Imperial College London

The Composites Centre

From water-epoxy slurries to hierarchical composites

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Team MAST

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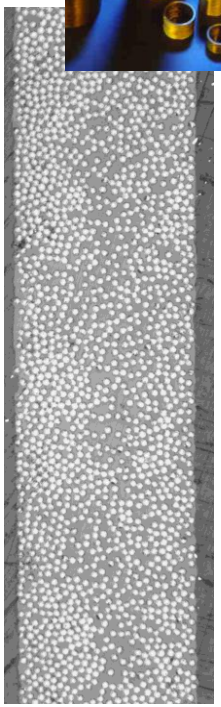
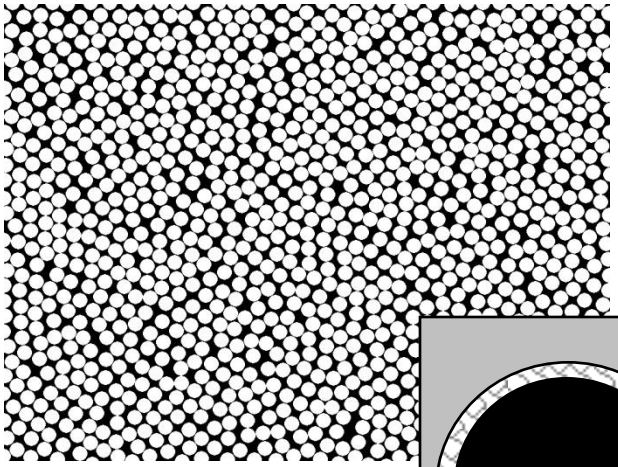
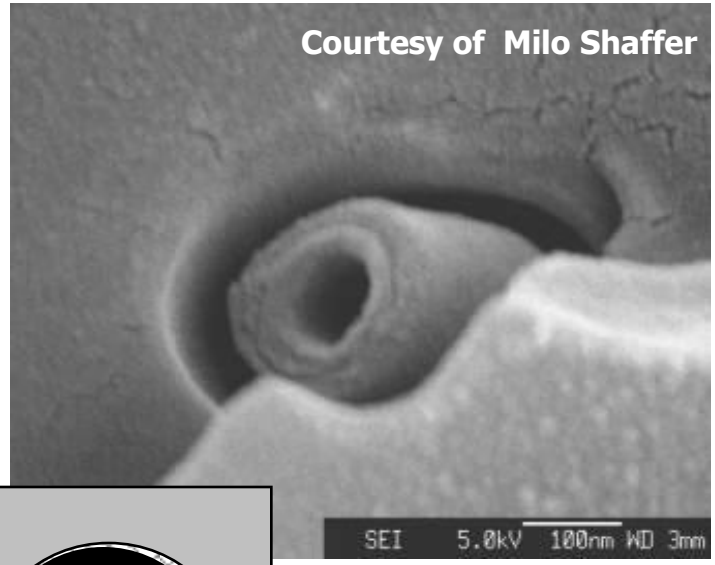
Outline

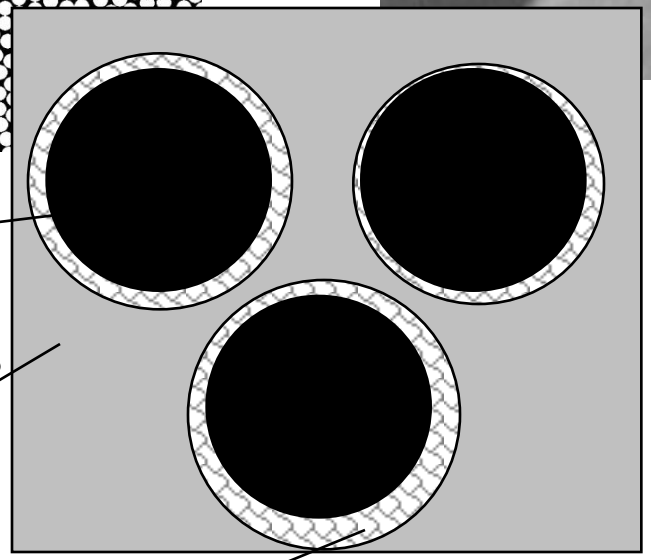


- What are we aiming for?
- What are Hierarchical Composites?
- Challenges to be tackled
- The “hairy fibre” route
- The nanocomposite route to thermoset hierarchical composites
- Summary

What Are Composites?







No wetting →
No interface

Fibre

parallel strength, E-modulus

Matrix

transverse strength, HT-properties

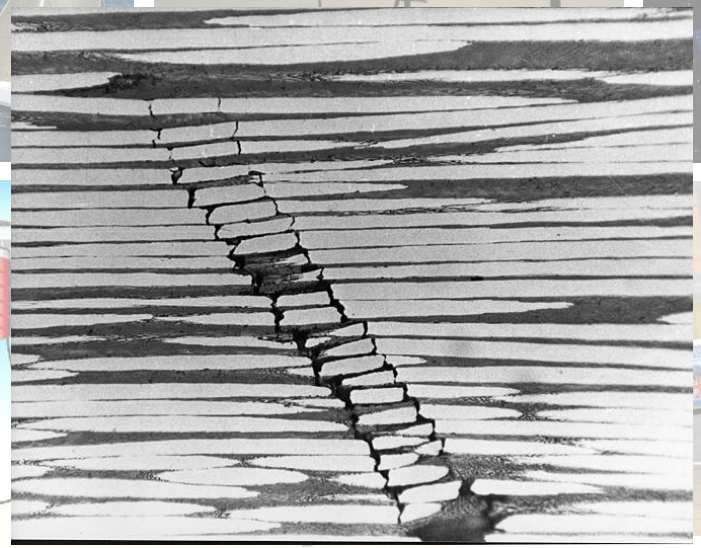
Interphase

adhesion, load transfer & overall performance

Interface

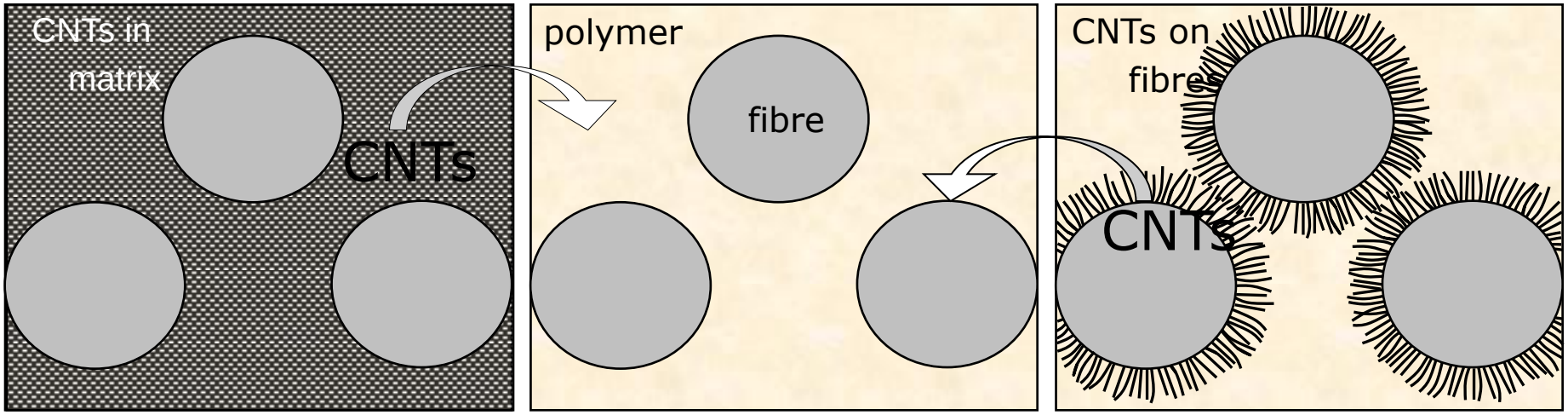
Fibre reinforced polymer composites

- current state of the art
- excellent in-plane properties
- ‘weak’ compression and interlaminar properties



Objective: hierarchical composites!

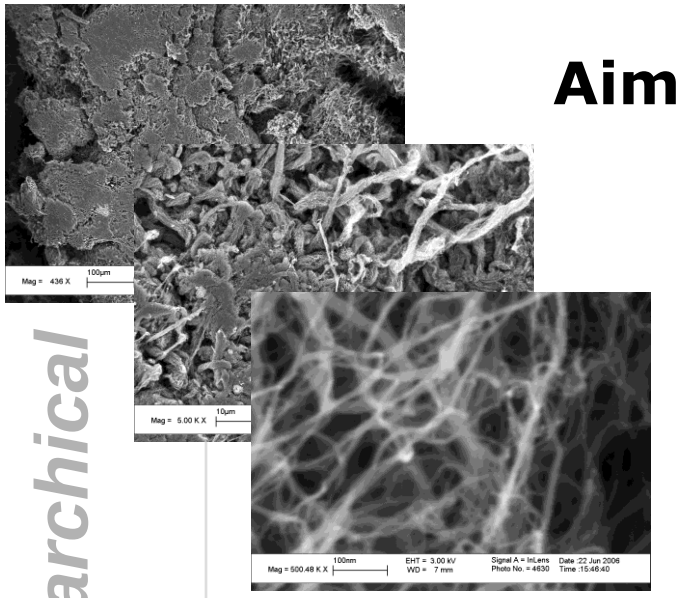
Hierarchical composite structures with CNTs:



Incorporation of nano-reinforcement should improve:

- through-thickness properties & other critical properties

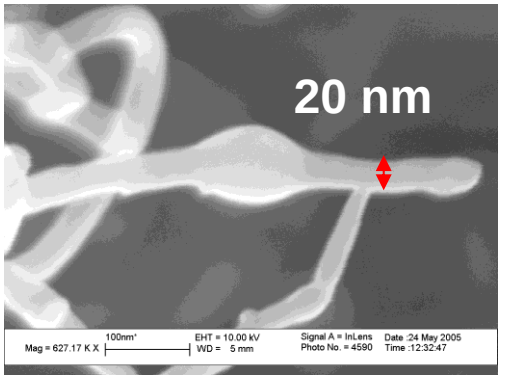
Challenges to be addressed



Aim reinforce matrix with "nano"-sized "filler"

Challenges:

1. disperse CNTs
2. tailor adhesion
3. maintain processability of matrix or identify new processing routes



En route to hierarchical composites

further reinforce nanocomposite matrix with conventional fibres to create

high performance hierarchical composites

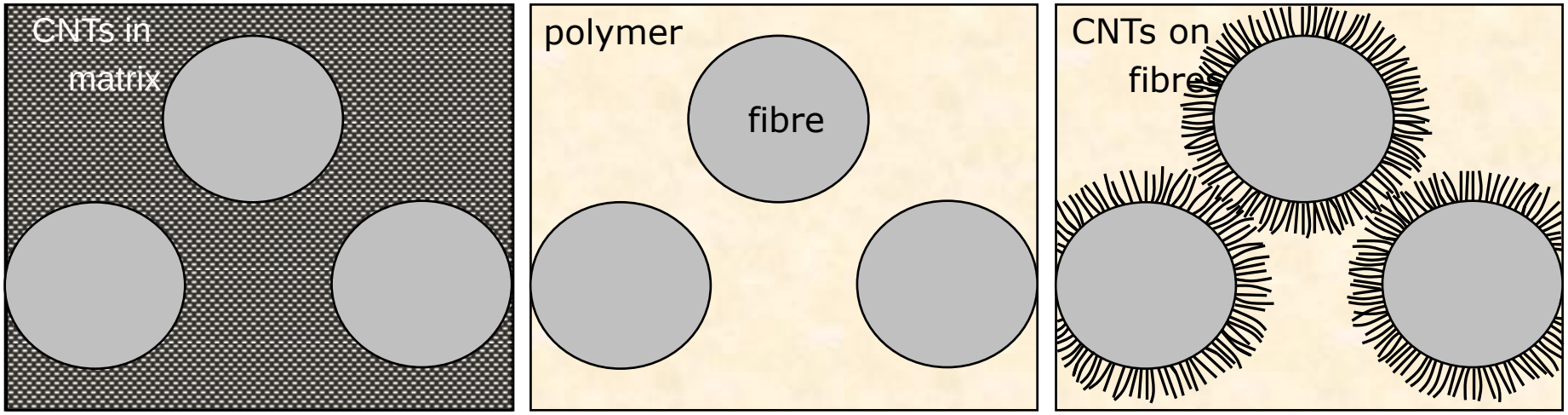
with improved mechanical properties, thermal stability and reduced through life costs

Motivation

- Achieve good CNT dispersion for high (10+wt%) loadings
- “Upgrade” cheap epoxy resins
- Identify route to scale-up fabrication
- Demonstrate improvements in fracture toughness and critical engineering properties
- Can it be combined with hairy fibre research?
  - Ultra-high CNT loadings

Objective: hierarchical composites!

