

# **Exposure assessment of a weaving process of CNT-coated yarn by applying carbon analysis**

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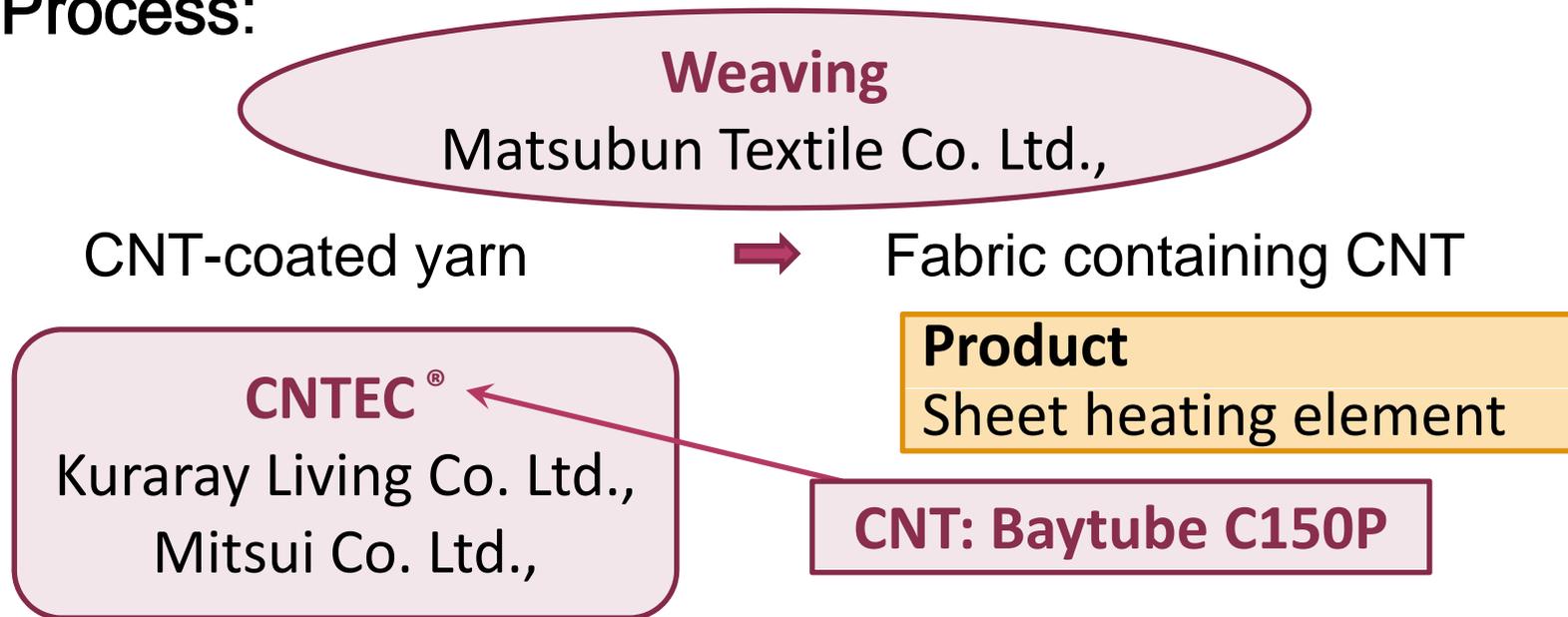
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2 Kuraray Living Co., Ltd

3 Shinshu University

# Objects

- Exposure assessment to CNT or CNT containing particles in a workplace of downstream users
- **Process:**



(in this presentation CNT means MWCNT)

# Procedure of monitoring

- Deciding the sampling points
  - where higher stress is given to the yarn both airborne and wipe

- **Real-time monitoring** by SMPS, CPC, OPC
- **Sampling for SEM observation (Qualification)**
- **Sampling for carbon and mass measurement (Quantification)**

