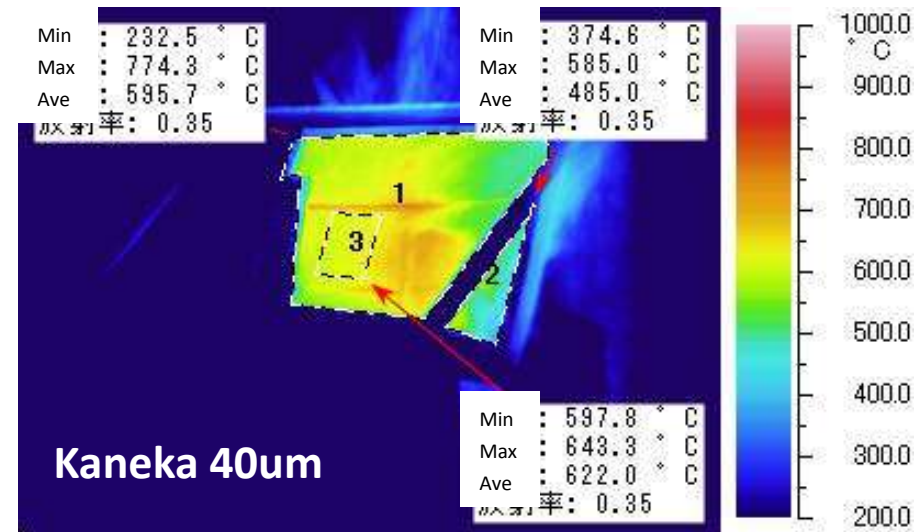
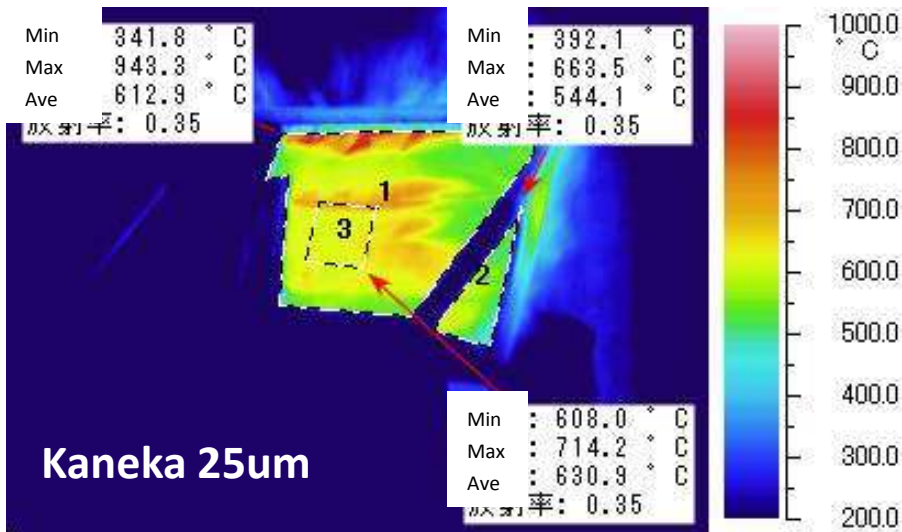
40um



25um material can withstand 8min., and 40um material can withstand 10min.

Burn-through Test Results

After 4 min.




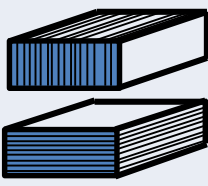

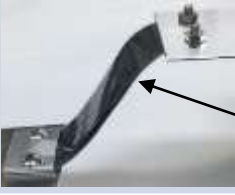


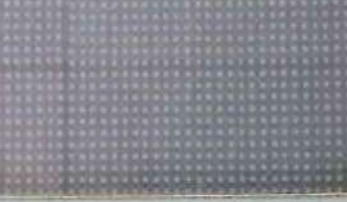
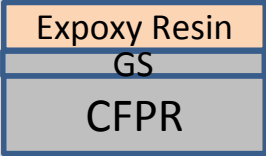


Kaneka material and competitive material are equivalent with regard to flame penetration.

However, Kaneka material has better insulative properties.

