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Track 2 Nanotechnology in Materials Science,
2-1 Nanomaterials and nanocomposites

3rd International Conference and Exhibition on
Materials Science & Engineering
October 06-08, 2014 San Antonio, USA

Damage Sensing of Nanocomposites for Smart Paste Applications

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Contents

- Objectives
- Introduction
- Experimental
- Results and Discussion
- Conclusions

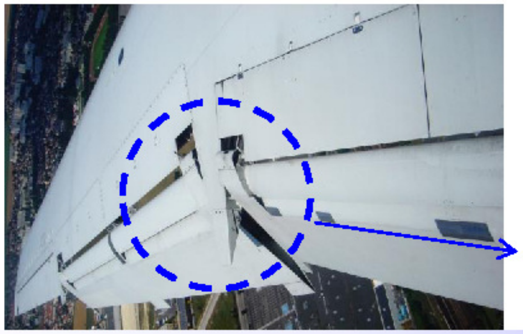
Objectives: Smart Structure

Damaged structural parts due to cracking and de-lamination

How to prevent and to monitor damage of structural materials?

Smart paste
(CF composites, Conductive materials)

Need to find de-lamination, crack, and micro craze



Detection of fatigued fracture parts

Where?

Interface, joint, body, etc.

When?

Ongoing, in situ

How?

How much stress applied?
How much propagated?

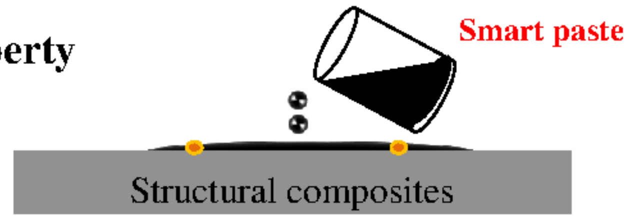
Why?

Inside cracking, External stress, Environment, etc.

Objectives: Smart Pastes

• **Goal property**

(1)

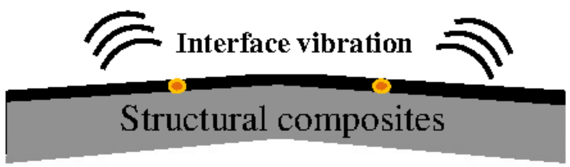


Easy casting, Good adhesion, Good wetting

(3)

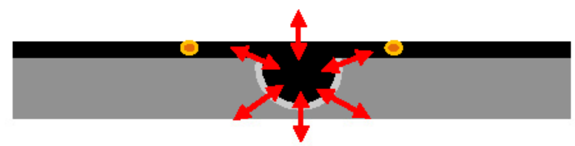
Good fixed electrical connection, <u>enhancing electrical signal</u>

(2)



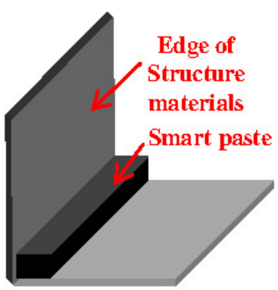
High fatigue and delamiation resistance

(4)

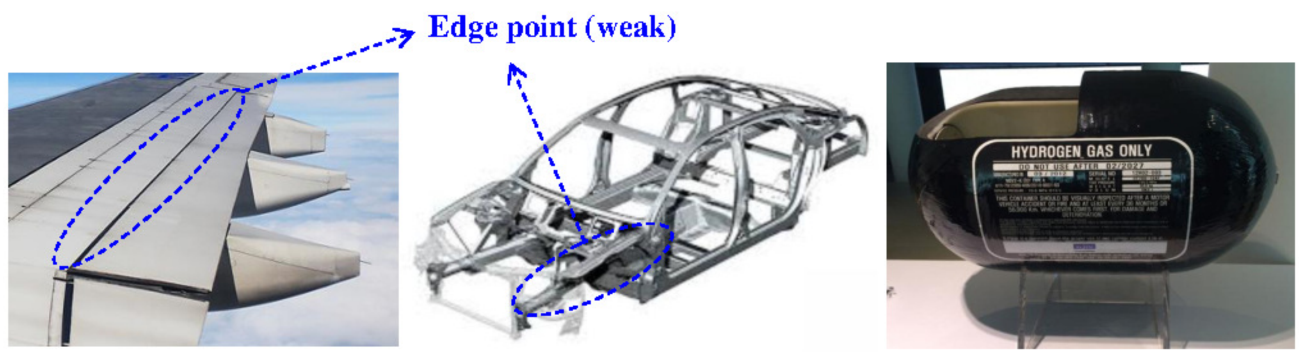


Reduced crack propagation, Increased stress transfer at cracking point

• **Application parts**



Angle point



Aerospace | Automobile | CFRP CNG

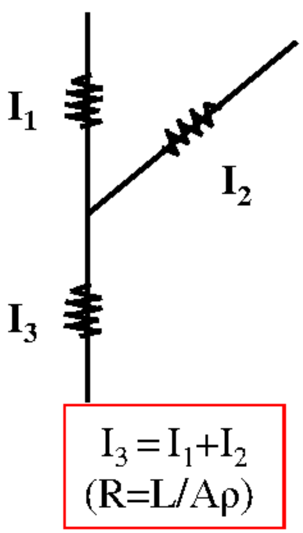
Background

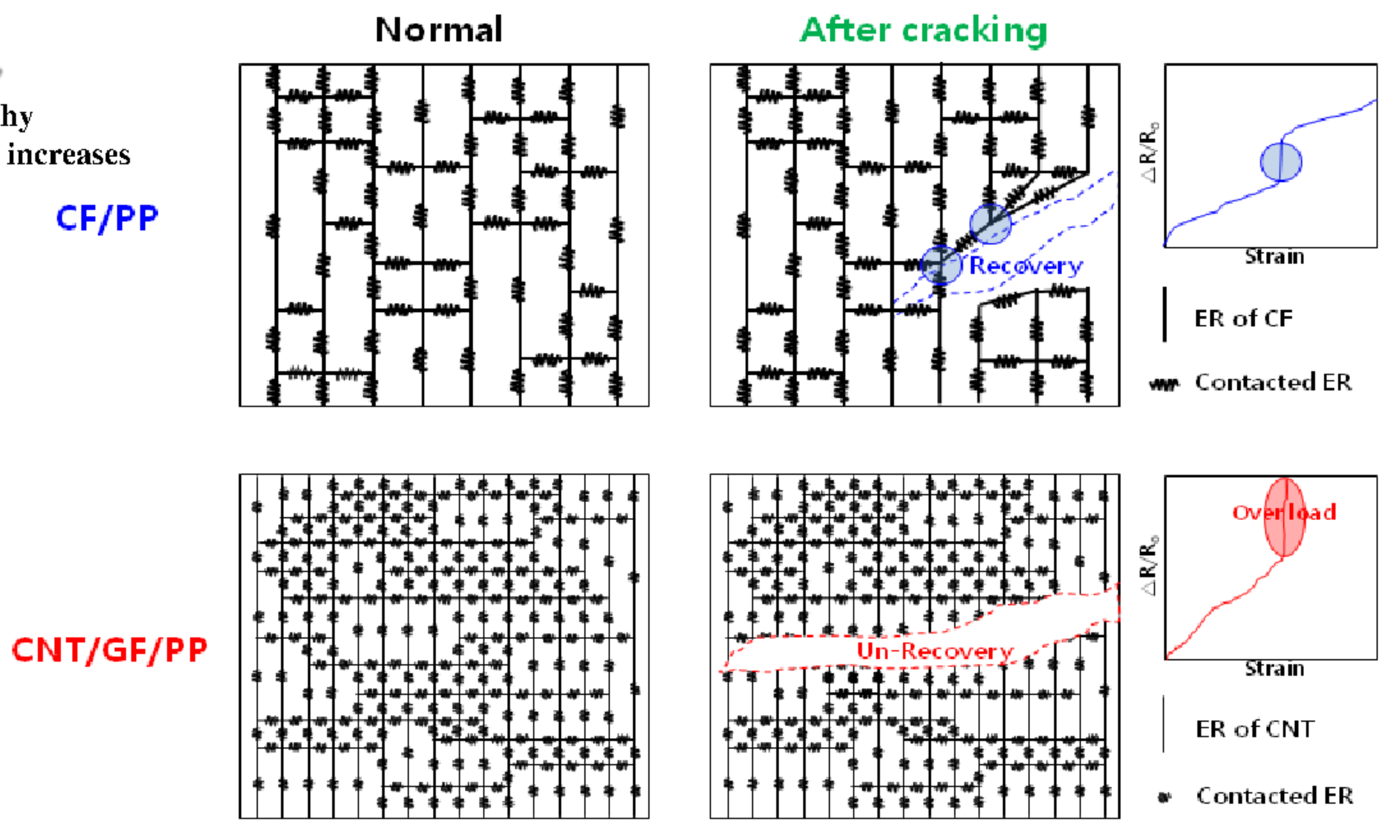
Basic principle for measuring electrical resistance (ER)

- using Kirchhoff's law ER can be measured for damage detection due to ER change due to electro-circuit disconnection and fracture
- Prediction of internal micro-damage prediction

Kirchhoff's Law

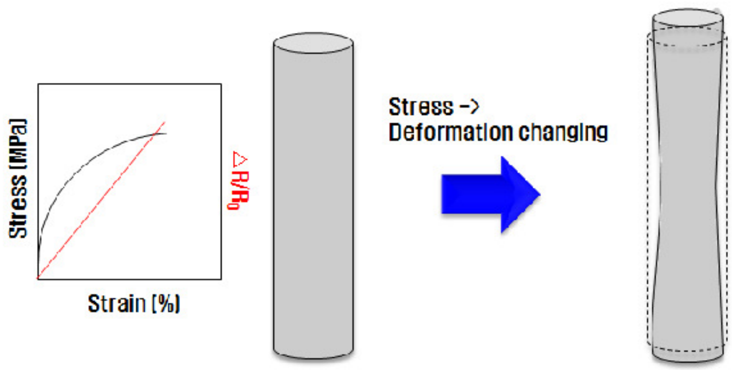
: theory explaining why electrical resistance increases from damage



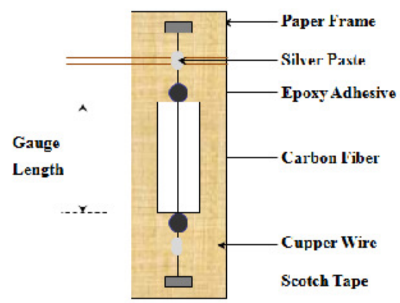


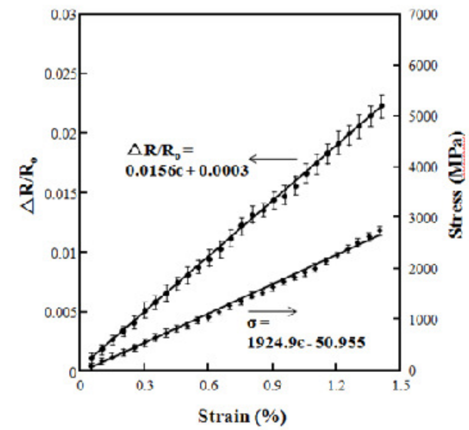
Introduction

Single material (self-sensing)

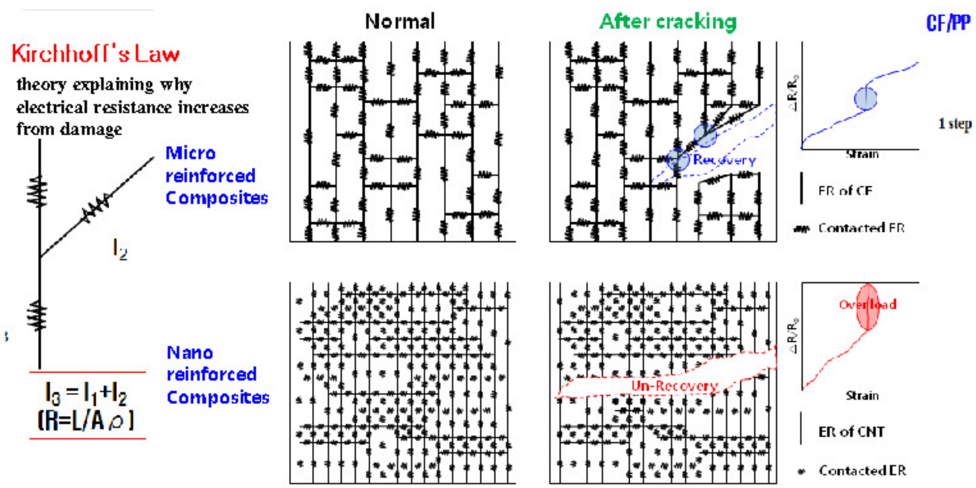


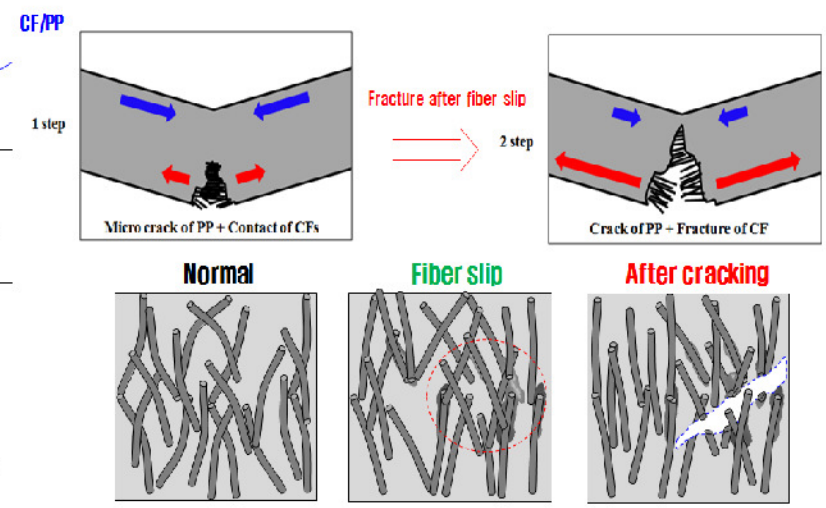
What is basic theory for ER measurement?





