量子理工学概論IV
講義1

ナノカーボンの調製と
エネルギー・環境デバイスへの応用

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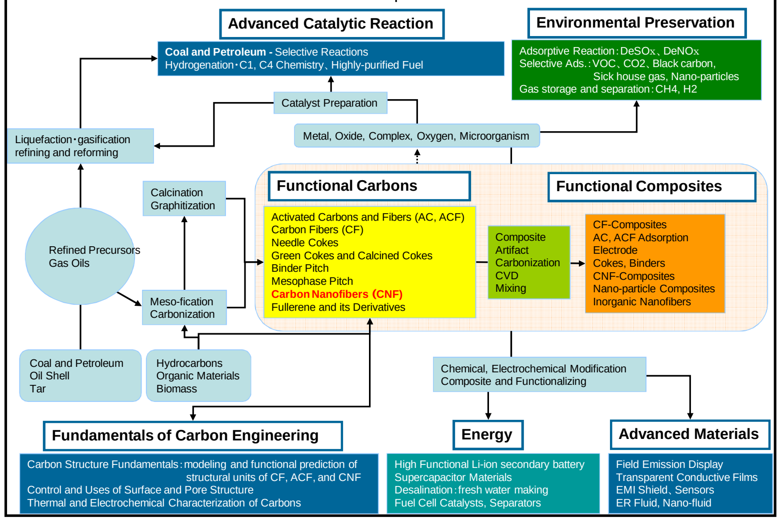
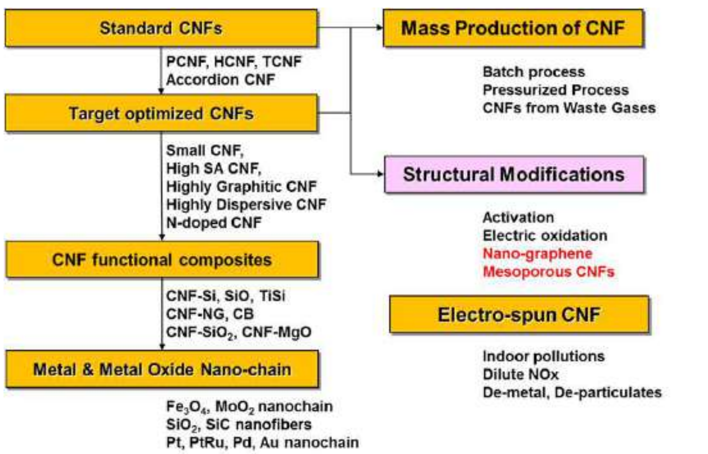
Research scope of Yoon's Lab

- Synthesis and applications of functional materials
- Energy and environmental engineering

- Outline and Interrelation of Research Topics



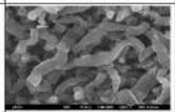
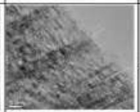
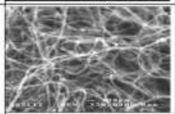
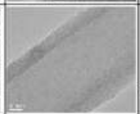
Flow chart of CNF studies in Yoon Lab



Short introduction of carbon nanofiber study in Yoon Lab

- Carbon nanofiber general
- Preparation and structural analysis of carbon nanofibers

Standard CNFs

Sample	SEM	TEM	Properties	Applications	Etc.
KNF-SPR Platelet Nano-rod			Platelet high grapht. deg. 80 – 400 nm, SA 90 m^2/g d_{002} 3.36Å, Lc(002) 30 nm	Catalyst support	70 g//day
KNF-SH Herring-bone			Herringbone high surface area 70 ~ 500 nm, SA 150 m^2/g d_{002} 3.45Å, Lc(002) 3 nm	Composite filler	100 g/day
KNF-ST Tubular Highly graphitic			Tubular thin walls, open tips high grapht deg. 20 ~ 50 nm, SA 90 m^2/g d_{002} 3.37Å, Lc(002) 13 nm	Composite filler	20 g//day
KNF-FM Tubular Small diameter			tubular, hollow 5~15 nm, 4 -7 walls	Composite filler Catalyst support	20 g/day

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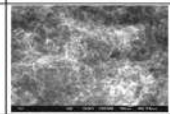
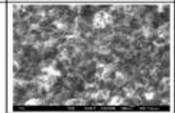
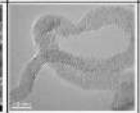
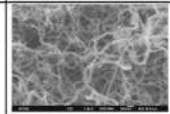
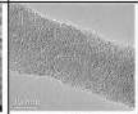
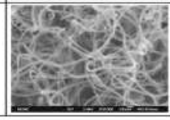
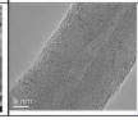
CNF (Small & Middle Diameters)

Sample	SEM	TEM	Properties	Applications	Product
KNF-CM Small Highly dispersive			Herringbone, hollow 7 ~ 20 nm	Composite Catalyst support FED	20-30 g/ day
KNF-CC Small			Herringbone 7 ~ 15 nm	Composite Catalyst support	15-20 g/ day
KNF-NM Middle			Herringbone 10~60 nm (30~40)	Composite Catalyst support	50-70 g/ day
KNF-NF Middle linear			Herringbone 20 ~ 50 nm Straightness	Composite Catalyst support	50-70g/ day

CARBON 42 (8-9): 1773-1781 2004

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Structural variety of CNFs

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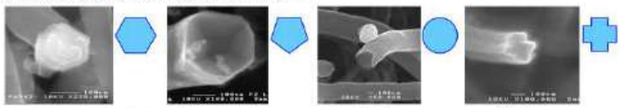
Typical classification of CNF Structure

- graphene ((002) layers) alignment to the fiber axis, TEM observation

< Simple cases of CNF structure >

- However, complicated structure is often found.
- The morphological diversity confirmed simply by SEM observation cannot be neglected, considering possibly their different physical properties.

Various cross sections of CNFs



Polygonal Circle Cross

Different Surface Characteristics

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Surfaces of PCNF

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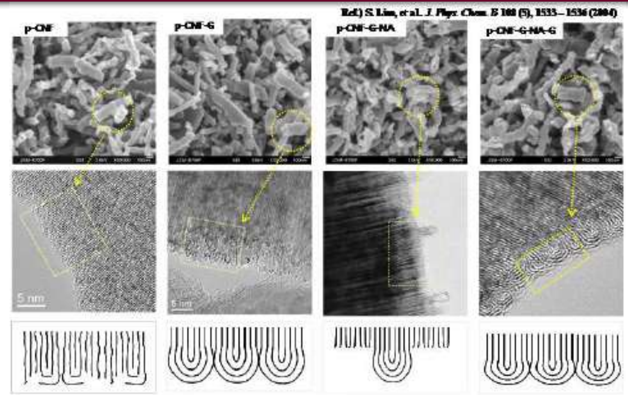

According to the graphitization degree,
we found some difference at edge plane by TEM analysis

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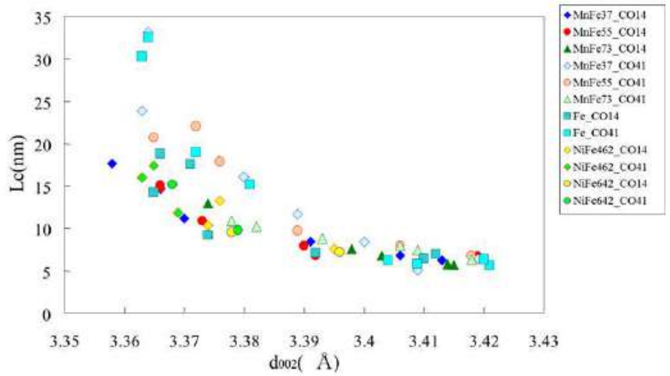
J PHYS CHEM B 108 (5): 1533-1536 FEB 5 2004

Control of Graphitic Properties of TCNFs



Carbon, 42, 1279-1283, 2004

Highly graphitic CNFs

- CNF of similar graphitic properties with Natural Graphite
- CNT usually shows low graphitic properties
- Conductive materials or supports for heterogeneous catalysts

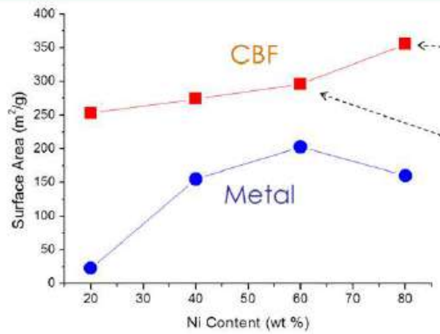
GPCNF-N

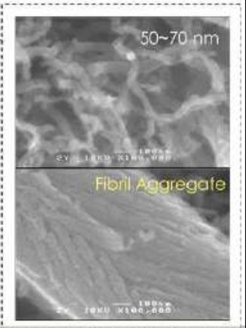
PCNF, HCNF
↓ 黒鉛化
GPCNF
↓ 硝酸処理
G-PCNF-N
↓ B黒鉛化
BA-GGPCNF-N

	Preparation conditions	d_{002} (nm)	Lc(002) (nm)
PCNF	Fe catalyst, 620, CO/H2 : 4/1	0.3365	72
G-PCNF	2800℃ heat treatment of PCNF	0.3364	83
G-PCNF-N	30% HNO3 treatment of GPCNF for 50℃, 8hs	0.3362	152
GG-PCNF-N	2800℃ heat treatment of GPCNFN	0.3362	106
BA-G-PCNF	Boric acid added heat treatment of PCNF	0.3359	115
BA-GG-PCNF-N	30% HNO3 treatment of GPCNF for 50℃, 8hs Boric acid added heat treatment	0.3357	377
BC-G-PCNF	Boron carbide added heat treatment of PCNF	0.3354	178
BC-GG-PCNF-N	30% HNO3 treatment of GPCNF for 50℃, 8hs Boron carbide added heat treatment	0.3354	167

J. Phy.Chem. C to be submitted

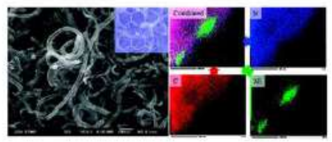
Control of surface area

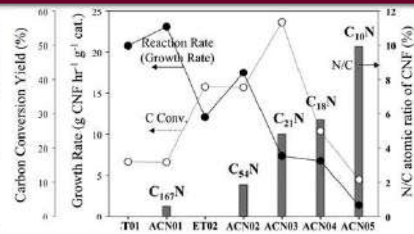




- CBF fibers 250 ~ 350m²/g, Metal fibers 20 ~ 200 m²/g
- CBF fibers shows 2~10 times higher SA than Metal fibers.
- SEM of CBF fibers with SA around 300 m²/g: small fibrils, fibril aggregate, and rough surface one like activated one.