Kaneka's Graphite Materials for Air Craft

➤ New Graphite Materials (under development)

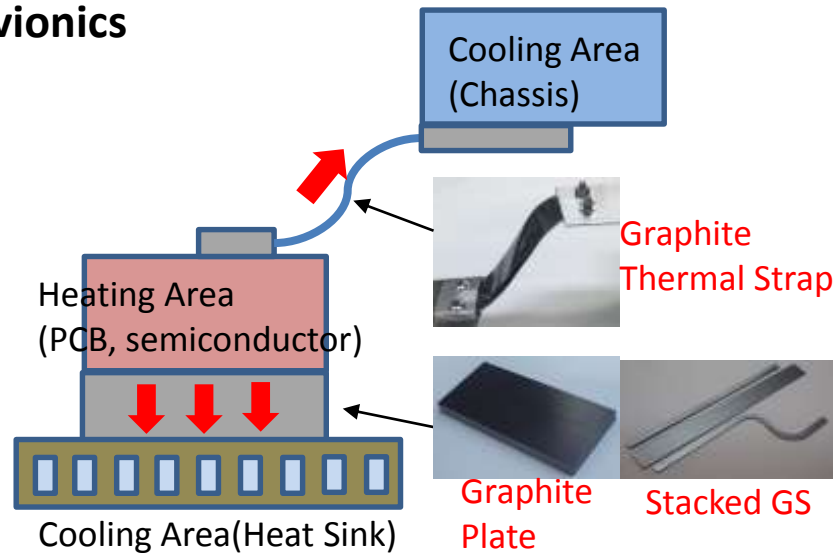
	Composition	Feature
Stacked GS	 	<ul style="list-style-type: none"> • High thermal transfer in XY or XZ axis • Free form
Graphite Plate	 	<ul style="list-style-type: none"> • All graphite • High thermal transfer in XY or XZ axis
Graphite Thermal Strap	  Multilayer Structure	<ul style="list-style-type: none"> • High thermal transfer • Flexible • Light weight (comparable to Cu)
Metalized Graphite Sheet	 	<ul style="list-style-type: none"> • High electrical conductivity • Electromagnetic shield
GS/CFRP composite	 	<ul style="list-style-type: none"> • High Thermal conductivity (comparable to CFRP without GS) • High Strength