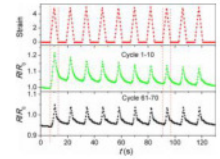
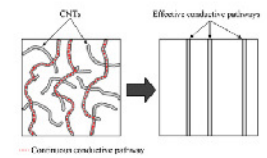
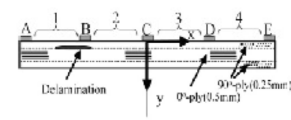
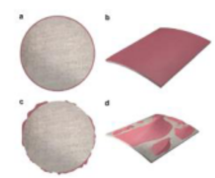
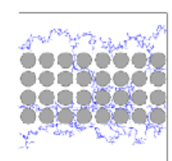
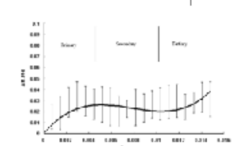
Composites materials (Multi-sensing signals)





References

damage sensing papers

Year	Journal	Author	University	Title	Data
2013	Composites Science and Technology	R. Zhang, H. Deng, R. Valenca, J. Jin, Q. Fu, E. Bilotti, T. Peijs	Queen Mary University of London, UK	Strain sensing behaviour of Elastomeric composite films containing carbon nanotubes under cyclic loading	
2012	Composite Part B	J. Wen, Z. Xia, F. Choy	The University of Akron, U.S.A.	Damage detection of carbon fiber reinforced polymer composites via electrical resistance Measurement	
2012	Composites Part B	Y. Shindo, Y. Kuronuma, T. Takeda, F. Narita, S.Y. Fu	Tohoku University, Japan	Electrical resistance change and crack behavior in carbon nano tube/ polymer composites under tensile loading	
2010	Composites Science and Technology	J. Rausch, E. Mader	Leibniz Institute of Polymer Research Dresden, Germany	Health monitoring in continuous glass fibre reinforced thermoplastics: Manufacturing and application of interphase sensors based on carbon nanotubes	
2008	Composites Science and Technology	C. Li, **T.W. Chou**	University of Delaware, U.S.A.	Modeling of damage sensing in fiber composites using carbon nanotube networks	
2008	Composites Science and Technology	E. Sevkat, J. Li, B. Liaw, **F. Delale**	The City College of New York, U.S.A.	A statistical model of electrical resistance of carbon fiber reinforced composites under tensile loading	

Damage self-sensing for conductive composite materials

Damage self-sensing using conductive composite materials

1) **Background**

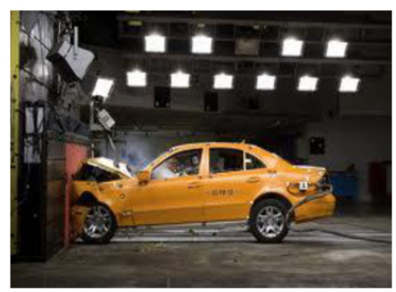
- increasing in conductive materials, e,g, CNT, CFRP in nonconductive GF/PP composites
- New NDE for detecting durability evaluation of inter- and external parts
- Importance of dispersion of nano- and micro-particles such as carbon nanotube (CNT)

<Plastic module>

<Audio Case>

Conductive nanocomposites (high modulus, light Weight)

<CF Composites examples>



Damage evaluation methods for new materials parts (rapid, accurate)

<New materials for car>

Experimental: Materials

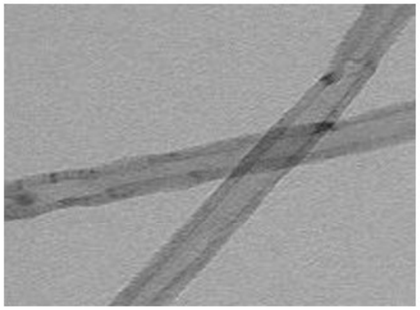
❖ Structural Materials

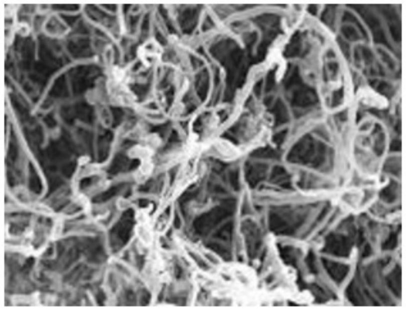
Injection method using LFP of CF 30wt%/PP made by Hyundai EP

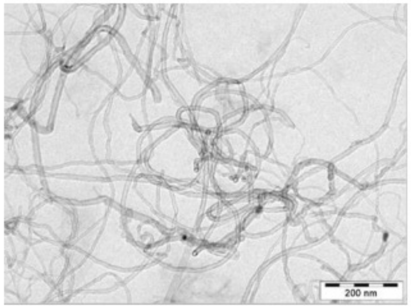
- Matrix: Homo PP, LG Chemical Co., LTD., Korea
- Reinforcement: CF 15LT, Mistubishi Rayon Co., LTD., Japan (15mm length, 12k)

❖ Reinforced materials

- **CNT:** M095, Carbon Nanotech Co., LTD., Korea, Purity 95%, CVD method,
  Diameter :5 ~ 20 nm, Length : ~10 μm, Aspect Ratio : > 500



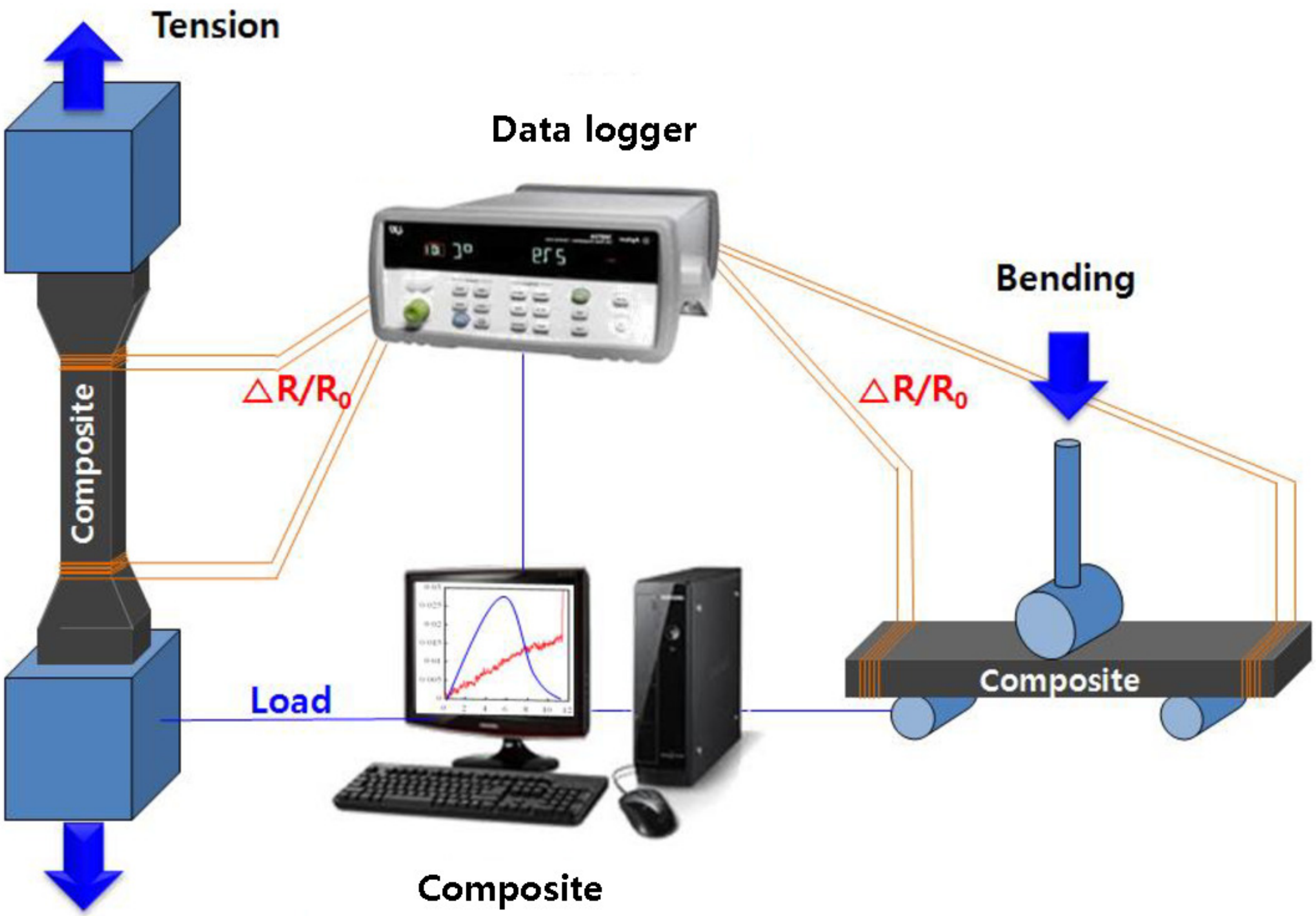




❖ Matrix

- **Epoxy:** YD-128, Kukdo Chemical Co., LTD., Korea, Bisphenol A type epoxy
- **Hardener**: T-403, Kukdo chemical Co., LTD., Korea, Amino type
  G-640, Kukdo chemical Co., LTD., Korea, Polyamide type

