

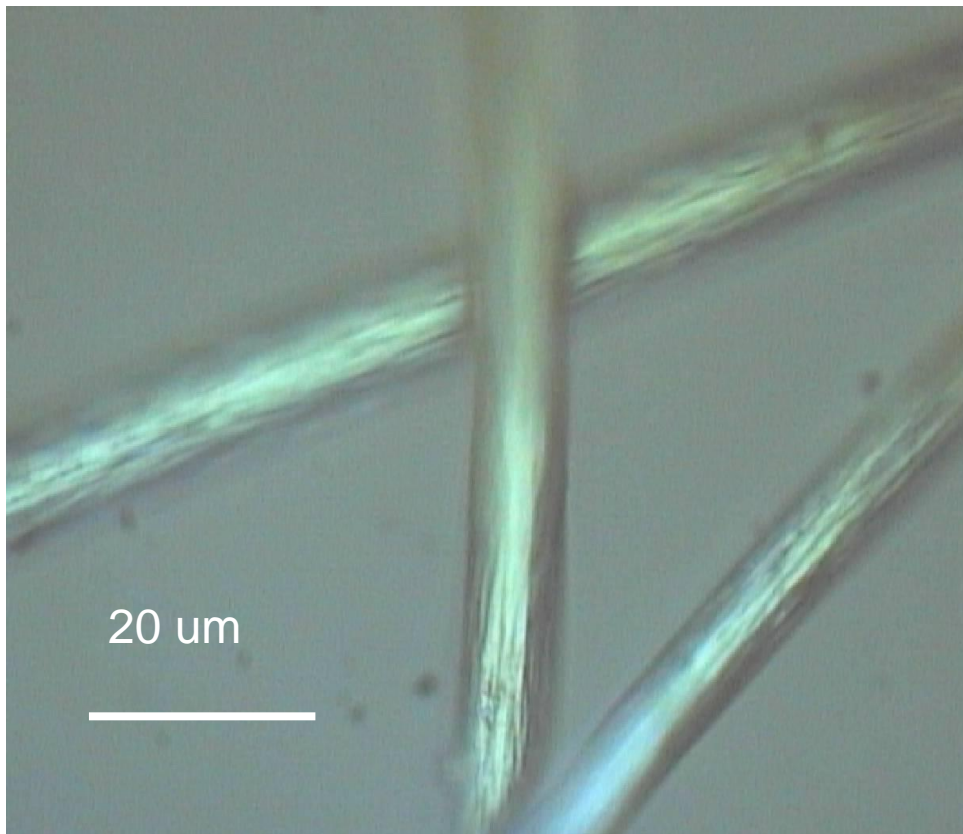
# Confocal Raman microscopy for noncontact and nondestructive characterization of carbon fibers

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## Carbon fibers



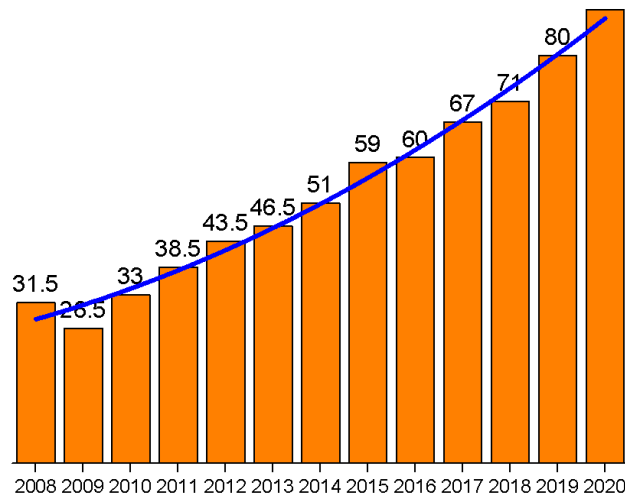
Carbon (graphite) fiber is a new material consisting of fibers about 5–10  $\mu\text{m}$  in diameter and composed mostly of carbon atoms.

## Production of carbon fibers

Carbon fibers are mainly made from polyacrylonitrile (PAN). Converting PAN to carbon fibers is a multi-step process including graphitization.

The higher the level of graphitization, the higher the amount of ordered carbon, allowing for higher grade fiber to be produced.