Kaneka's Graphite Materials for Air Craft

➤ Potential application

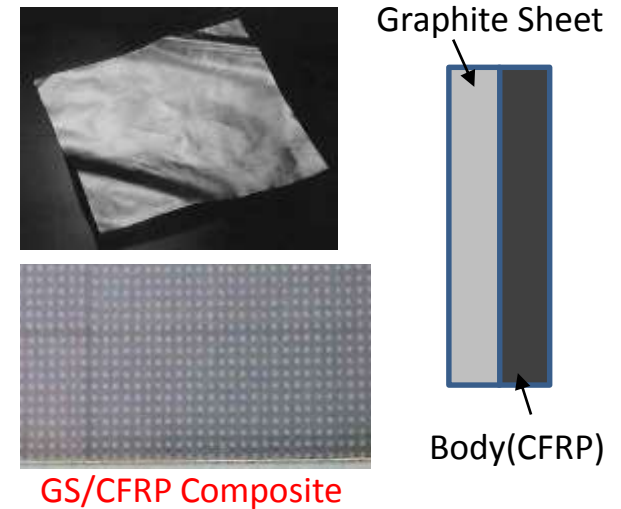


Avionics



Heat dissipation of the equipment

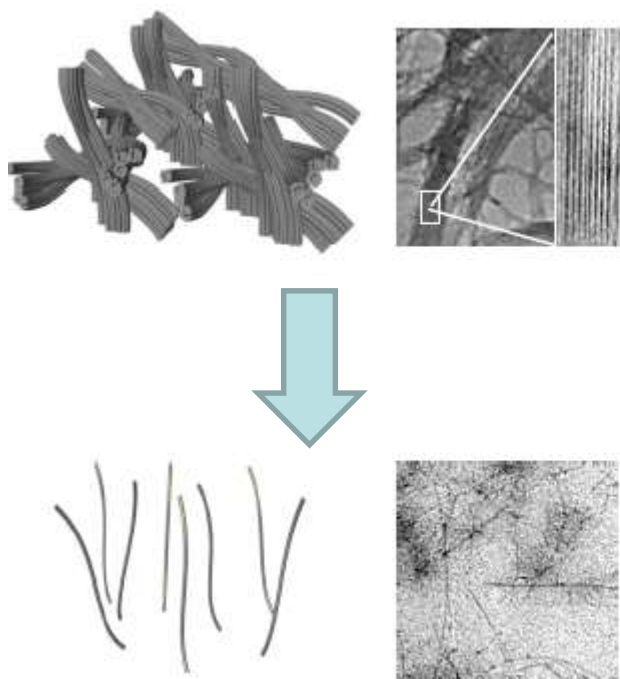
Body



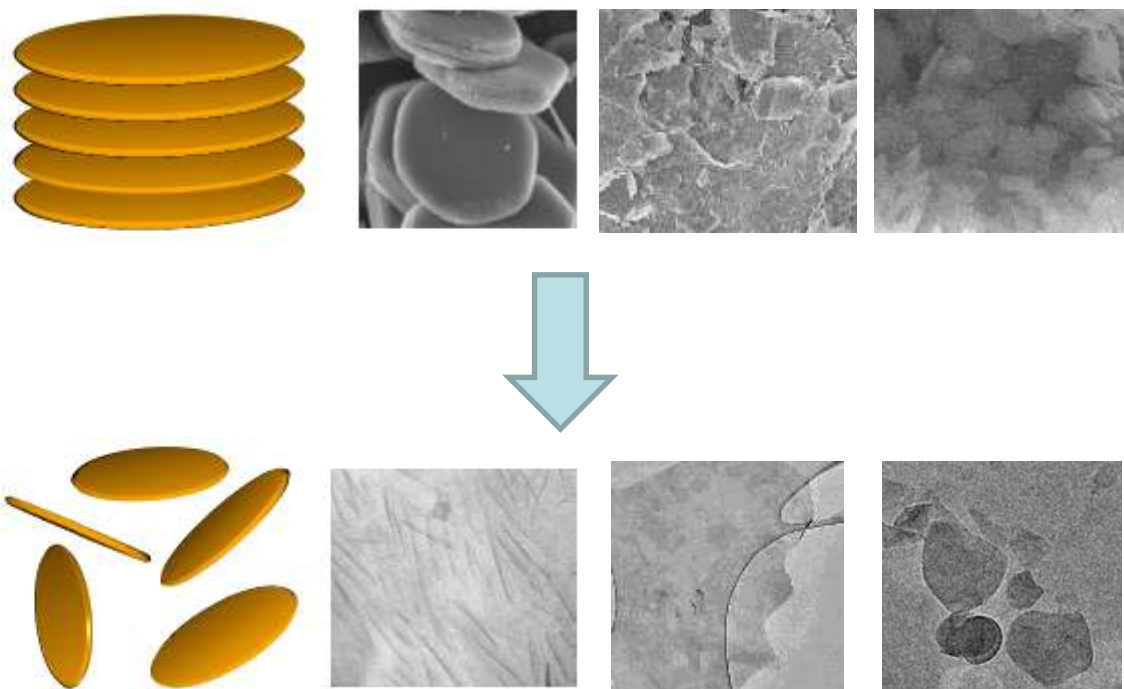
High Flame Resistance of Aircraft Body

Nanomaterials

1-D
carbon nanotubes

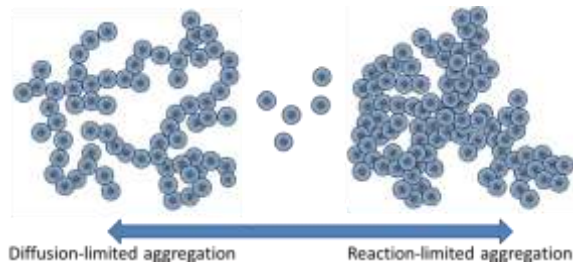


2-D
Clay, graphene, BN

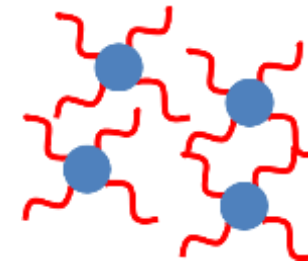


Nanocomposite/Hybrid Research at KMR

Nanocomposites



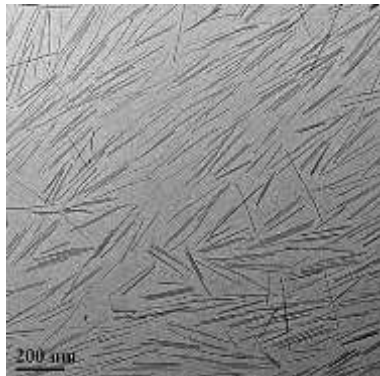
Organic-inorganic hybrids



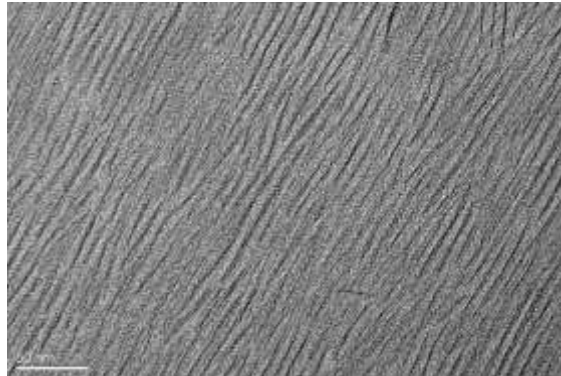
Nano-particle dispersion



Exfoliation

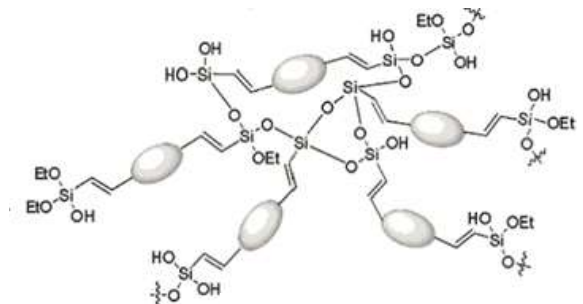