Mechanical test on ER measurement

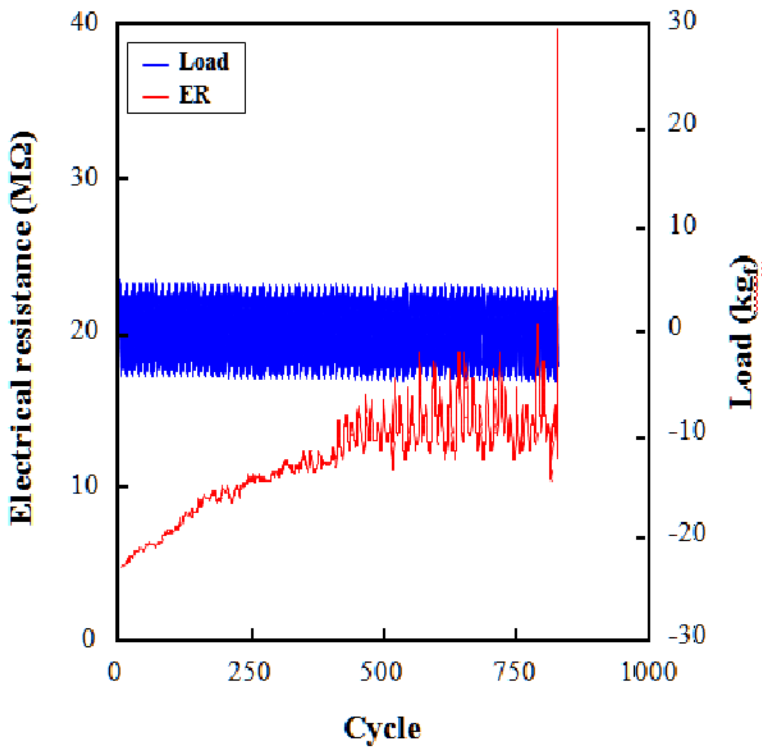


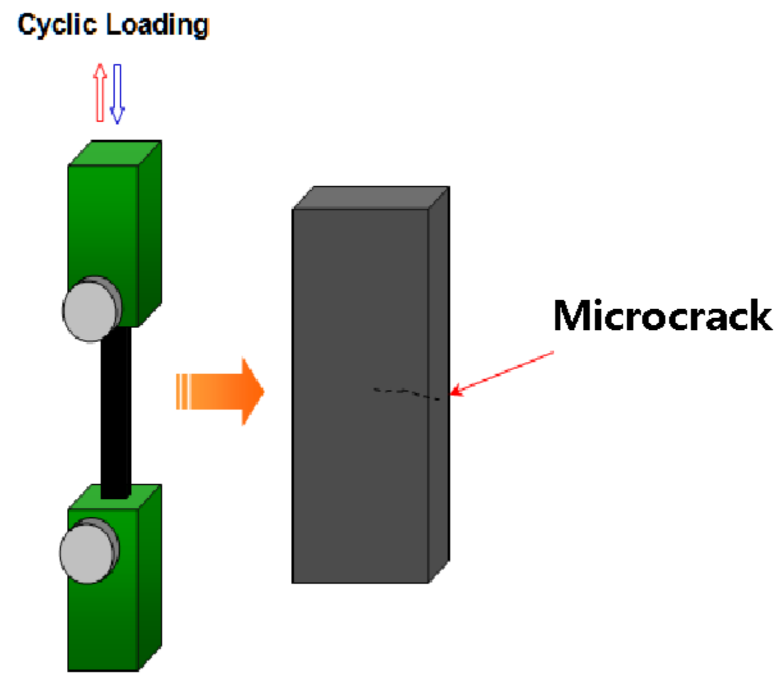
Big plate specimen


Tensile specimen


3 pt-Bending specimen

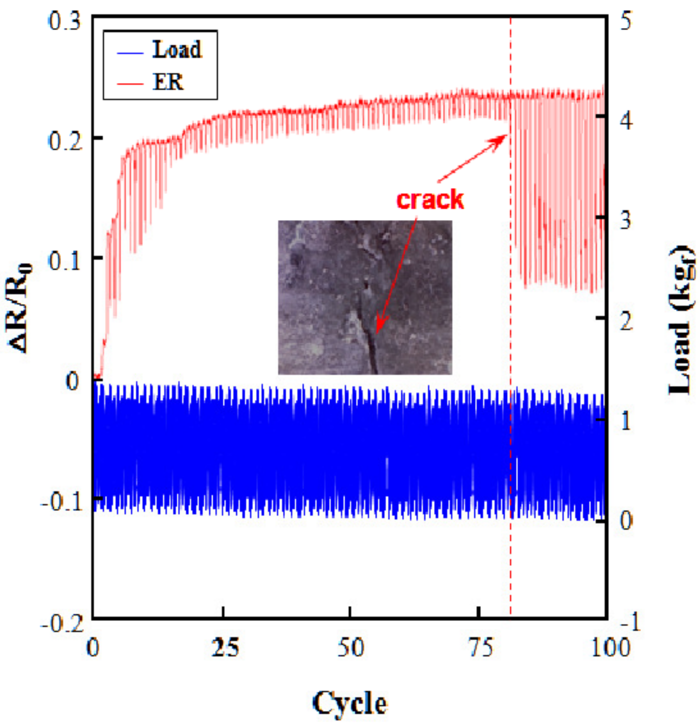
Fatigue test of CNT/CF-PP composites

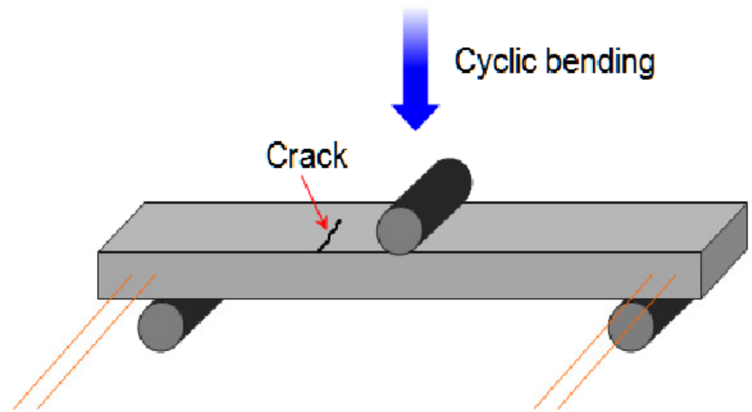




Condition
-Speed: 1mm/min
-Extension: 1mm

Cyclic bending test of CNT/CF-PP composites



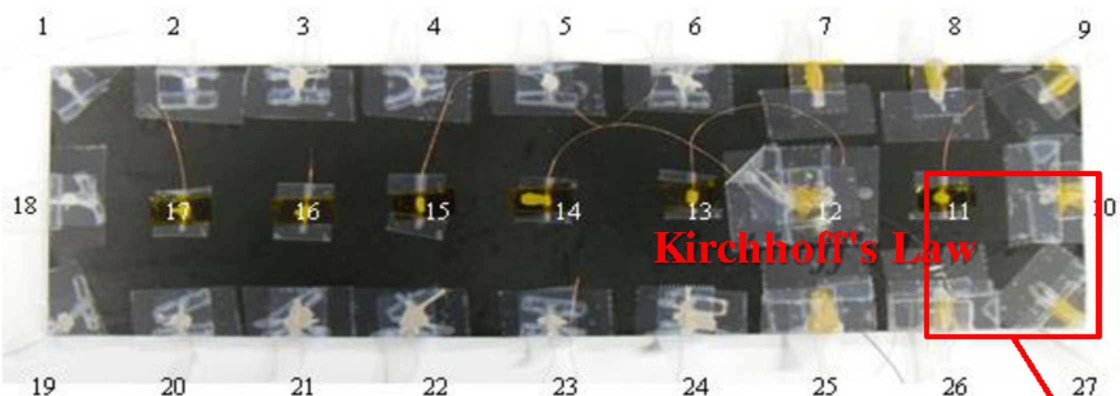


Test condition:

- Specimen: CNT-PP
- Concentration: 0.5wt%
- Test speed: 20mm/min
- Extension: 2mm

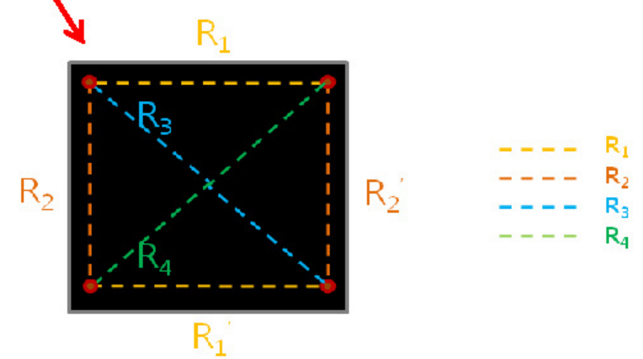
Dispersion evaluation obtained from electrical resistance change

- **measuring sectional electrical resistance → predict dispersion degree of conductive nanomaterials**
- **microdamage prediction for practical structural parts**



Length: 350 mm
Width: 100 mm
Thickness: 4 mm

Measurement model



- Specimen with conductive CF chops reinforced PP composites which were dispersed non-uniformly during manufacturing processing
- electrical connection with uniform intervals

Dispersion evaluation obtained from ER change

- measuring sectional ER → predicting dispersion conductive nanomaterials
- microdamage prediction for practical structural parts

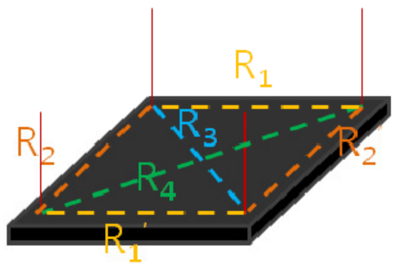
Method of arrange for data

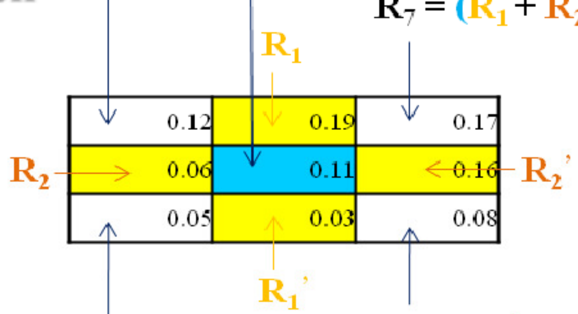
$R_9 = (R_1 + R_2 + R_5)/3$

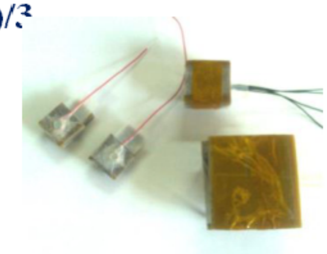
$R_5 = (R_3 + R_4)/2$

$R_7 = (R_1 + R_2' + R_5)/3$

1 cell







$R_6 = (R_1' + R_2' + R_5)/3$

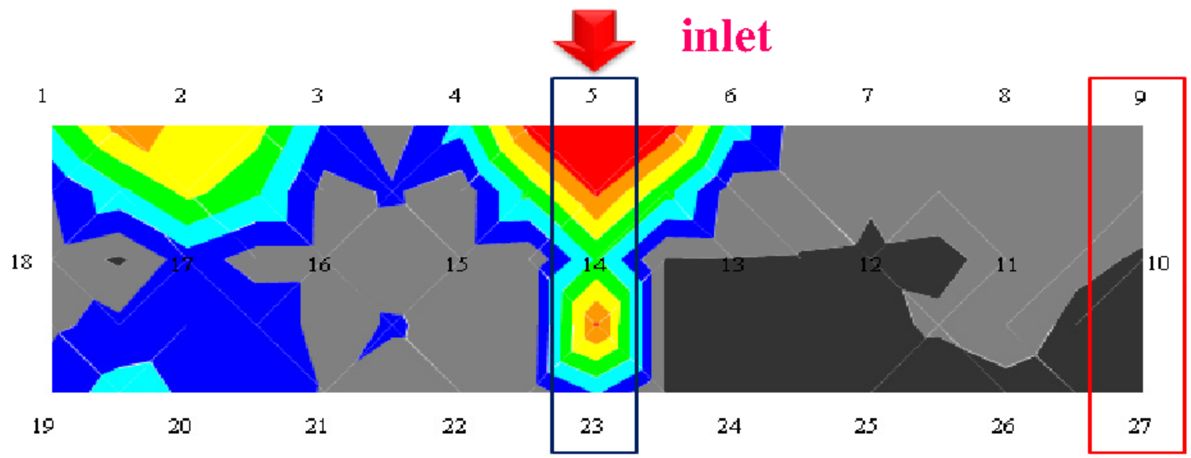
$R_8 = (R_1' + R_2 + R_5)/3$

Full data

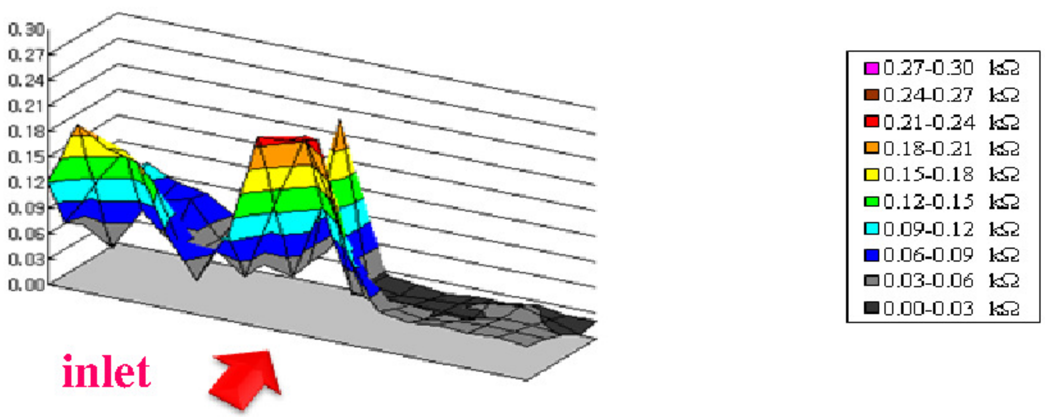
0.12	0.19	0.17	0.16	0.08	0.04	0.10	0.22	0.22	0.23	0.11	0.05	0.04	0.04	0.04	0.04	0.04
0.06	0.11	0.16	0.13	0.05	0.06	0.04	0.13	0.22	0.13	0.04	0.04	0.03	0.04	0.04	0.04	0.04
0.05	0.03	0.08	0.05	0.05	0.05	0.04	0.04	0.12	0.03	0.03	0.03	0.03	0.03	0.03	0.03	0.03
0.06	0.07	0.08	0.09	0.05	0.06	0.05	0.04	0.21	0.03	0.02	0.02	0.03	0.03	0.04	0.03	0.02
0.08	0.10	0.09	0.08	0.06	0.05	0.04	0.04	0.09	0.03	0.02	0.02	0.02	0.02	0.03	0.02	0.02

