

RAMAN SPECTROSCOPY OF MACROSCALE FIBERS COMPRISED OF CARBON NANOTUBES OF DIFFERENT LENGTHS UNDER UNIAXIAL STRAIN

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Carbon nanotube (CNT) fibers are considered an excellent material for high performance engineering applications, such as electrical or aerospace devices, due to their low weight, high modulus and high tensile strength. The modulus and tensile strength of CNT fibers are thought to depend on the structure of the fibers, because the stress of the fibers must be distributed effectively and uniformly to the CNTs. The goal of our research is to quantitatively assess which CNT sources are suitable for making CNT fibers. We used a wet-spinning technique to produce fibers from different CNT sources and measured them by *in situ* Raman spectroscopy while changing the values of uniaxial strain. We investigated the stress distribution of CNT fibers by analyzing the total strain, the G' peak downshift, and the G' band broadening of each fiber's Raman spectra. Furthermore, to determine the magnitude of uniaxial strain on CNTs in the fiber, a strain transfer factor (STF) was used, which was calculated from the G' peak shift in their Raman spectra. We compared the CNT fibers derived from CNT sources of different lengths and alignments in terms of G' shift, G' band broadening and STF. Our findings will provide insight into the relationship between the structure and the mechanical properties of CNT fibers.

Raman Spectroscopy of Macroscopic Fibers Spun from Carbon Nanotubes under Tensile Strain

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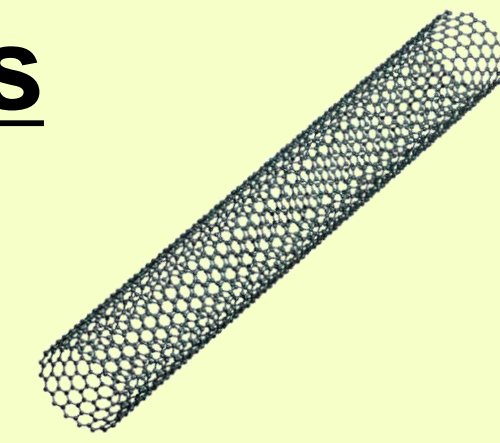
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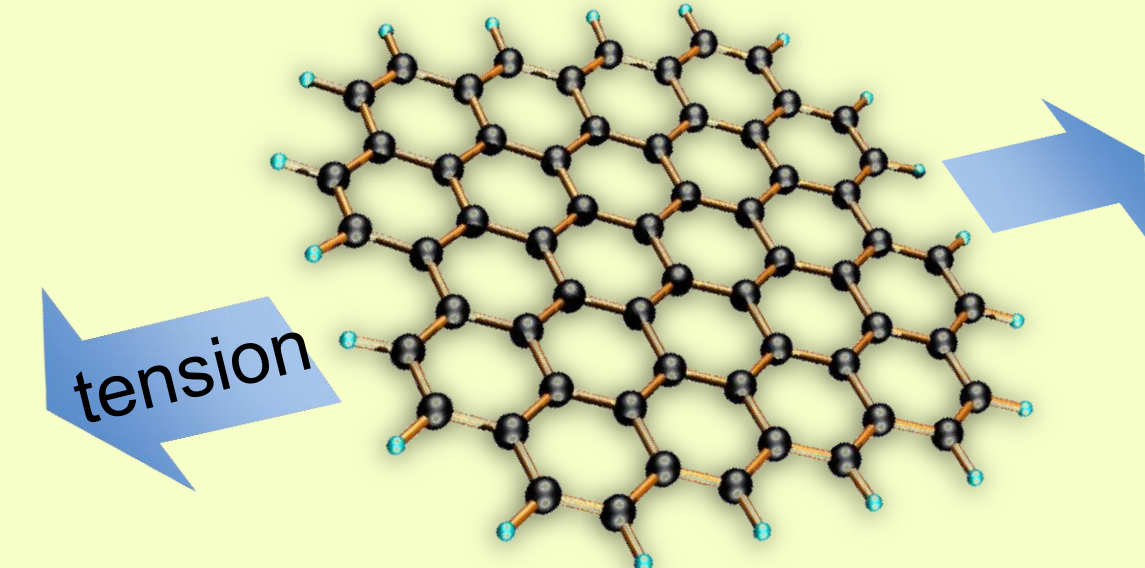
Introduction

Carbon Nanotube (CNT) Fibers

- Exceptional mechanical strength
- Tensile strength > 37 GPa
- Young modulus ~ 640 GPa



Raman Spectrum vs. Mechanical Property



- Increase of tension
- Elongation of the C-C bonds
 - Lower vibration frequency
 - Downshift of G' peak

In a CNT fiber G' band characteristic shows:

- how effectively tensile stress transferred to each CNT (extent of G' peak shift)
- how uniformly tensile stress distributed to each CNT (broadening of G' peak)