- Analysis of the data

# Views around the loom

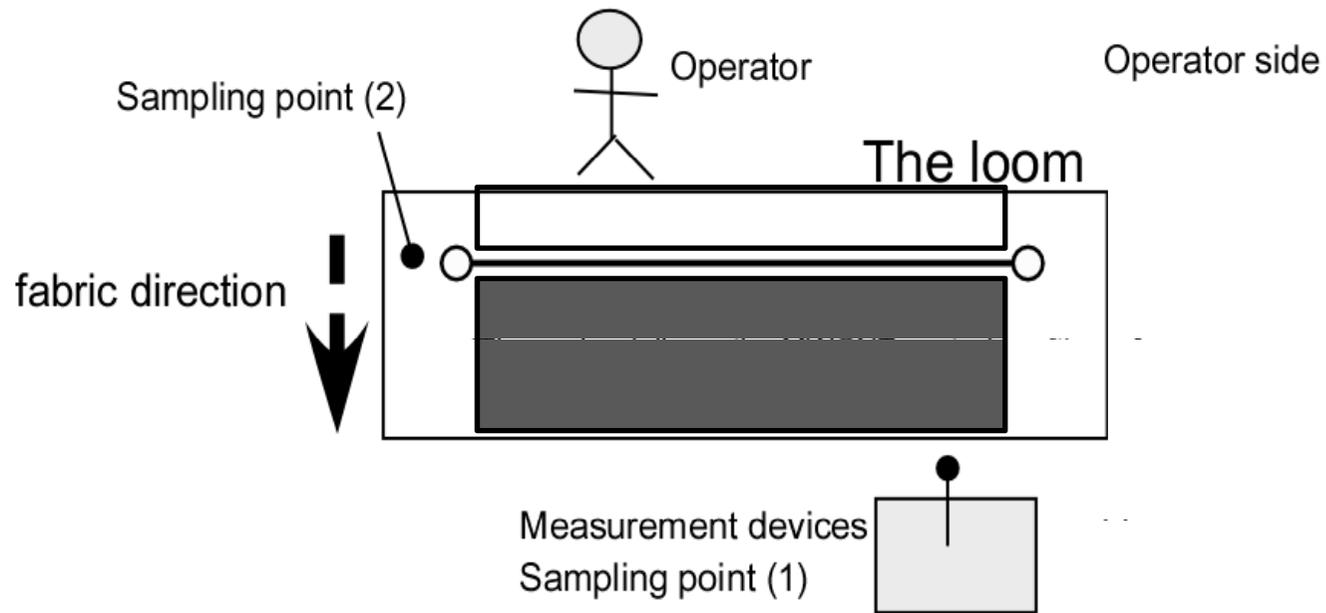
From operator's side



Rear side



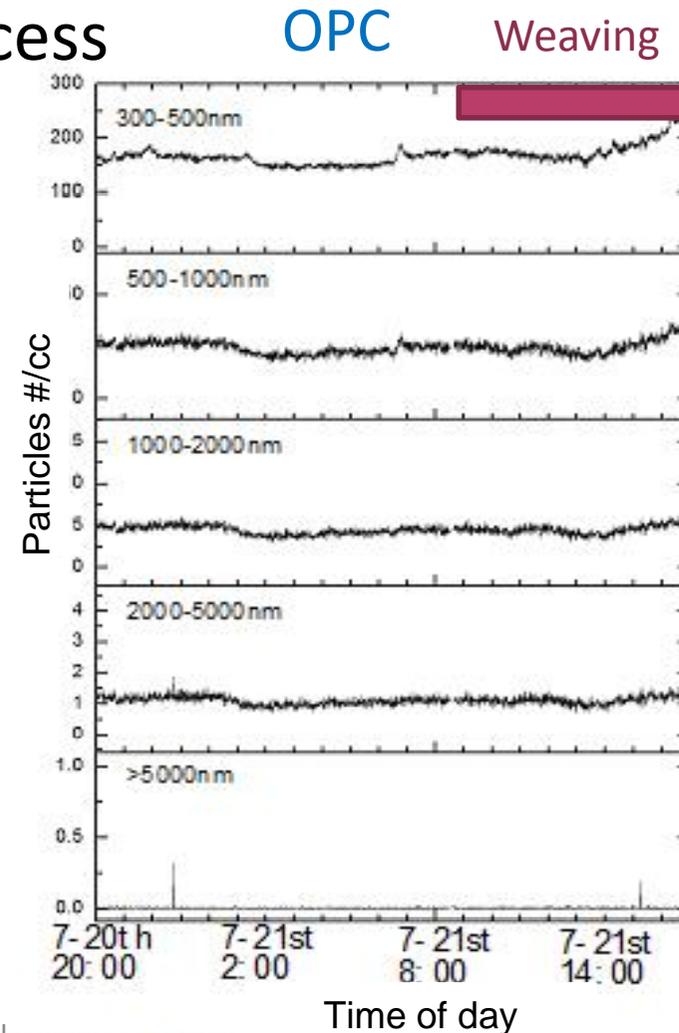
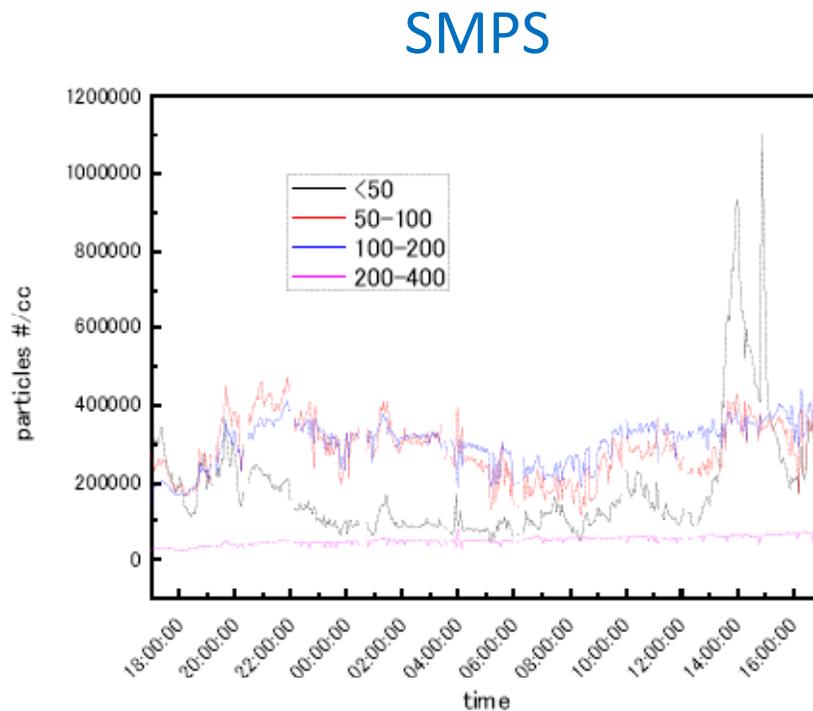
# Sampling points



- Number of the loom weaving CNTEC 1
- Number of the looms weaving polyester yarn >15

# Results: Real-time monitoring

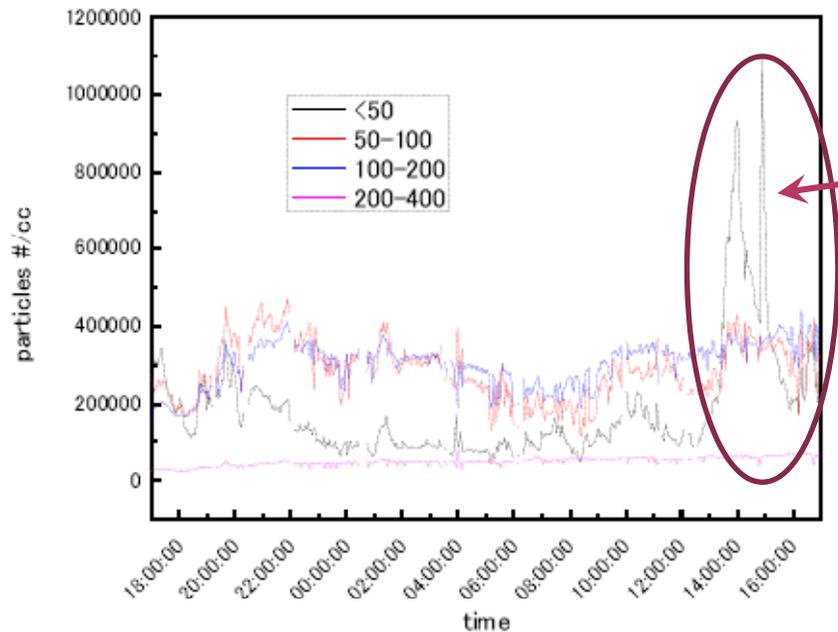
No relationship between number concentration and weaving process