Image of dispersion distribution by electrical resistance change

- **measuring sectional electrical resistance → predicting dispersion conductive nanomaterials**
- **microdamage prediction for practical structural parts**

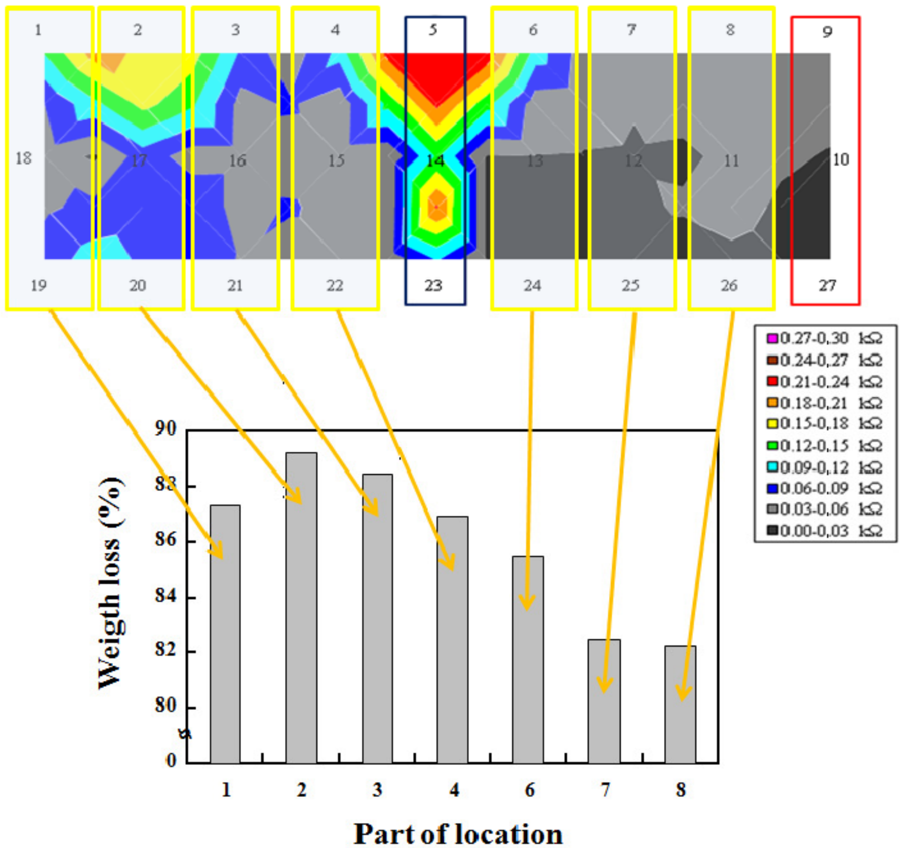


Condition
- Check length: 50 mm
- 2 probe method
- CF 10%
- Average : 0.067 kΩ
- S.D : 0.055



Dispersion evaluation from electrical resistance change and their proof by measuring burn weight

- Burning and weighting for each sectional parts
- ---> similar dispersion distribution result of electrical resistance



- Furnace heating condition: 400°C, 1 hour
- Specimen size: 20 x 40 x 3 mm

- Weighting loss % by burning after 400C for 1 hour
- Identical piece parts from same injection molding CF/PP 10% specimen but different CF amount exists
- Similar results as imaged ER plot

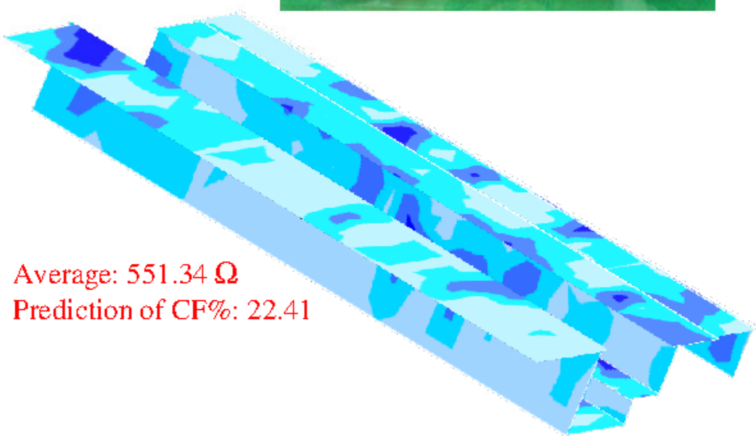
Hyundai Sunroof CFRP frame – measuring electrical resistance change

sectional dispersion distribution

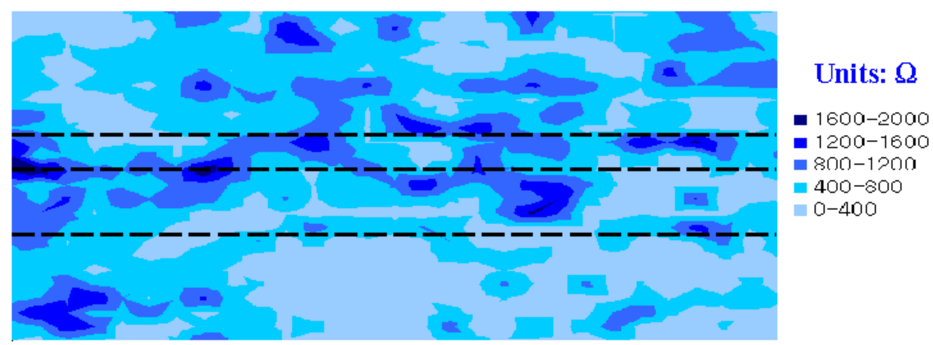
1) Total sunroof
2) Part of center bar
3) Part of side bar

Electrical resistance distribution for Side bar

- 20 wt% CF/PP-PA
- Sunroof parts side bar



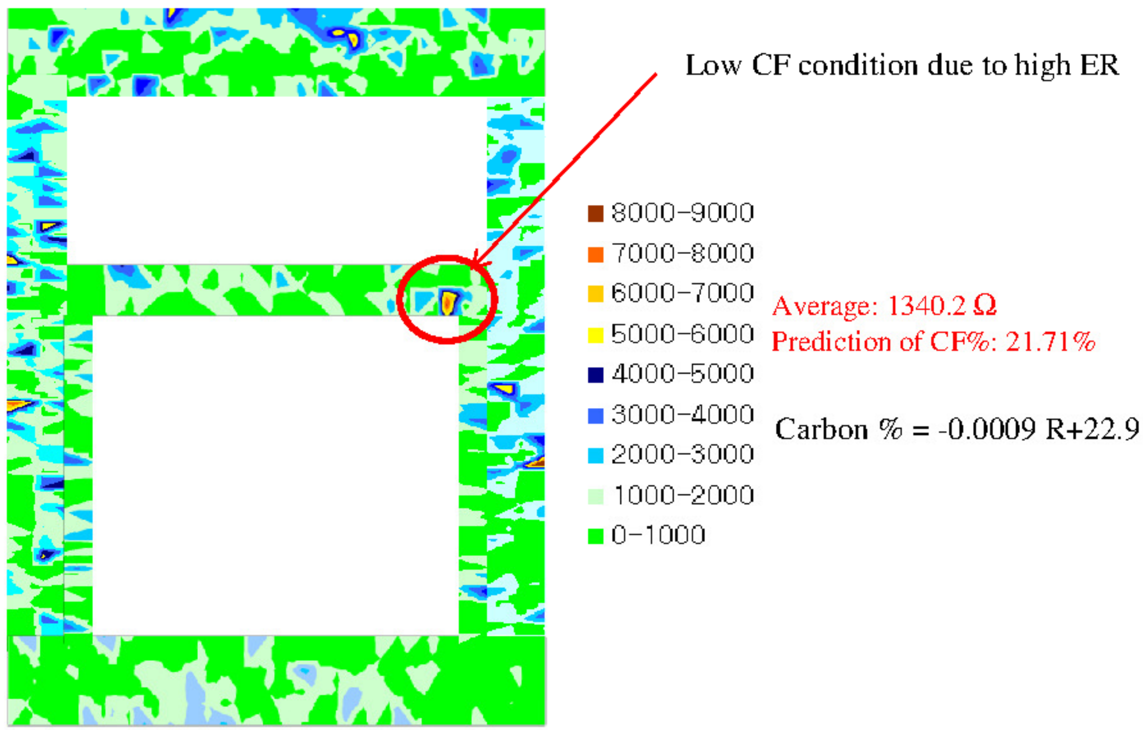
Average: 551.34 Ω
Prediction of CF%: 22.41



- Metal back bone embedded with CF 20wt%/PP-PA
  Deficiency of CF at bent corner compared to other regions

Electrical resistance distribution for sunroof

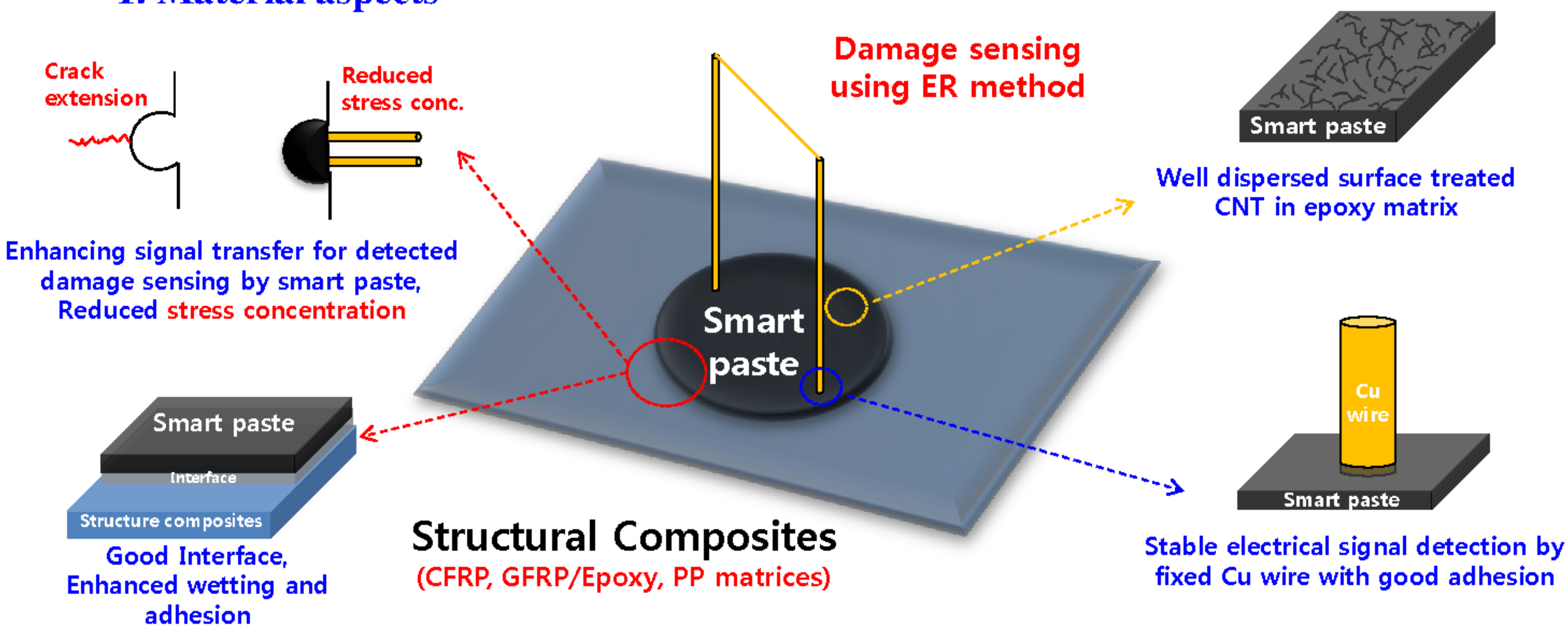
- **20 wt% CF/PP-PA**
- **Sunroof total parts**



Smart pastes for (enhancing) damage sensing

Why we study smart paste for damage-sensing?