N-doped CNFs

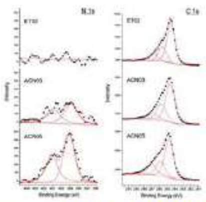




Synthesis Conditions Using Acetonitrile As the Nitrogen Source and Corresponding Sample Names

Conditions (at 530 ° C)

sample	C_2H_4 (g) mL/min	H_2 (g) mL/min	He (g) mL/min	N-source (liq.)[a] μL/min	N/C (cal.) atom %
ET01	160	40	0	0	0.0
ET02	40	40	160	0	0.0
ACN01	160	40	0	35	4.6
ACN02	40	40	120	35	14.5
ACN03	0	40	160	35	50.0
ACN04	0	40	160	70	50.0
ACN05	0	0	200	35	50.0



Langmuir, 25(14), pp. 8268-8278(2009)

Preparation (Fixed Bed Method)

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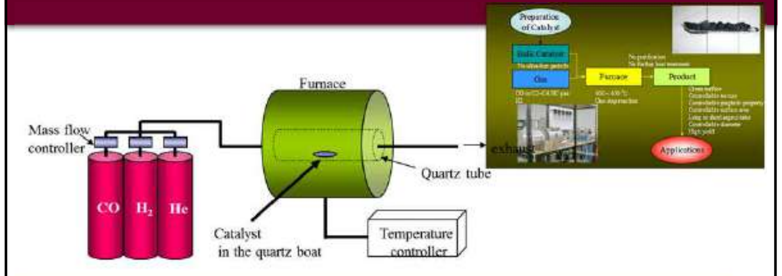

Catalyst : Transition metals, Their alloys or supported catalyst
Catalyst preparation method : co-precipitation

1) Best, R. J. and Russell, W. W., J. Amer. Soc. 76, 838(1954)
2) Sinfelt, J. H., Carter, J. L., and Yates, D. J. C., J. Catal. 24, 283(1972)

Reduction : H_2/He(1/9, 200sccm//4.5 cm diameter tubular furnace, 2h
Reaction : $CO(C_2H_4)/H_2$ (4/1 & 1/4v/v%), 200 sccm// 4.5 cm tubular furnace
Reaction Time & temperature : 1~4 hs, 400 ~ 680 ℃

CARBON 42 (3): 591-597 2004

Catalysts for CNF Preparation

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Mono-metal
- Fe, Co, Ni
- Fe, Co, Ni / Supports

Support: Alumina, Silica >>> MgO

Bimetallic Catalyst
- Fe, Co, Ni / Fe, Ni, Mn, Cu, …/Supports

Trimetallic Catalyst
- Fe, Co, Ni / Fe, Ni, Cu, Mn / Cr, Al, …/Supports

Functions of Second or Third Metals ?

JOURNAL OF PHYSICAL CHEMISTRY C, 112, 10050-10060, 2008

Main Catalyst | **2nd Catalyst** | **3rd Catalyst**

Fe
Fe:Mg=8:2
収率: 1.2倍

Cr
Fe:Cr:Mg=6.4:1.6:2
収率 : 4.6倍
繊径 : 40nm
Tubular

Mo
Fe:Cr:Mo:Mg=6:1:1:2
収率 : 27.8倍
繊径 : 20nm
Tubular

Mn
Fe:Mn:Mg=6:2:2
収率 : 1.1倍

Co
Fe:Mn:Co:Mg=4:2:2:2
収率 : 11.6倍
繊径 : 50nm
不均一 CNF

Cu
Fe:Cu:Mg=6:2:2
収率 : 2.0倍

Co
Fe:Cu:Co:Mg=6:1:1:2
収率 : 60.2倍
繊径 : 180nm
Herringbone CNF

Ni

Co
Fe:Ni:Co:Mg=7:0.5:0.5:2
収率 : 60.2倍
繊径 : 120nm
Tubular

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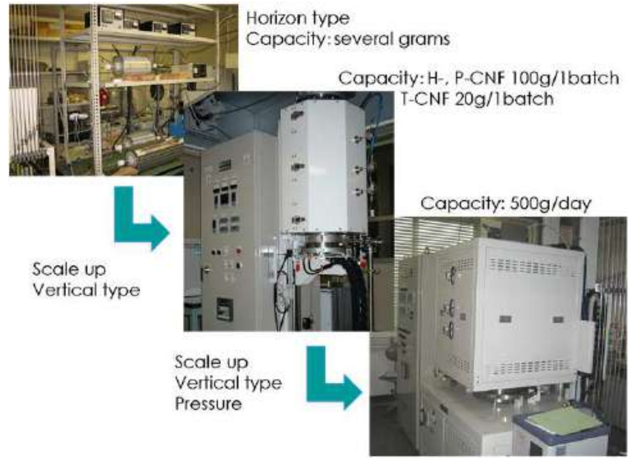
Mass Production of CNFs



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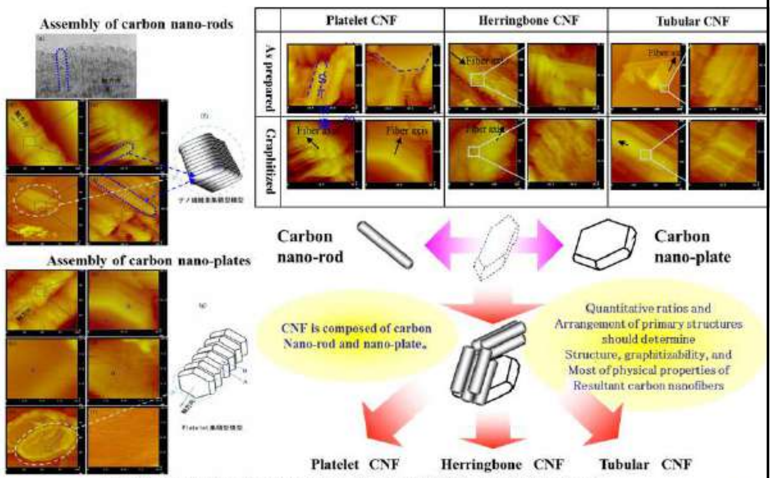
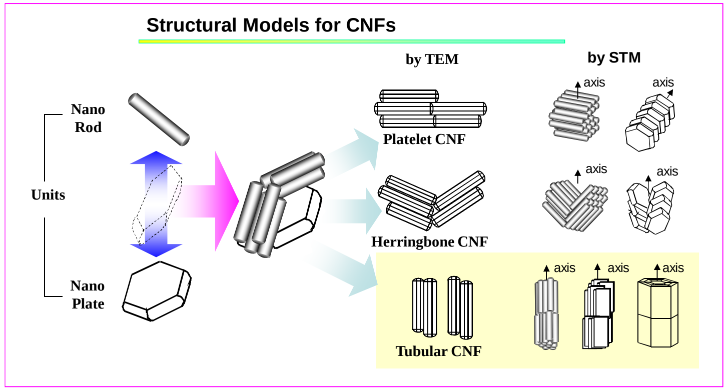
Primary Structures of Various CNFs



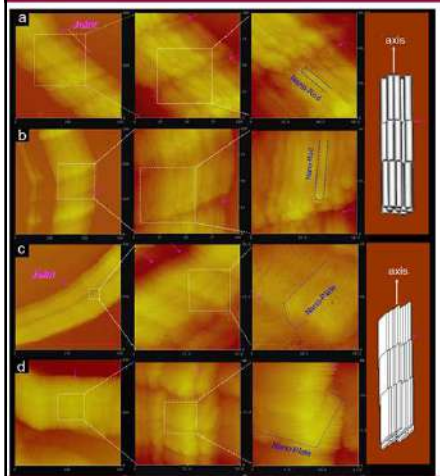
⇨ Various structures and surfaces of CNFs are determined by the arrangements of primary structural units

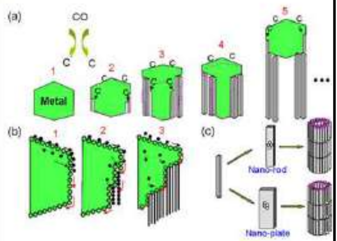
Recent new finding by us :
Platelet and herringbone CNFs are constructed by two types of structural units, a ***nano-rod*** type and a ***nano-plate*** type.