# Results: Real-time monitoring

No relationship between number concentration and weaving process

## SMPS

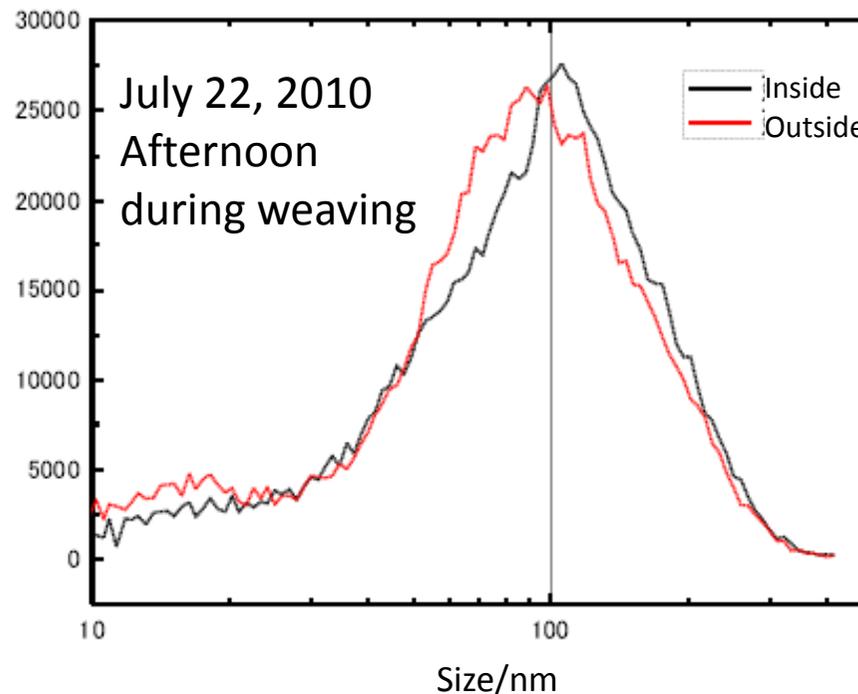


Where is the origin?

# Results: Size distribution

- Size distribution and concentration of nanoparticles are similar at inside and outside
- Nanoparticles are observed in the afternoon

Origins of nanoparticles are outside,  
photochemically generated secondary  
particles

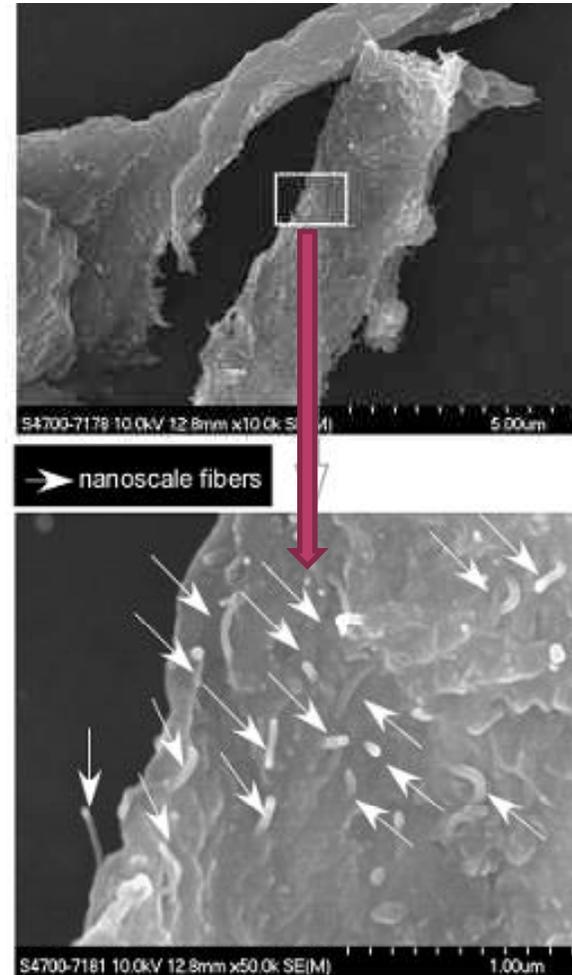


# Results: SEM observation

- During weaving
  - Micron-size CNTEC fragments
  - Nanosize particles exist?

- How to determine CNT and CNT containing particles?