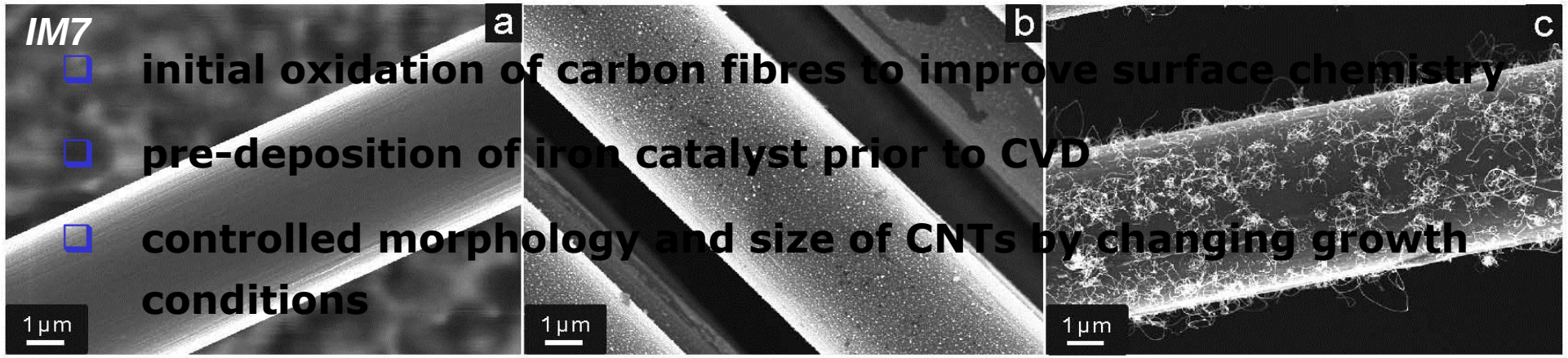
Hierarchical composite structures with CNTs:



Using "hairy" fibres to deliver nano-reinforcement should improve:

- dispersion & alignment of nano-reinforcement
- interfacial area & bonding
- through-thickness properties & other critical properties

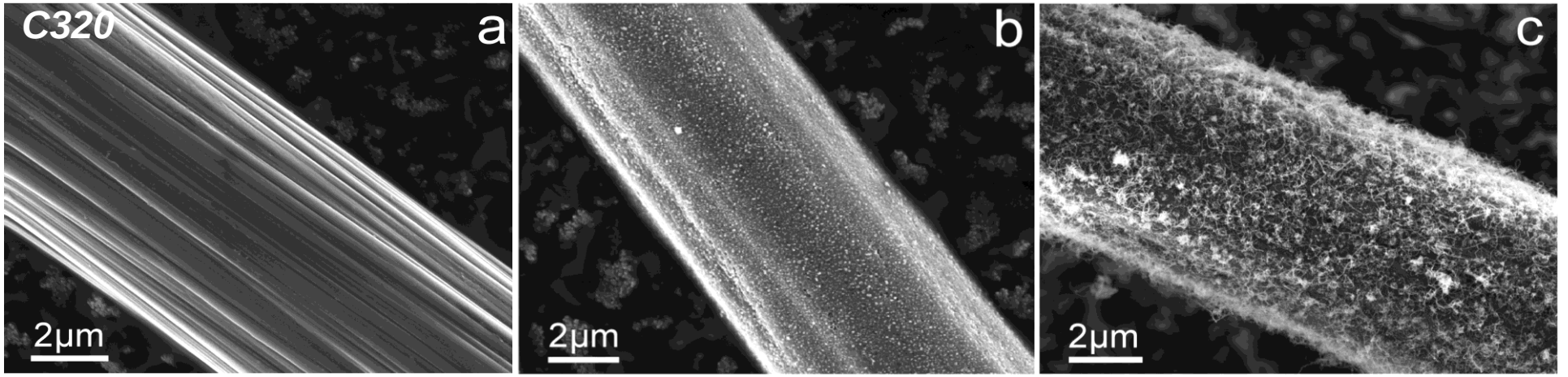
CNT-grafted carbon fibres



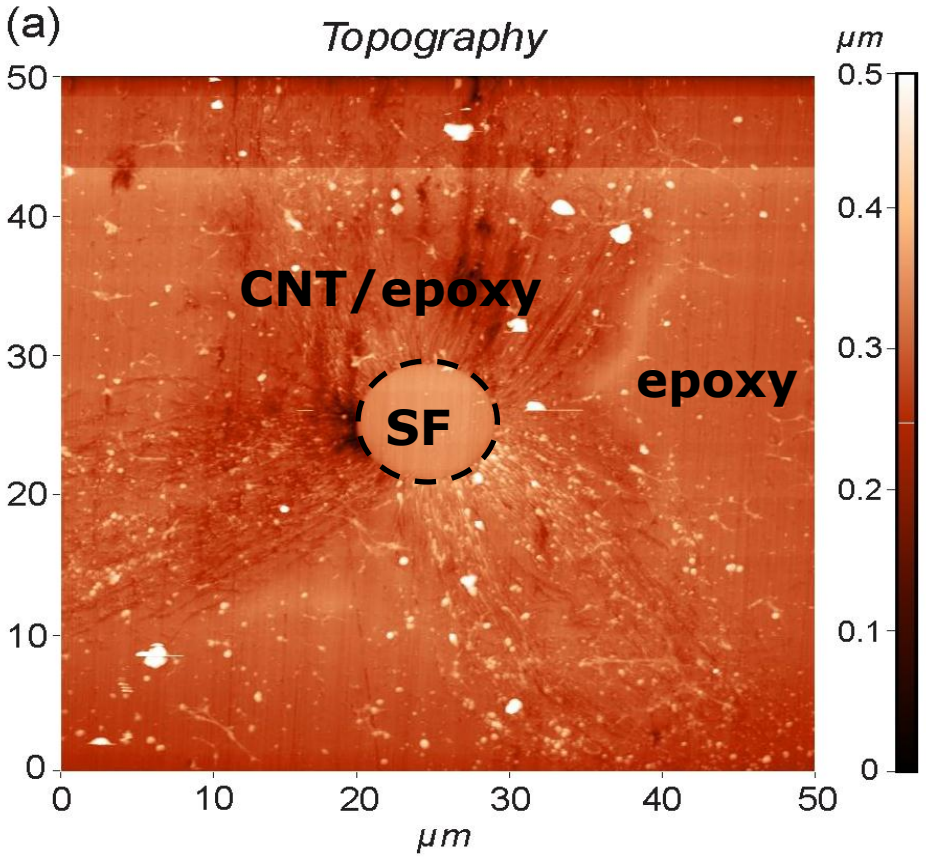
- initial oxidation of carbon fibres to improve surface chemistry
- pre-deposition of iron catalyst prior to CVD
- controlled morphology and size of CNTs by changing growth conditions

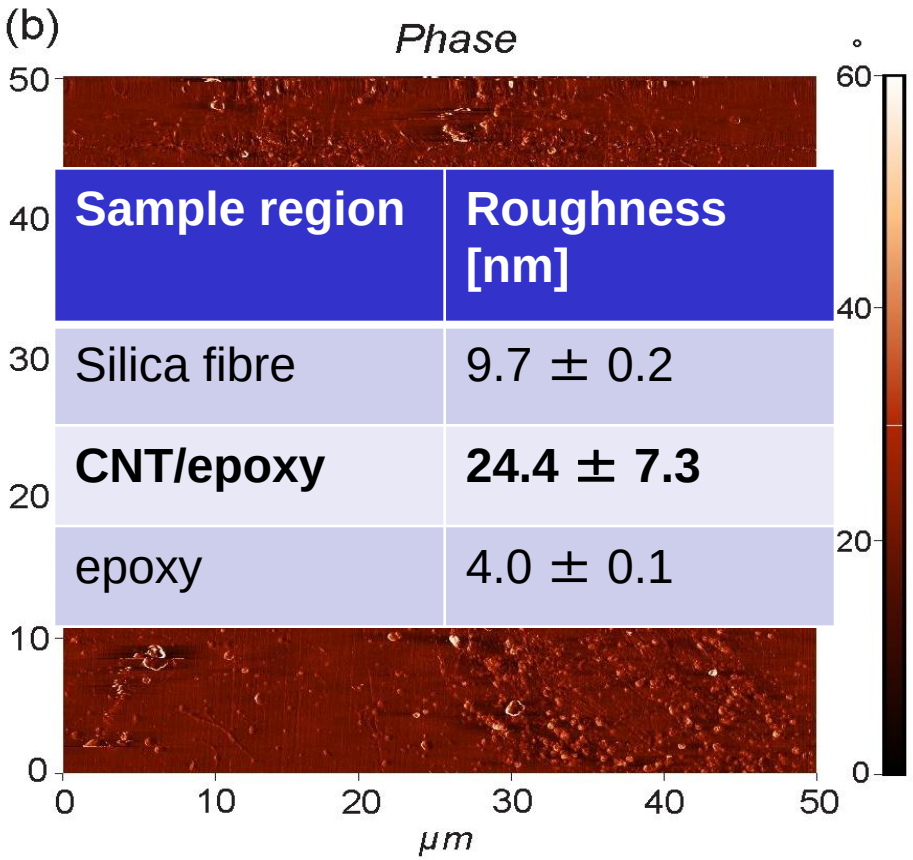
catalyst loading

CNT growth



Atomic force microscopy (AFM)