At the present time the role of carbon fibers  
is increasing in the world



Global demand for carbon fibers in 2008–2020 (in thousand tonnes).  
[<http://www.statista.com/statistics/282243/global-demand-for-carbon-fibre/>]

## Global demand for carbon fibers increases due to

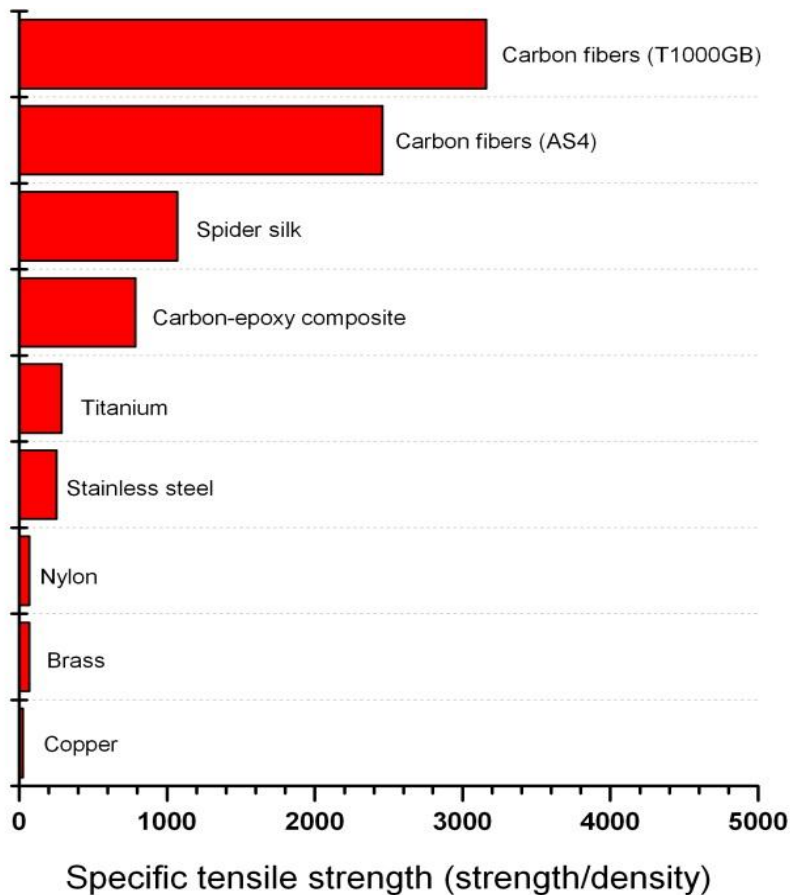
- Their high strength and stiffness potential
- Low weight
- High chemical resistance
- High temperature stability
- Low thermal expansion

## Specific strength (strength-to-weight ratio)

	Tensile strength (MPa)	Density (g/cm <sup>3</sup> )	Specific strength (kN·m/kg)
Carbon fibre (AS4)	4300	1.75	2457
Stainless steel (304)	505	8.00	63.1

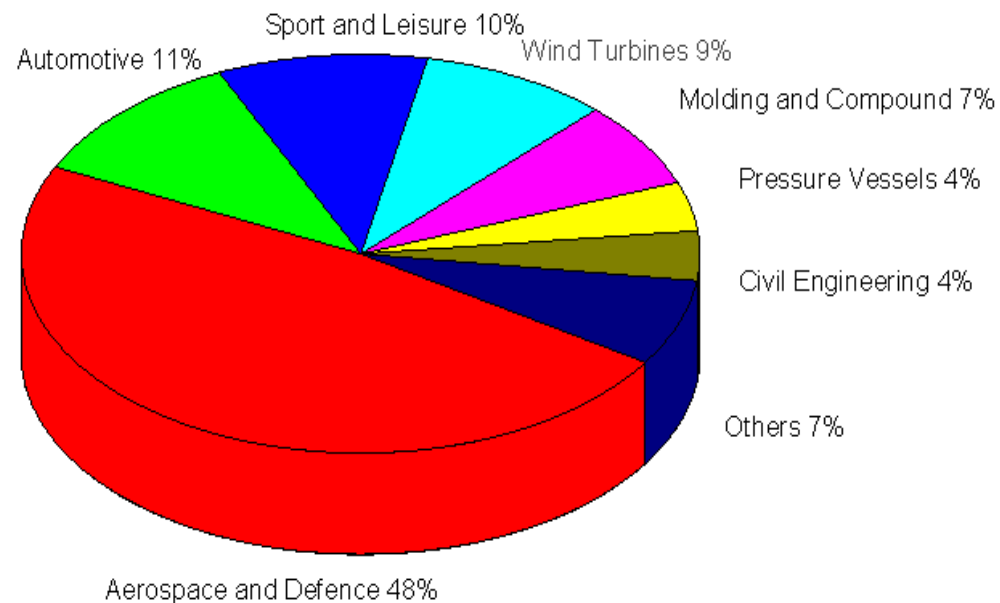
As can be seen, carbon fibre has a tensile strength almost 8.5 times greater than that of steel, yet is 4.5 times less dense.