S-H. Yoon et al., Carbon 42 (2004),3087.
Carbon 43 (2005),1828.

Schematic model of nucleation and growth process of CNTs





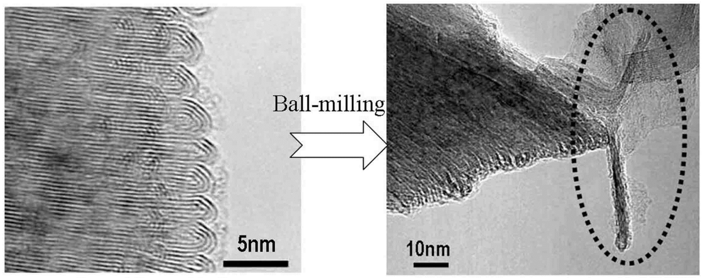
STM images of the pristine (a, c) and graphitized (b, d) carbon nanotubes, which indicate that both pristine and graphitized CNTs consist of rod (a, b) and plate (c, d) shape structural unit

Separation of structural units from CNFs

- From PCNF to Nano-graphene

Separation of structural unit (Nano-rod)

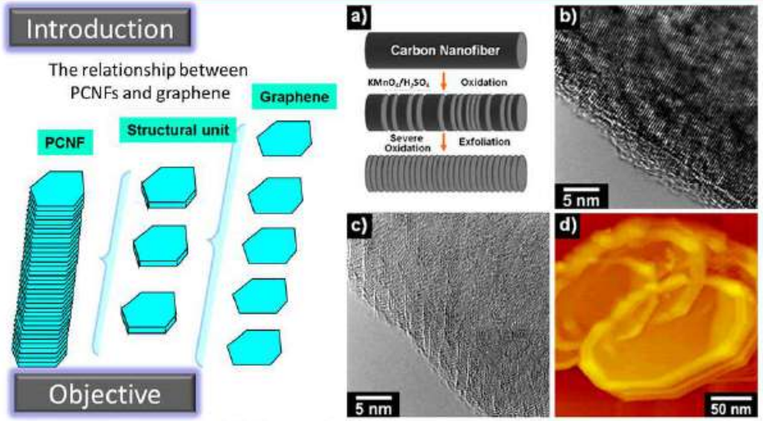
Graphitized platelet CNF



Closed Loop Ends

A Rod Unit Separated

Separation of structural unit (Nano-platelet)



Using oxidation and exfoliation methods to transversely isolate structural unit of PCNFs for further understanding of CNFs' structure.

@ *ACS Nano* 2011, *5(8)*, 6254–6261.

CNF preparation using waste industrial gases

- Effect of poison gases: H_2S, COS
- Effect of oxidative gases: CO_2, H_2O
- Effect of different reactive gases
  - (a) C_2H_4
  - (b) CO
  - (c) CH_4

Carbon 2011 (Shanghai)

Waste Industrial Gases

Composition of gases (R.T.)

Gas	vol. %
H_2S	~ 1
CO_2	~ 8
H_2O	~ 2
(a) C_2H_4	~ 30
(b) CO	~ 24
(c) CH_4	~ 20
C_3H_6	~ 15
N-containing gases	trace

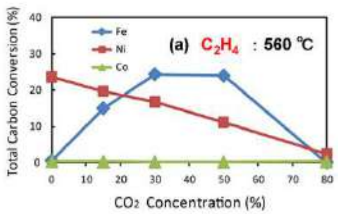
Problems

① H_2S …… Poison gas → Removal (1)

② CO_2, H_2O } Oxidative gases → CO_2 Effect on CNF growth

③ CH_4, C_2H_4, C_3H_6, CO } Different reactivity

(2) Effect of oxidative gas

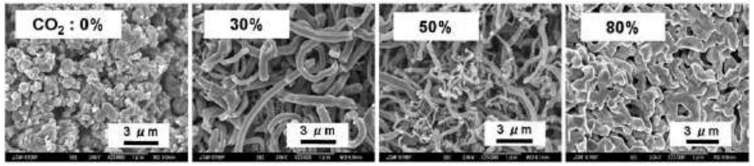


CO2 effect to carbon conversion on the various catalysts at 560 ℃

- Catalyst: Fe, Ni, Co
- Amount: 30 mg
- Gases: C_2H_4 / H_2 (1 / 1) + CO_2 (0 ~ 80%)

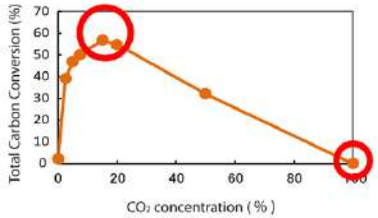
Total carbon conversion (%)

$$= \frac{\text{CNF weight (g)} \times 100}{\text{Carbon amounts of gases(g)}}$$



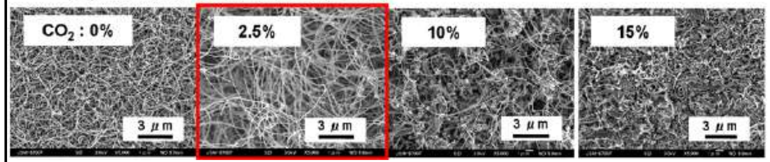
CNF images which are prepared at different amount of CO_2

Gas : C_2H_4, Catalyst : FN64, Temperature : 560℃



CO_2 Effect on the CNF preparation using FN64

- Catalyst: FN64
- Temperature: 560 ℃
- Amount: 100 mg
- Gases: C_2H_4 / H_2 (1 / 1) + CO_2 (0 ~ 100%)

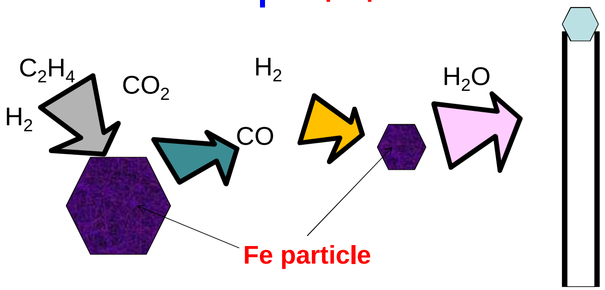


Conjectured reaction mechanism

1. Ethylene+H_2+CO_2 → CO+CH_4+…
2. CO spillovers to Fe

1. CO+H2 to Fe surface → **Growth of CNF**

Disproportionation reaction



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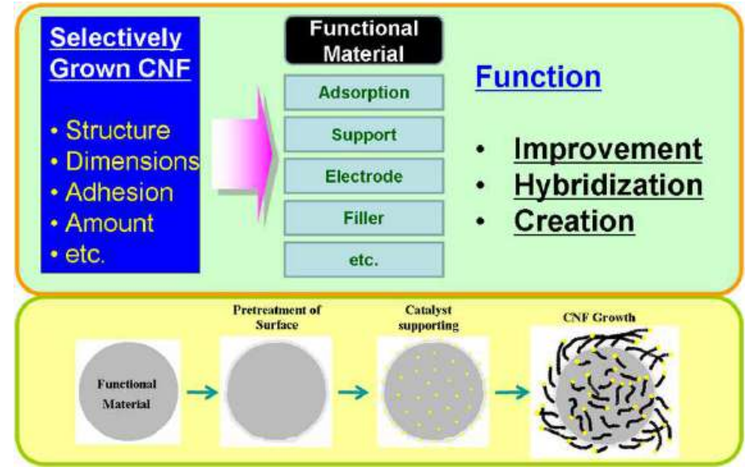
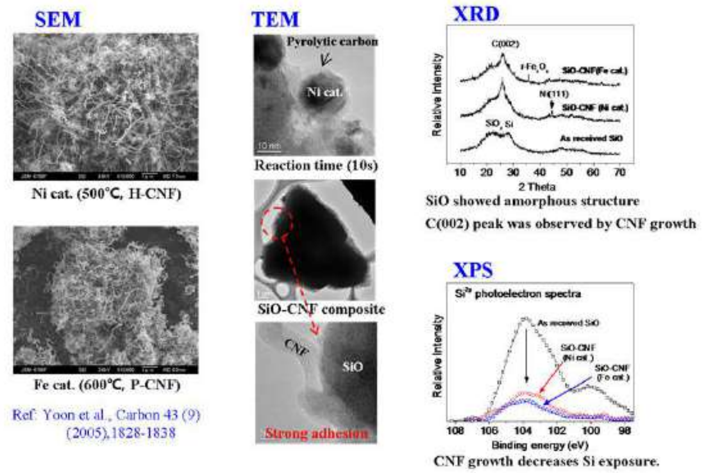
CNFs for Battery Study