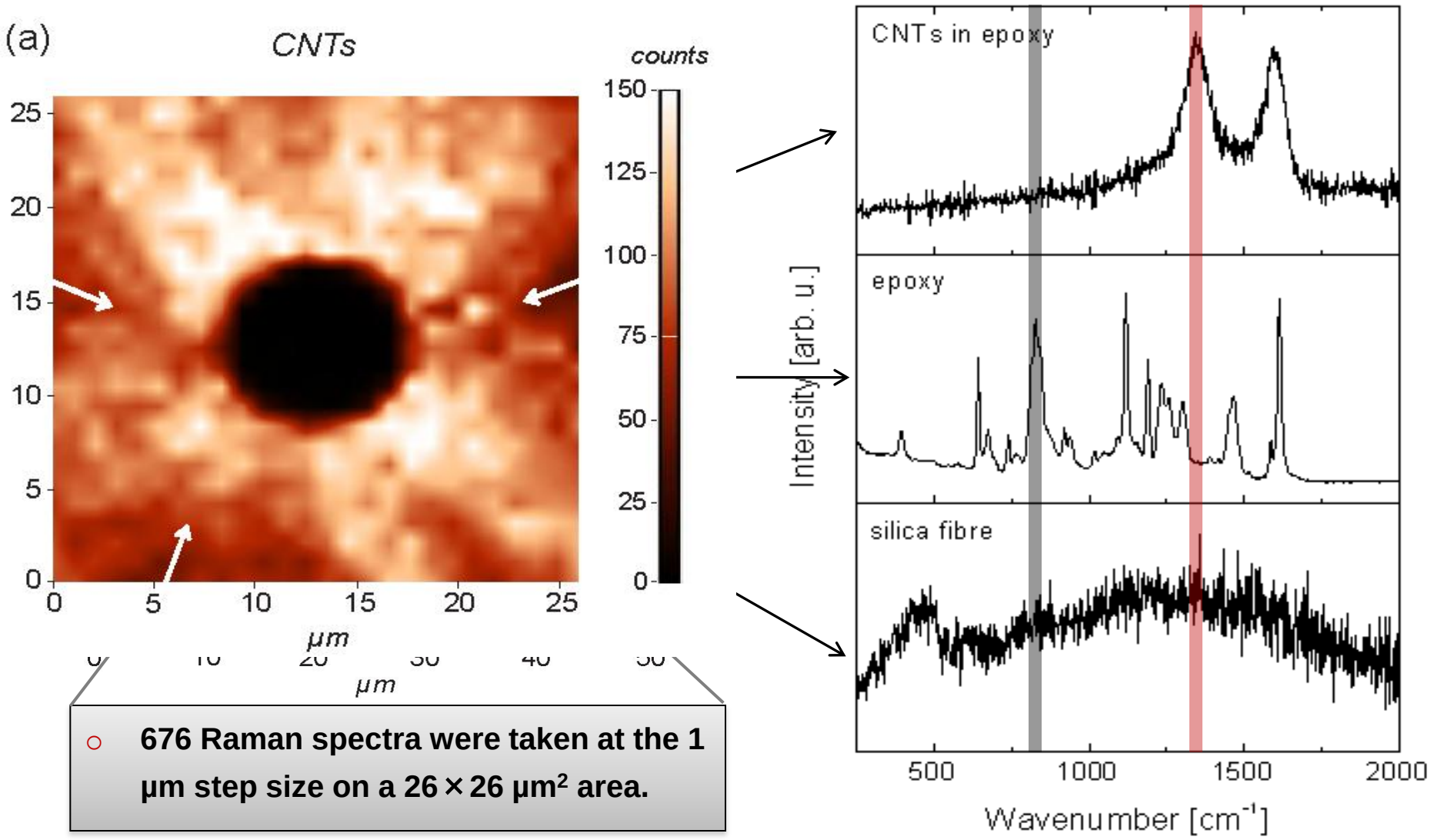


Sample region	Roughness [nm]
Silica fibre	9.7 ± 0.2
CNT/epoxy	**24.4 ± 7.3**
epoxy	4.0 ± 0.1

- AFM images showed variations in surface roughness and material properties.

Ref.: H. Qian et al. *Nanoscale,* 3 (2011) 4759.

Raman mapping

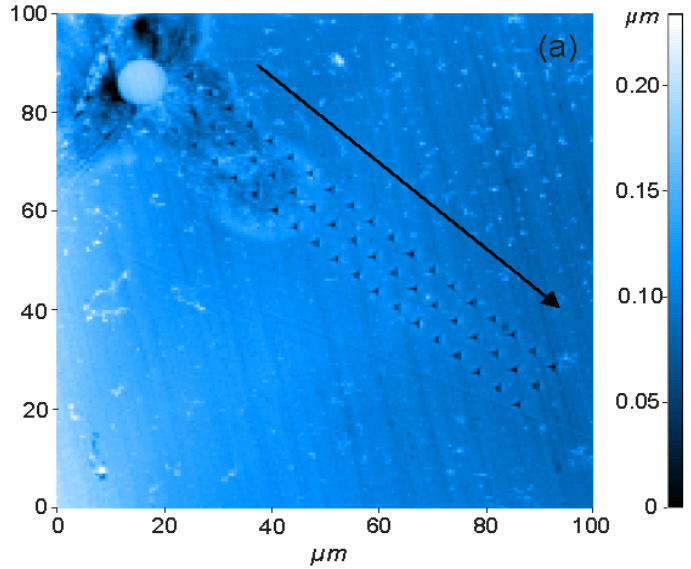


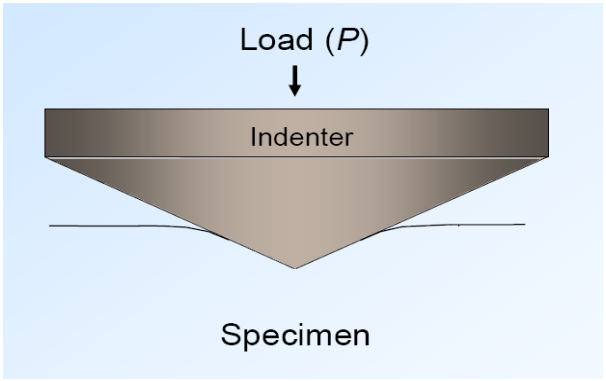
- **676 Raman spectra were taken at the 1 μm step size on a 26 × 26 μm² area.**

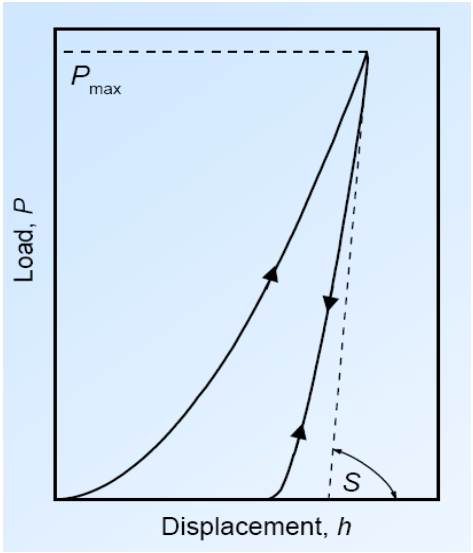
Ref.: H. Qian et al. *Nanoscale,* 3 (2011) 4759.

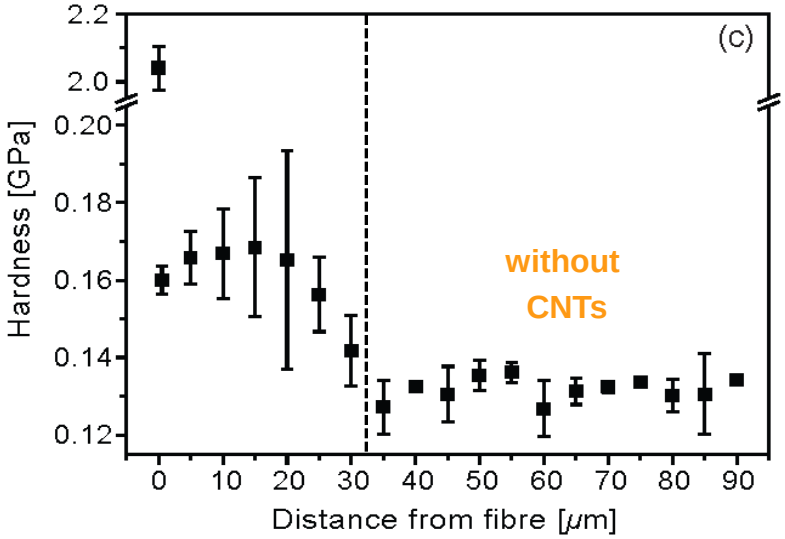
Nanoindentation

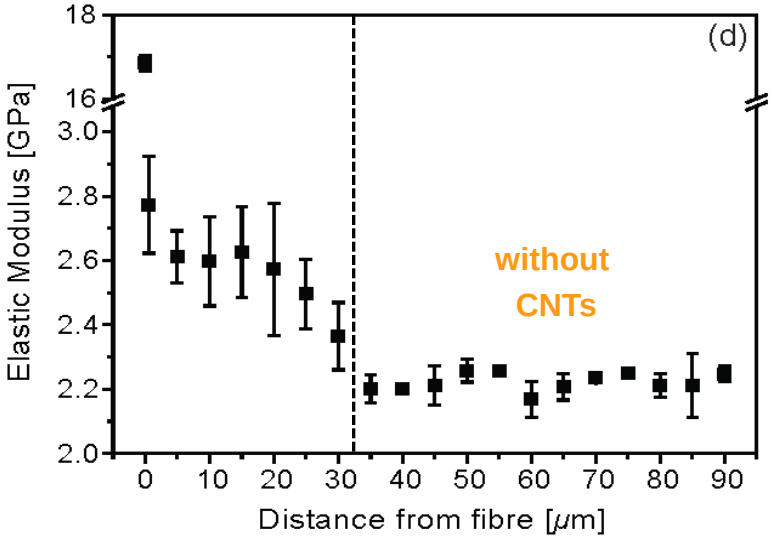
CNT-g-silica fibres











Ref.: H. Qian et al. *Nanoscale,* 3 (2011) 4759.

Summary ‘Hairy Fibres’

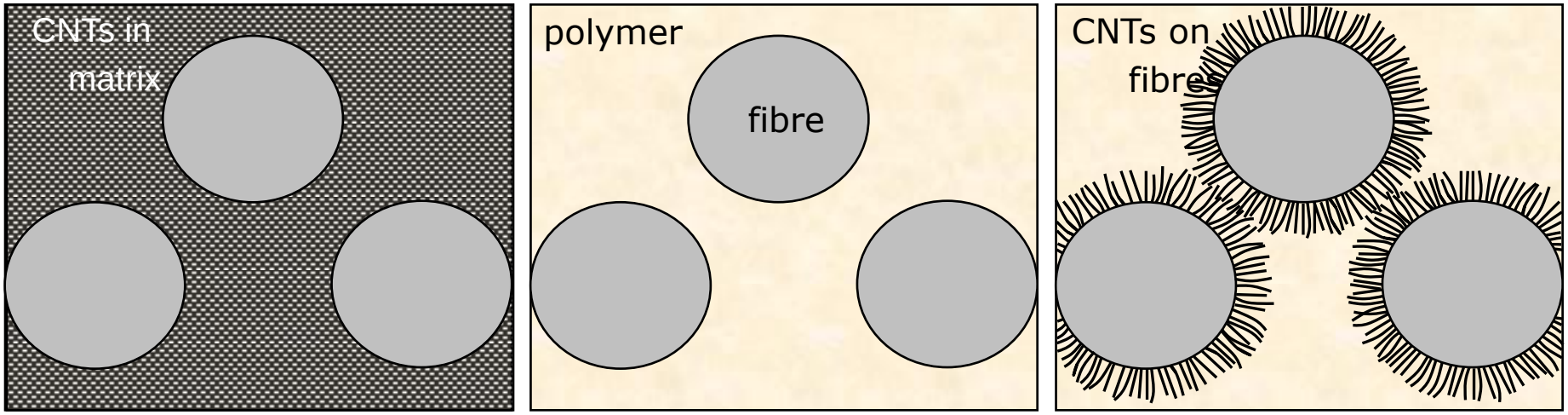
Key findings:

- successful CNT growth on different fibres
- significant increase of surface area
- unaffected fibre modulus, but a decrease of fibre tensile strength
- good wettability of hairy fibres
- 30-150% improvement of IFSS
- increase of hardness and modulus of the surrounding matrix

Ref.: H. Qian, E.S. Greenhalgh, M.S.P. Shaffer, A. Bismarck, *J. Mater. Chem.* 20 (2010) 4751-62.