## Specific tensile strength of various materials



Comparison of carbon fiber properties with other engineering materials.

## Global carbon fiber demand by application in 2014



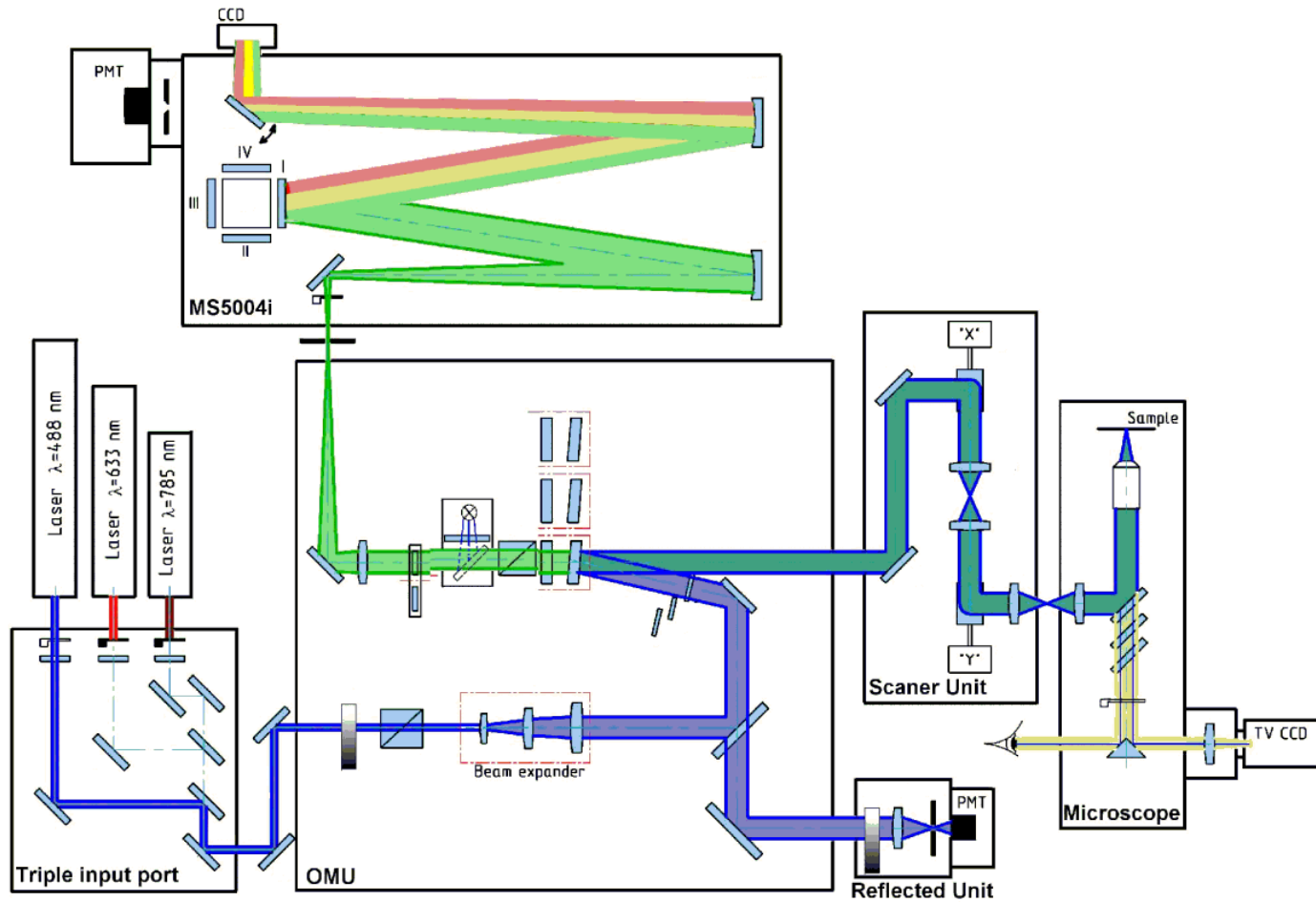


## Instrumentation 3D Raman Confocal Microscope Confotec®

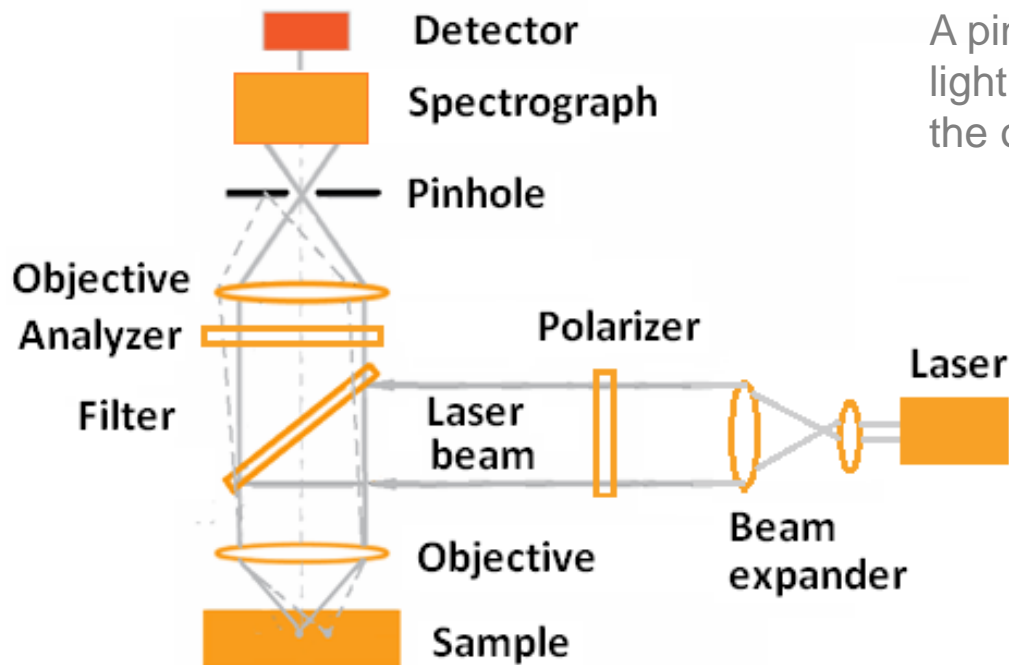


Confocal Raman spectroscopy is a well-established non-destructive and a very powerful analysis technique. It is rapid and high sensitive analysis method.