Concept of CNF composites

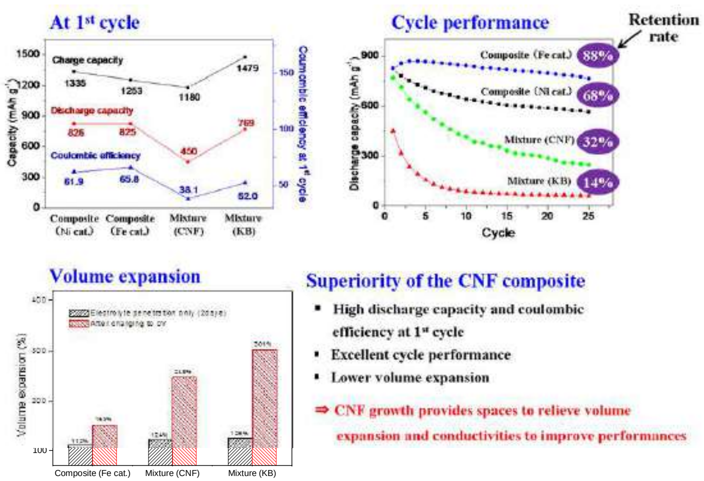


Preparation and Analysis of SiO-CNF Composites



Electrochimica Acta, 55, 5519-5522 (2010)

Comparison *bet.* Composite *and* Mixture

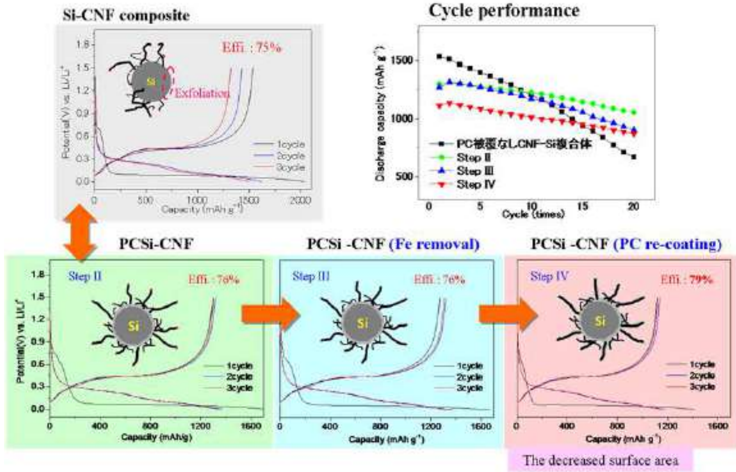


Superiority of the CNF composite

- High discharge capacity and coulombic efficiency at 1st cycle
- Excellent cycle performance
- Lower volume expansion

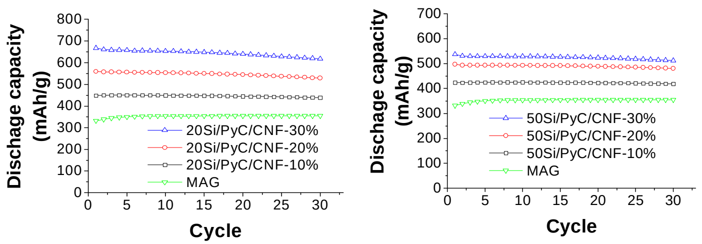
⇒ CNF growth provides spaces to relieve volume expansion and conductivities to improve performances

Cycle performances of PCSi-CNF composite



CARBON, *48*, *3381-3391*, *2009.*

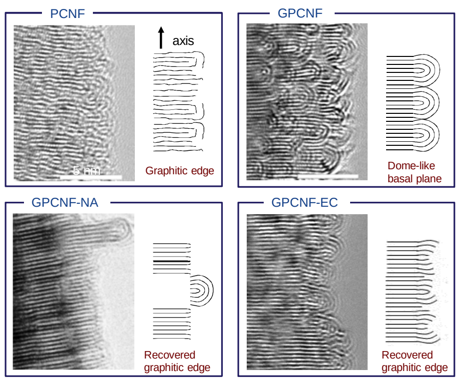
Si-CNF composite / Graphite Hybridization



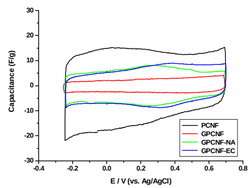
Carbon 2011 (Shanghai)

CNF for Capacitor Study

Surface-modified PCNF series



Cyclic voltammogram of GPCNF series



Elemental analysis of GPCNF series

Samples	Elemental analysis (wt%)			
	H	C	N	O (diff.)
PCNF	0.33	98.15	0.05	1.47
GPCNF	0.10	99.90	0	0
GPCNF-NA	0.15	99.12	0.06	0.67
GPCNF-EC	0.13	98.50	0	1.37

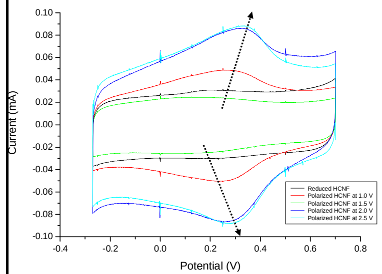
Langmuir, 22 (22), 9086 -9088, 2006

Functional Groups vs. capacitance

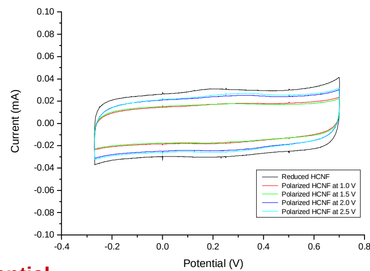
Polarized anodic HCNF by binderless polarization condition in 30 wt% H2SO4

Polarized HCNF under binderless condition in 30 wt% H_2SO_4

Anode (＋ electrode)



Cathode (－ electrode)



*** According to increase of the potential,**

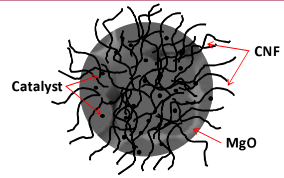
in anode, EDLC and psudocapacitane increased.
in cathode, capacitance decreased slightly.

Carbon 49 (1), pp. 96-105 (2011)

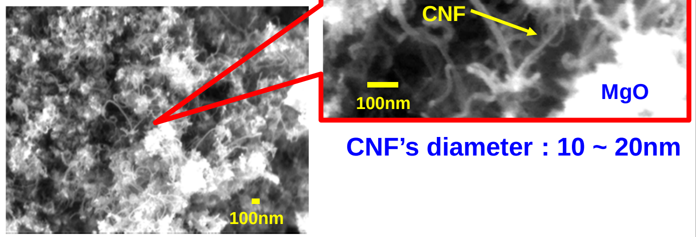
Ceramic applications

- Increasing the strength of refractory through the small amount addition of CNF-MgO composites

CNF coated MgO (CNF-MgO)

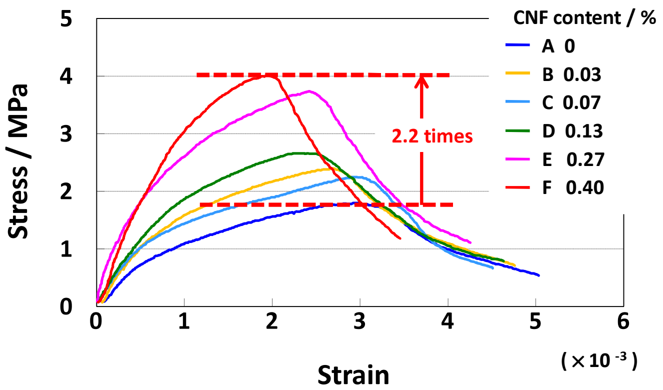


Model of CNF-MgO



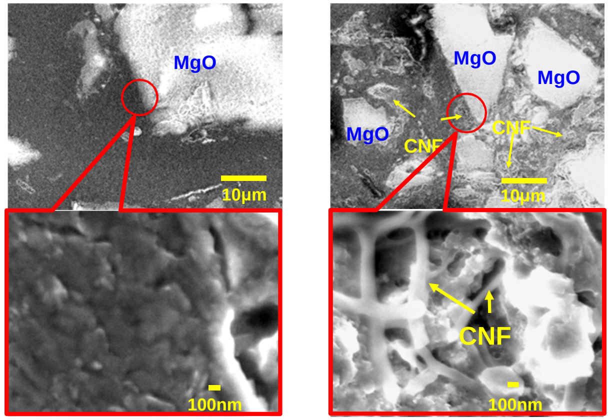
CNF's diameter : 10 ~ 20nm

Stress – strain curves



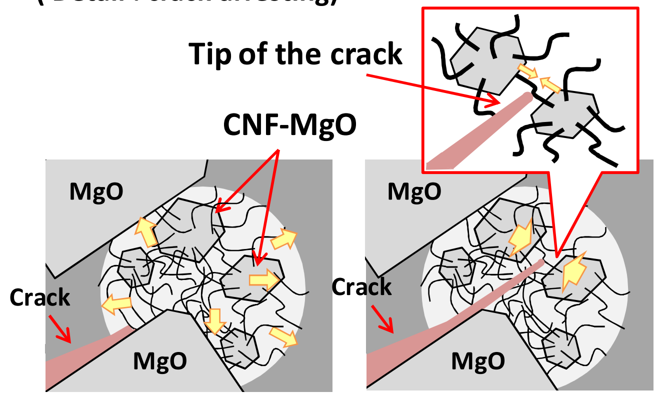
Scanning electron microscopy

Specimen A (CNF : 0%) F (CNF : 0.40%)



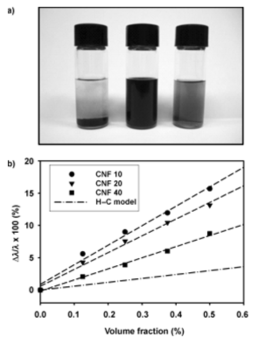
Mechanism of strengthening – Ⅱ

(Detail : crack arresting)