Alignment



Polymer-based nanocomposites

NP-based hybrids

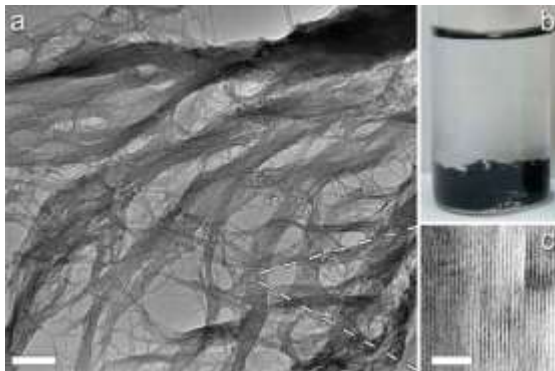


Molecular hybrids

ZrP assisted exfoliation of single-walled CNTs (SWCNTs)

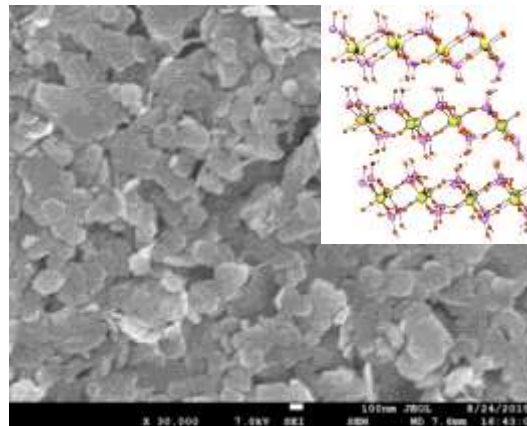
SWCNT aggregates

They form bundles in microscale and cannot be dispersed in solvent



To solve this issue, we developed a method to disperse CNT by ZrP nanoplatelets

ZrP: synthetic clay, high purity, small size distribution, mature exfoliation technique
~100 nm in diameter

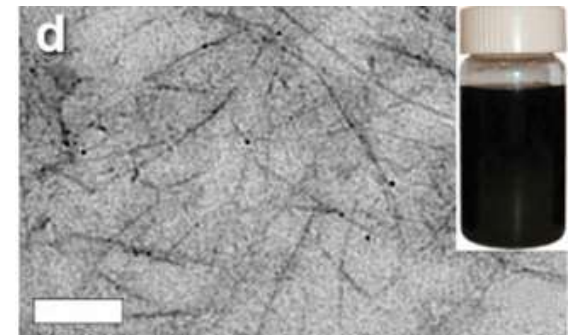


Types of CNTs used for this process:

- HiPco SWCNTs
- Arkema MWCNTs
- SouthWest NanoTechnologies (SWeNT)