STF (Strain Transfer factor)^[1]

Quantitative Index of efficiency of stress distribution to CNTs:

$$\alpha = \frac{\Delta G'_{\text{fiber}}}{\Delta G'_{\text{CNT}}} \quad \Delta G'_{\text{fiber}} : \text{Downshift rate of a strained fiber} \\ \Delta G'_{\text{CNT}} : \text{Downshift rate of a strained CNT (37 cm}^{-1}\text{/}\%)^{[2]}$$

Results reported in past for CNT fibers^[1]

Fabrication Method: Twisted from a solid CNT film

CNTs: CVD growth

STF: $\alpha = 0.045$

G' peak FWHM broadening of 1.25 cm⁻¹ per 1% strain, indicating that not all CNTs are under strain due to the loose mesh structure.

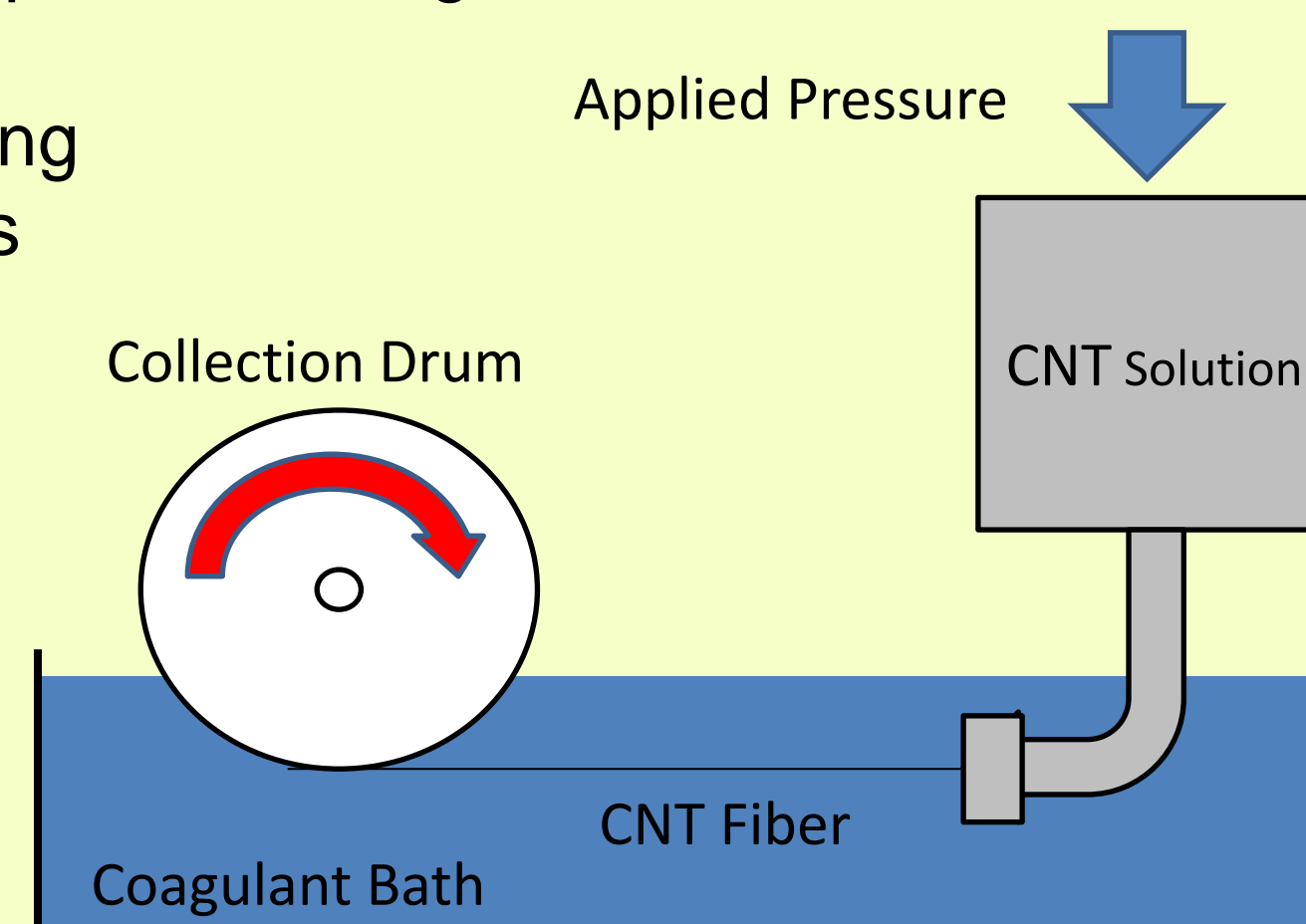
Wet Spinning Technique^[3]