Objective: hierarchical composites!

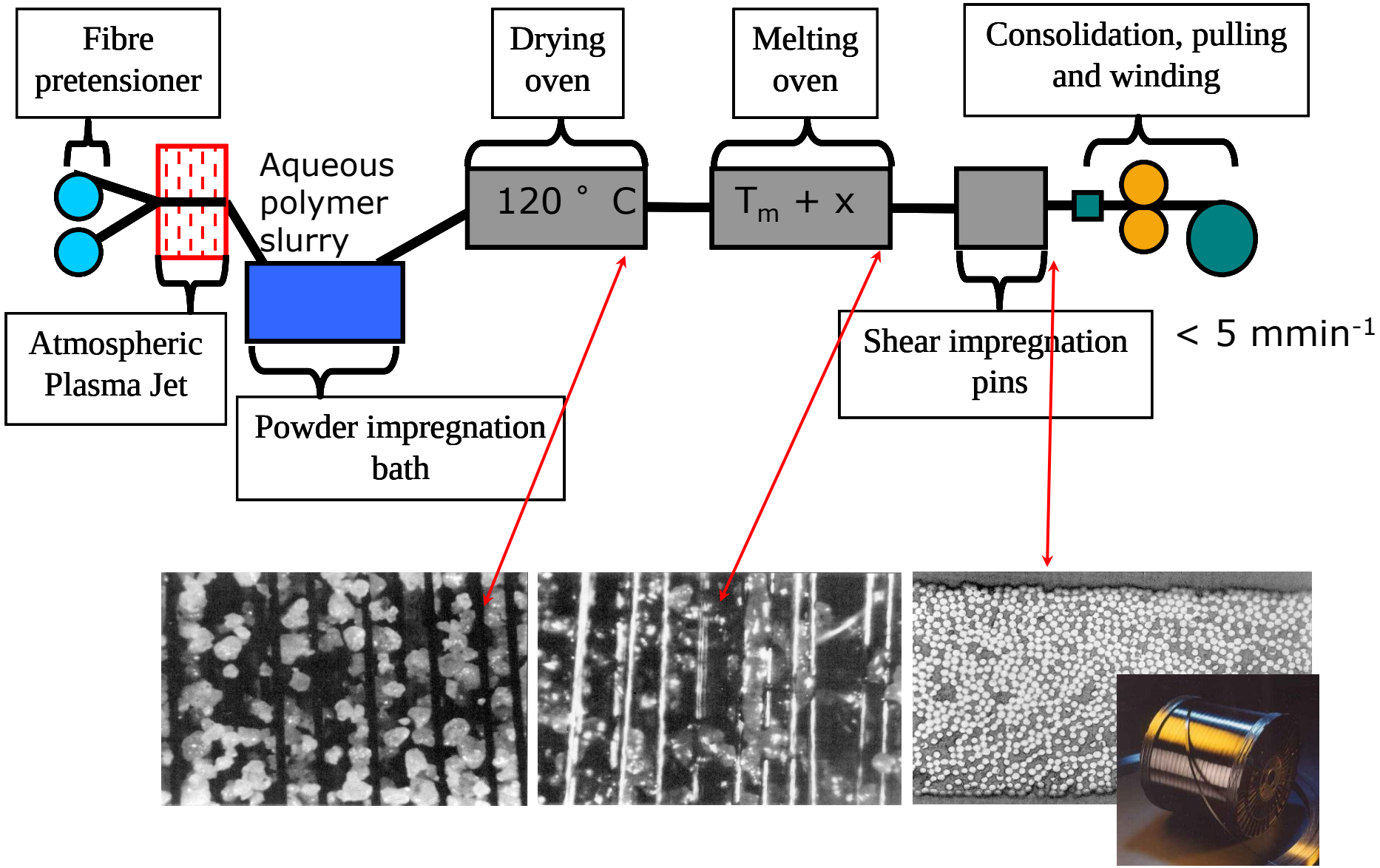
Hierarchical composite structures with CNTs:



Using nanocomposite powder impregnation route should:

- allow to disperse nano-reinforcement throughout composite
- be easy and fast to process
- through-thickness strength & other critical properties

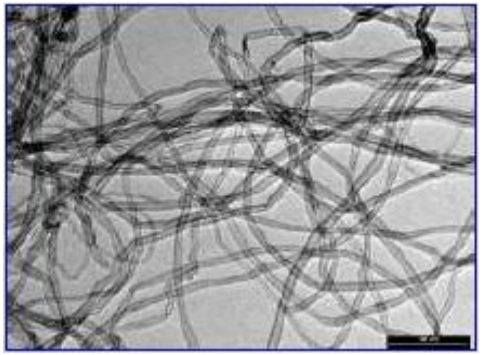
Continuous Processing



Ref.: J. Kärger et al. *Sulzer Technical Review*, 2 (1999) 4.

Materials

- Carbon Nanotubes (CNTs)
  - Nanocyl NC 7000
- Epoxy Matrix and hardener
  - EPIKOTE 1001 epoxy
  - DICY hardener
- Carbon Fibres
  - AS4C – GP (Hexcel)



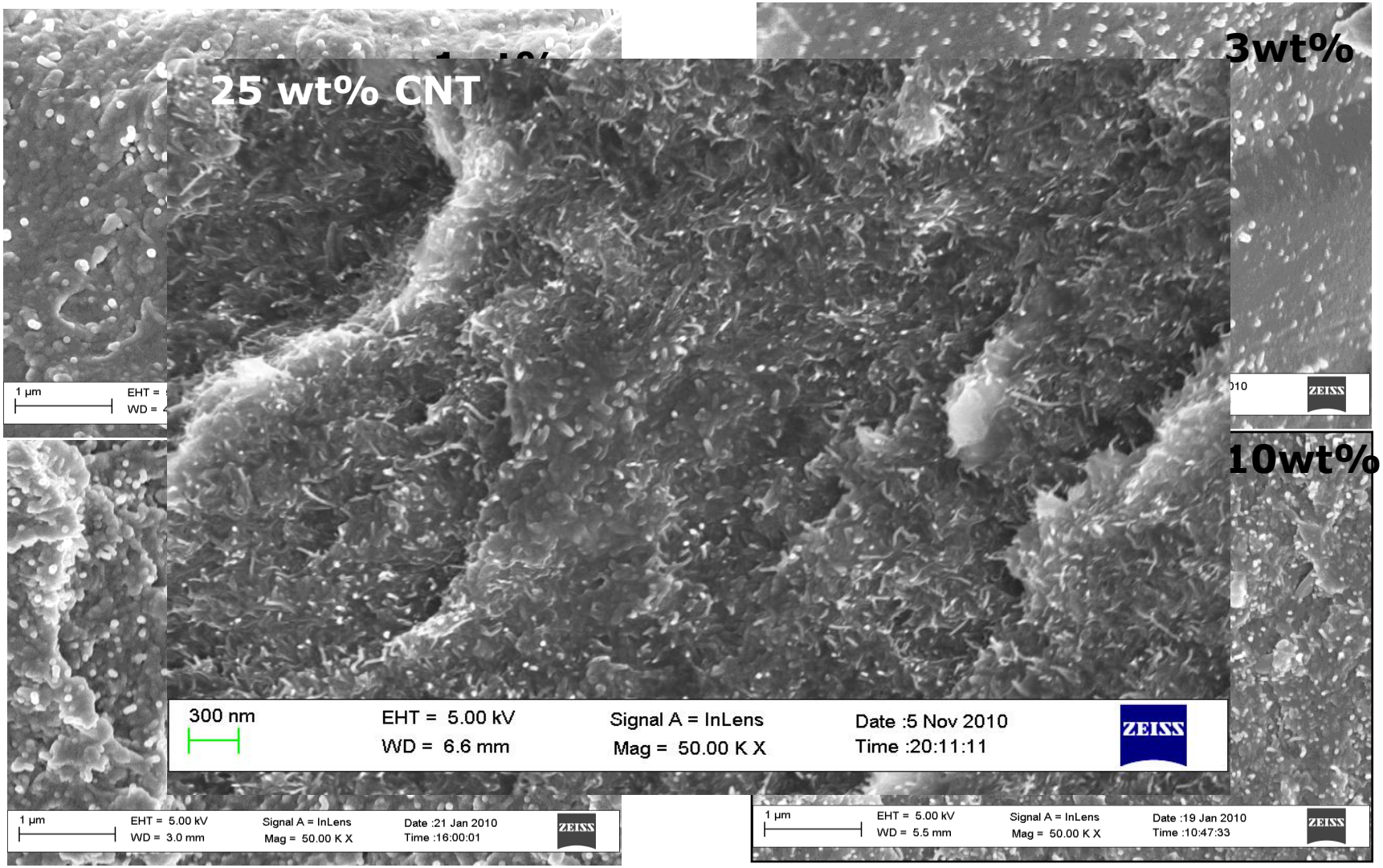
Nanocomposite production

- **Looking beyond traditional thermoset mixing**

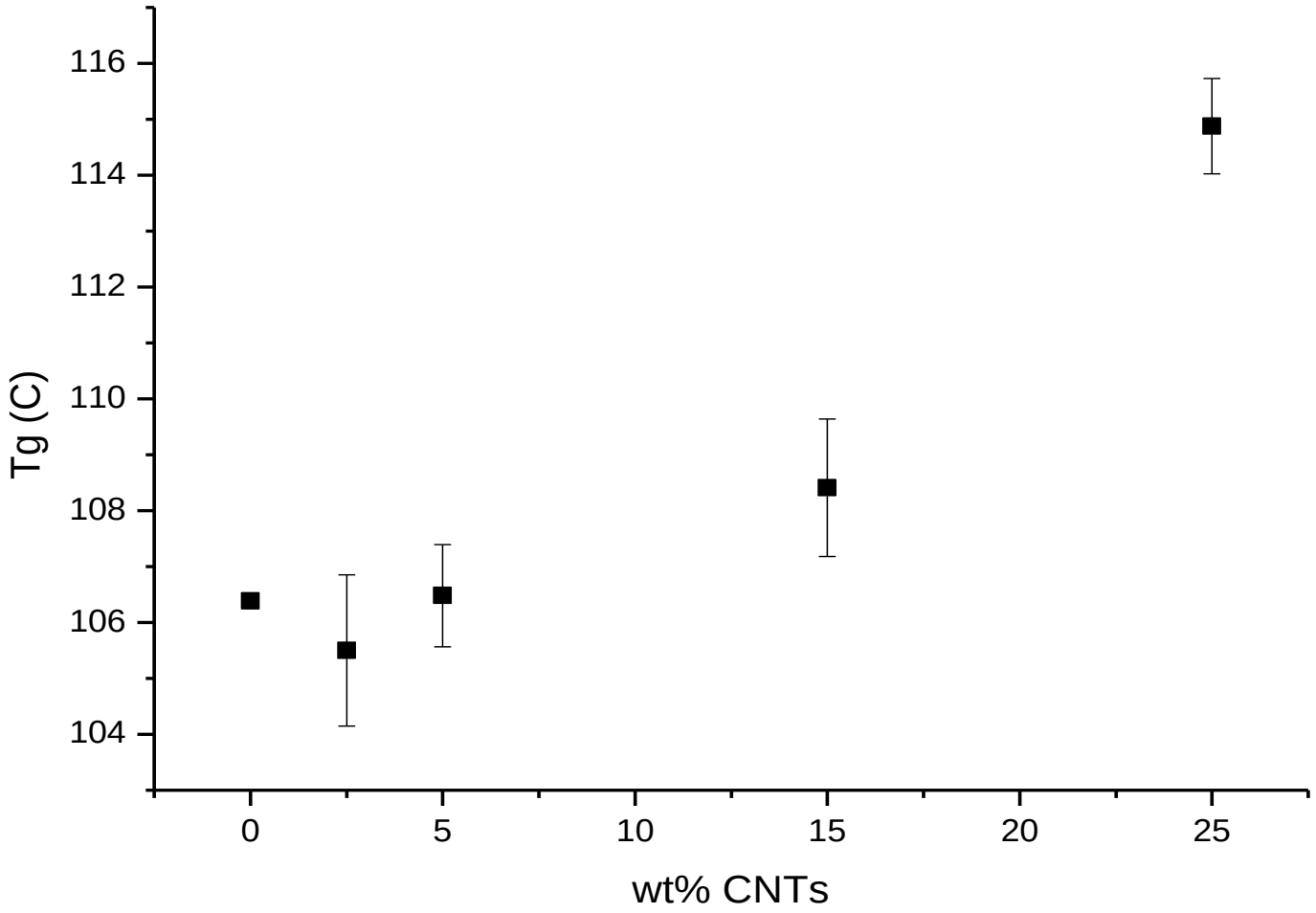
- **Extrusion**
  - Constrained system
  - High shear forces
  - Easily scalable to continuous production