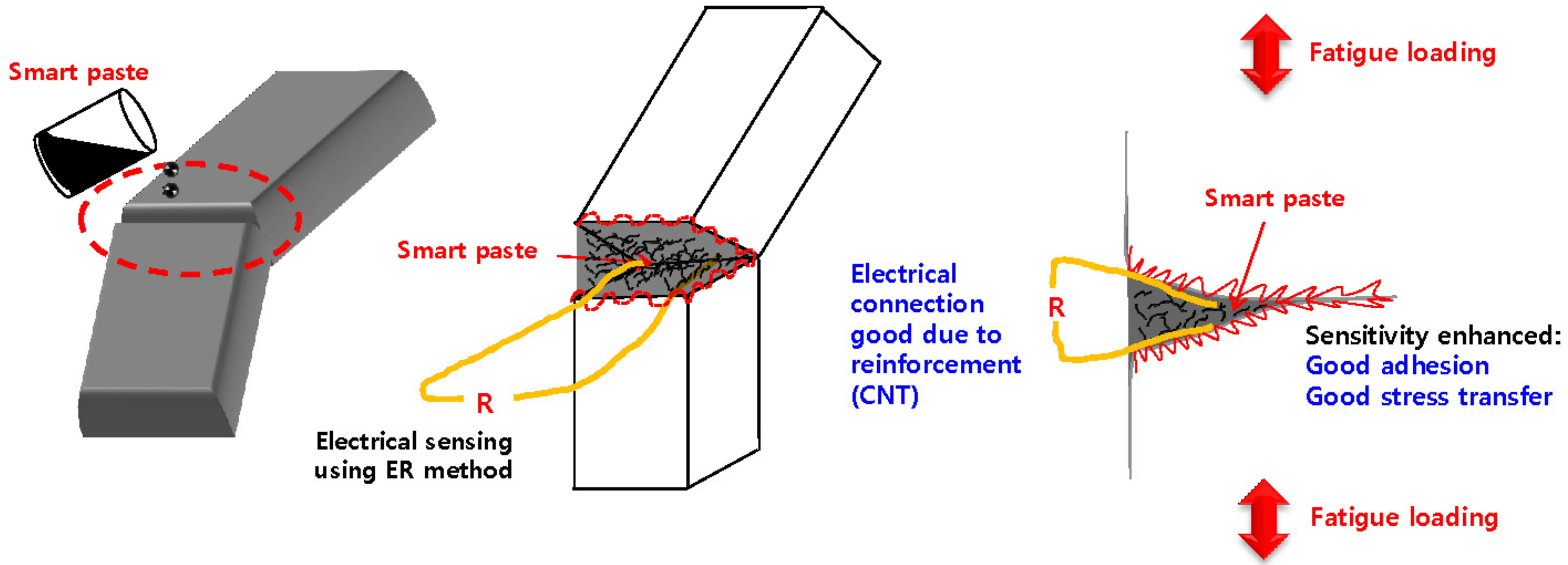
1. Material aspects



- Smart paste can be applied to enhance damage sensing for conductive CFRP, nonconductive GFRP (or even concrete structure)
- Smart paste was attached to surface of constructing materials
- Durable damage detection under severe situations

Why we study smart paste for damage-sensing?

2. Where to use?



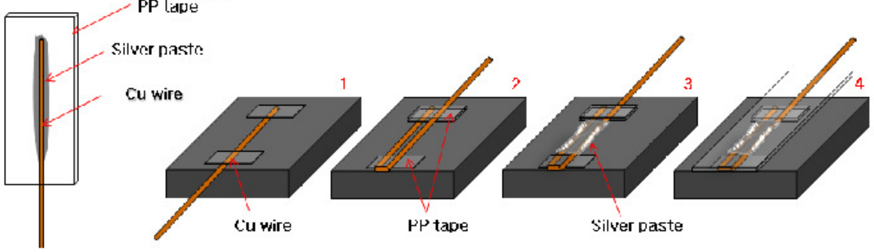
- Smart paste can be applied to structure materials for damage sensing by ER for damage location and crack propagation inside structural materials.

Why study smart paste for damage-sensing?

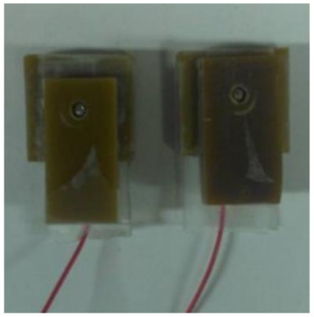
3. Electrical connection point factor

Connection methods for ER damage sensing method

• Adhesive type

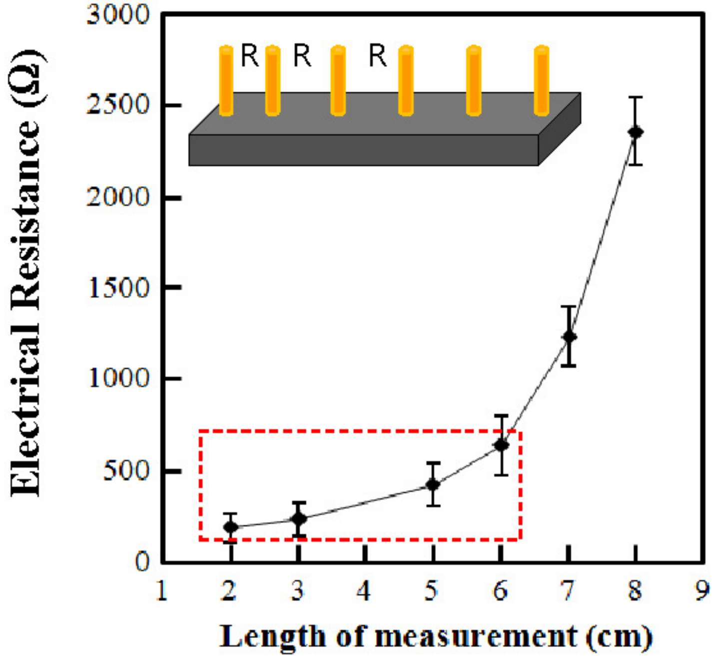


• Probe type



• Probe type

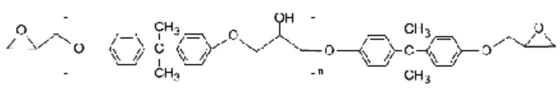
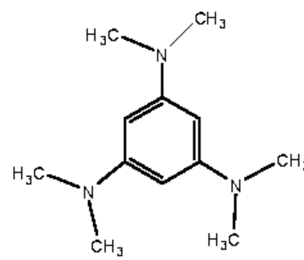
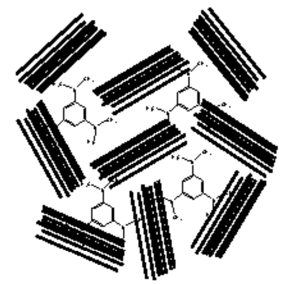
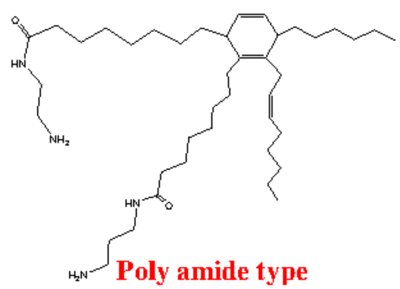
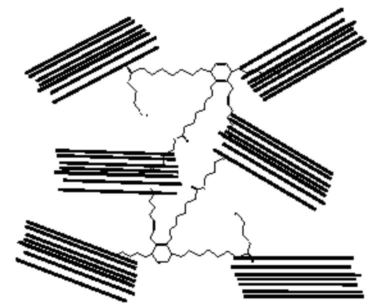
Test gap effect of ER damage sensing



- Smart paste was uniform size for damage sensing test method. (2cm length of smart paste)
- Electrical connection noise of smart paste was much smaller than neat adhesive or probe type methods.

Experimental

using 2 different hardeners

Company	Type: Resin	Type: Hardener	Product	etc.
Kukdo Chemical Inc., Co., Korea	 Bisphenol A type YD-128	 Amino type T-403		
		 Poly amide type G-640		

CNT: M095, Purity 95%, CVD method, Carbon Nanotech, Korea

Bulk size nano-dispersion manufactured system

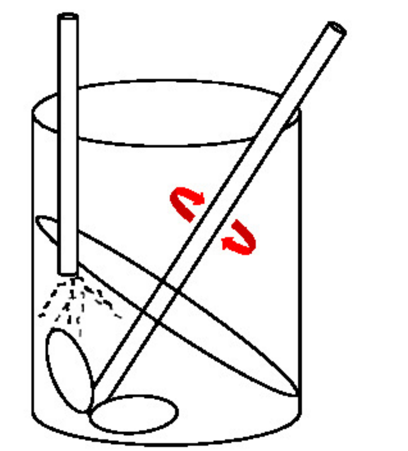
Comparison of volume
Between CNT paste and CNT powder

10wt% CNT paste
1g

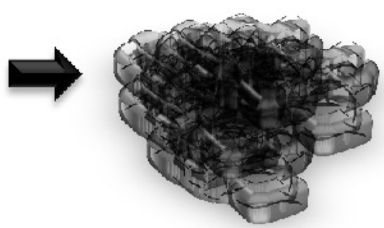
>

CNT powder
0.1g

(1) Manufacture 10wt% CNT paste



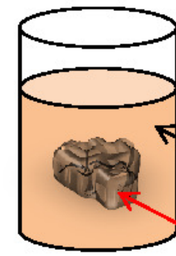
Inserted 10g CNT powder
in epoxy (Bisphenol A type,
Kukdo chemical Co., YD-128)



10wt% CNT paste

- 2 hours
- Stirrer: 500rpm
- Sonication: Amplitude 50%, cycle 1s
- 100g resin, 10g CNT

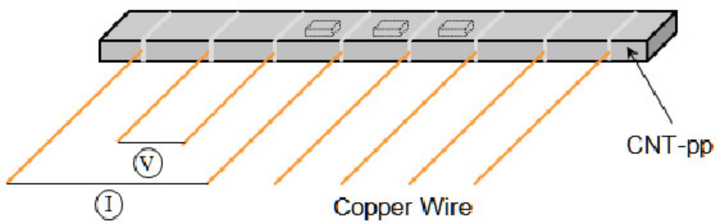
(2) Manufacture 1wt% Smart paste (dilute)

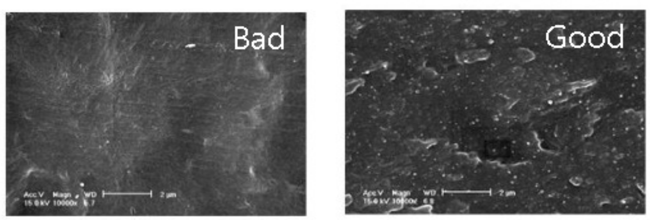


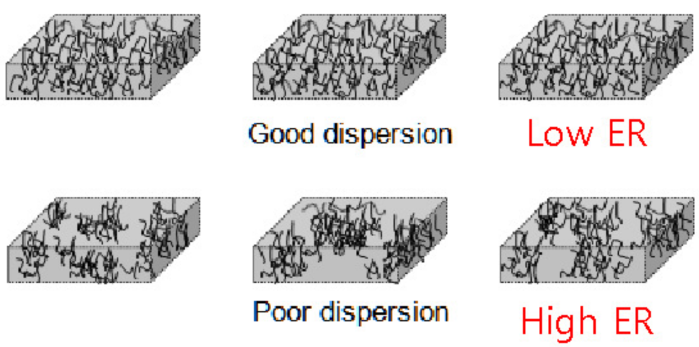
- Easy mixing
- Easy manufacture of nanocomposites
- Improvement of working condition

Testing method and model

(1) Dispersion evaluation using electrical resistance method



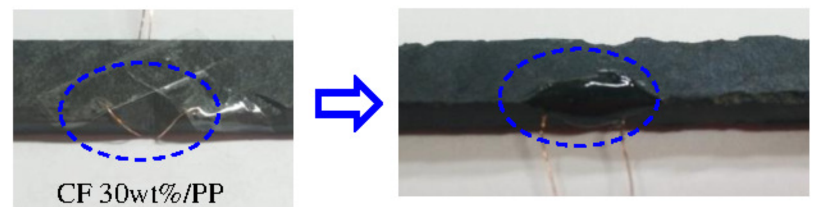




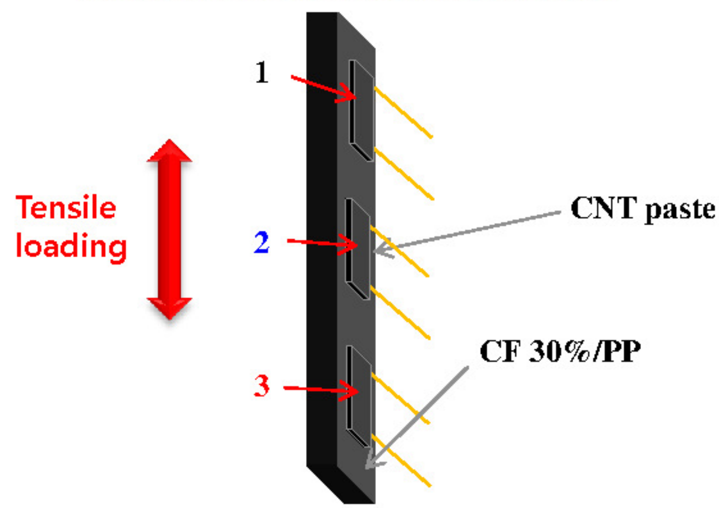
(2) Damage sensing with crack extension using electrical resistance method