## Confotec® Raman spectroscopy set-up

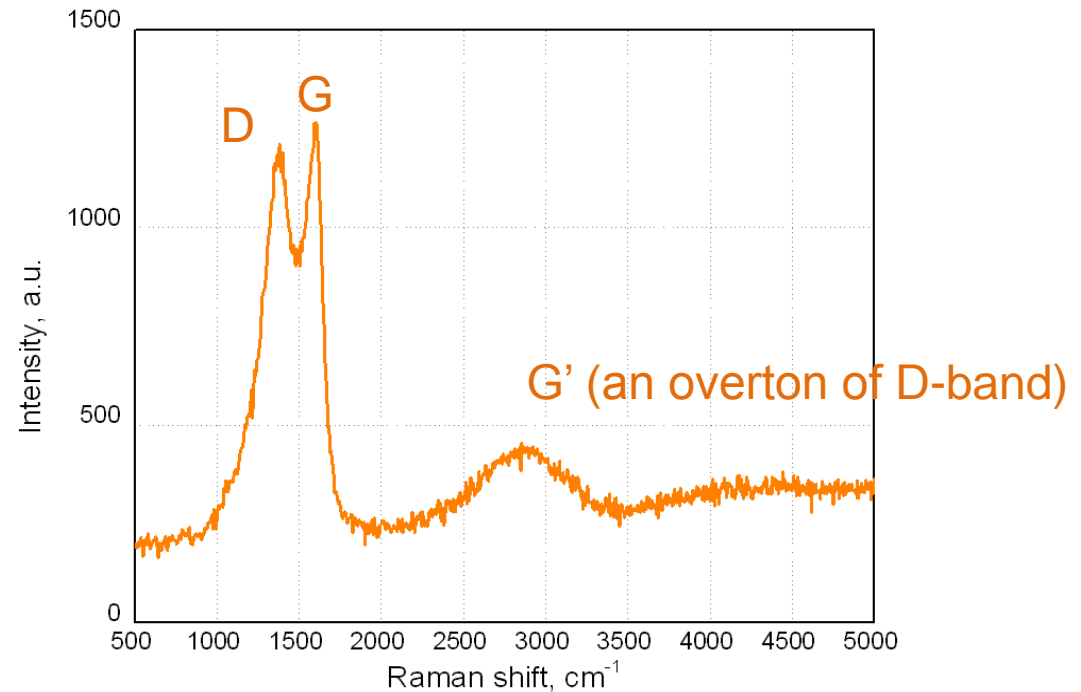


## Confocal Raman Detection



A pinhole blocks the scattered light which is coming from the out-of-focus points.