CNT dispersion

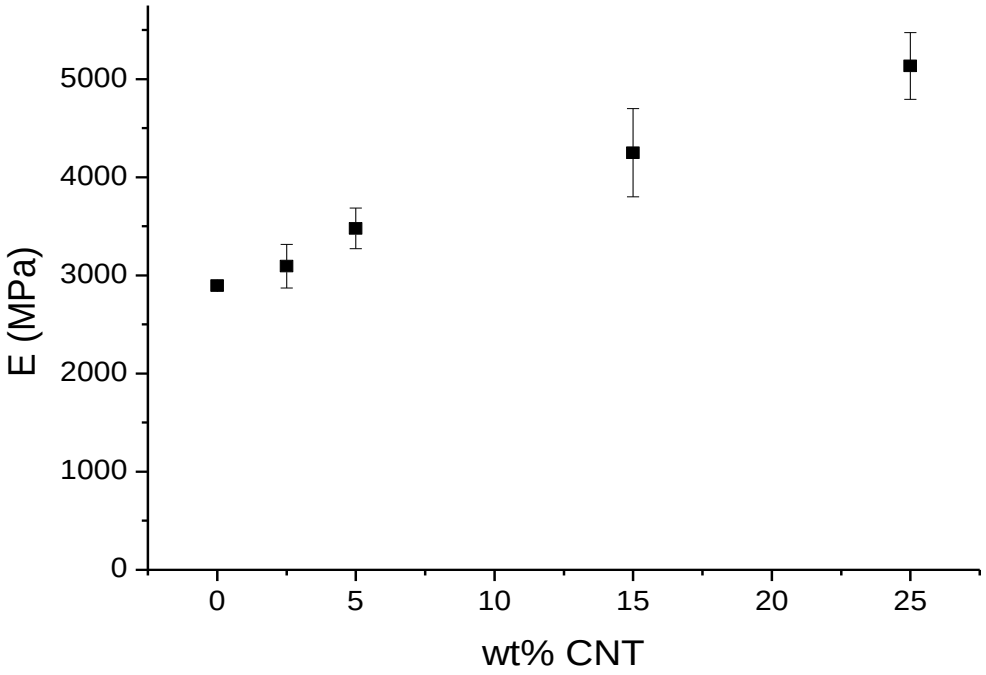
- Very good CNT dispersion and distribution

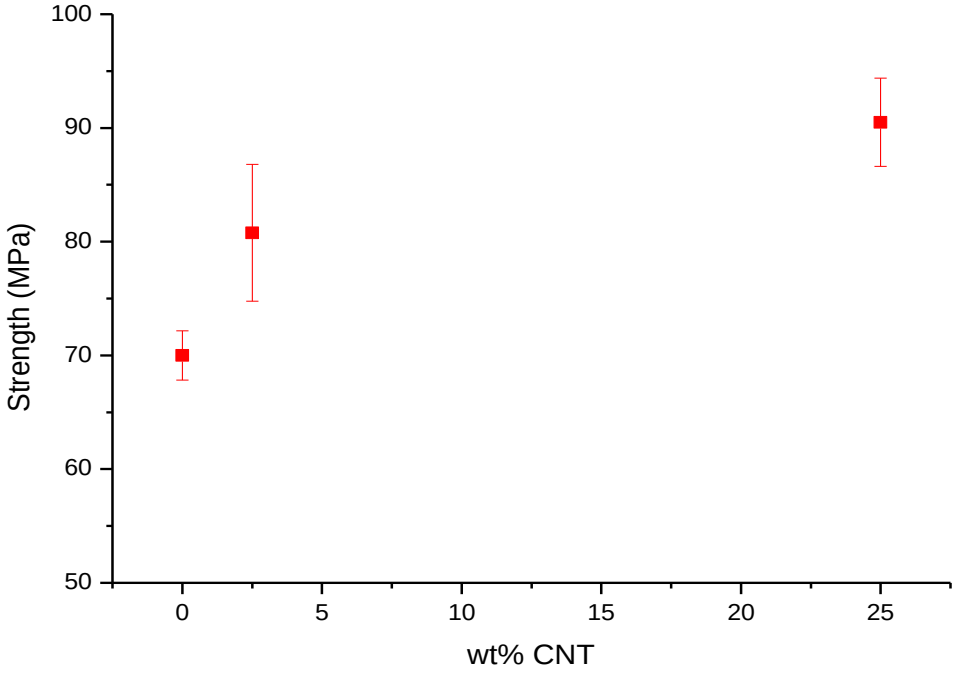


Glass transition of modified matrix



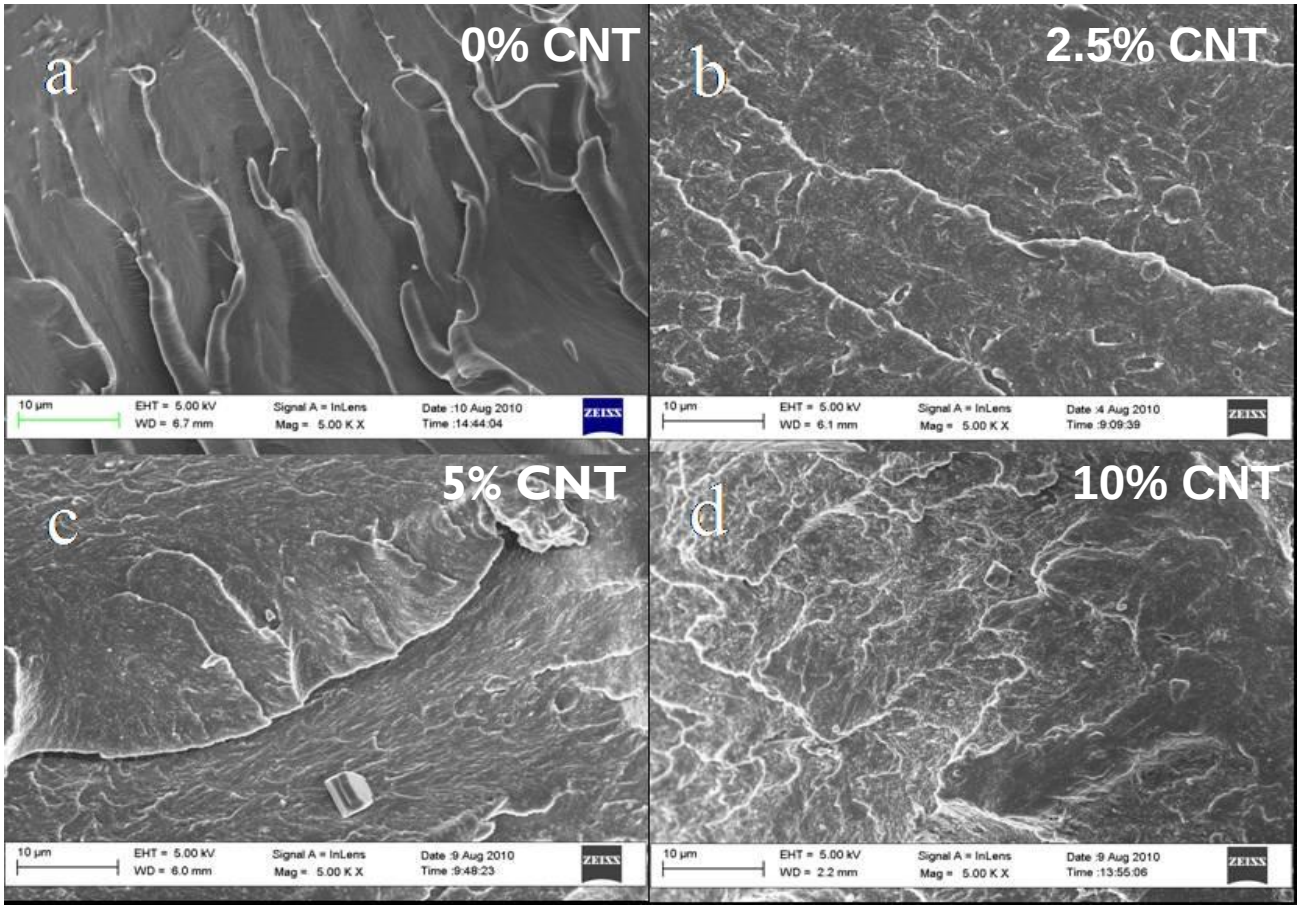
Nanocomposite mechanical properties





- Polymer matrix is stiffened
- Increasing strength indicative for good CNT/matrix interface