We used a solution-based technique for making CNT fibers in order to achieve:

- Scalable/Continuous producing
- High packing density of fibers
- Better alignment of CNTs

We used 2 types of CNT sources:

- HiPco SWNT
- Longer single-/double-walled CNTs (SWNTs/DWNTs)



Motivation for our Raman measurements

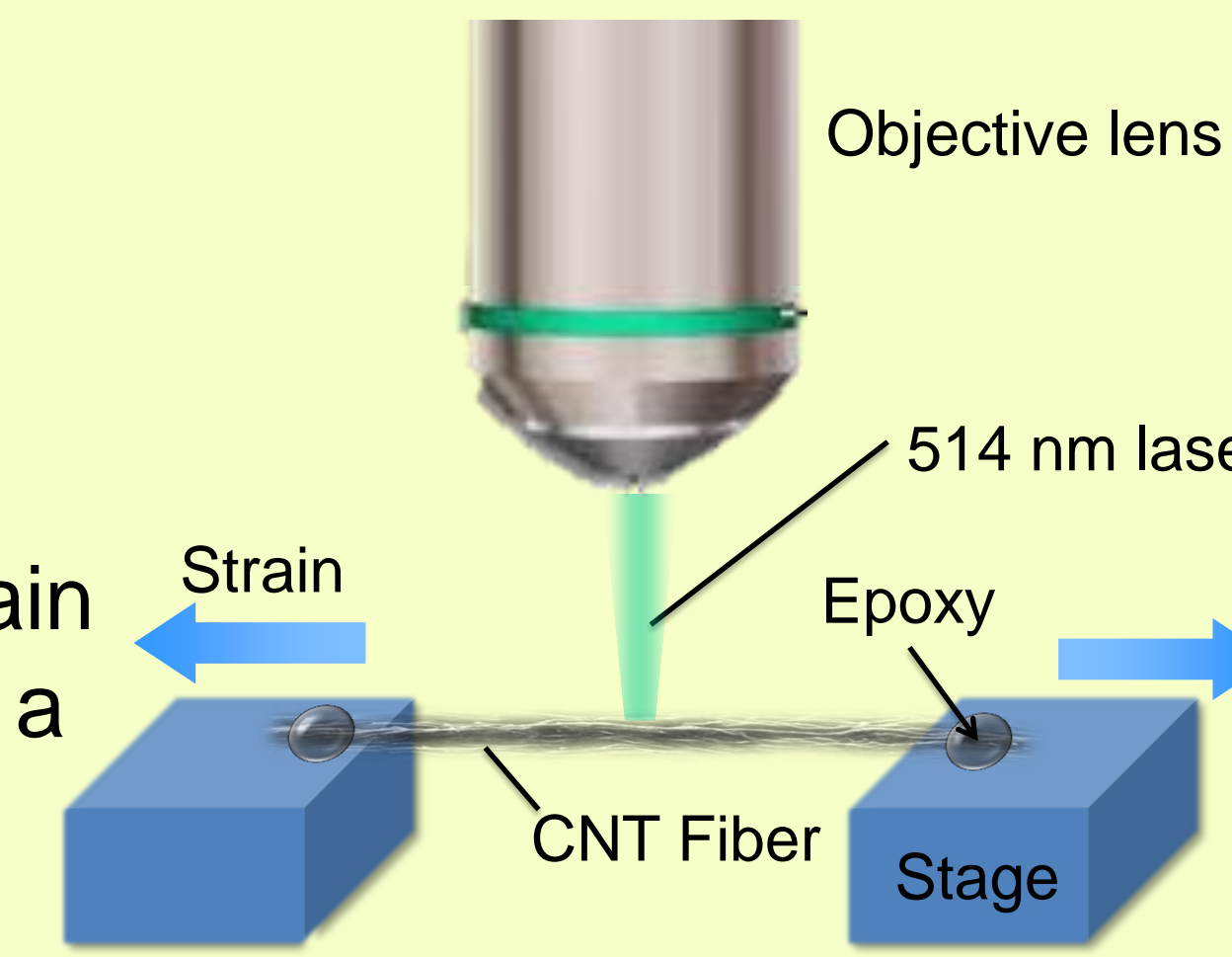
- CNT packing/length should affect strain transfer
- Compare fibers from long vs. short CNTs
- Compare STF of wet spun vs. twisted fiber