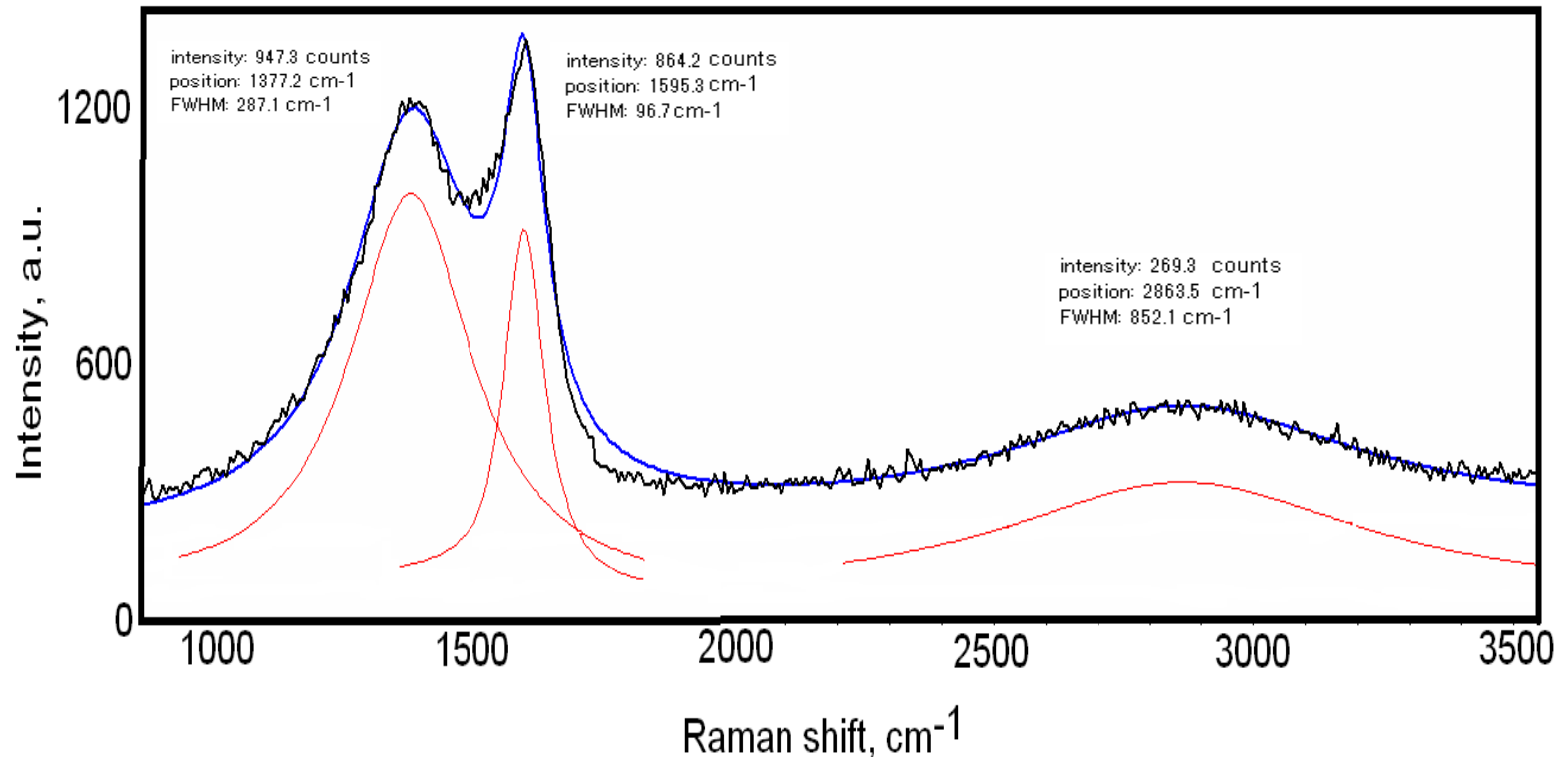
## Raman spectra of carbon fibers



The disorder-induced band (D-band) occurs at about  $1372 \text{ cm}^{-1}$ .  
The G-band at  $1595 \text{ cm}^{-1}$  is related to C–C vibrations in Graphite.

The ratio of the intensities of the D-band to the G-band is commonly used to characterize different kinds of disordered  $\text{sp}^2$  carbon.