Nanocomposite fractography

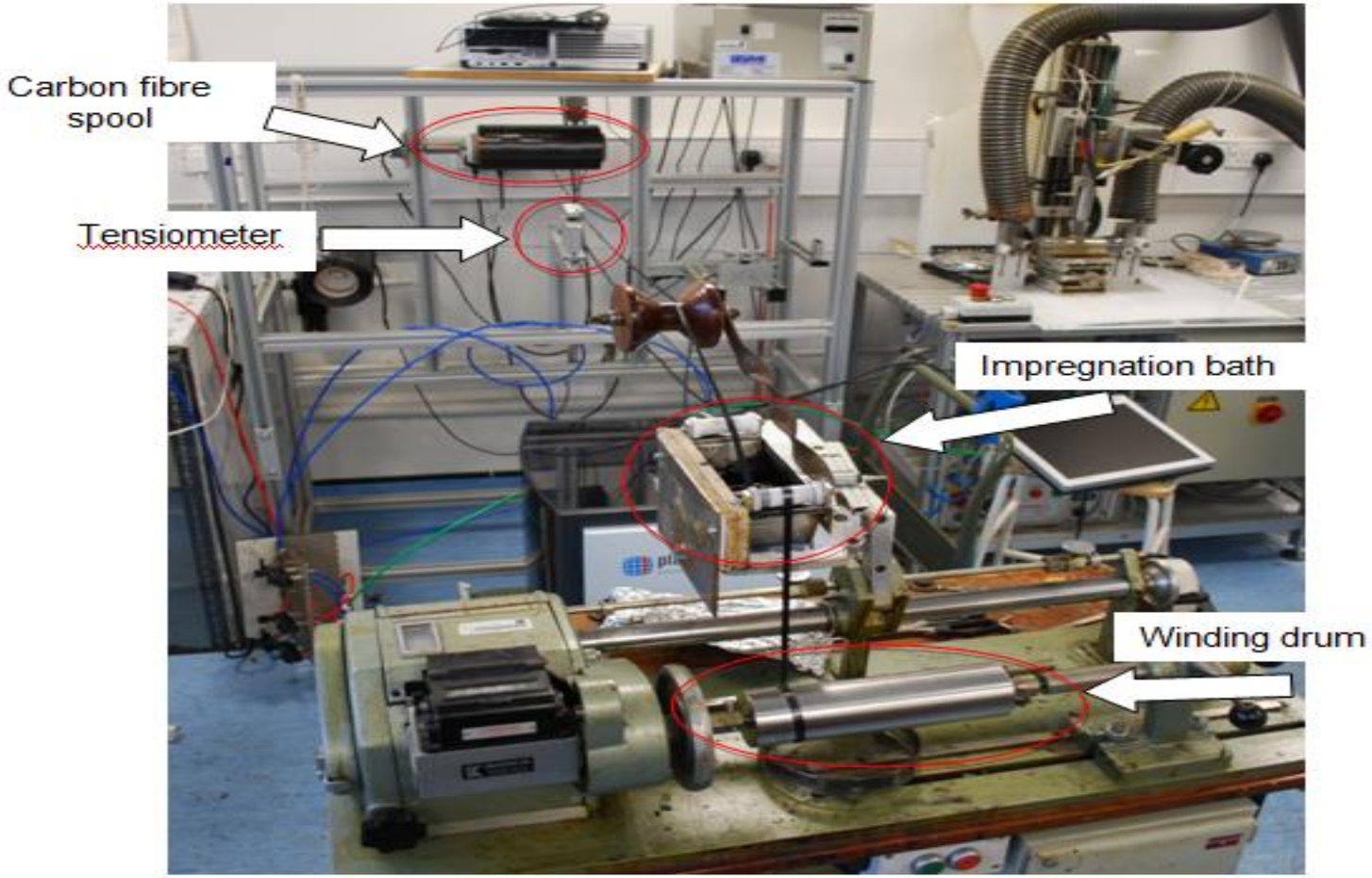


- Rougher surfaces with increasing CNT loading indicate toughening

Hierarchical composite production

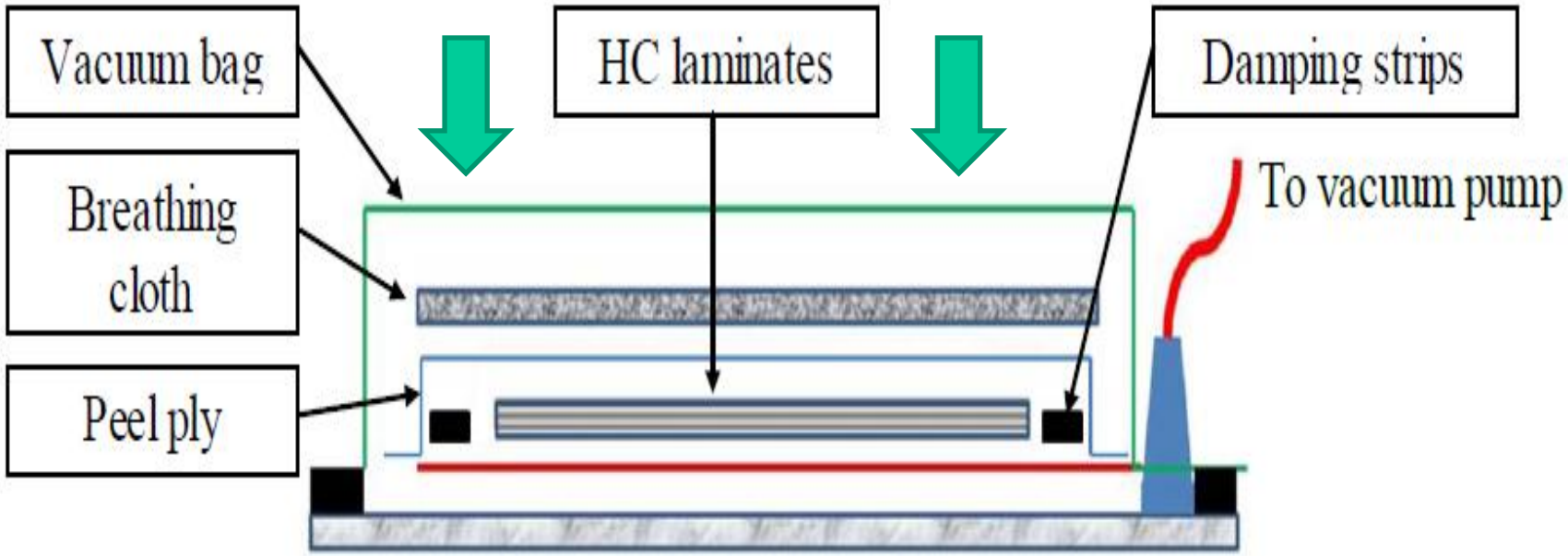
- Wet impregnation avoids issues with traditional CFRP processing
- Cryo-milling produces the precursor for powder pregging
- Demonstration of ply production