Artificial crack

Fill a crack point with CNT epoxy paste

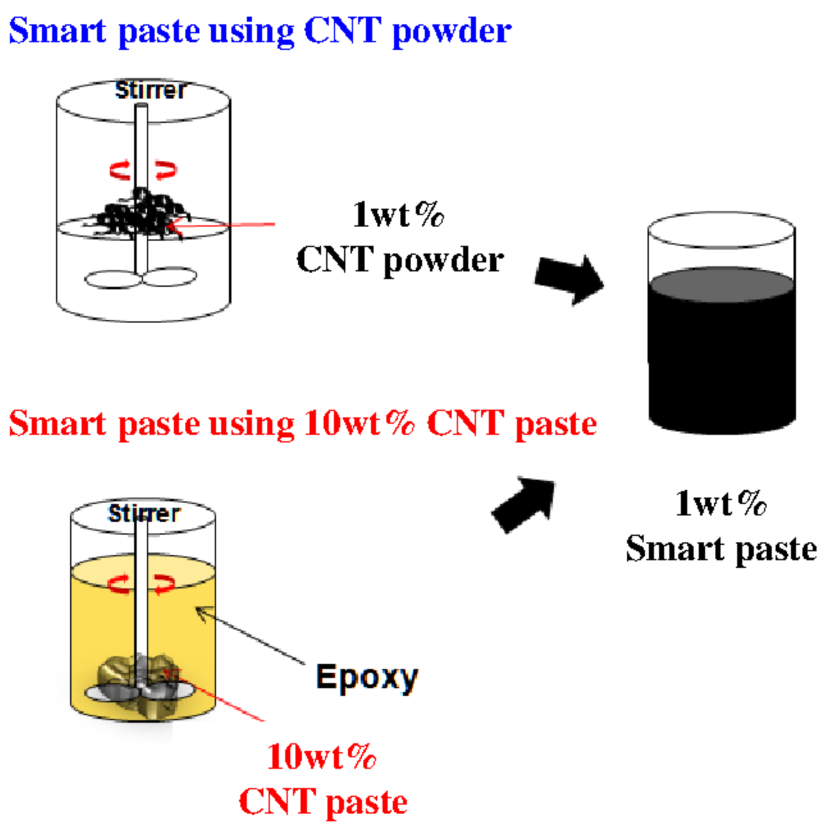


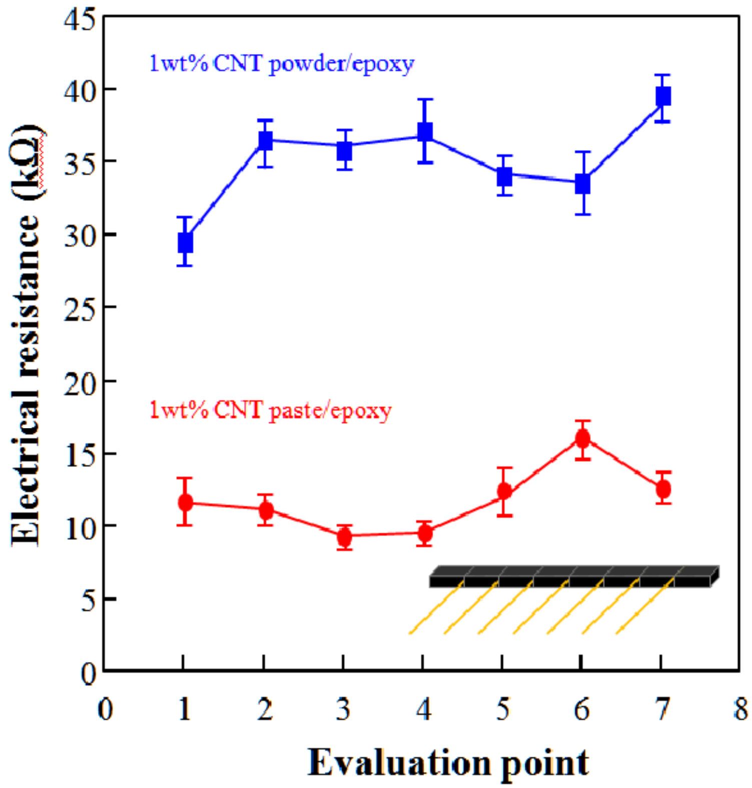
(3) Damage sensing with crack location using electrical resistance method



Results and Discussion

Simplification of manufacture method

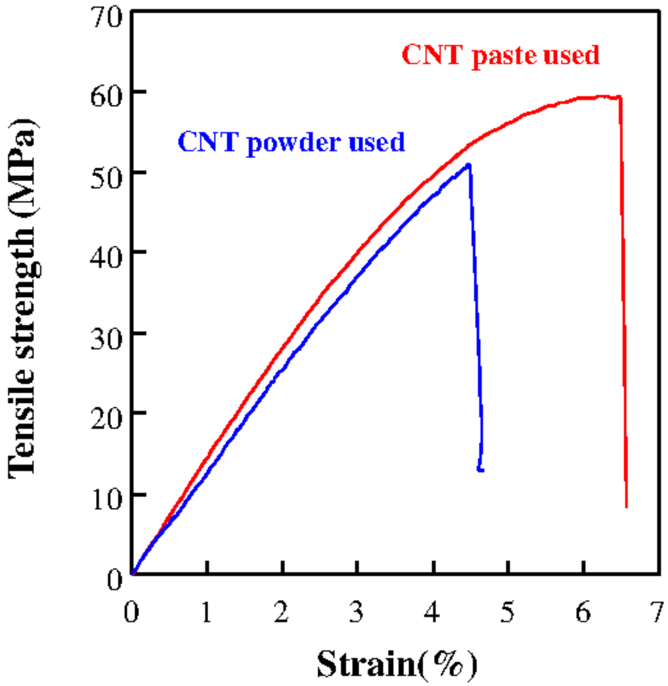


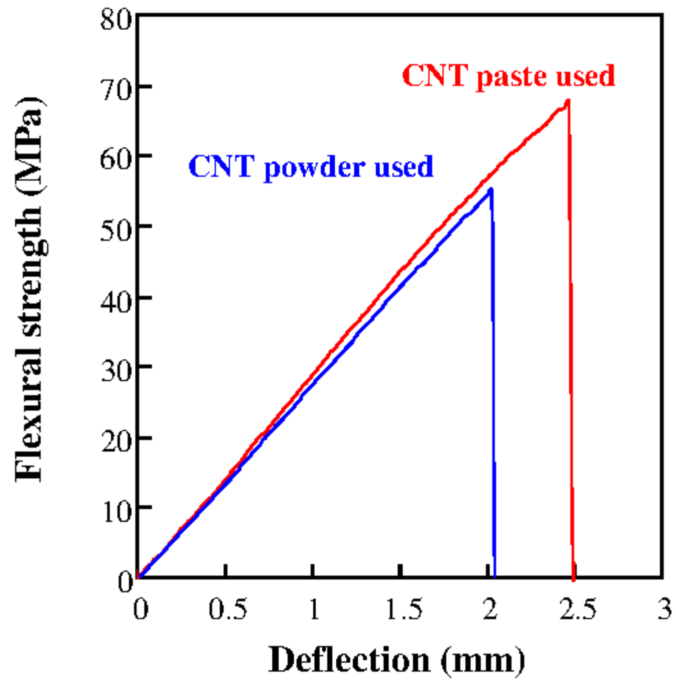


- High concentration of CNT paste was useful to manufacture for smart paste than CNT powder.
- 10wt% CNT paste was good dispersion condition better than CNT powder. Due to ahead of time, 10wt% CNT paste was treated sonication method.

Simplification of manufacture method

Mechanical test of smart paste with different manufacture and testing methods

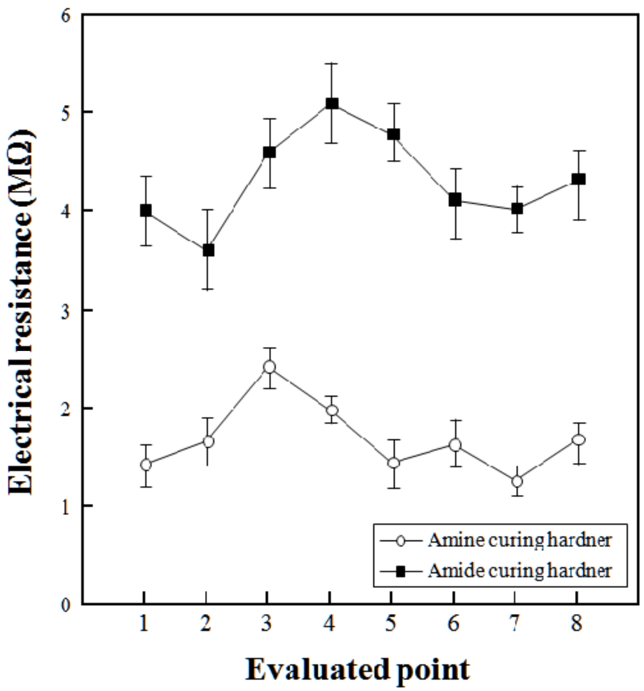




- 10wt% CNT paste used smart paste was better than CNT powder used smart paste due to good dispersion condition

Optimum condition of epoxy conformation

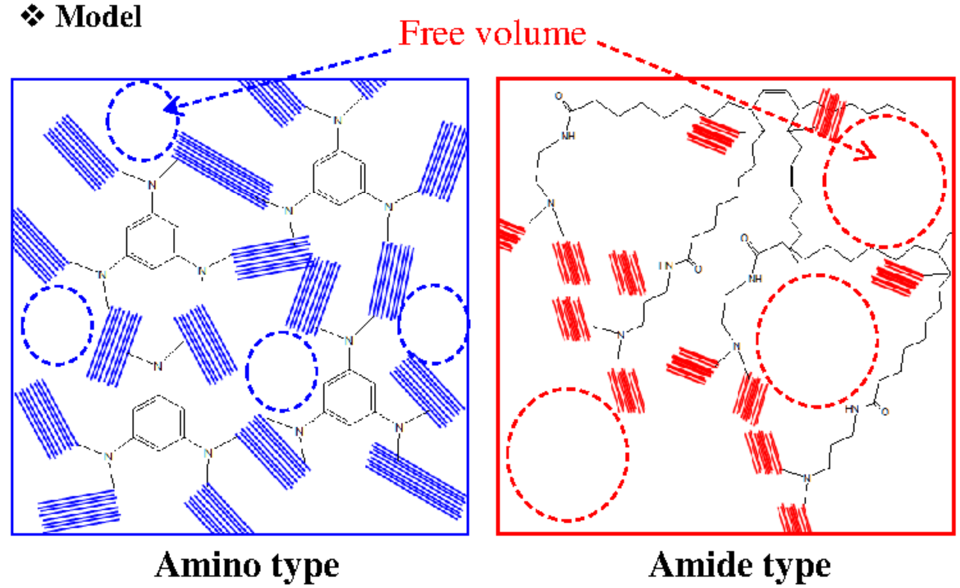
Dispersion test of smart paste with different epoxy hardeners



❖ **Condition**

- CNT: M090 1wt%
- YD128
- Hardener: amine type: T-403
  polyamide type: G640

❖ **Model**



- Amino type epoxy condition of smart paste was optimum for dispersion condition.
- Amino type epoxy smart paste was small size of free volume. Nanoparticle dispersion in epoxy was optimum. (Good electrical sensing and useful damage sensing)