→ Carbon analysis

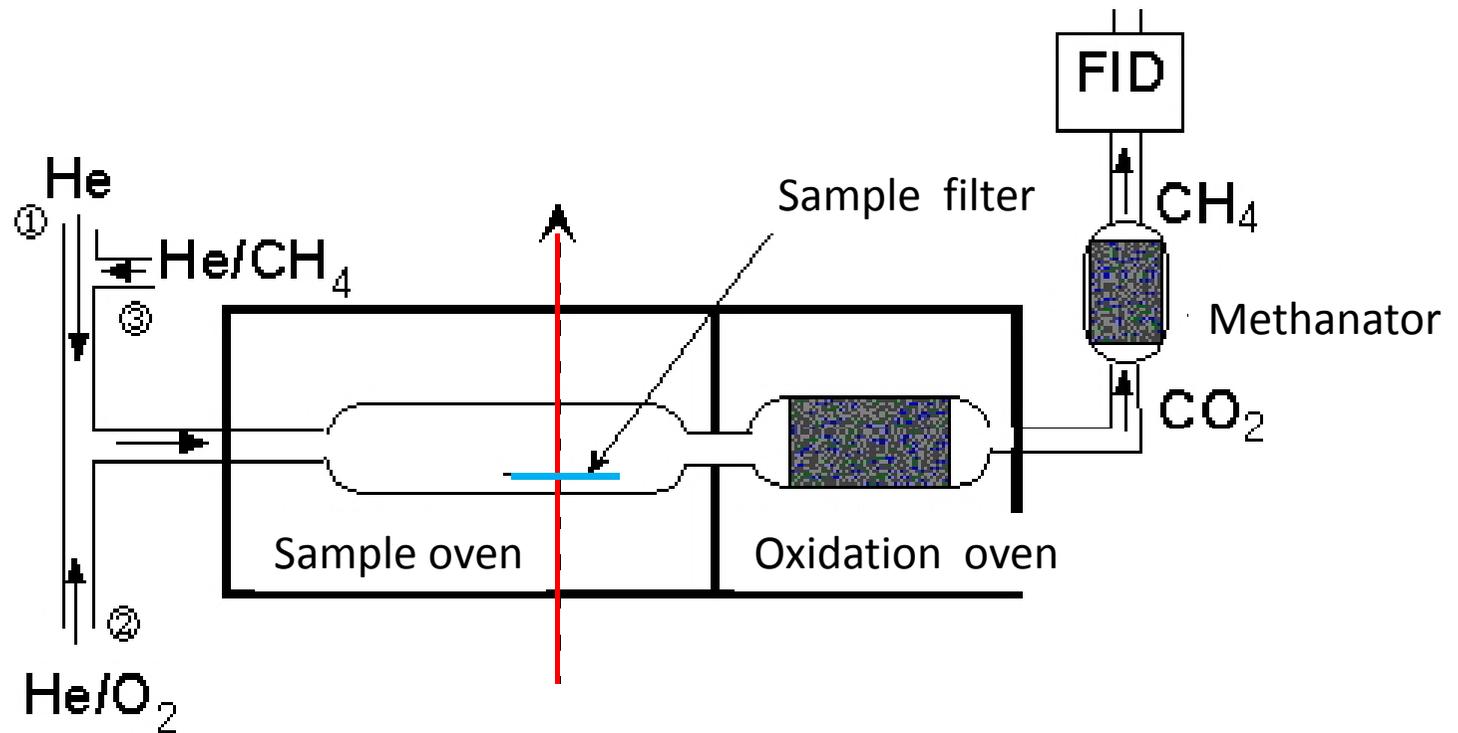


x1000

x5000

with weaving

# Carbon monitor



# Protocol for carbon analysis

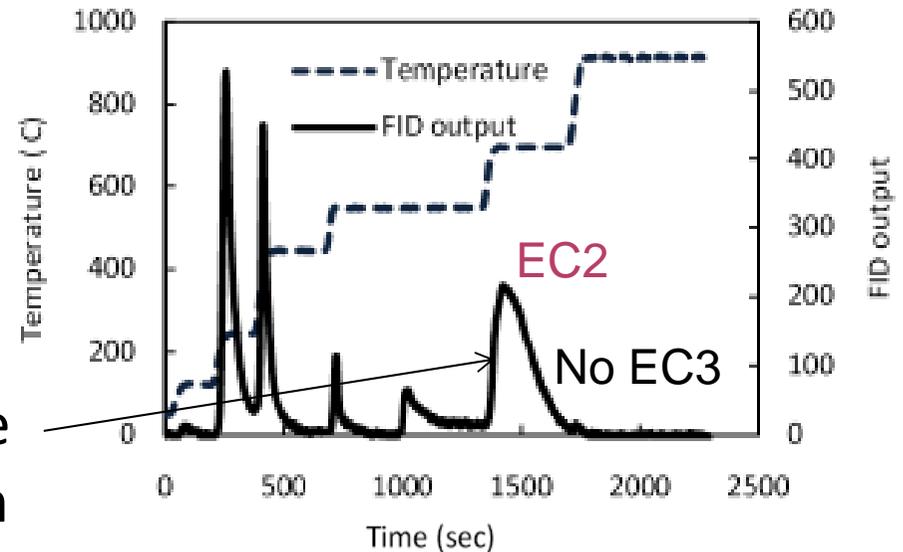
	Time (sec)	Oven temperature(°C)	Atmospheric gas
OC1	180	120	He
OC2	180	250	He
OC3	300	450	He
OC4	300	550	He
EC1	360	550	He/5% O <sub>2</sub>
EC2	360 - 600	700	He/5% O <sub>2</sub>
EC3	600	920	He/5% O <sub>2</sub>

Graphitic  
carbon

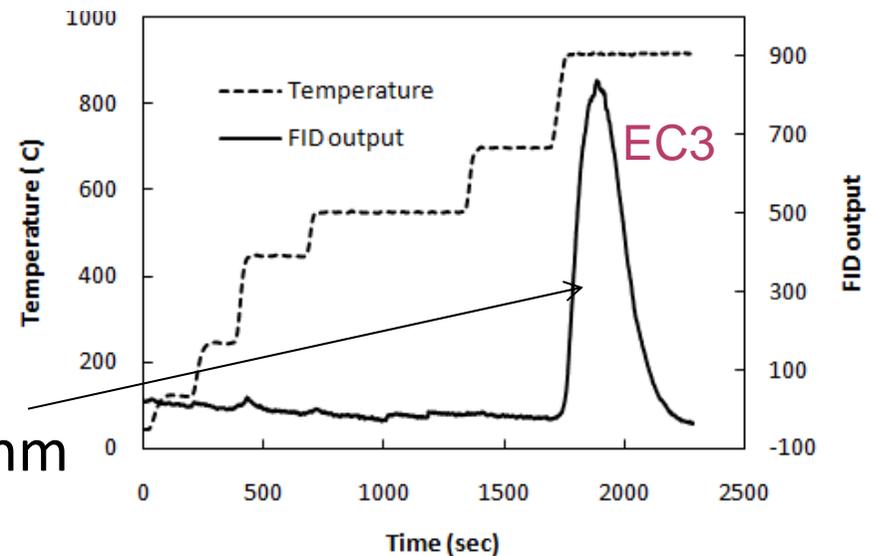
OC: Organic carbon, EC: Elemental carbon