Production of thermosetting HCs



Filament winding process for HCs

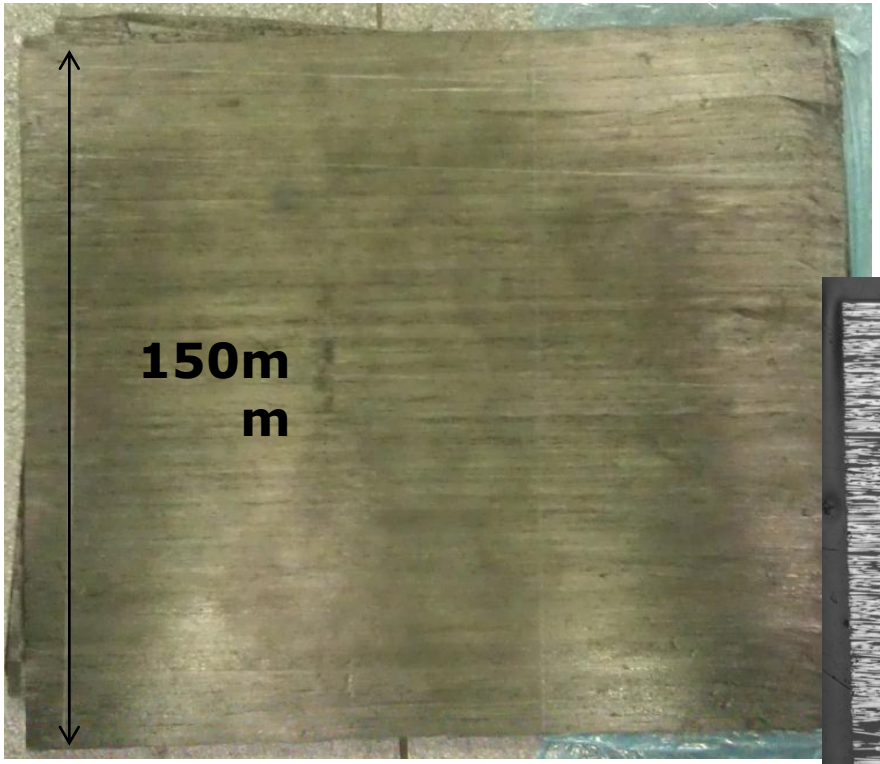
Production of thermosetting HCs

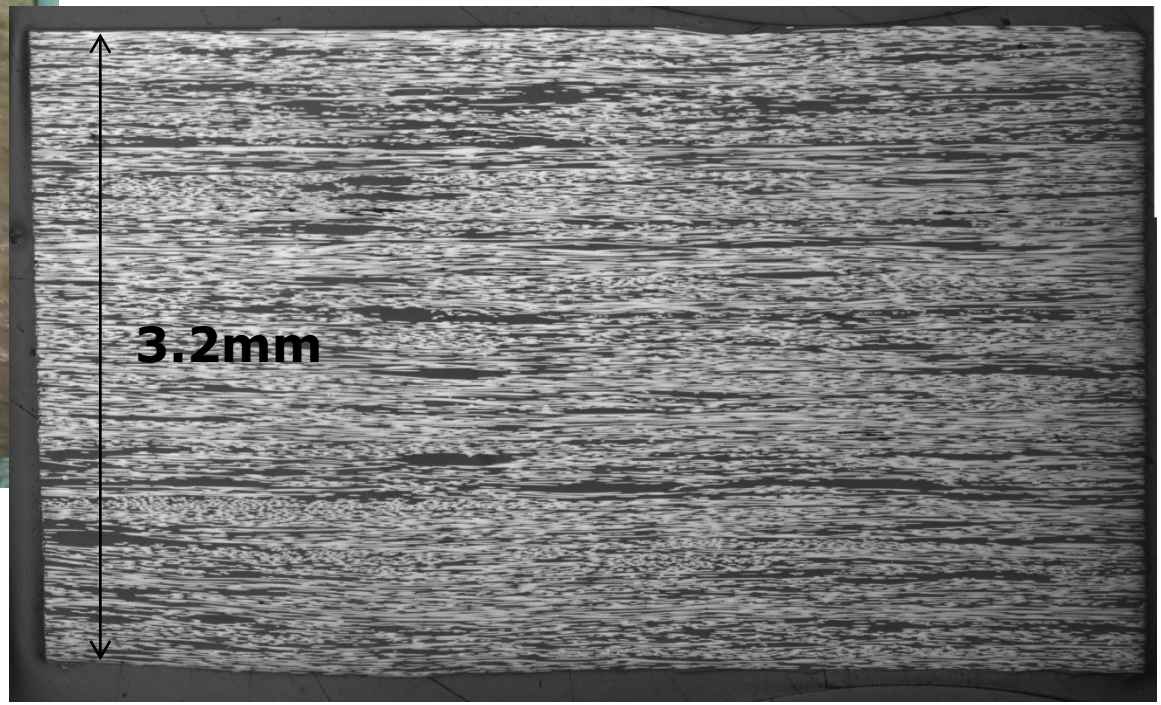


Vacuum assisted hot pressing

Hierarchical composites

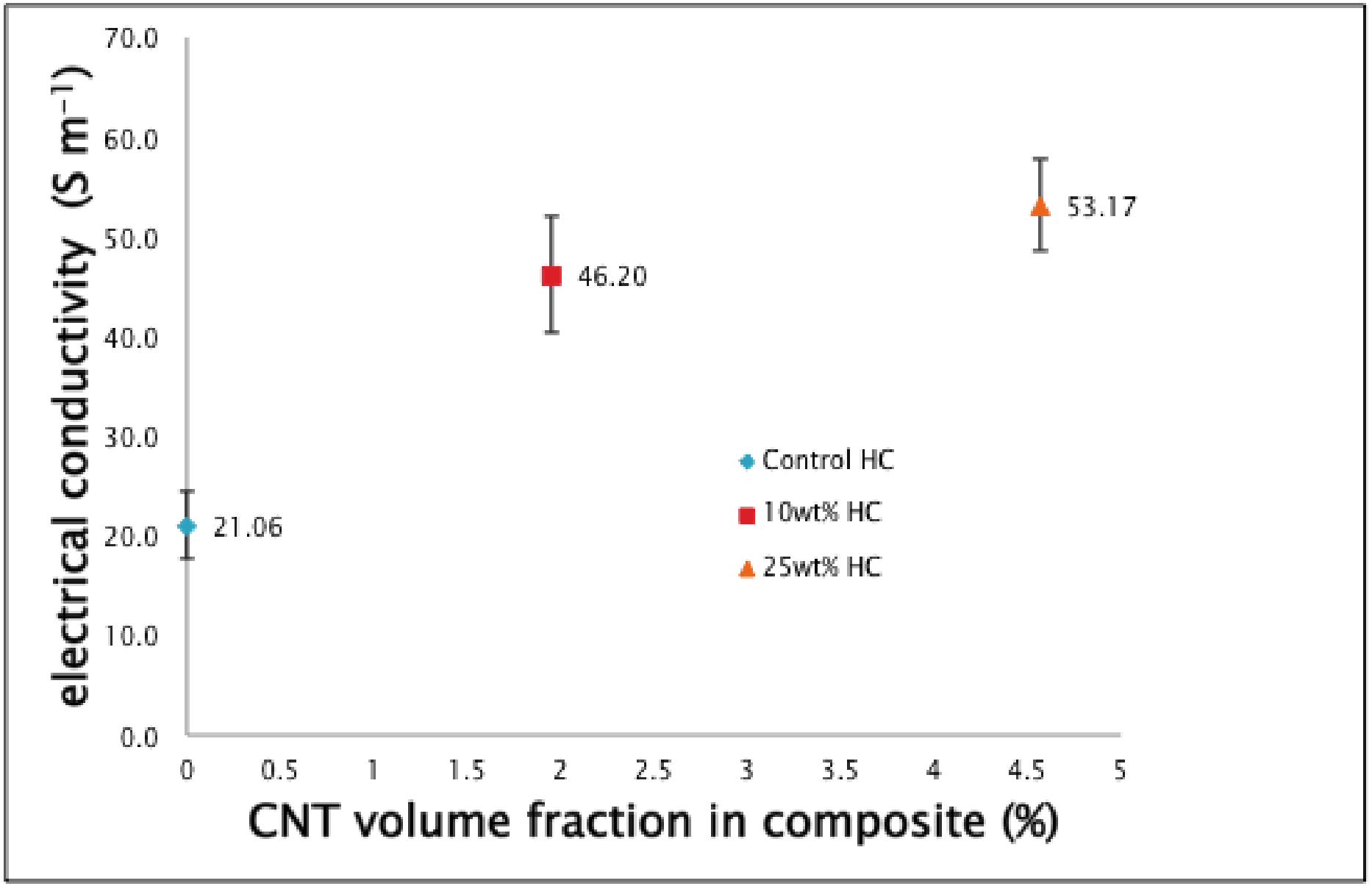
3-4 mm laminates with low voidage (< 2%)