Raman Spectroscopy and Analysis

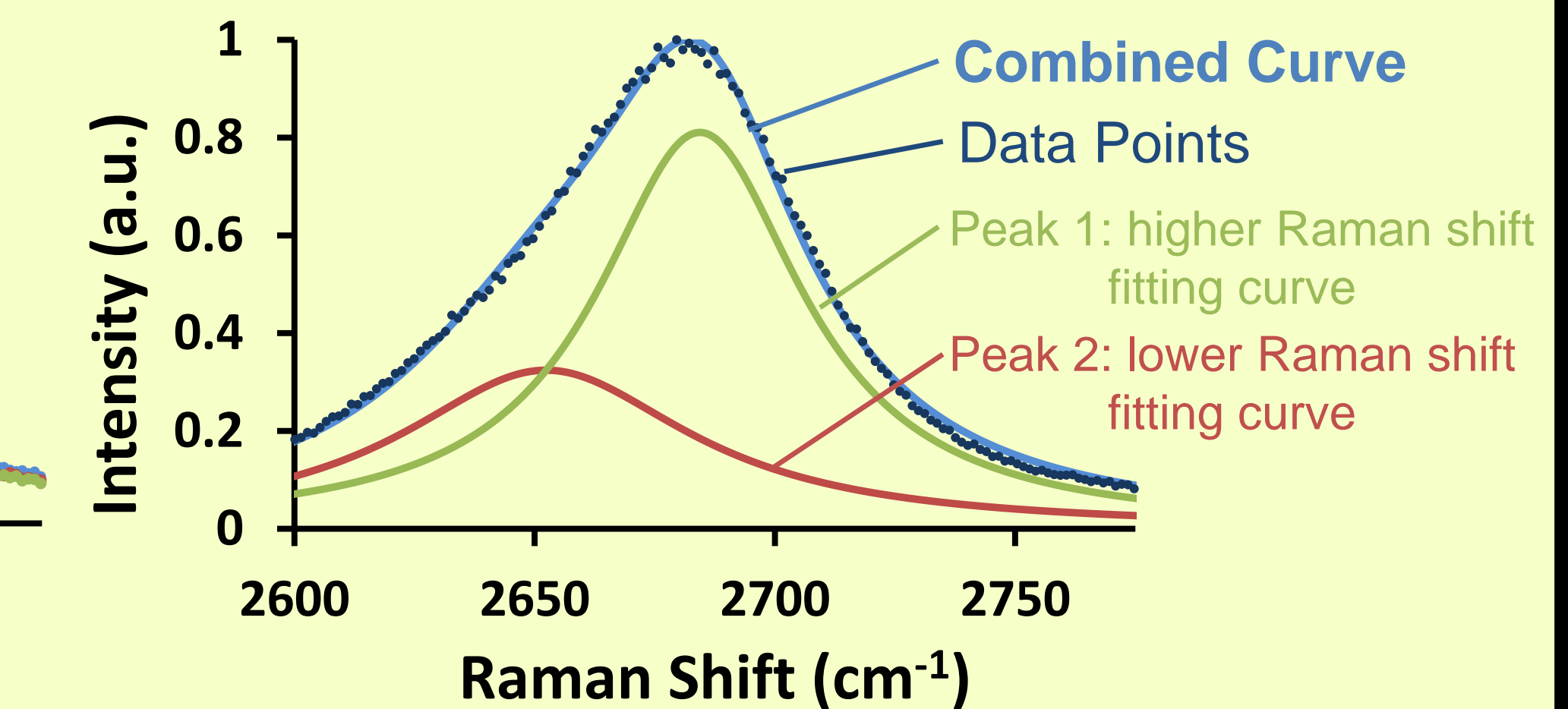