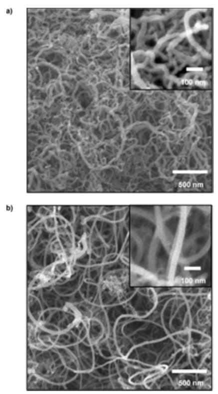


CNFs for Nanofluid

CNF AS A Novel Nanofiller for Nanofluid Applications



a) Photograph of CNF-10-water suspensions. Left: pristine CNFs (0.5 vol %); middle: TCNFs from plasma oxidation for 30 min (0.5 vol %); right: TCNF-water suspension diluted 20 times. b) TC enhancement of nanofluid containing various contents of CNFs. The dot-dashed line indicates the theoretical prediction for TC enhancement based on the Hamilton-Crosser (H-C) equation

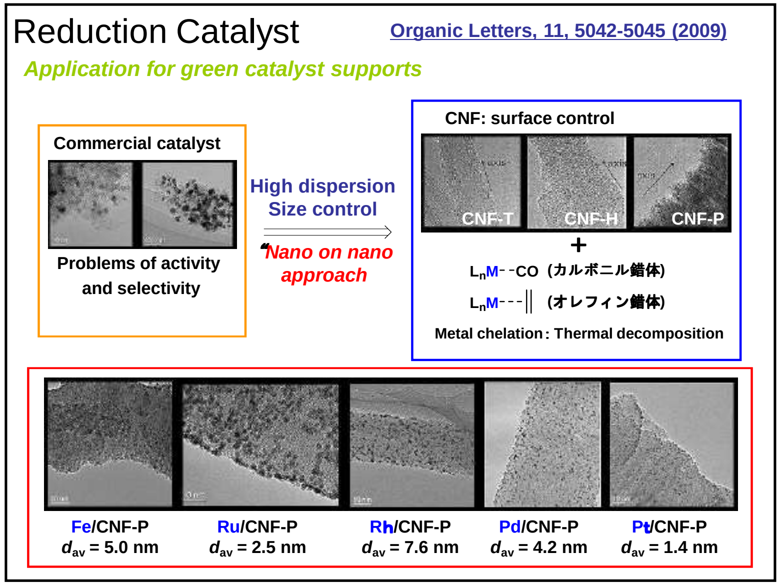


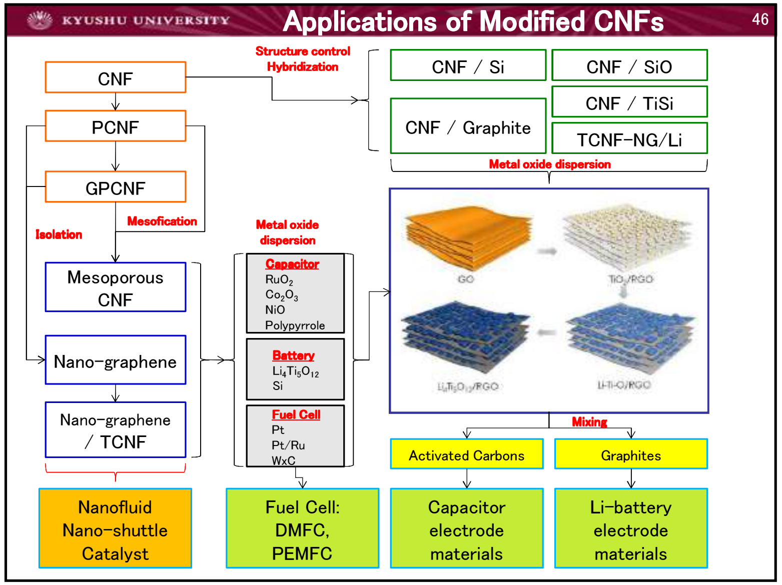
SEM images of a) pristine CNFs and b) TCNFs (CNF-10).
Insets: higher-magnification SEM images

Small, 3, Issue 7, Date: July 2, 2007, Pages: 1209-1213

CNFs for Green Organic Chemistry

- *CHEMISTRY-AN ASIAN JOURNAL* 2 (12): 1524-1533 2007
- *JOURNAL OF SYNTHETIC ORGANIC CHEMISTRY JAPAN*, 67, 7, 724-734, JUL 2009
- *Organic Letters*, 11, 5042-5045 (2009)



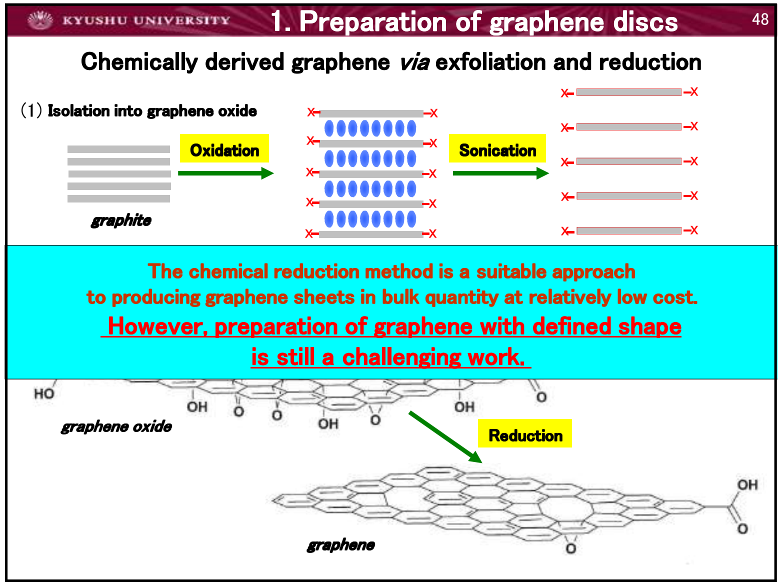


Outline

1. **Preparation of graphene discs**
2. **Preparation of mesoporous CNFs**
3. **Preparation of partially unzipped CNF**
4. **Electrochemical applications**

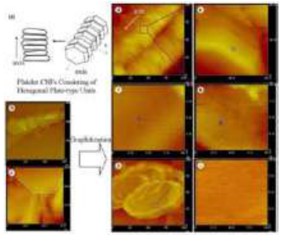
References

- Long, D.; Hong, J.Y.; Li, W.; Miyawaki, J.; Ling, L.; Mochida, I.; Yoon, S.-H.; Jang, J. *ACS Nano* **2011**, *5(8)*, 6254-6261.
- Long, D.; Li, W.; Qiao, W.; Miyawaki, J.; Yoon, S.-H.; Mochida, I.; Ling, L. *Chem. Commun.* **2011**, *47(33)*, 9429-9431.
- Long, D.; Li, W.; Qiao, W.; Miyawaki, J.; Yoon, S.-H.; Mochida, I.; Ling, L. *Nanoscale* **2011**, *3(9)* 3652-3656.
- Long, D.; Li, W.; Miyawaki, J.; Qiao, W.; Ling, L.; Mochida, I.; Yoon, S.-H. *Chem. Mater.* **2011**, *23(18)*, 4141-4148.



Preparation of uniform graphene disc

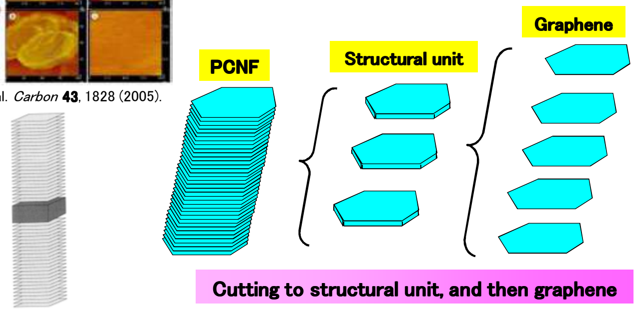
Step-by-step cutting of graphenes of platelet carbon nanofiber (PCNF)



Yoon SH et al. *Carbon* **43**, 1828 (2005).

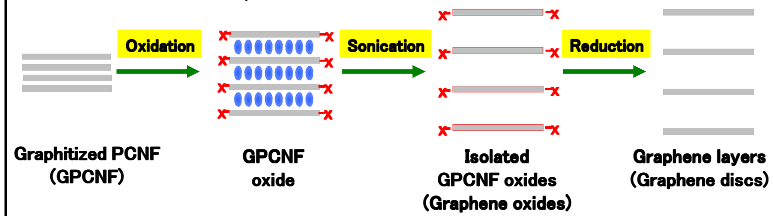
PCNF consists of nano-sized platelet structural units stacked perpendicular to fiber axis.

The plate unit has the thickness of 2-3 nm consisted of 6-10 graphene layers.