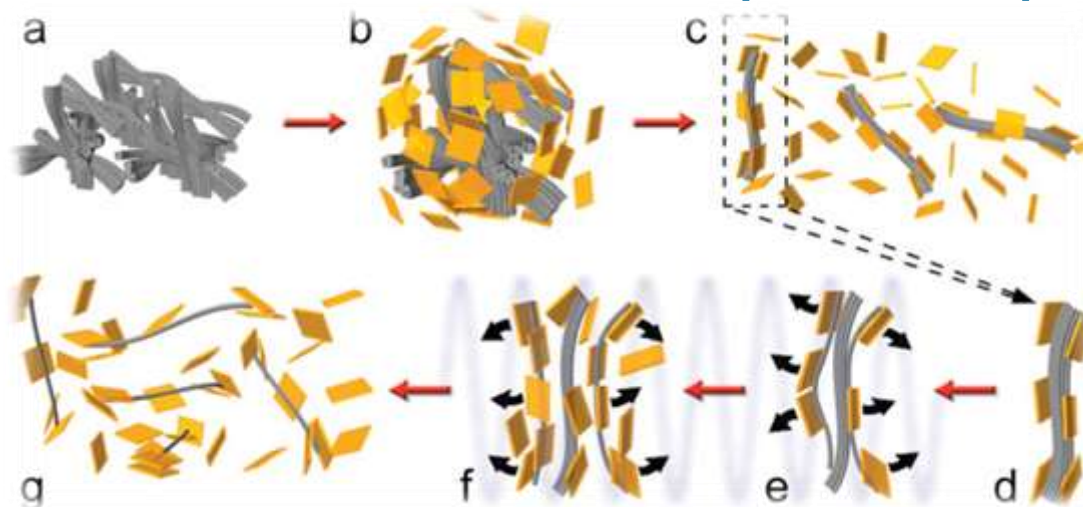
SWCNT dispersion

SWCNTs have been de-bundled and show good dispersion in water

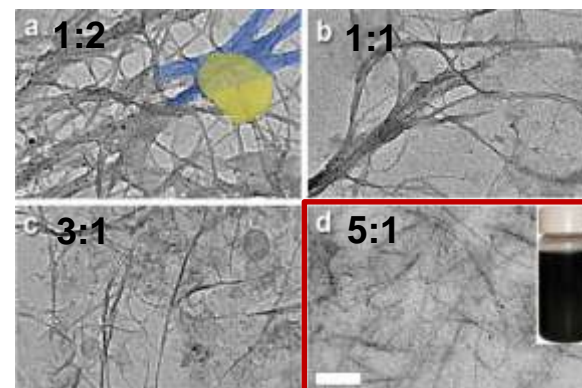


ZrP aqueous solution

ZrP assisted exfoliation of single-walled CNTs (SWCNTs)



Different weight ratio between ZrP and SWCNTs

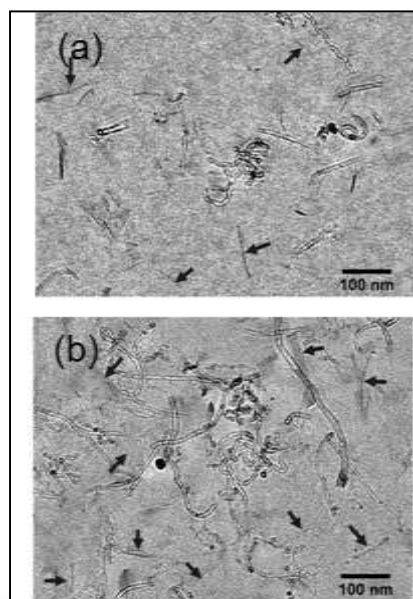
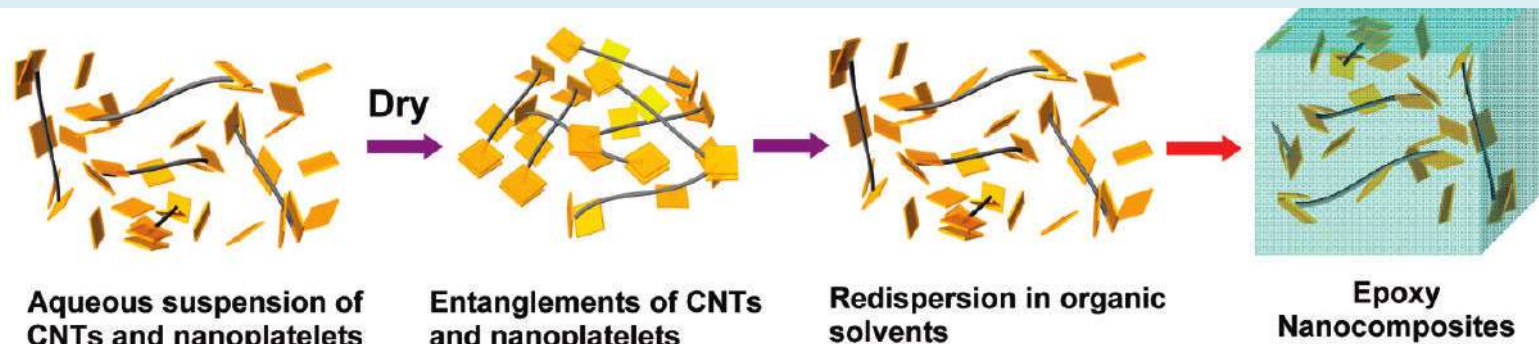


- a) Large aggregates of pre-treated(oxidized) SWCNT are in an aqueous medium.
- b) ZrP nanoplatelets are added and electrostatically bind to the SWCNT bundles.
- c) SWCNT aggregates are then broken up into smaller bundles.
- d) One specific example of the bind between ZrP and SWCNT.
- e) Nanoplatelets with an ultrasonic wave progressively pull individual tubes and small bundles from bundles.
- f) Nanoplatelets continue to pull small bundles from larger bundles.
- g) Mechanism continues until the nanotubes are pulled into an individually dispersed state.

**Good dispersion is achieved with a ZrP-to-CNT weight ratio of 5:1, but ZrP could be removed afterward.
More than 95% of CNT could be dispersed.
This method also applies to multi-walled carbon nanotube (MWCNT).**

Preparation of epoxy hybrid nanocomposites based on carbon nanotubes and ZrP

Transfer MWCNT/ZrP aqueous suspension into organic solvent and prepare epoxy nanocomposites



Microstructure of epoxy nanocomposites with (A) 0.2wt% of MWNTs and 1.0 wt % of ZrP nanoplatelets and (B) 0.4wt% of MWNTs and 2.0 wt % of ZrP