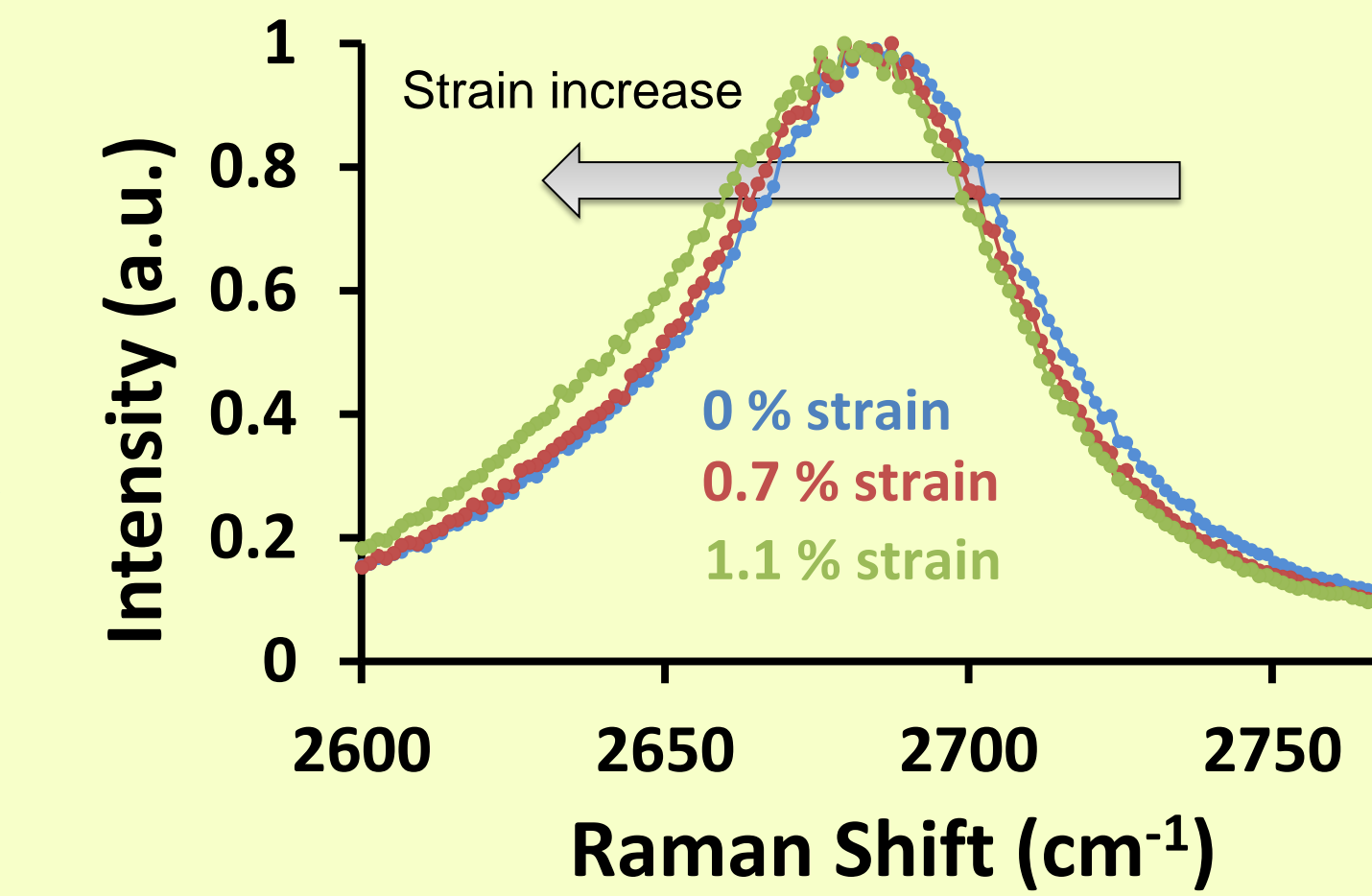
Measuring