Optimum condition of epoxy conformation

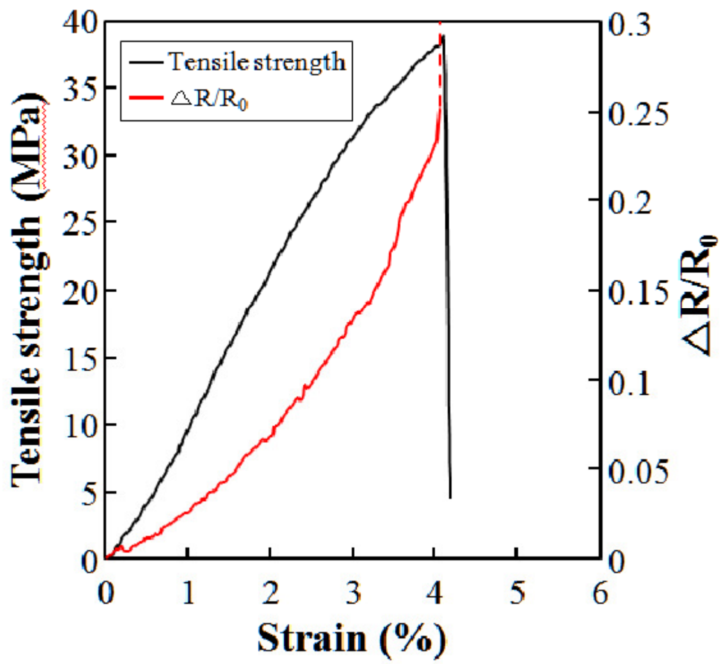
Dispersion test results of CNT/epoxy pastes with different hardeners

Epoxy	Hardener	Conc.	Units	1	2	3	4	5	6	7	Average	S.D	C.O.V.
YD-128	G-640	1wt%	MΩ	4	4	5	6	9	6	7	5.9	1.8	0.3
		2wt%	MΩ	131	272	153	144	155	92	81	144	62	4
	T-403	**1wt%**	**MΩ**	**1.2**	**1**	**0.9**	**1.3**	**1.6**	**1.4**	**1.2**	**1.2**	**0.2**	**0.2**
		2wt%	kΩ	173	209	239	327	375	281	275	268.4	68.9	0.3

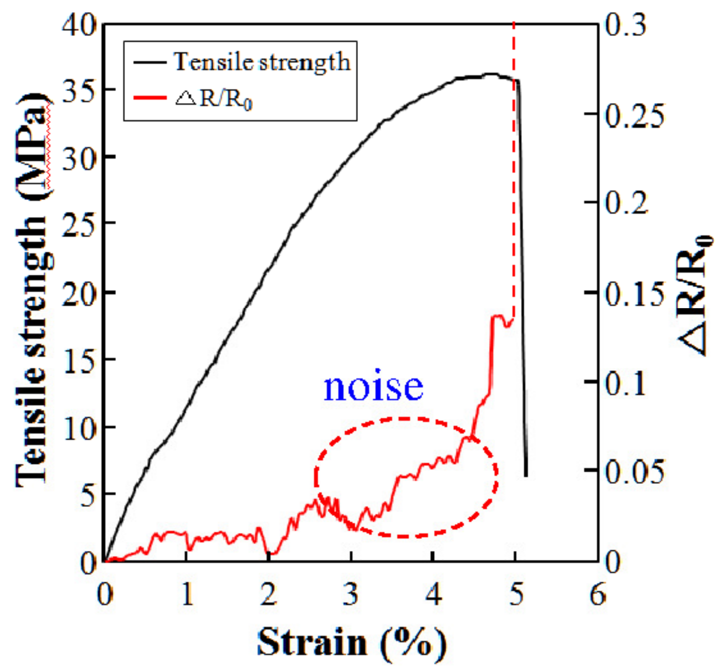
- Epoxy : Hardener = 100:50

- 1~2MΩ electrical resistance condition was optimum for damage sensing using 1wt% CNT concentration and amino-type T-403 hardener.

Tensile loading sensing test of CNT/epoxy pastes with different hardeners

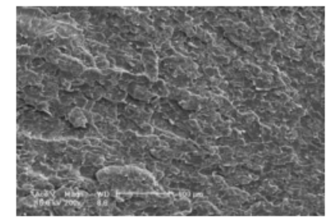


Amino type



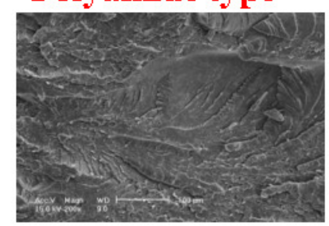
Polyamide type

Amino type



Hard fracture case

Polyamide type

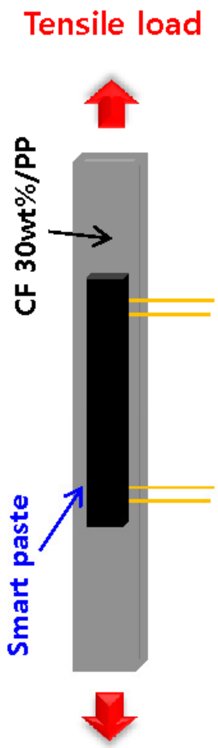


Soft fracture case

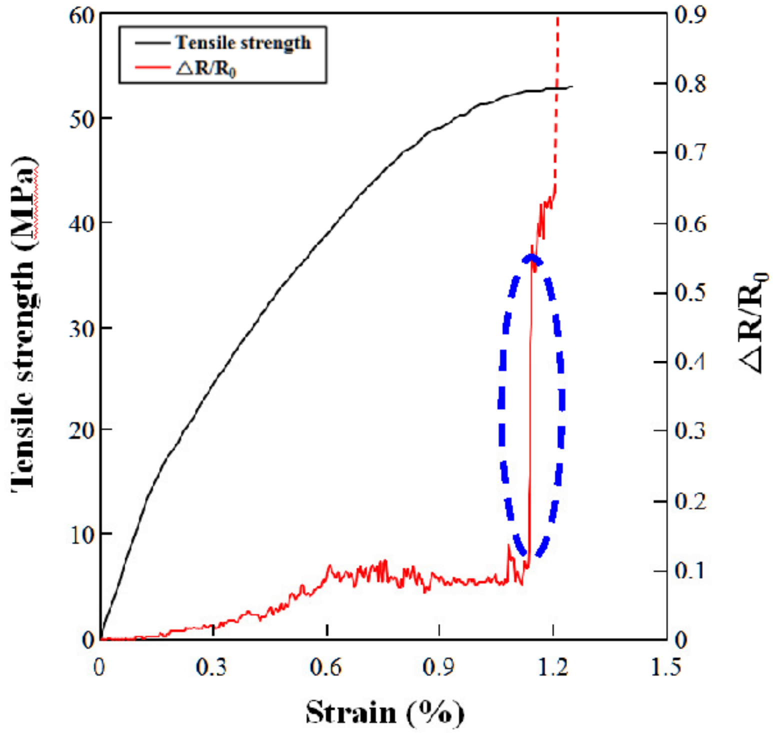
- Amino type smart paste showed uniform electrical resistance as tensile loading.
- Polyamide type smart paste showed un-uniform electrical resistance with many electrical noise.

Tensile loading sensing test

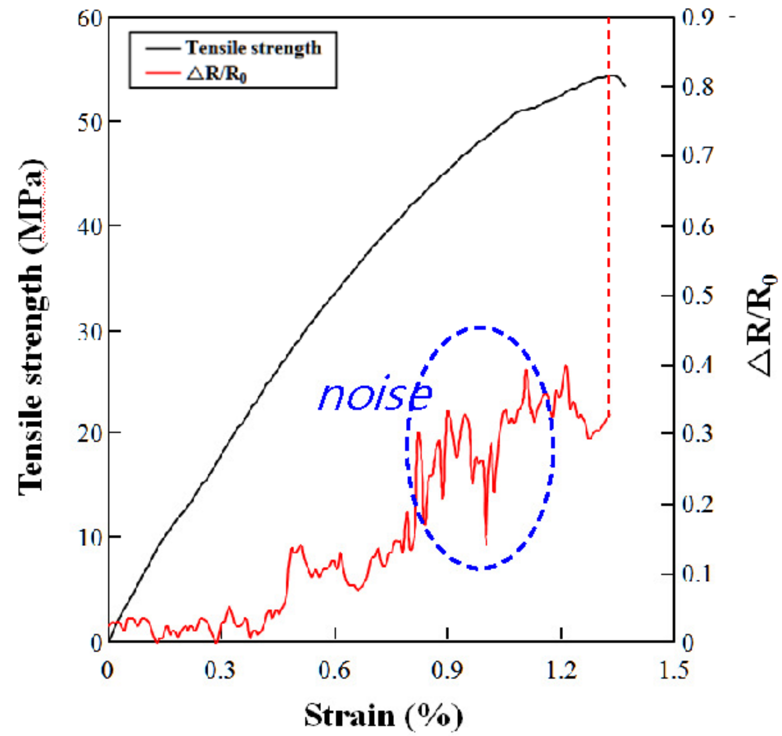
Tensile test of CNT paste coated CF 30wt%/PP



Condition
- 1mm/min
- 2mm thickness
- 20mm length



Amino type

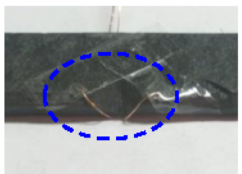


Polyamide type

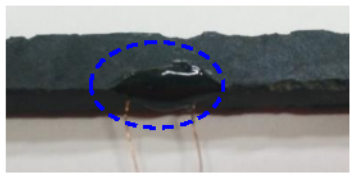
- Amino type smart paste was investigated to uniform electrical signal jumping results.
- Electrical jumping results means as soon as possible fracture at body.

Crack extension sensing of CNT/epoxy pastes

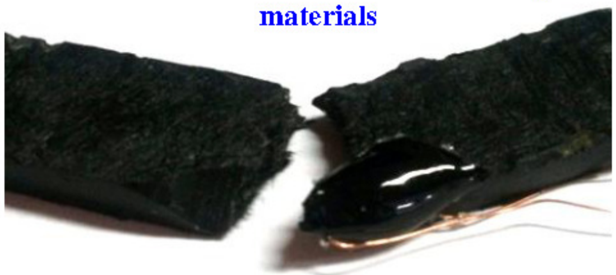
Artificial crack

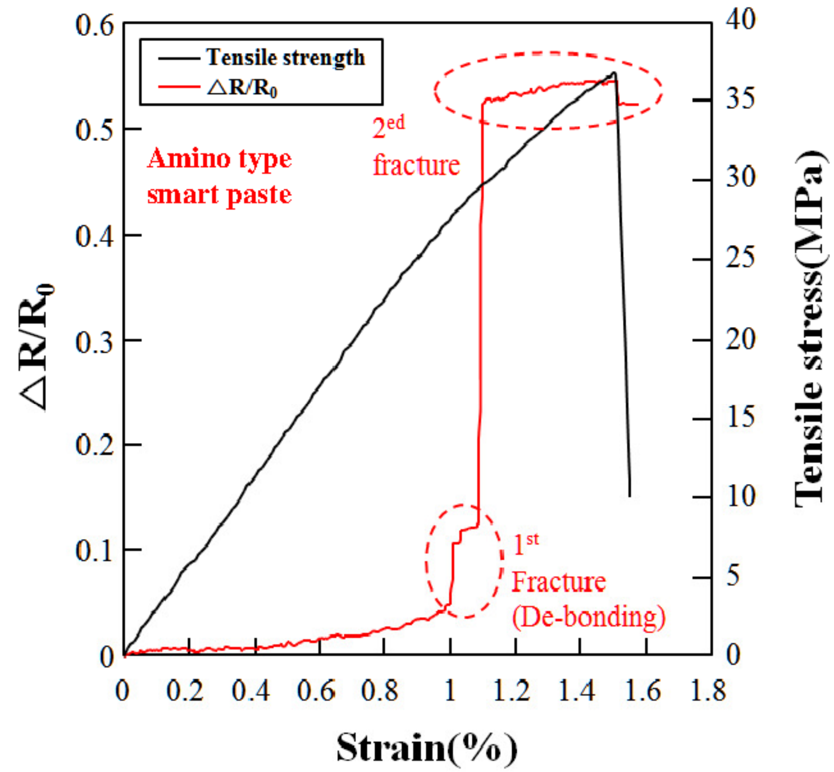


Fill a crack point with CNT paste



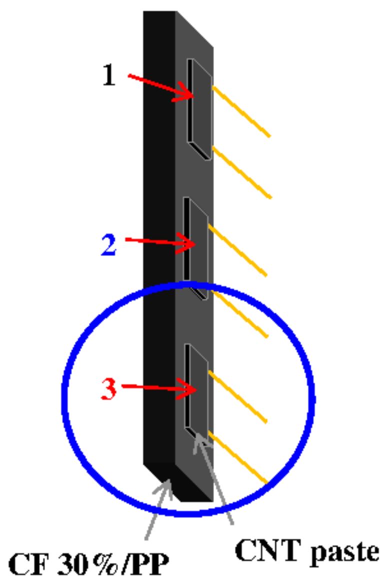
Fracture was started from artificial crack point of materials