Cutting to structural unit, and then graphene

Oxidation, exfoliation and reduction of GPCNF



(1) Oxidation of GPCNF to PCNF oxide by the Hummer's method

Dispersion of 1g GPCNF in 100 mL of 98% H_2SO_4

Addition of 1～7 g $KMnO_4$

Stirring at 0°C for 30 min and then 40°C for 30 min

(2) Washing with assistant of centrifuge

Washing with water and H_2O_2

Centrifugation at 4800 rpm

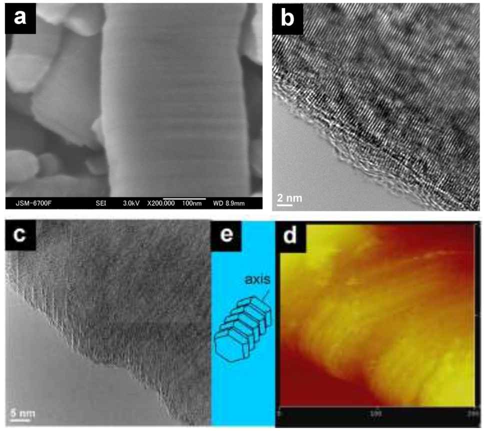
Washing with HCl and acetone

(3) Exfoliation through strong sonication

(4) Reduction

Chemical reduction or Mechanical reduction

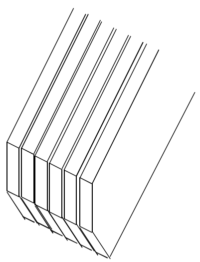
Structure of graphitized PCNFs



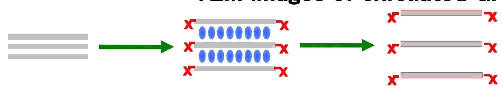
GPCNF (2800°C)

S_{BET} = 50 m^2/g

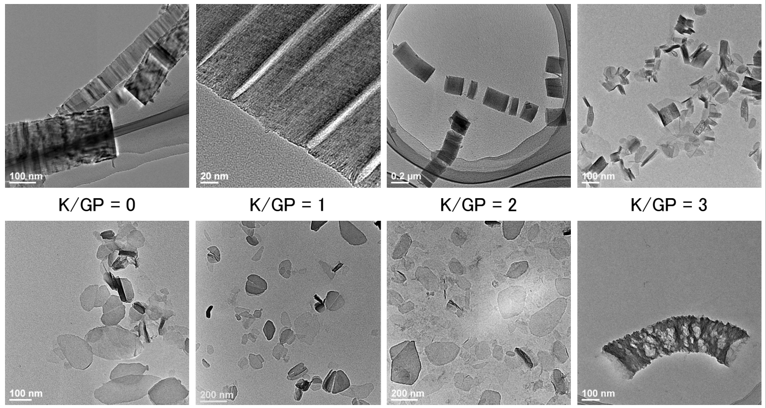
d_{002} = 0.3476 nm



TEM images of exfoliated GPCNFs



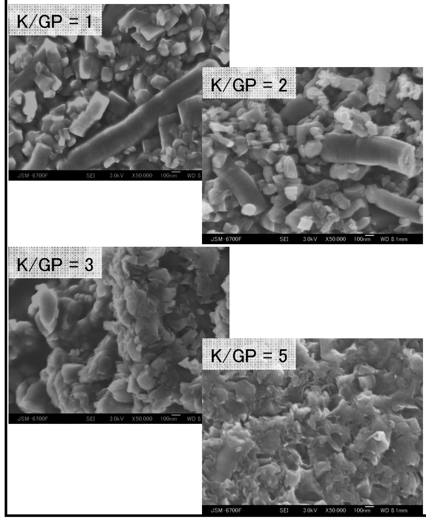
K/GP = $KMnO_4$/GPCNF ratio

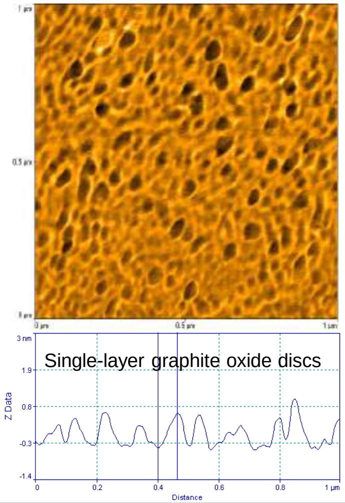


K/GP = 0 K/GP = 1 K/GP = 2 K/GP = 3

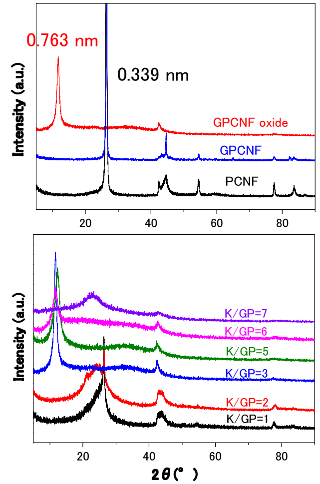
K/GP = 4 K/GP = 5 K/GP = 6 K/GP = 7

SEM and AFM images of exfoliated GPCNFs

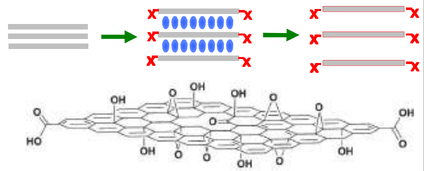


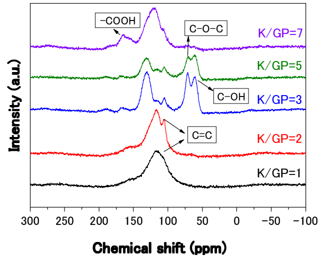


XRD patterns and ^{13}C–NMR spectra of oxidized GPCNFs



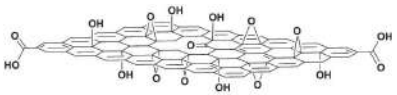
XRD patterns

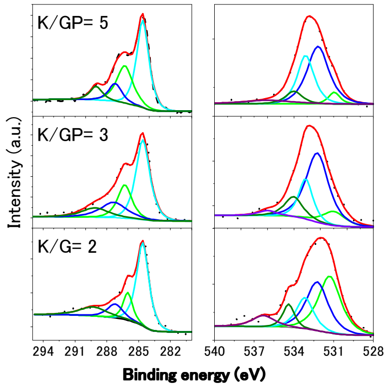




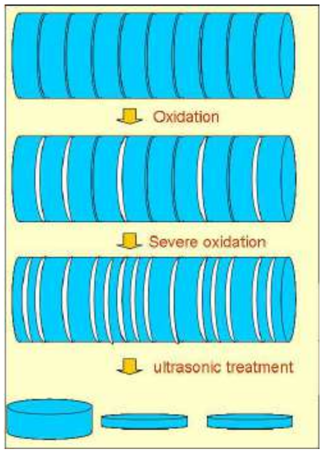
^{13}C solid–state NMR spectra

Step–by–step cutting model of GPCNF into graphene discs



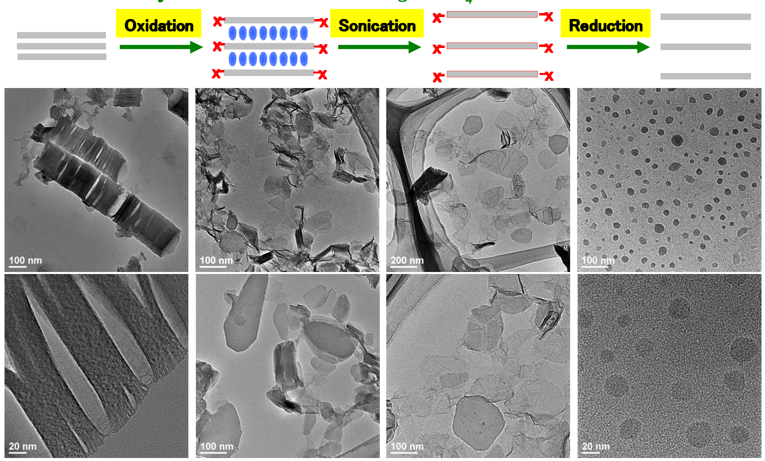


XPS spectra



Chemical reduction of GPCNF oxide

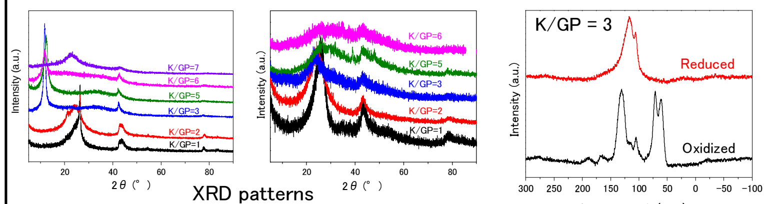
Hydrothermal reduction using $NaBH_4$ at 130°C for 5 h



K/GP = 1 K/GP = 3 K/GP = 5 K/GP = 6

Chemical reduction of GPCNF oxide

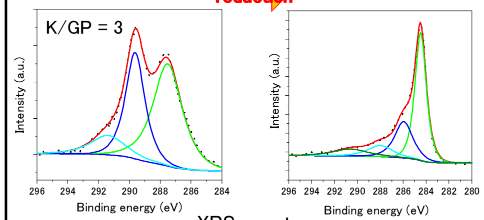
Hydrothermal reduction using $NaBH_4$ at 130°C for 5 h