We measured CNT fibers using Raman spectroscopy while changing tensile strain.

- 3 positions X 3 times each strain
- Fiber ends fixed with epoxy to a stage and separated by hand with a micrometer screw



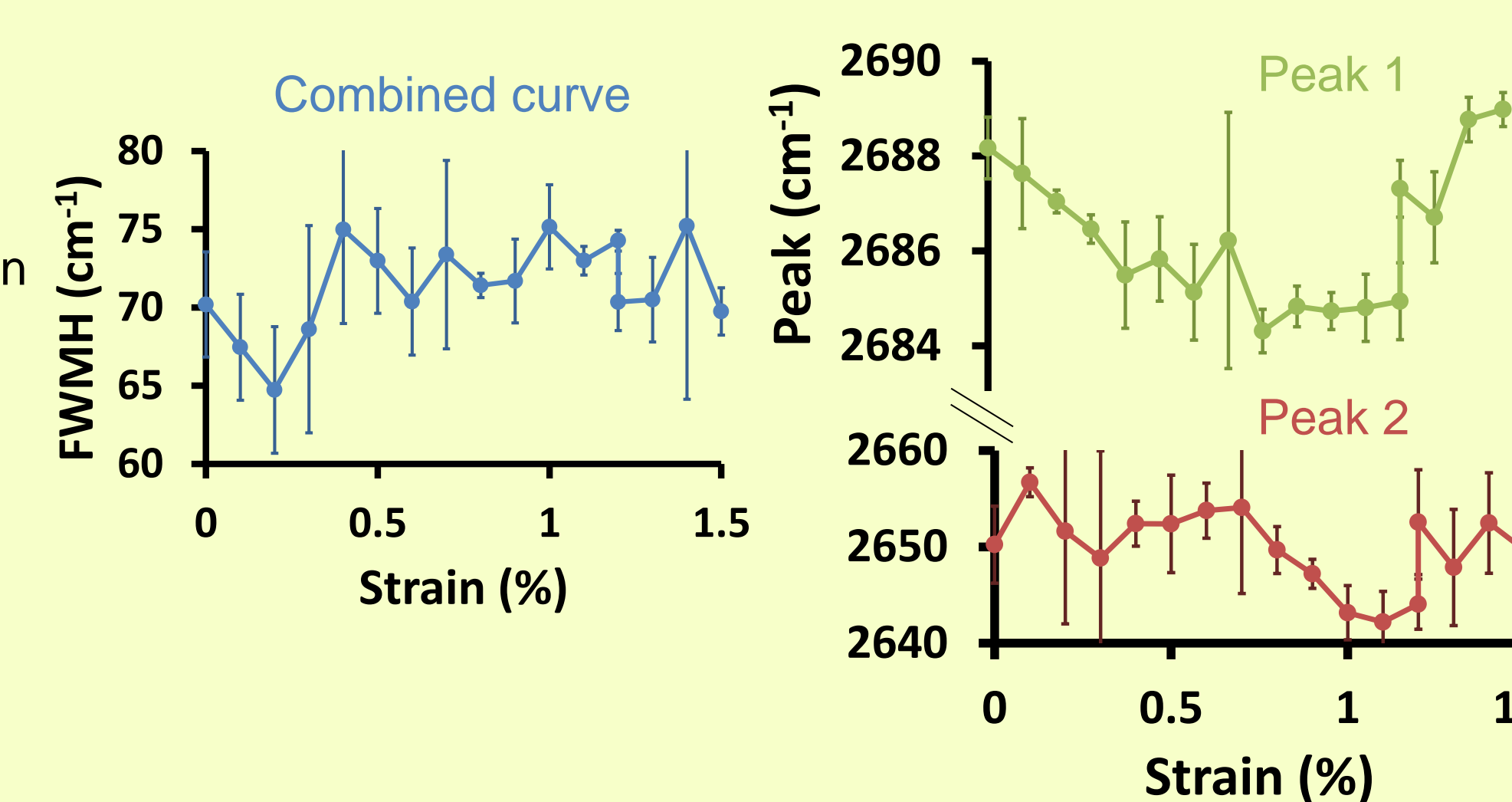
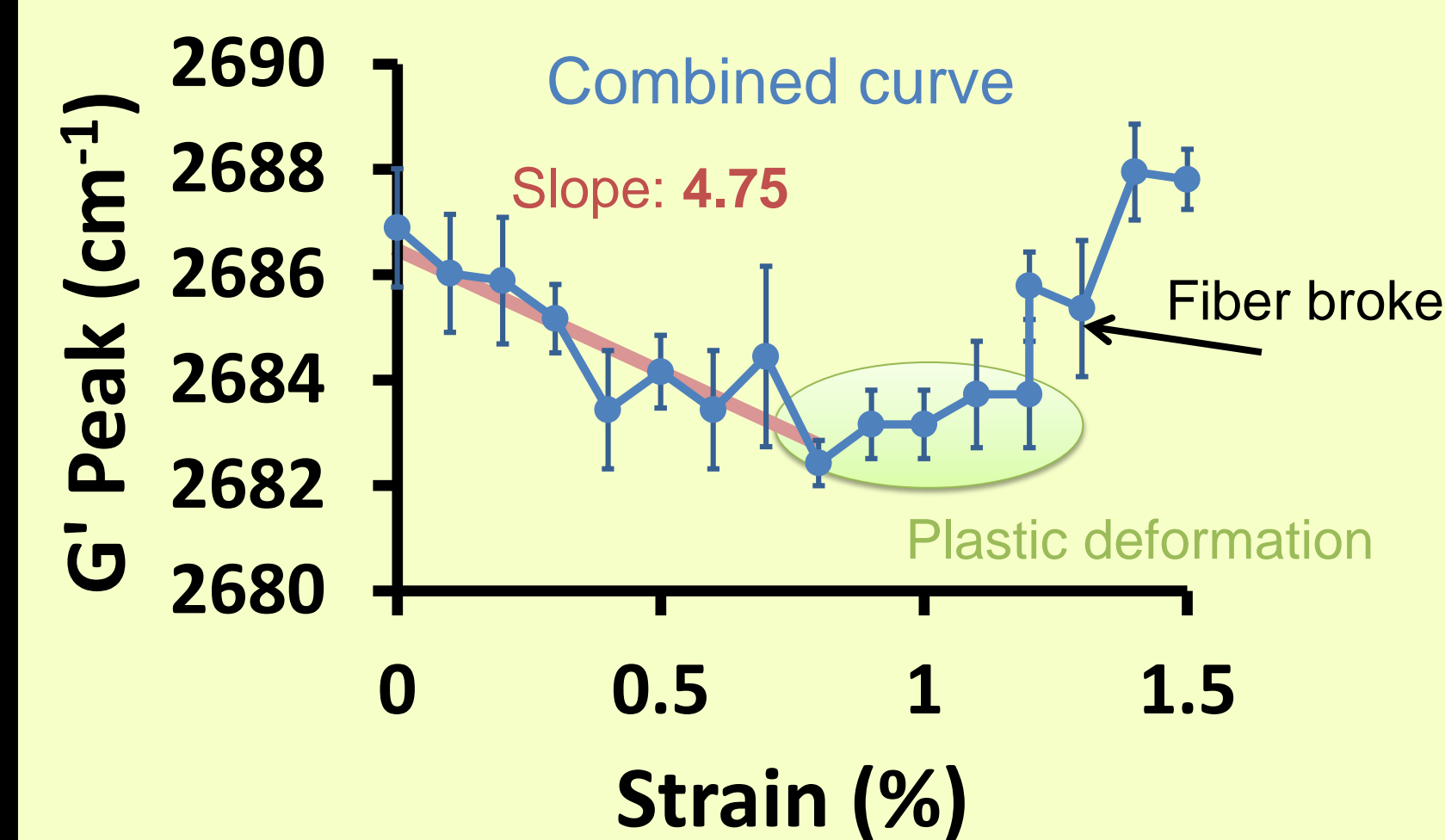
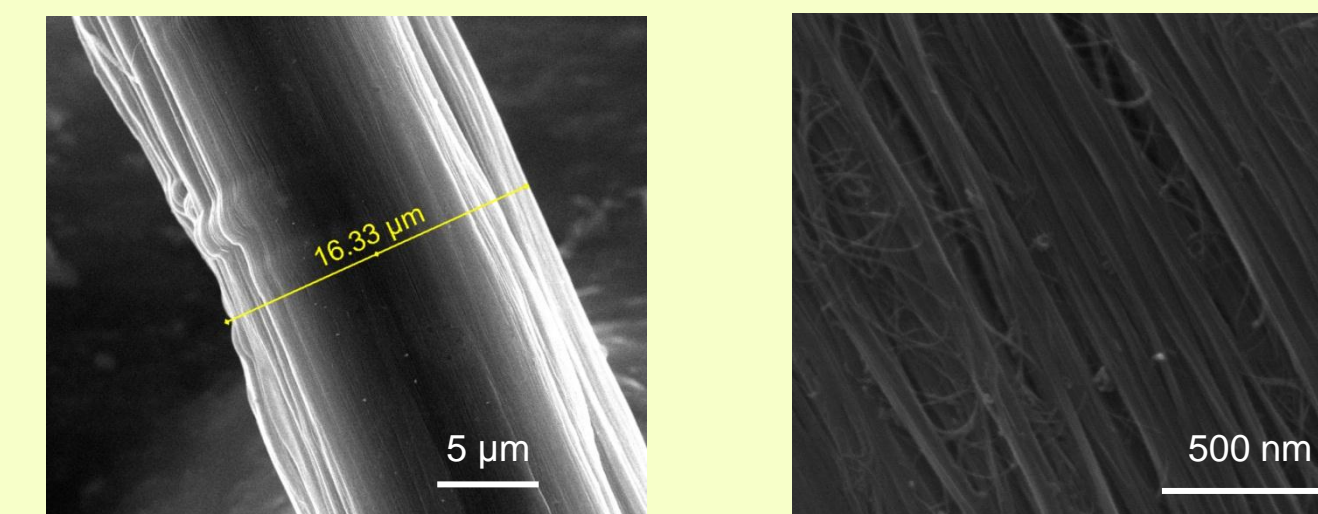
Analysis