## Lorentzian curve fitting of the D and G bands



- 1) total integral intensity ratio  $I_D/(I_D+I_G)$ ,
- 2) integral intensity ratio  $I_D/I_G$ ,
- 3) Raman peak intensity ratio  $R= I_D/I_G$

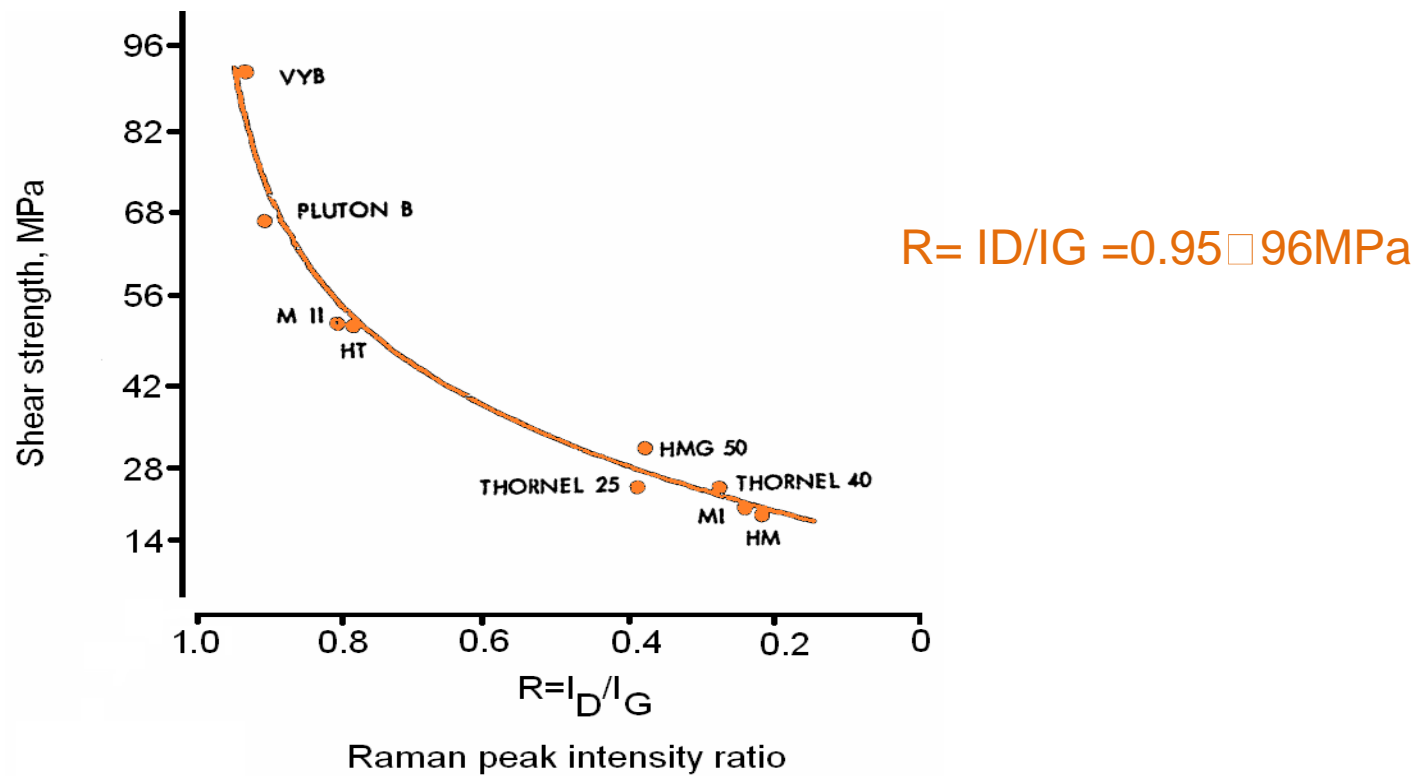
The Raman peak intensity ratio ( $R = I_D/I_G$ )  
as a function of burn-off