# Concept for distinguishing MWCNT

Ambient particle  
Graphitic carbon

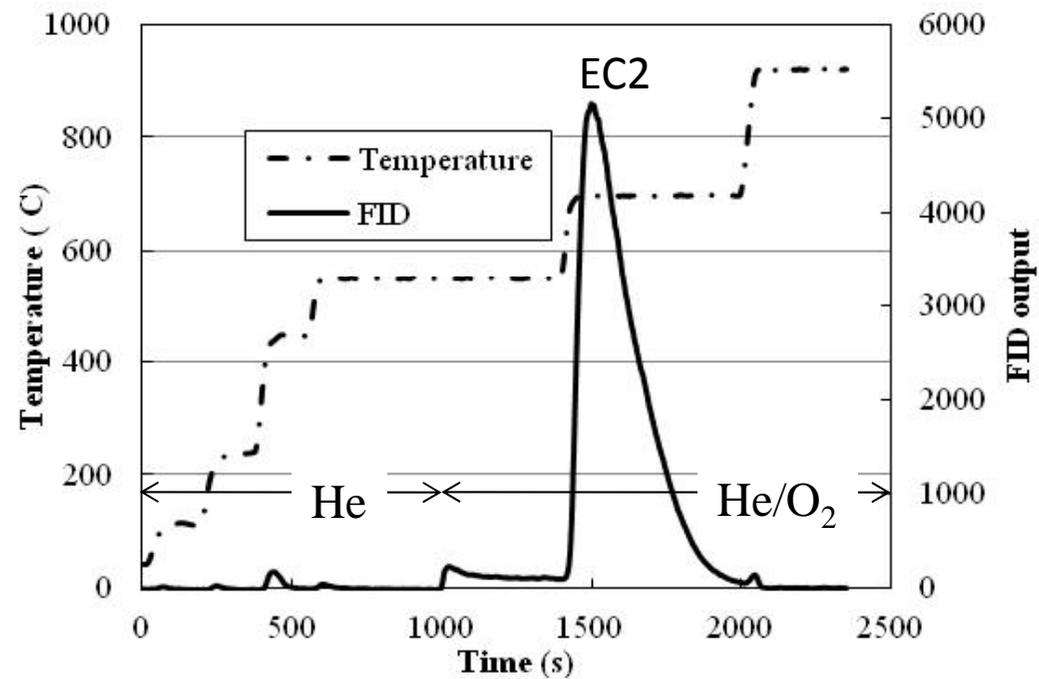


MWCNT  
Diameter >20 nm



# Baytube

## EC2 temperature setting at 700°C



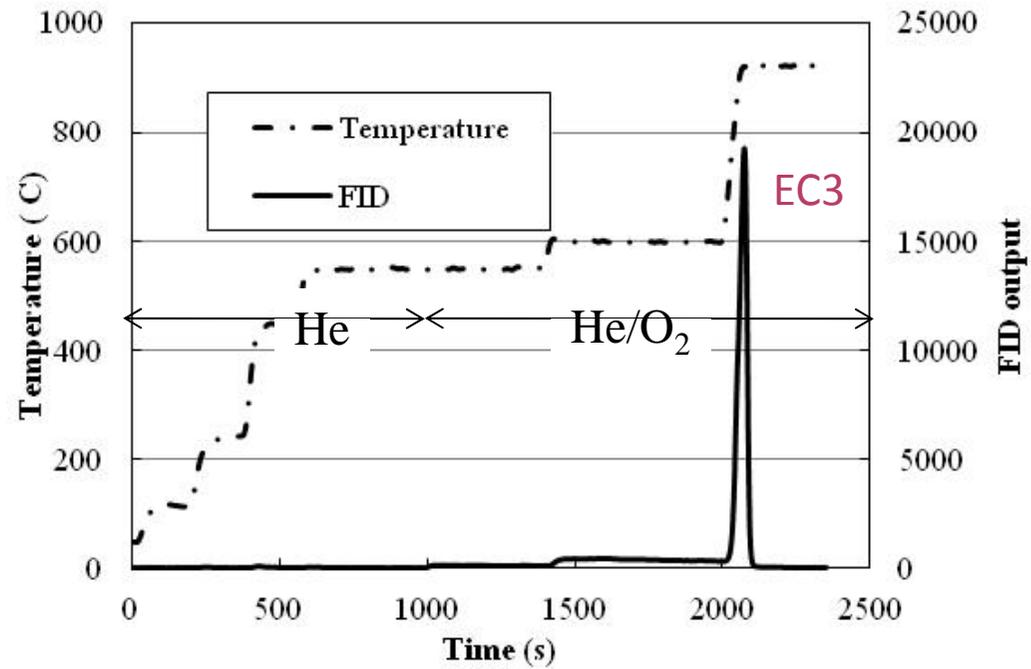
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EC2	360 - 600	600	He/5% O <sub>2</sub>
EC3	600	920	He/5% O <sub>2</sub>