Mechanical properties of epoxy nanocomposites

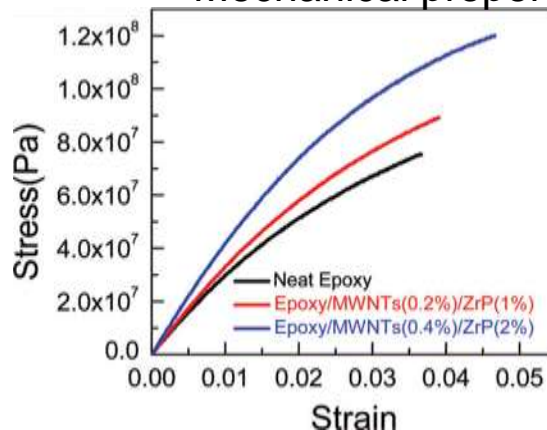


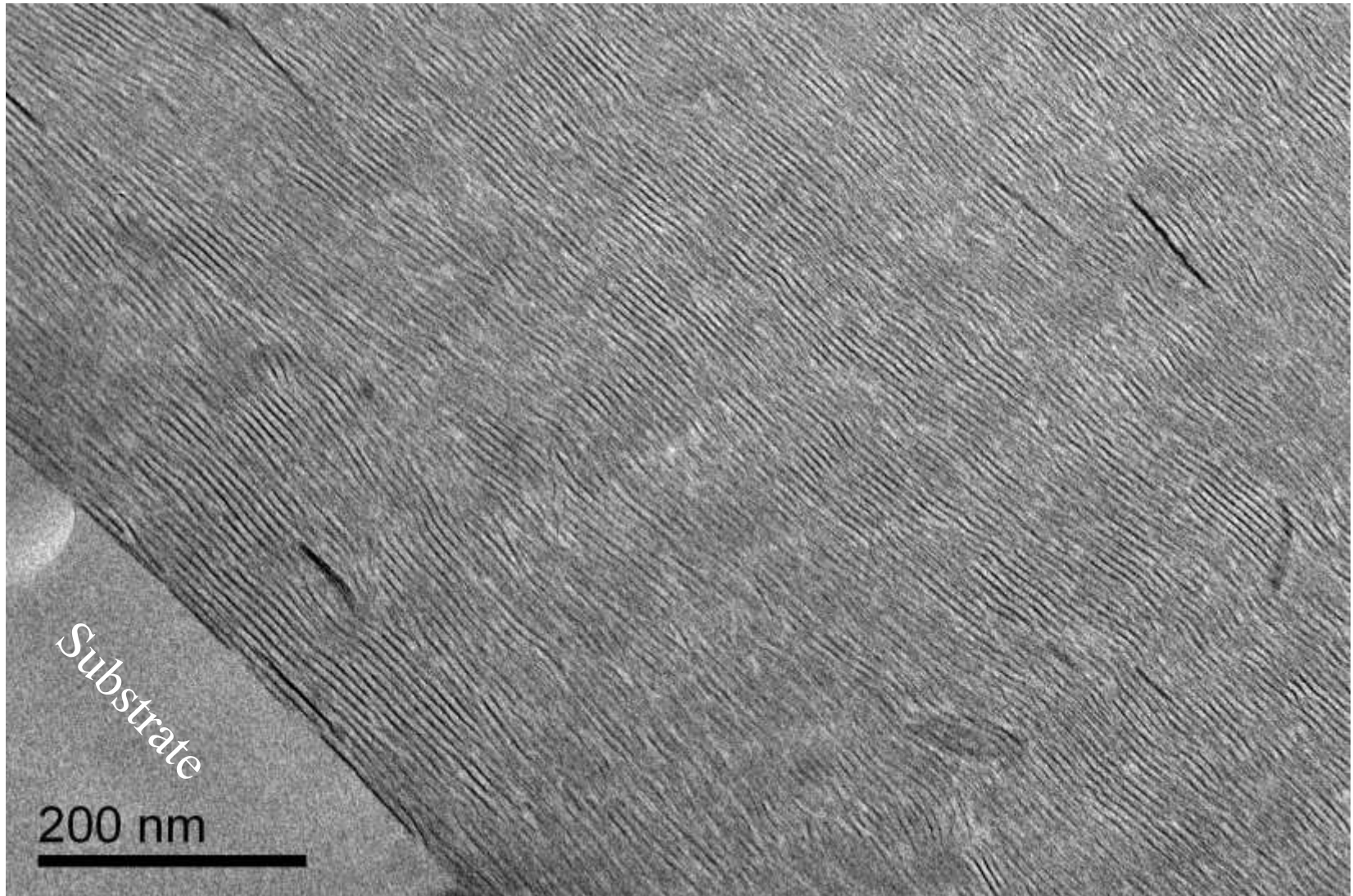
Table 1. Mechanical Properties of the Neat Epoxy and Epoxy Nanocomposites Containing Exfoliated ZrP Nanoplatelets and MWNTs

	neat epoxy	epoxy/ MWNTs(0.2%)/ ZrP(1.0%)	epoxy/ MWNTs(0.4%)/ ZrP(2.0%)
Young's modulus (GPa)	3.04 ± 0.04	3.40 ± 0.06	4.27 ± 0.07
tensile strength (MPa)	75.3 ± 4.2	83.1 ± 4.8	116 ± 5.5
elongation at break (%)	3.7 ± 0.1	3.9 ± 0.3	4.3 ± 0.4