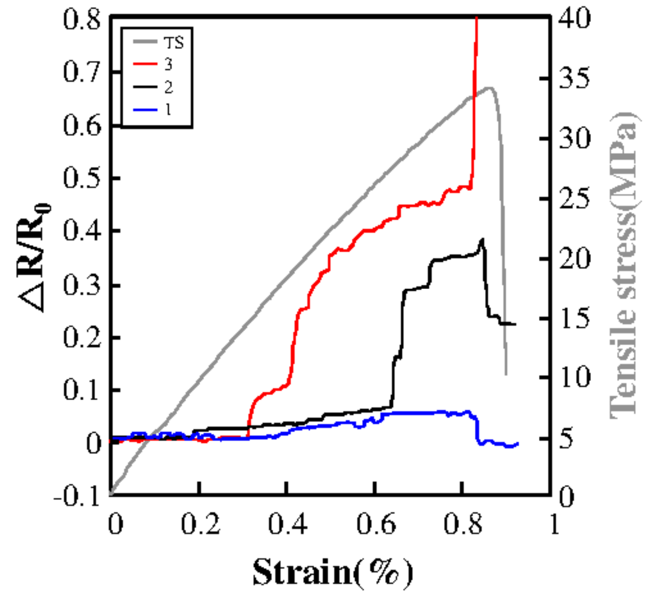
- Fracture 2 steps: Interface between smart paste and CF 30wt%/PP →Crack extension in CF 30wt%/PP → Fracture
- Electrical resistance change ratio jumping was predicted to fracture interface between smart paste and CF 30wt%/PP

Cracking location sensing of CNT/epoxy pastes

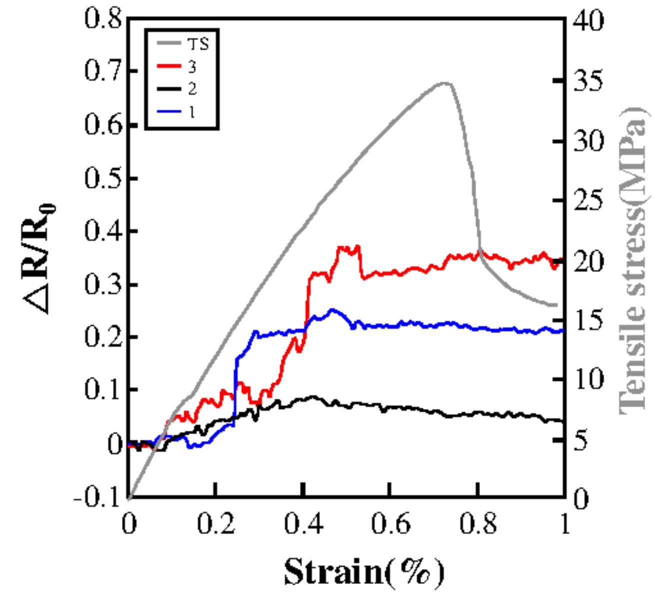


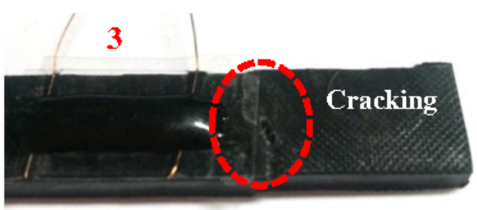
Generally, top point was fractured.

Amino type smart paste



Polyamide type smart paste





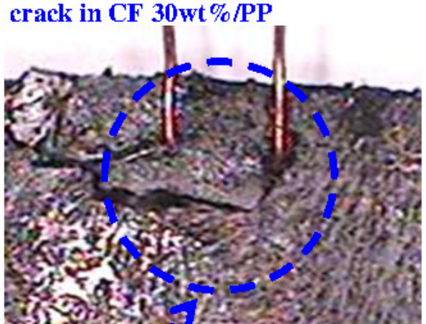
-Electrical jumping signal of amino type smart paste was easily observed due to fatal cracking.

- Polyamide type smart paste was un-uniform electrical jumping signal. (?)
- Good smart paste condition was amino type.

Crack blunting effect of CNT/epoxy paste

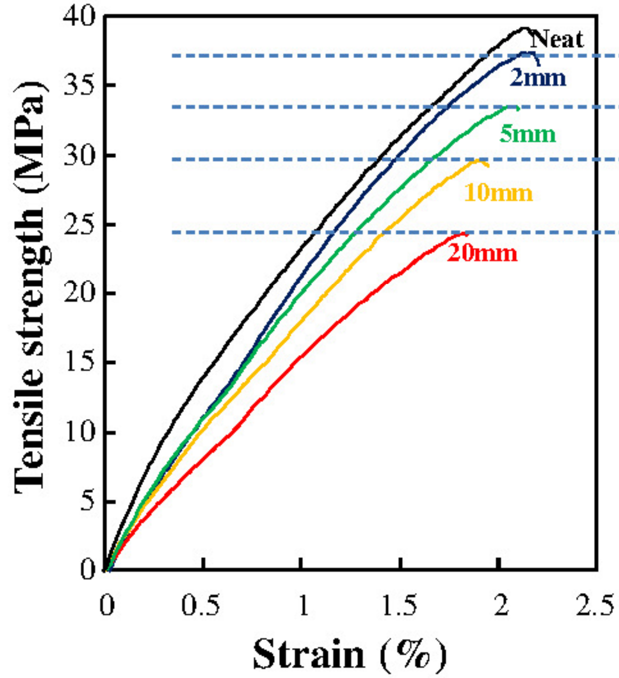
Perfect fractured
at artificial crack

Smart paste was filled artificial
crack in CF 30wt%/PP

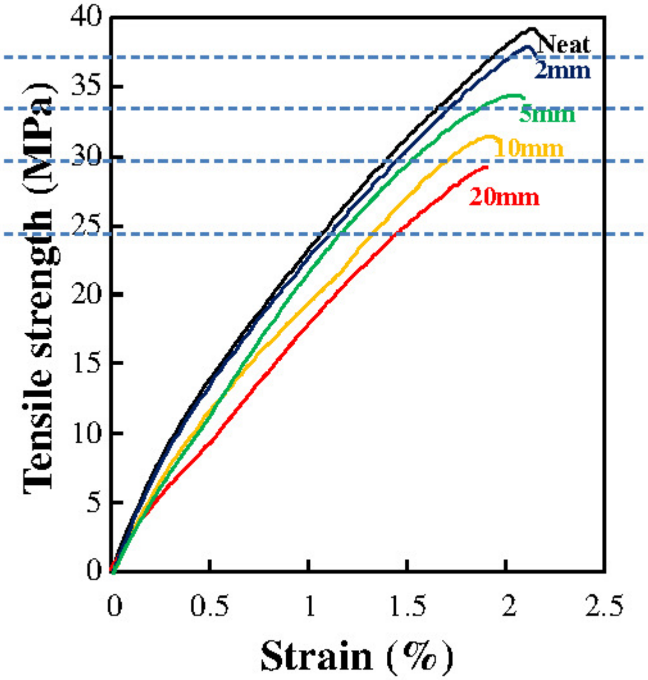


Reduced crack extension
at artificial crack

Artificial crack of CF 30wt%/PP



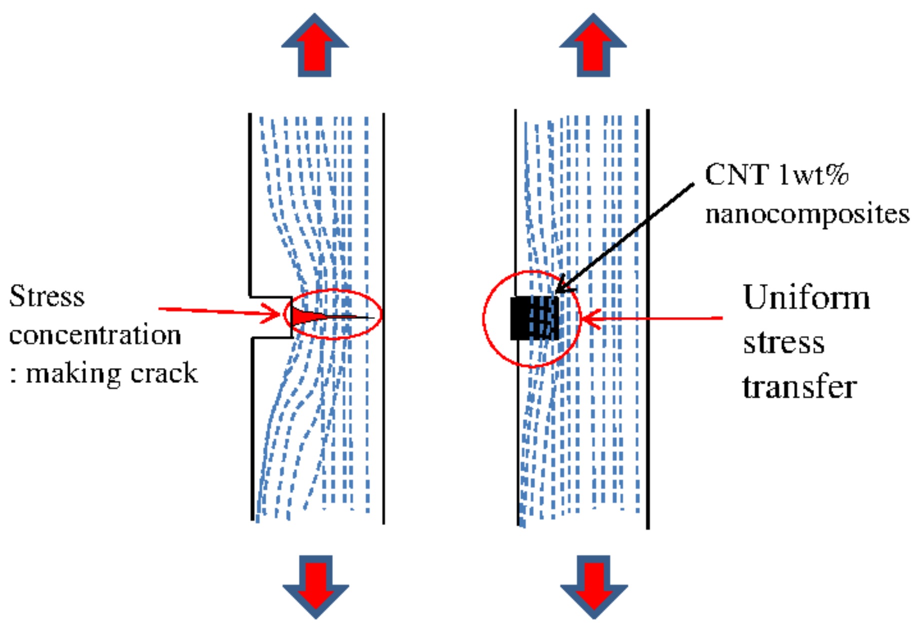
Smart paste was filled at
artificial crack of CF
30wt%/PP

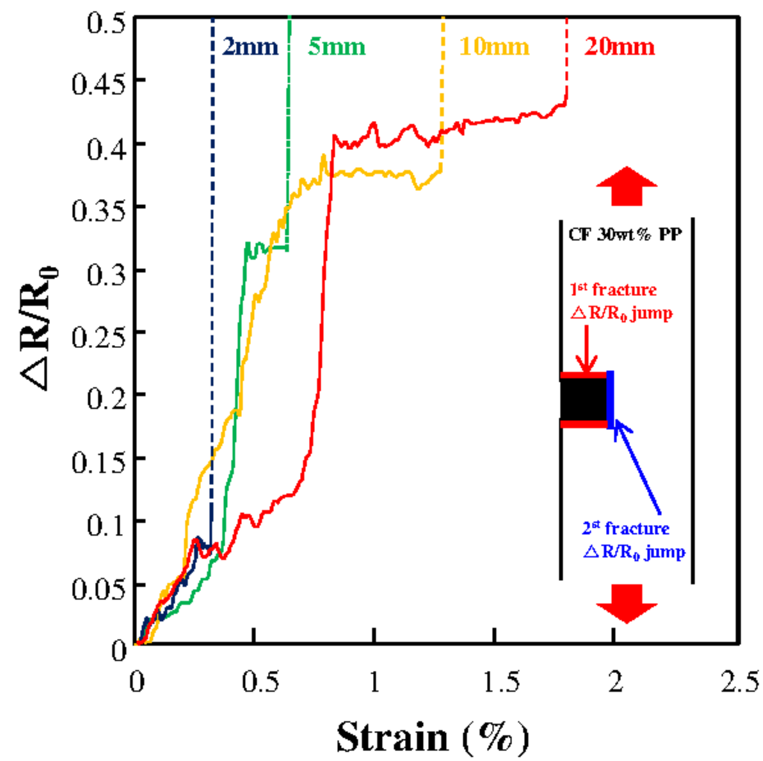


- Smart paste repairing effect was observed with a few stress more until fracture.
- Crack repairing effect increased with increasing crack size.

Crack reducing effect of CNT/epoxy pastes

Crack reducing effect of smart paste with crack size





- Large artificial crack size was good sensing to damage at crack point.
- Electrical jumping signal made by interface de-bonding between smart paste/CF 30wt% PP.
- **Electrical jumping signal was predicted to fracture in structure matrix as soon as possible after loading.**

Conclusions

- Conductive smart paste was composed of 10 wt% CNT in epoxy as paste and can be applied practically for detecting the fracture, cracking, shear slip in structural parts more durably and stably.
- Conductivity and dispersion condition of CNT paste with amino hardener was better than amide hardener due to different cross-linking density and free volume effects.
- Interfacial adhesion between smart paste and structural parts will be very important to transfer stress and electrical resistance signal stably from substrate through smart paste medium to monitoring machine.

Acknowledgements

- Defense Acquisition Program Administration and Agency of Defense Defense Development (ADD) (2012-2014), Korea.
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- Ministry of Knowledge Economy, KIAT (2012-2015)
- Defense Acquisition Program Administration, DAPA (2013-2018)
- Doosan Heavy Industries & Construction, Korea (2014-2015)
- Appreciation for Distinguished Professor Lawrence K. DeVries, Department of Mechanical Engineering, The University of Utah (2003-Now)

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