OC: Organic carbon, EC: Elemental carbon

# Baytube

## EC2 temperature setting at 600°C



# Sample preparation for TEM CNTEC embedded resin block

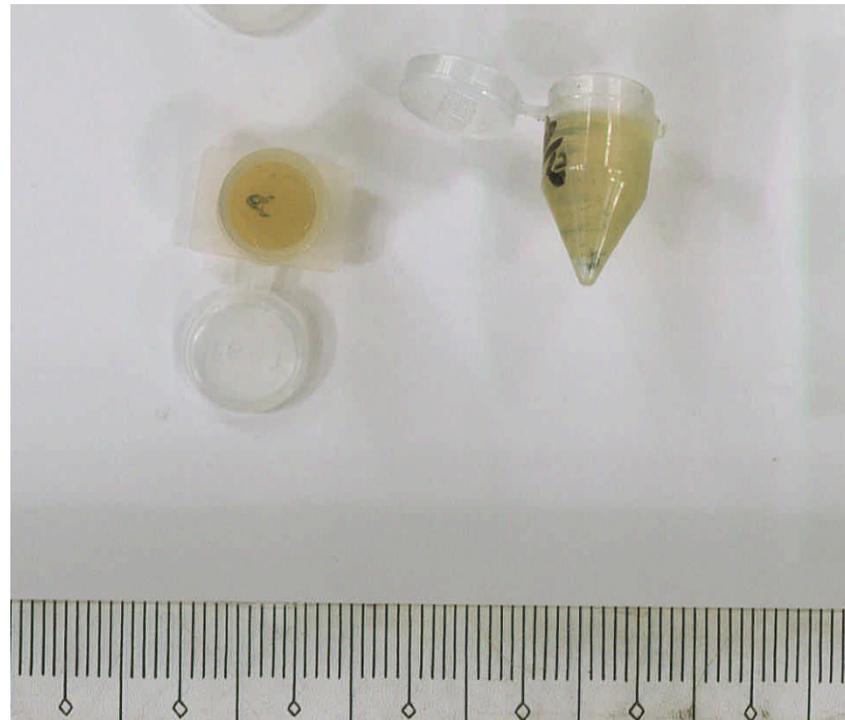
Before carbon analysis



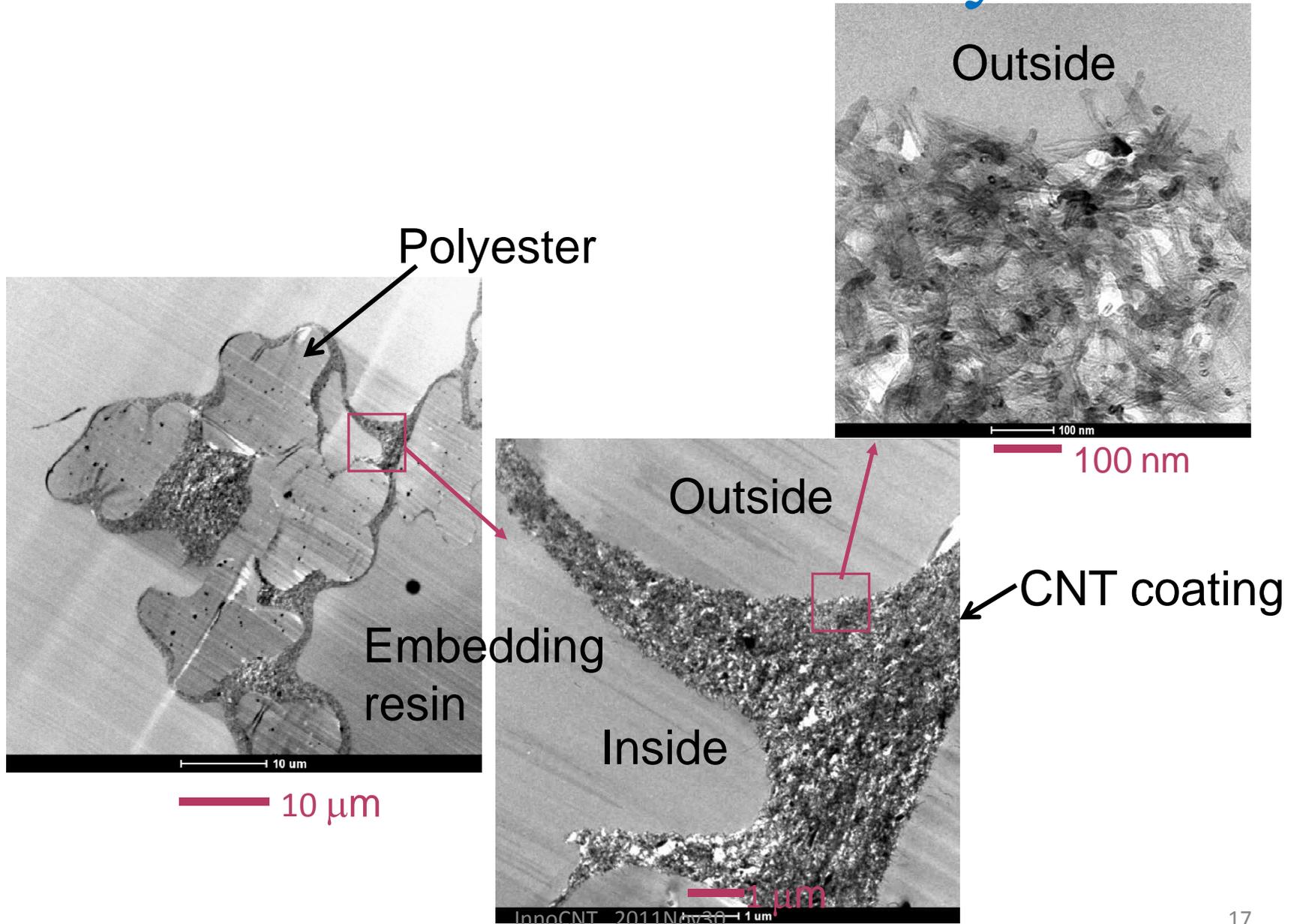
CNTEC



EC3 after EC2 burned

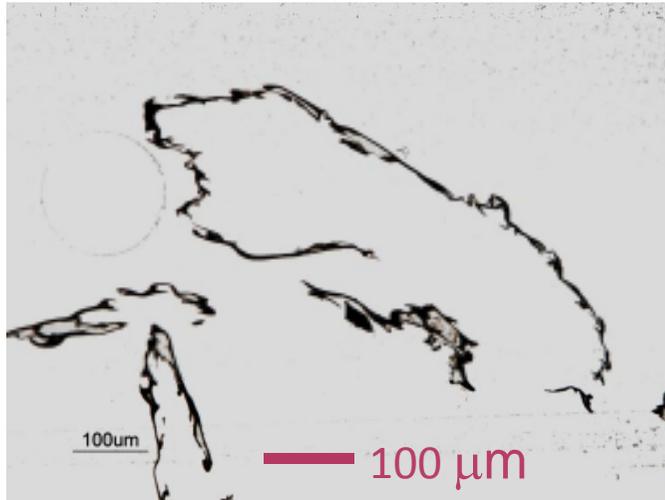


# CNTEC before carbon analysis

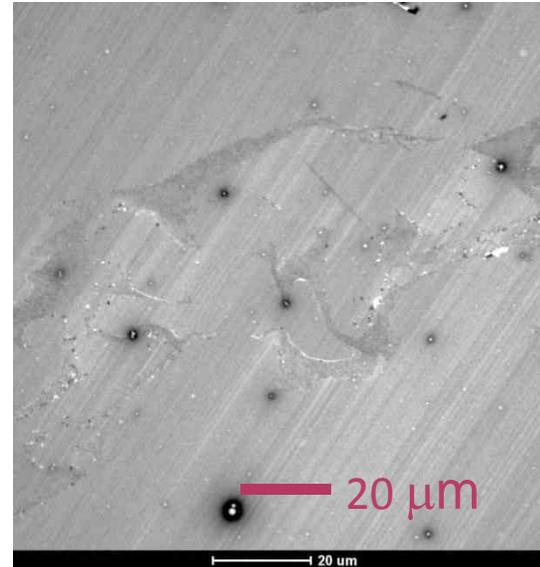


# EC3 left after EC2 burned

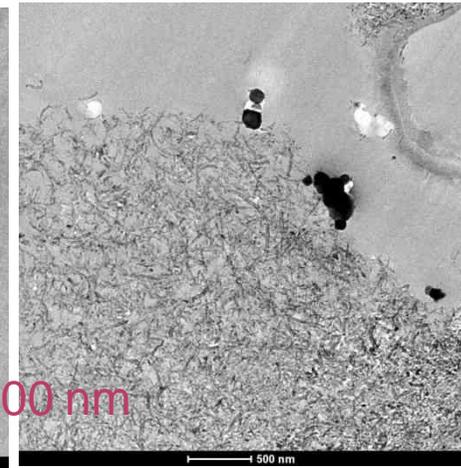
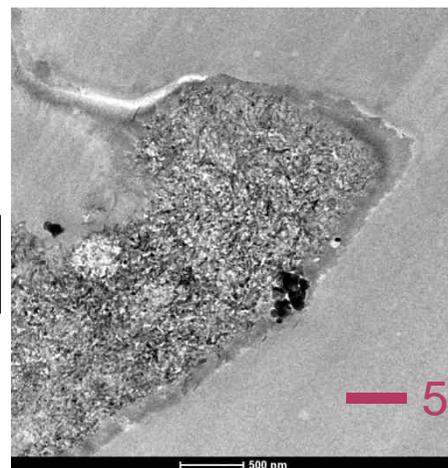
Optical micrograph