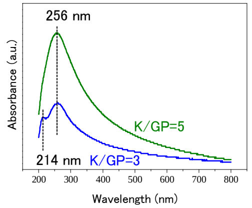
XRD patterns

^{13}C solid-state NMR spectra

GPCNF oxide → Hydrothermal reduction → Graphene disks



XPS spectra

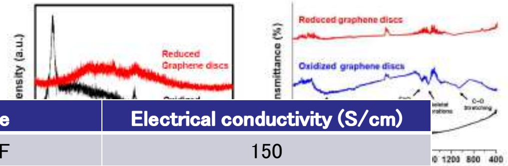


UVvis absorption spectra

Mechanical reduction of GPCNF oxide

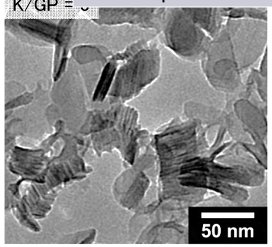
Wet milling method using nano-dispersion machine

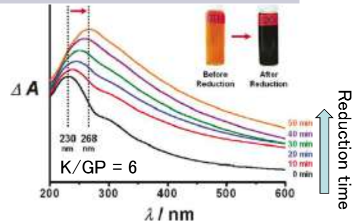
T.K.FILMIC
(18400 rp



Sample	Electrical conductivity (S/cm)
GPCNF	150
GPCNF oxide (K/GP = 6)	7.4 × 10^{-4}
Graphene discs	14

K/GP =

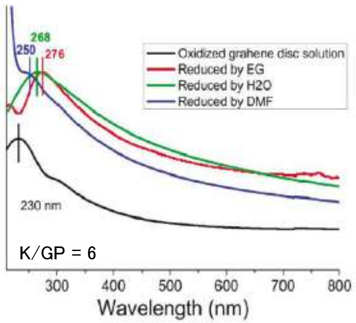




UVvis absorption spectra

Mechanical reduction of GPCNF oxide

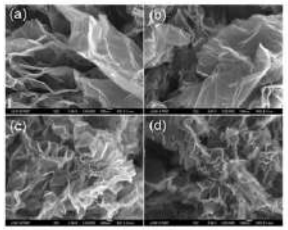
Influence of solvent during mechanical reduction

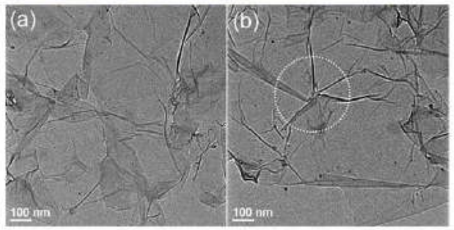


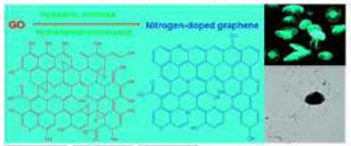
Concentration > 2.0 g/L

Nitrogen-Doped Graphenes by a Combined Chemical and Hydrothermal Reduction

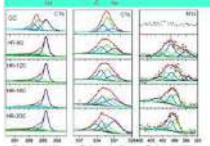
Langmuir, **2010**, *26* (20), pp 16096–16102







samples	C (wt %)	H (wt %)	N (wt %)	O (wt %)	N/C (at./at.)	O/C (at./at.)
GO	49.59	2.28	0.03	48.05	0.0018	0.72
HR-80	80.43	1.23	5.21	13.13	0.074	0.12
HR-120	80.76	1.15	4.57	13.52	0.064	0.12
HR-160	84.15	1.15	4.09	10.61	0.055	0.091
HR-200	86.12	1.23	4.01	8.64	0.051	0.077



			N distribution (at. %)				
samples	N/C (at./at.)	O/C (at./at.)	395.7 ± 0.3 eV	398.7 ± 0.3 eV	400.3 ± 0.3 eV	401.4 ± 0.3 eV	402–405 eV
GO	0	0.45					
HR-80	0.11	0.16	16	18	37	18	11
HR-120	0.095	0.13	11	27	24	21	17
HR-160	0.071	0.13	0	39	25	17	19
HR-200	0.052	0.098	0	42	28	16	14

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2. Preparation of mesoporous CNFs

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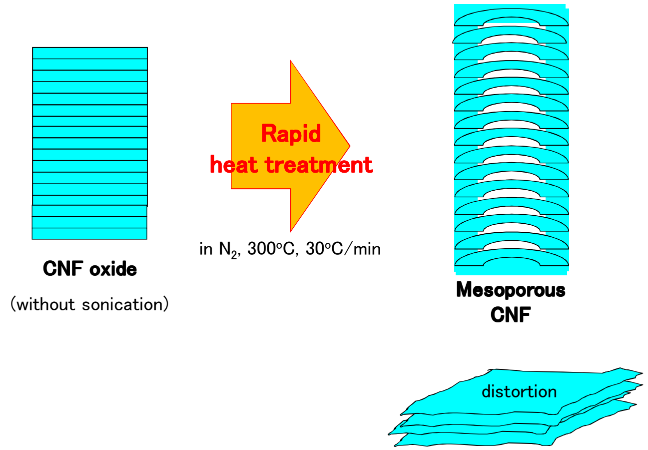
Thermal expansion of CNF oxide



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2. Preparation of mesoporous CNFs

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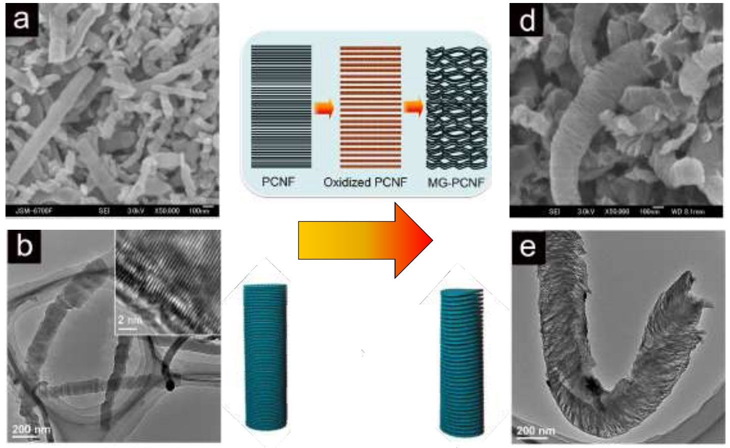
Preparation of mesoporous PCNFs from as-prepared PCNFs



As-prepared PCNF
S_{BET} = 56 m^2/g

Mesoporous PCNF (MG-PCNF)
S_{BET} = 355 m^2/g

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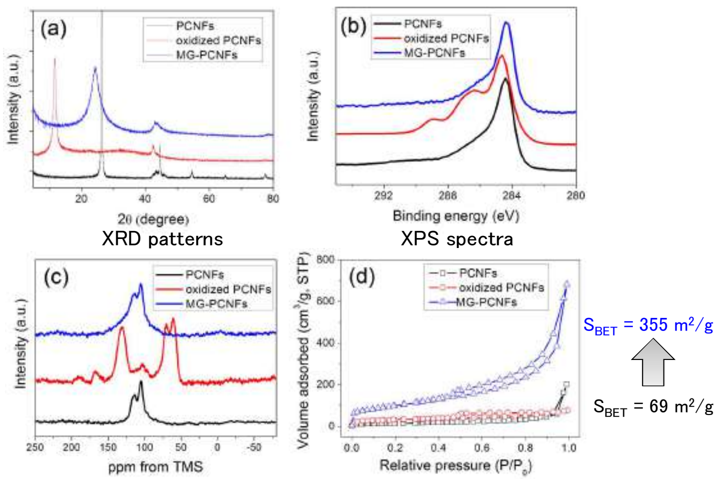
2. Preparation of mesoporous CNFs

63

Characterization of mesoporous PCNFs



XRD patterns

XPS spectra

^{13}C solid-state NMR spectra

N_2 adsorption isotherms at 77 K

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2. Preparation of mesoporous CNFs

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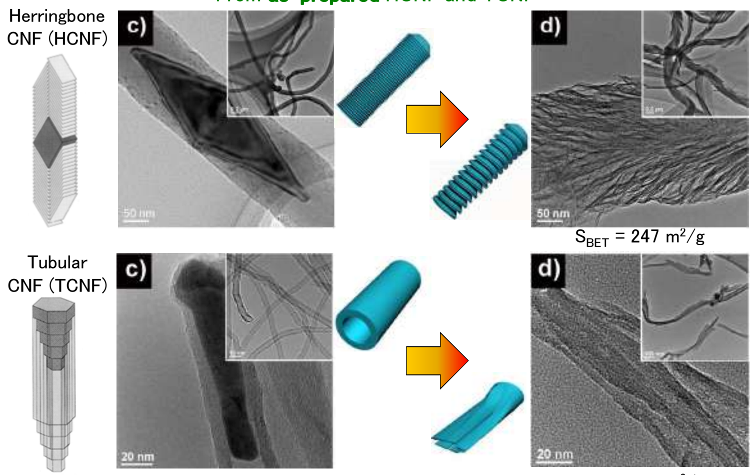
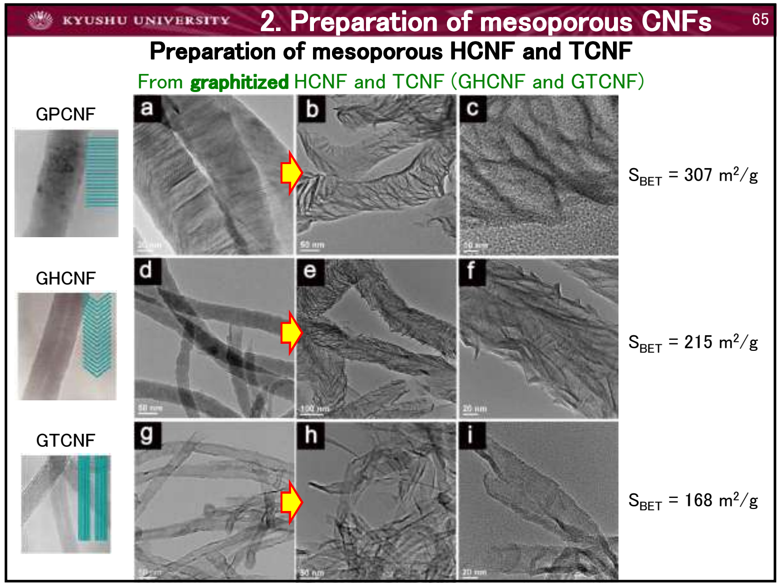
Preparation of mesoporous HCNF and TCNF