40%↑

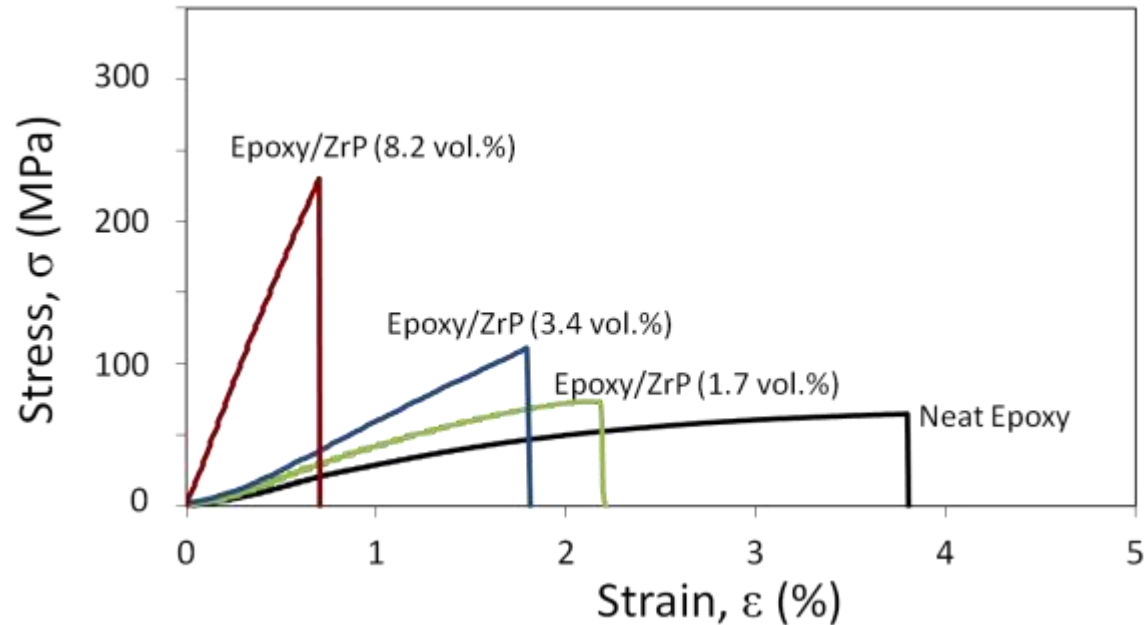
54%↑

TEM of Epoxy/ZrP Nanocomposites (4.5 vol.%)