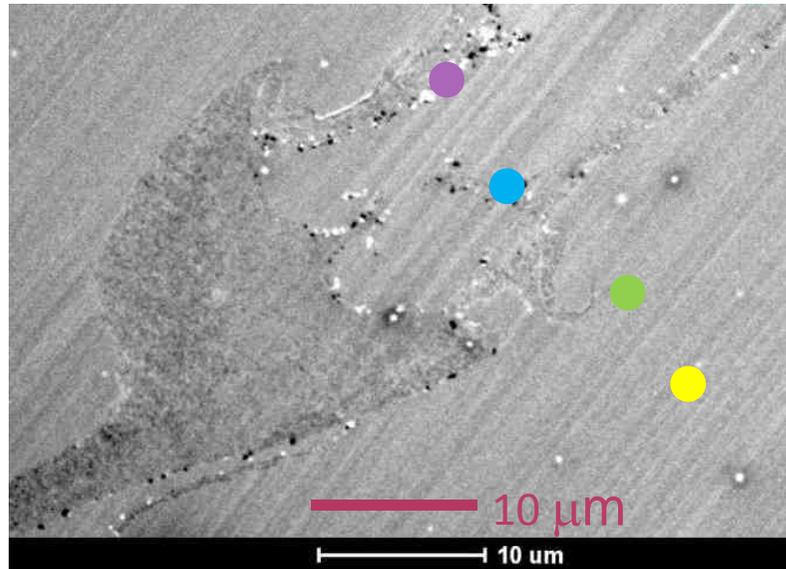
TEM



Polyester was not observed

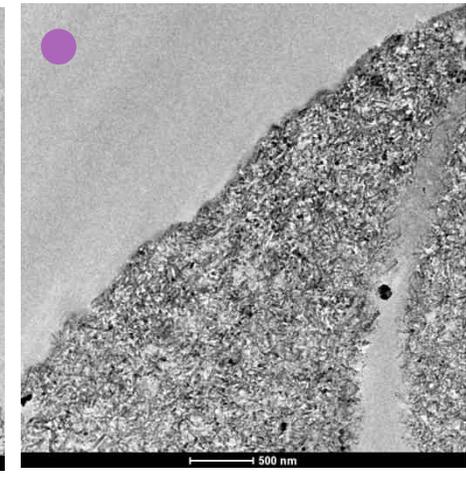
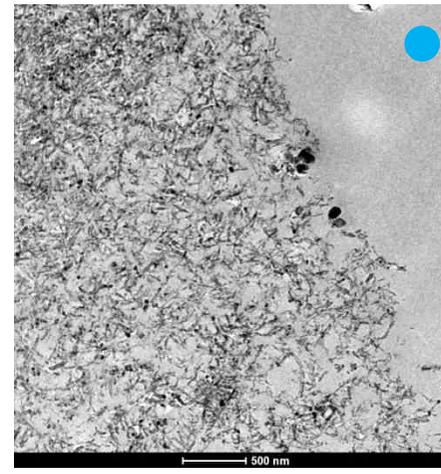
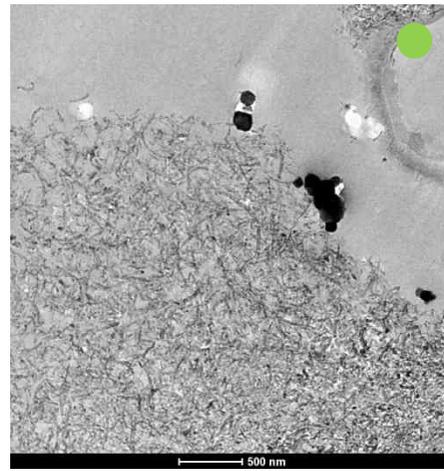
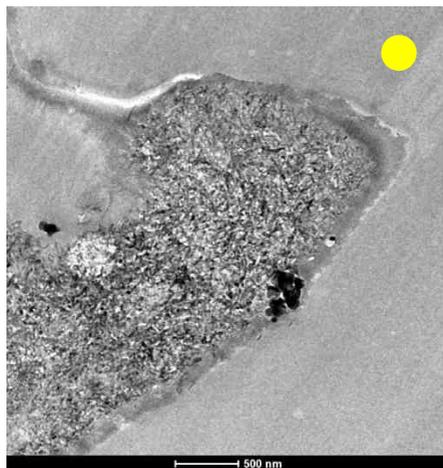