From **as-prepared** HCNF and TCNF

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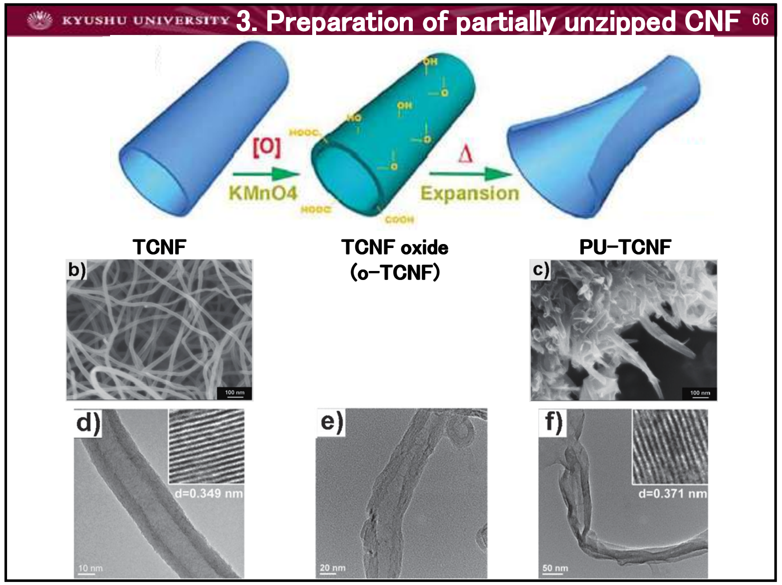
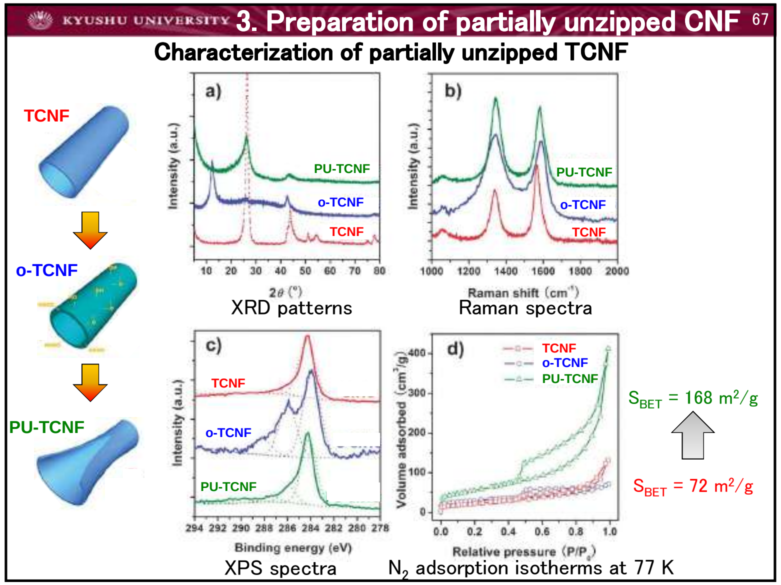
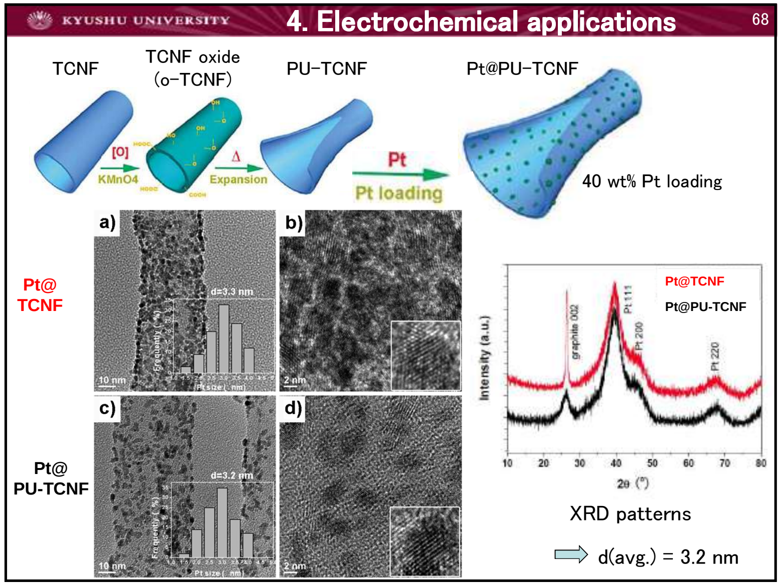
2. Preparation of mesoporous CNFs
65
Preparation of mesoporous HCNF and TCNF
From graphitized HCNF and TCNF (GHCNF and GTCNF)
GPCNF
GHCNF
GTCNF
a
b
c
d
e
f
g
h
i
S_{BET} = 307 m^2/g
S_{BET} = 215 m^2/g
S_{BET} = 168 m^2/g


KYUSHU UNIVERSITY
3. Preparation of partially unzipped CNF
66
[O]
KMnO4
Δ
Expansion
TCNF
TCNF oxide
(o-TCNF)
PU-TCNF
b)
c)
d)
e)
f)
d=0.349 nm
d=0.371 nm


KYUSHU UNIVERSITY
3. Preparation of partially unzipped CNF
67
Characterization of partially unzipped TCNF
TCNF
o-TCNF
PU-TCNF
a)
b)
XRD patterns
Raman spectra
c)
d)
XPS spectra
N$_2$ adsorption isotherms at 77 K
Binding energy (eV)
Relative pressure (P/P$_0$)
S_{BET} = 168 m^2/g
S_{BET} = 72 m^2/g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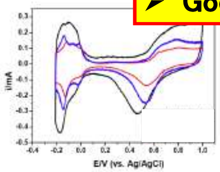
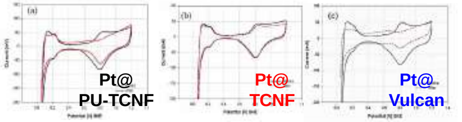
4. Electrochemical applications
68
TCNF
TCNF oxide
(o-TCNF)
PU-TCNF
Pt@PU-TCNF
[O]
KMnO4
Δ
Expansion
Pt
Pt loading
40 wt% Pt loading
a)
b)
c)
d)
Pt@
TCNF
Pt@
PU-TCNF
d=3.3 nm
d=3.2 nm
Pt@TCNF
Pt@PU-TCNF
XRD patterns
d(avg.) = 3.2 nm

4. Electrochemical applications

- ➢ Improved electrocatalytic performance
- ➢ Good durability

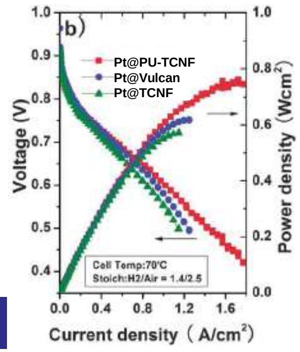


Cyclic voltammograms in 0.5 M H_2SO_4



Durability tests

Sample	Electrochemically active surface area, ESA (m^2/g)	Decay rate of ESA (%)
Pt@PU-TCNF	129	**38**
Pt@TCNF	64	**11**
Pt@Vulcan	71	**65**



Proton exchange membrane fuel cell (PEMFC) single-cell profiles

Conclusion

1. Special properties of CNFs are still promising for their commercial applications through the innovation of the performances of conventional carbons.
2. Very homogeneous nano-graphene and special fibrous mesoporous carbon can be obtained using CNFs as an effective precursor
3. We have to solve the problems of CNFs for the effective applications to the real market.

- From science to engineering
- Full understanding of the performances and costs of the conventional functional carbons

Acknowledgements

•This work was done within the category of CREST program of JST. We are sincerely appreciated to the financial support of JST.

•This work is supported by a grant from the Global-Center of Excellence (GCOE) in Novel Carbon Resource Sciences, Kyushu University. And, the authors are grateful for financial assistance provided by the Global-Centre of Excellence in Novel Carbon Resource Sciences, Kyushu University.

•The authors are very grateful to Dr. An Bai and Dr. Jang Sang-Min, Mr. Matsuo Yasunori, and Dr. Long Donghui for their contributions on this work.

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- ✓ Seong-Ho Yoon: Professor
- ✓ Isao Mochida: Professor of special appointment
- ✓ Jin Miyawaki: Assistant Professor
- ✓ 1 Guest Professor
- ✓ 2 Post-doctorates
- ✓ 2 Researcher for Analyses
- ✓ 9 Doctor course students
- ✓ 5 Mater course students
- ✓ 3 Secretary

Members for Nano-studies
- Faculties
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Thank you for attentions!