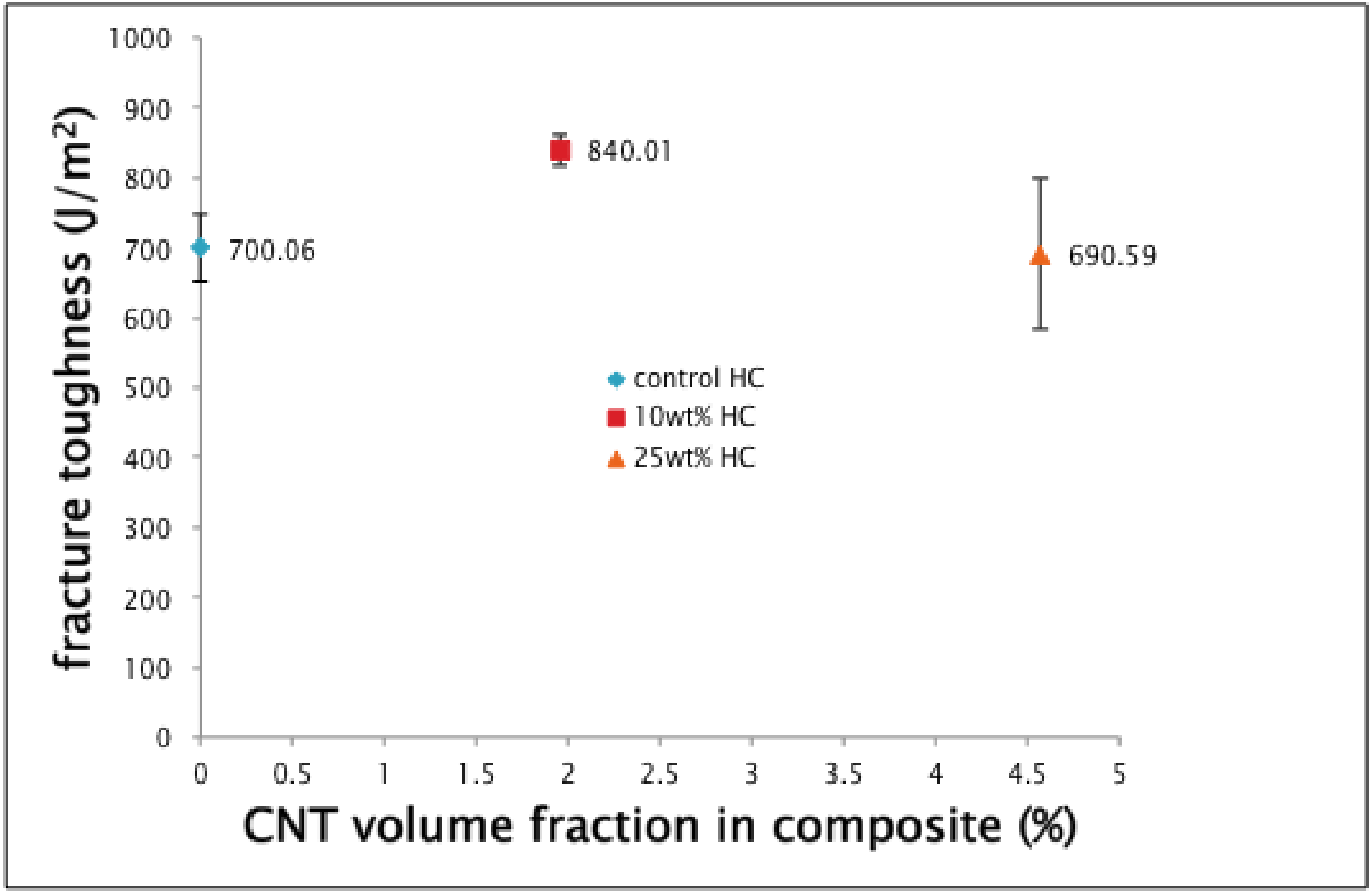


But issues: fibre waviness & straightness

Properties of Hierarchical composites

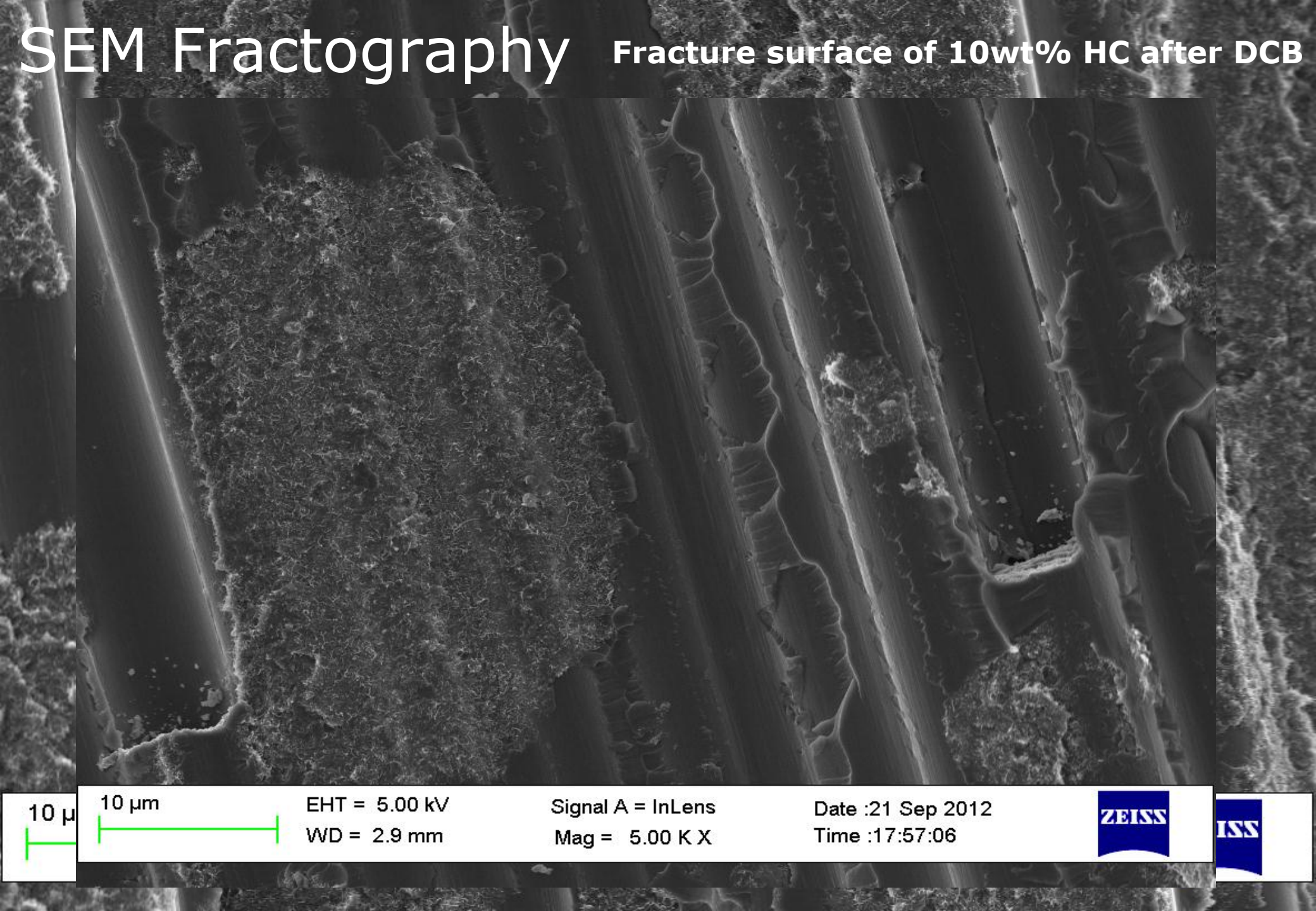


Properties of Hierarchical composites

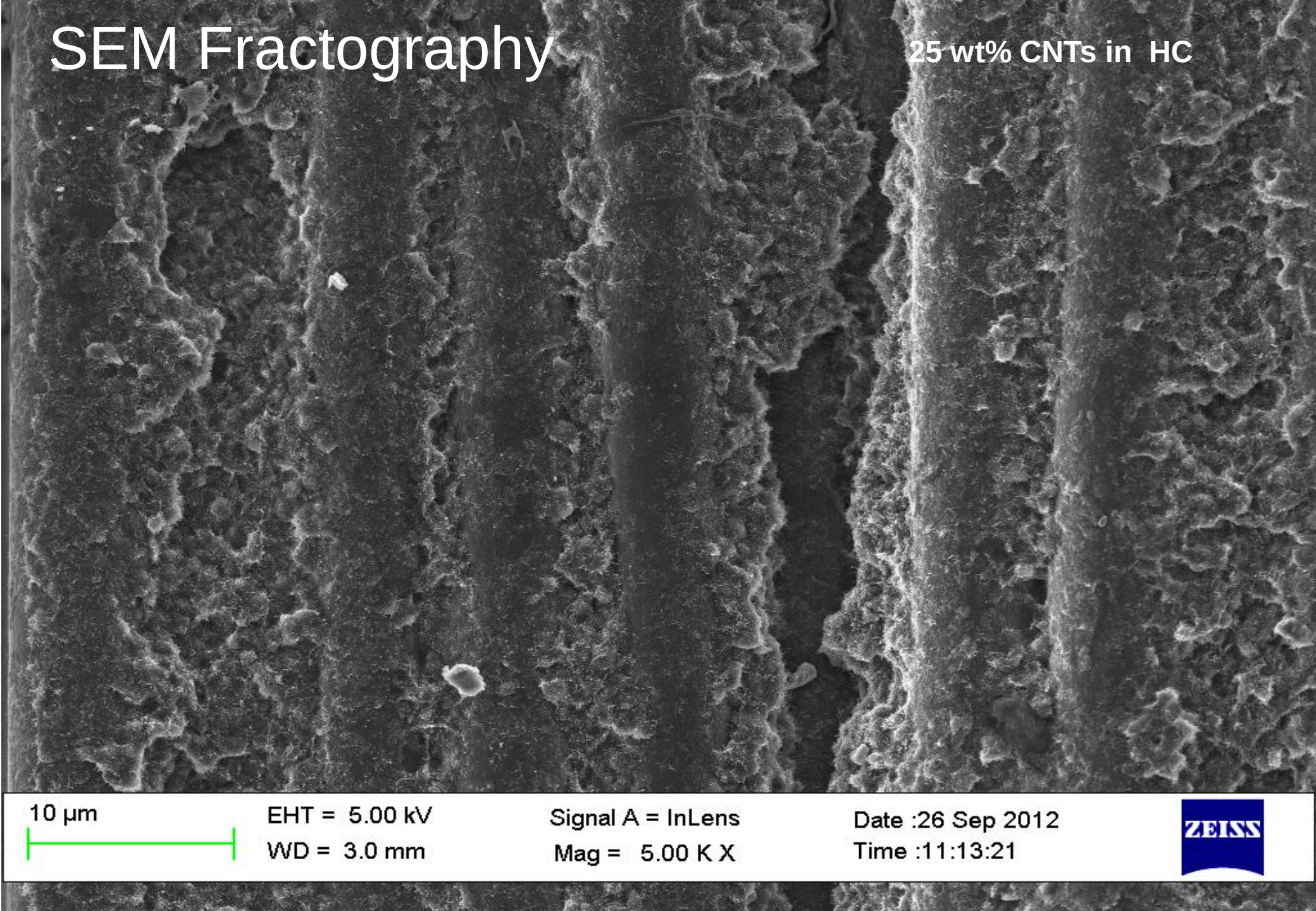


SEM Fractography

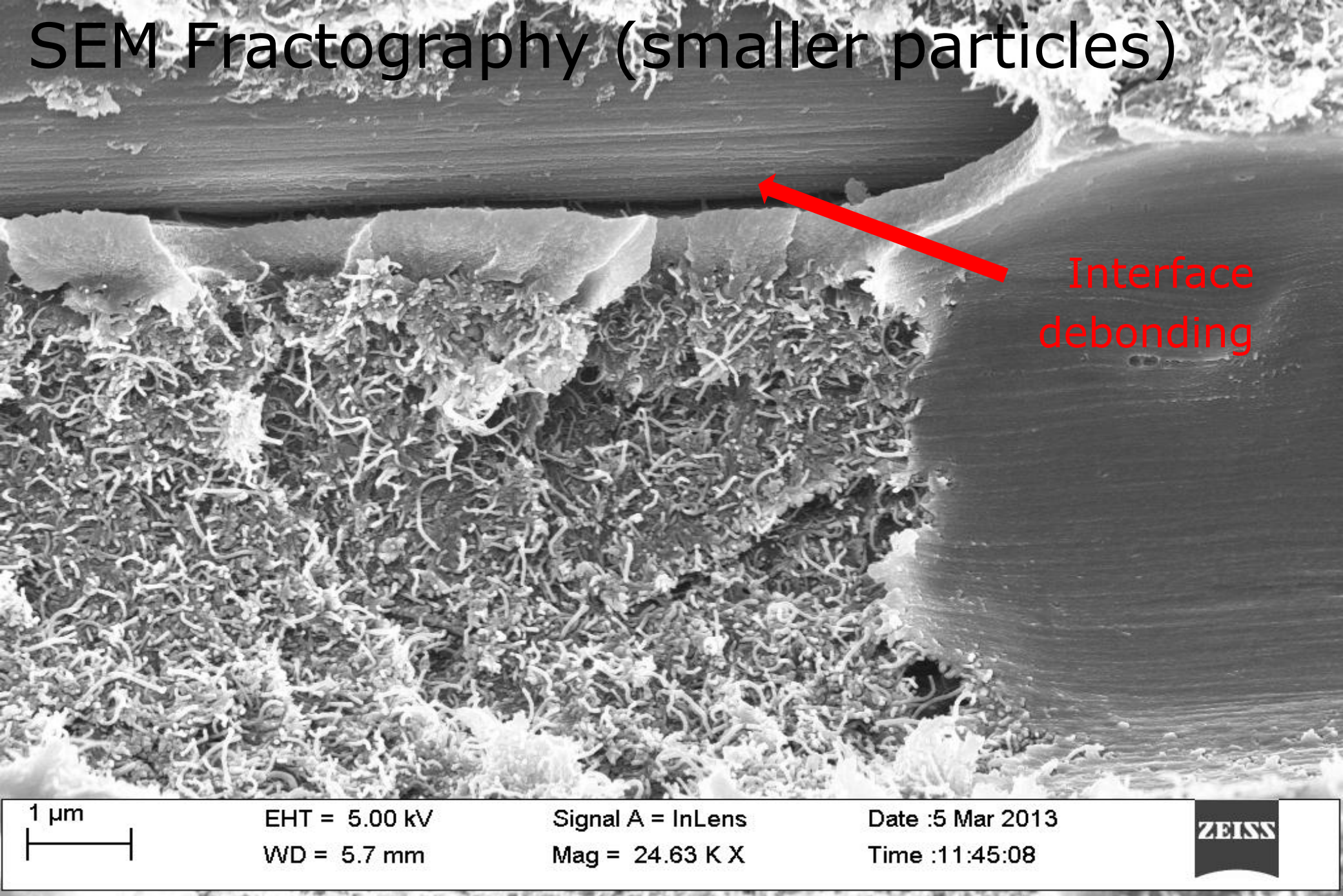
Fracture surface of 10wt% HC after DCB



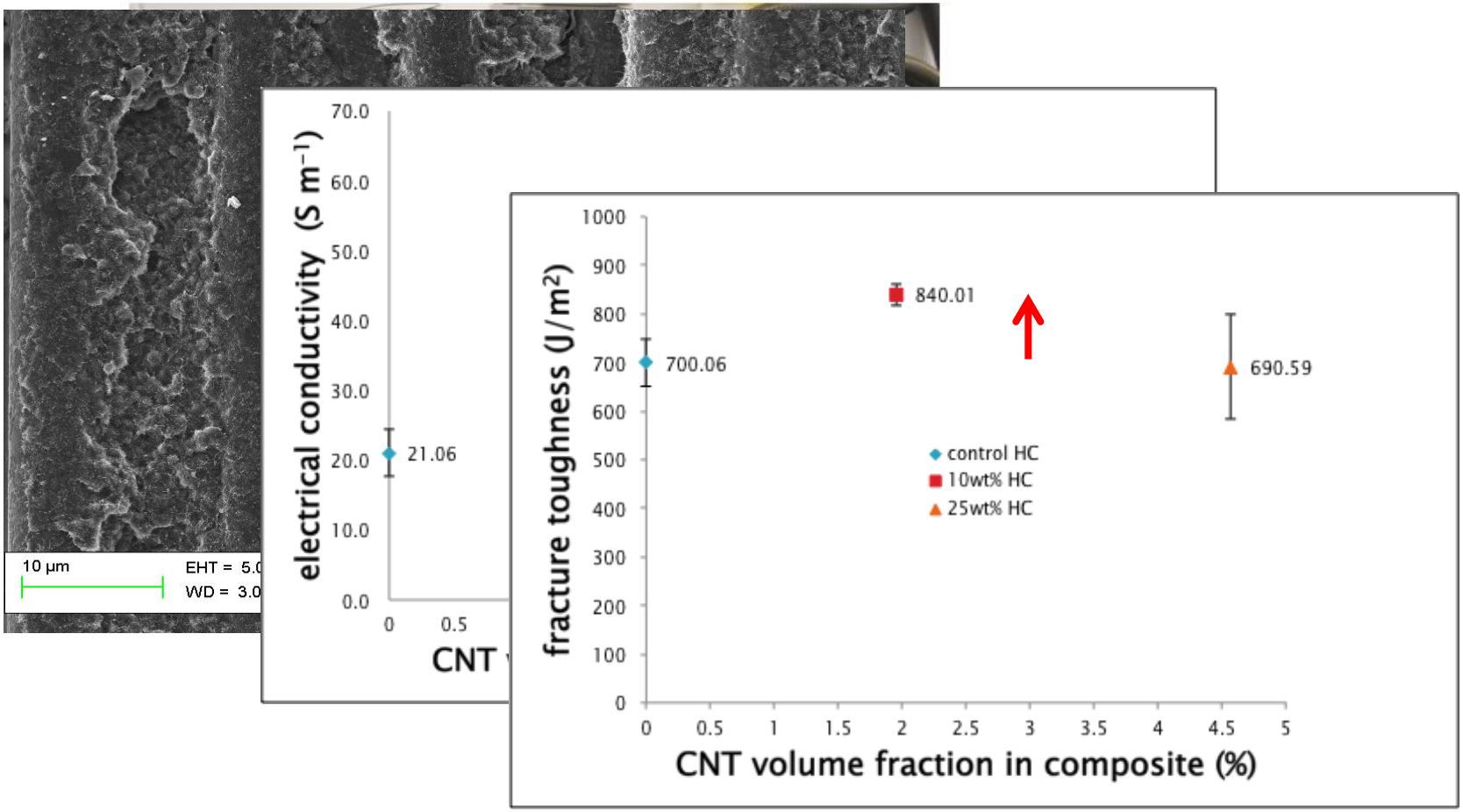
SEM Fractography



SEM Fractography (smaller particles)



Conclusion


10 µm
EHT = 5.0
WD = 3.0
electrical conductivity (S m⁻¹)
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0.0
0
0.5
21.06
CNT
fracture toughness (J/m²)
1000
900
800
700
600
500
400
300
200
100
0
0
0.5
1
1.5
2
2.5
3
3.5
4
4.5
5
700.06
840.01
690.59
control HC
10wt% HC
25wt% HC
CNT volume fraction in composite (%)

Thank You!

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