Results of Fibers from 2 types of CNTs

Fibers spun from long SWNTs/DWNTs

- Long SWNTs/DWNTs: Average length 2 - 7 μm
- Spun from chlorosulfonic acid solution
- Collected under tension to promote alignment



$$\alpha = 4.75/37 \approx 0.13$$

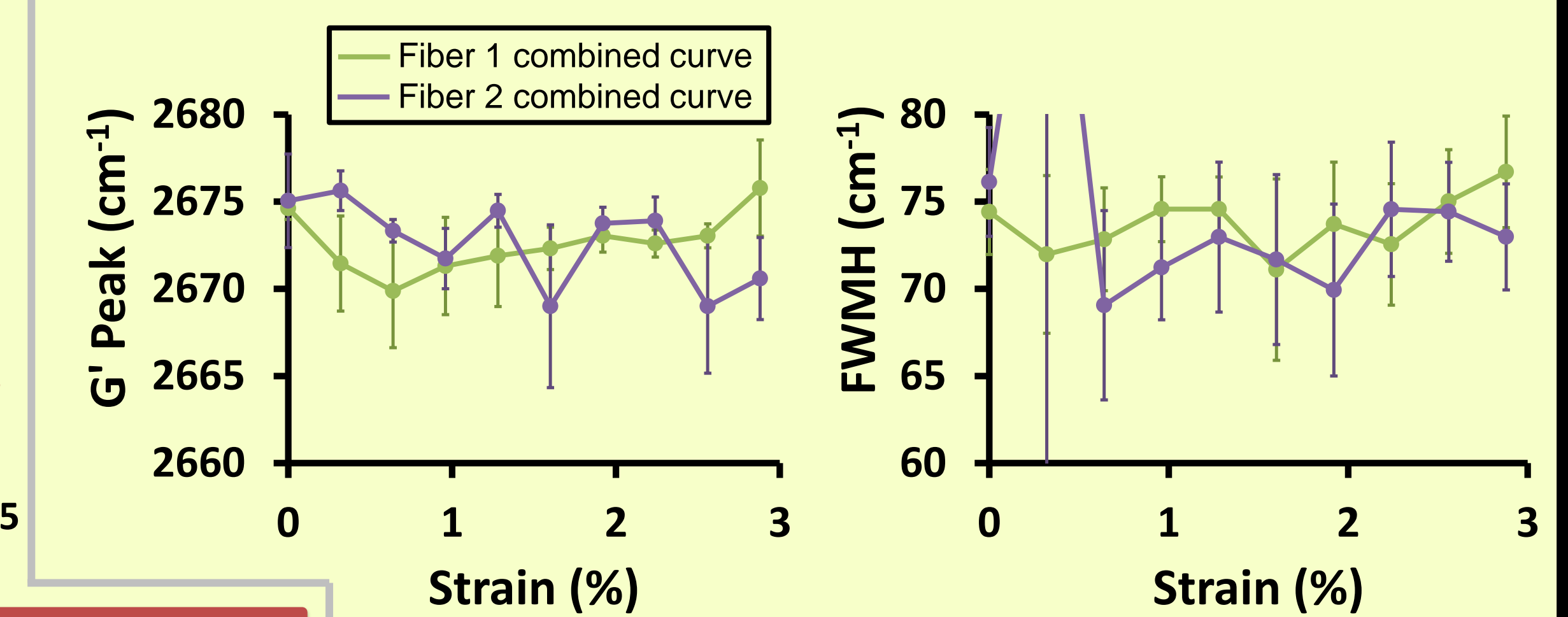
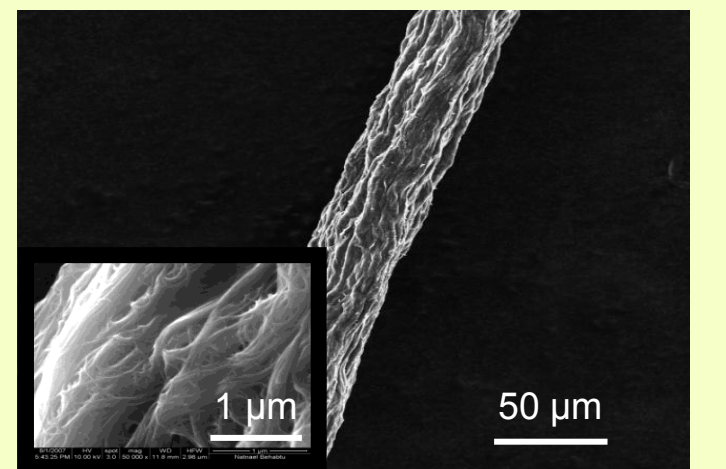
- STF of 6 fibers

Fiber	Slope	STF
# 1	5.86	0.16
# 2	28.8	0.78
# 3	4.22	0.11
# 4	4.75	0.13
# 5	2.47	0.067
# 6	4.58	0.12

- **STF: 0.13 (long SWNTs/DWNTs, wet-spun) > 0.045 (CVD, twisted-spun);** this is due to higher packing density and uniformity of CNTs in fiber.
- CNT fibers strain is followed by slipping/plastic deformation, then breaking which releases strain.
- Lorentzian Peak 1 is downshifted and Lorentzian Peak 2 has no clear trend.
- No clear broadening in the FWHM (given large error bars) – may indicate all CNTs equally strained.

Fibers spun from HiPco

- HiPco: Average length ~ 1 μm
- Spun from sulfuric acid solution
- Collected under no tension, which means less alignment of CNTs



- No clear trend in either peak position or FWHM of fibers spun from HiPco.
- Shorter CNTs and less alignment leads to much lower STF compared to other wet-spun fibers.

Conclusions

- Fibers spun from longer/more aligned CNTs vs. shorter and less aligned CNTs (HiPco) have a higher STF.
- STF is higher, and tensile stress is shared more uniformly in long SWNTs/DWNTs wet spun fiber than in fiber twisted from a solid CNT film; this reflects the improved alignment, packing, and uniformity achieved in the wet-spinning fiber process.
- There is a possibility that only one peak of the double Lorentzian fit is shifted; this might imply only a certain type of vibration is affected.

Acknowledgments

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