# EC3 left after EC2 burned



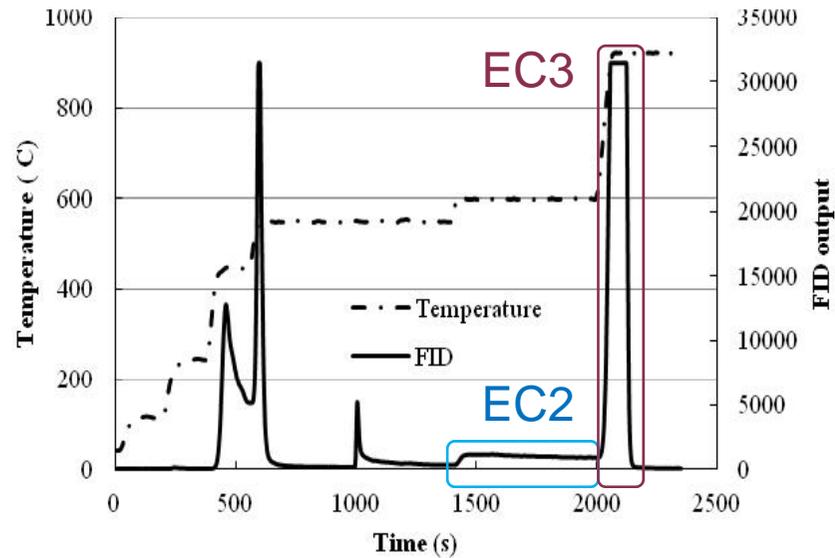
After burning,

- CNT becomes thinner
- Density of CNT coating becomes less dense

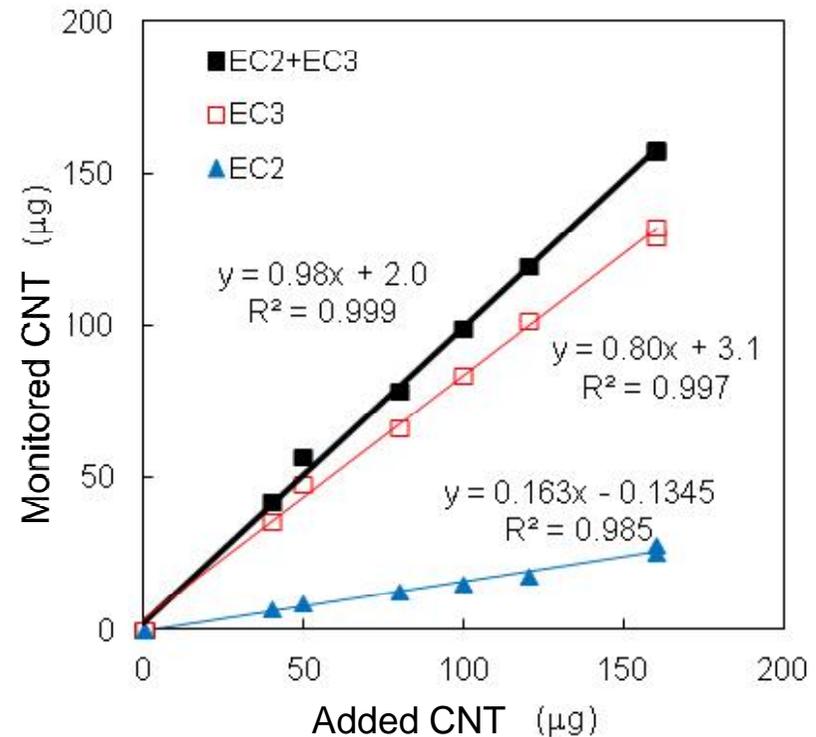


100 nm

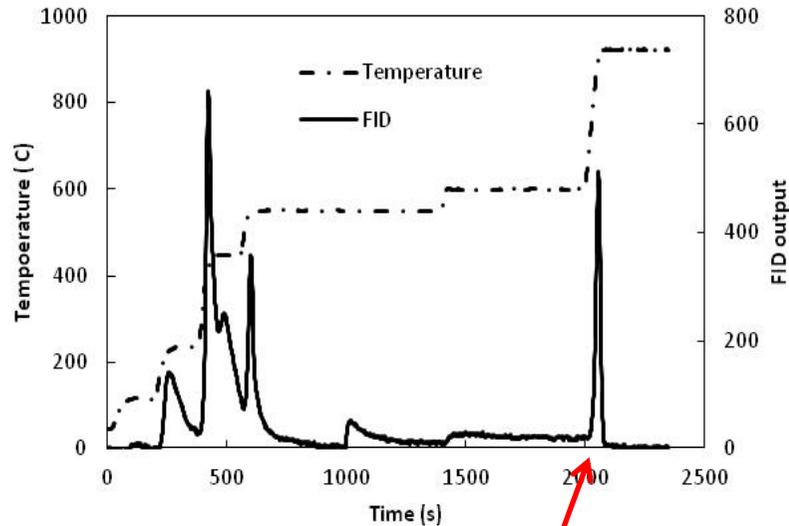
# Calibration curves by coating solution



Thermogram of CNT coating solution

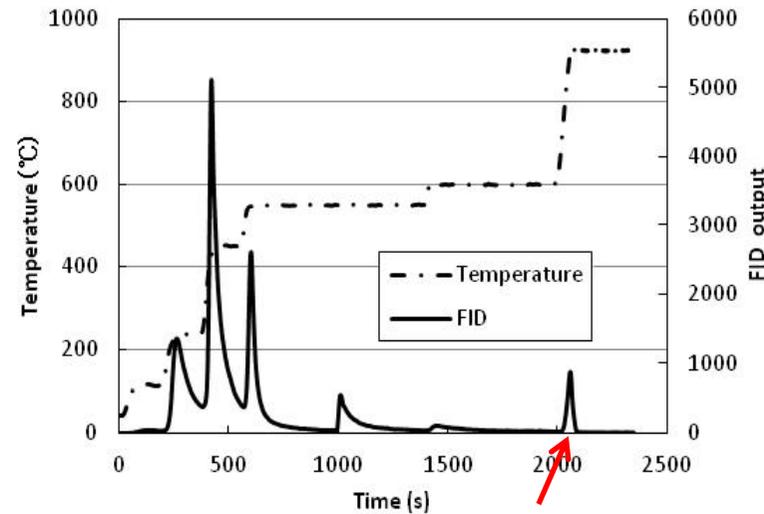


# Typical thermograms of collected samples



MWCNT

Sample: area sampling  
collected at sampling point 2,  
close to the weaving surface  
Size: 2.5–6.6  $\mu\text{m}$



MWCNT

Sample: wiped sample  
collected on the surface  
near physical stress given  
to CNTEC

# MWCNT concentrations

- MWCNT mainly contained in fragments of CNTEC
- EC3 concentration ( $\mu\text{g}/\text{m}^3$ )
  - Background (night) 1.2  
other looms for polyester yarn working
  - Background (working time) 2.7  
far from the CNTEC loom, affected by outside air
  - Sampling point 1 4.4 – 4.6  
2 m away from the loom
  - Sampling point 2 5.3  
close to the weaving surface
- Respirable ( $<4 \mu\text{m}$ ) mass concentration 66  
personal sampling

# Summary

- Real-time monitoring did not show any relationship with the present process.
- MWCNTs are quantified by carbon analysis with IMPROVE method.
- MWCNTs in CNTEC can be determined by EC3.
- EC3 can be an index of MWCNT, even if perfect separation of MWCNT from polyester and ambient particles is difficult.
- SEM/TEM observation is necessary.
- Protocol of carbon analysis should be considered for each target CNT because there are varieties of MWCNTs.