Highly aligned ZrP nanoplatelets in epoxy matrix

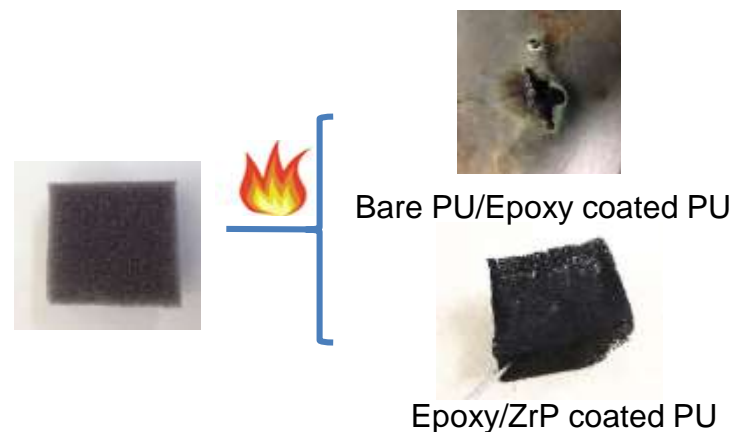
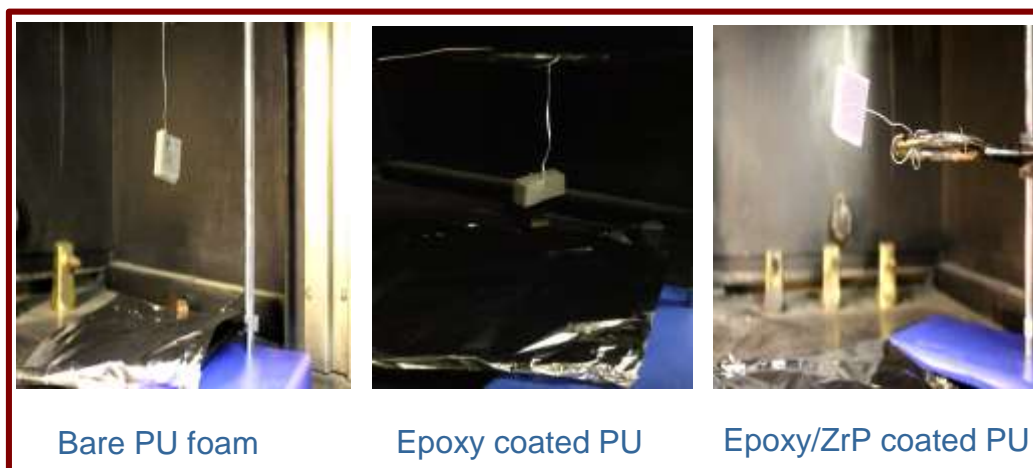
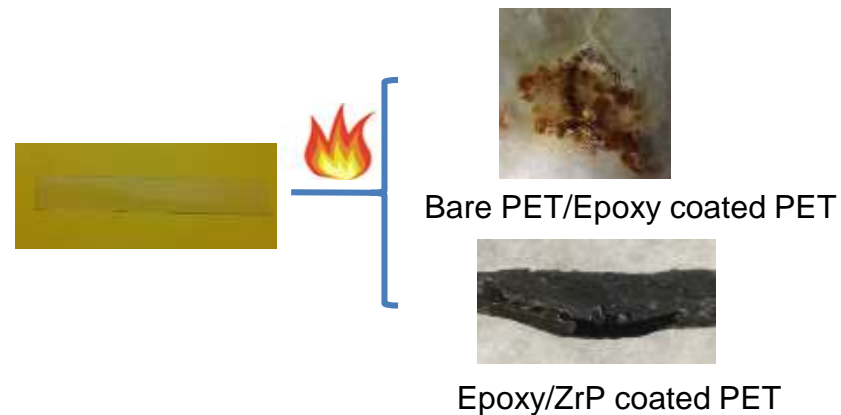
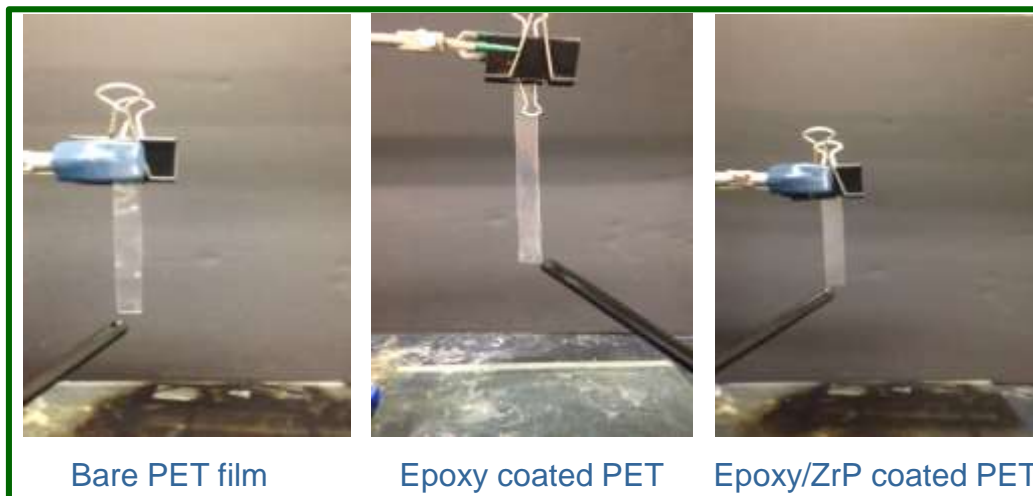
Tensile Properties of Epoxy/ZrP Films



	Pure Epoxy	Epoxy/ZrP (1.7 vol. %)	Epoxy/ZrP (3.4 vol. %)	Epoxy/ZrP (8.2 vol. %)
Young's Modulus, GPa	2.7 ± 0.4	4.4 ± 0.9	8.1 ± 2.3	22.0 ± 4.0
Tensile Strength, MPa	55 ± 5	79 ± 9	135 ± 25	210 ± 40
Elongation at Break, %	3.8 ± 0.5	2.1 ± 0.3	1.6 ± 0.3	0.8 ± 0.2

Note: Tensile tests were conducted using an RSA-G2 (TA Instruments) with a tensile fixture. The tensile tests were conducted in controlled strain mode with a constant linear rate of 0.05mm/s.

Smectic epoxy/ZrP coated PET & PU



Melt-dripping issue of PET film and polyurethane (PU) foam is effectively solved via application of epoxy/ZrP (4.6 vol.%) coating.

Summary

- **Polyimide**
- **Toughening Agent (CSR)**
- **Graphite Sheet**
- **Nanocomposites**

Kaneka

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