Burn-off (%)	$R = I_D/I_G$
0	0.8616
39	0.8667
59	0.9142
79	0.9338

$R = I_D/I_G = 0.95$  □ Burn-off > 79%

J.-S. Roh, Carbon Letters, Vol. 9, No. 2 (2008) 127-130

## Shear strength as a function of the Raman peak intensity ratio



## The degree of structural disorder in the fibers and the integrated intensity ratio (ID/IG)

The surface crystalline size (La) of the carbon fibers may be obtained by the following the equation

$$La=C/(I_D/I_G),$$

where La is the surface crystalline size and C is equal to 44 Å

Because the ID/IG integrated intensity ratio is 3.254, the surface crystalline size is 13.52 Å.

The slight distortion in graphitic structure of carbon fibers occur through the **Breaking of aromatic bonds and reduction of surface crystallinity.**

Sudhir Tiwari, J. Bijwe and S. Panier, Strengthening of a Fibre-Matrix Interface: A Novel Method Using Nanoparticles, Nanomaterials and Nanotechnology, 2013, vol 3, Art.3 :2013

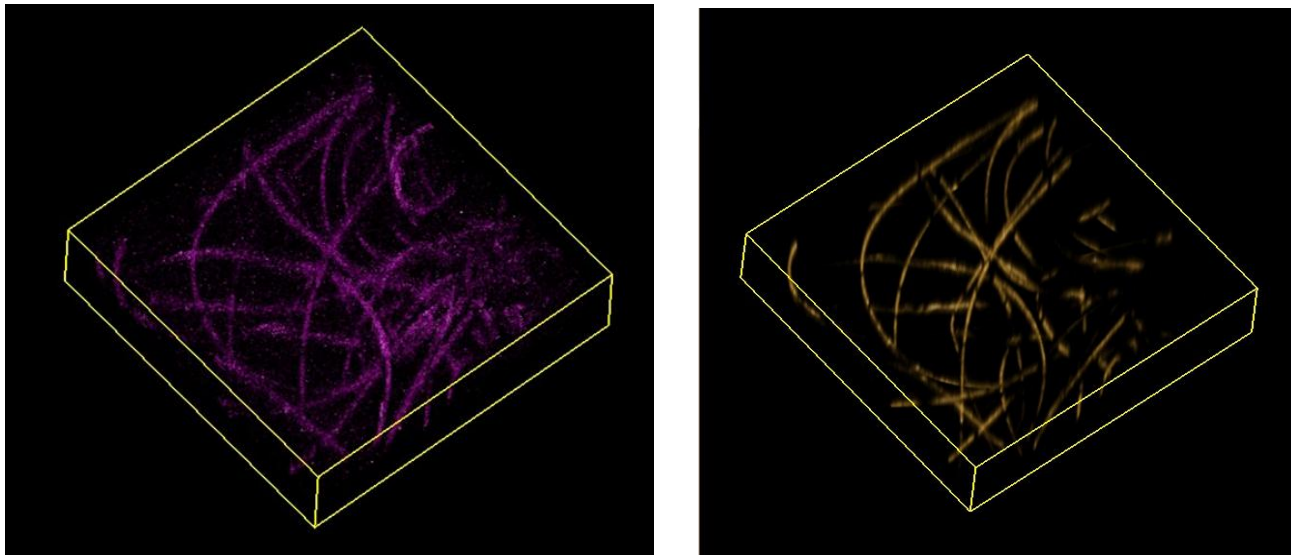


Raman bands present in the spectra of carbon fiber are strain sensitive

With an increase in applied tensile strain, Raman bands shift to lower frequencies and tend to broaden in peak width.

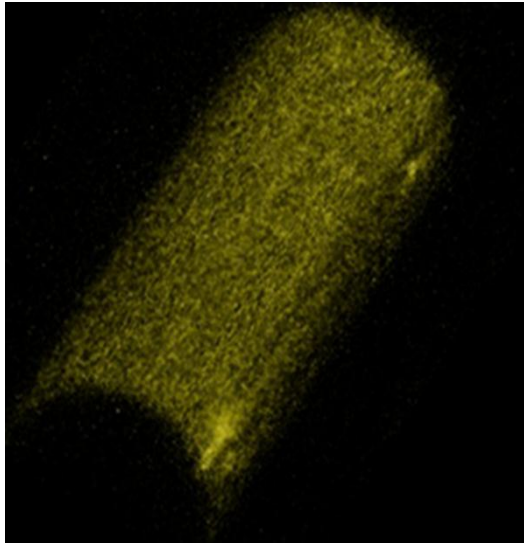
With applied compressive strain the Raman bands will shift to higher frequencies

Raman confocal image of carbon fibers (a),  
Laser confocal image of carbon fibers (b).

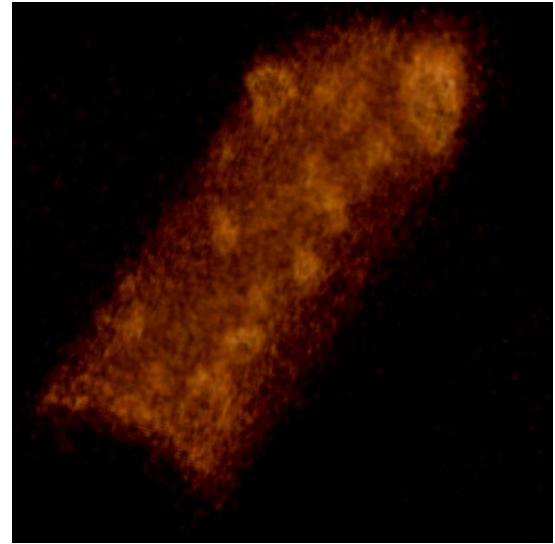


680 x 680 x 30  $\mu\text{m}$  (250 x 250 x 70 points)

G-band



D-band



Rayleigh

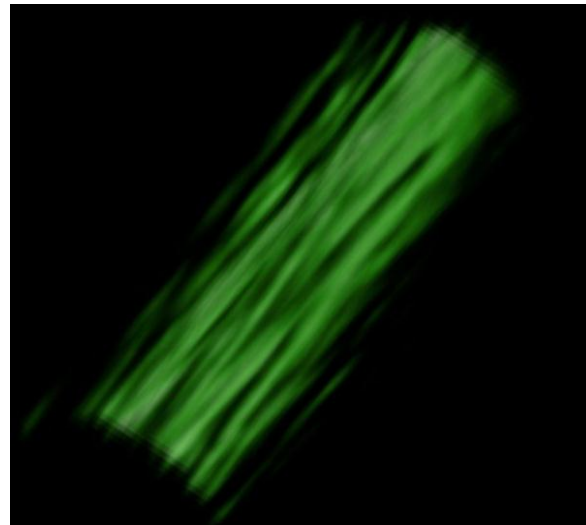


Image size is 21 x 21 x 7 $\mu$ m (250 x 250 x 70 pixels)

## Summary

Raman microscopy is a powerful method for the structural characterization of carbon fibers.

The characteristics and properties of carbon fibers have been examined By Raman Spectroscopy.



Thank you very much for your attention!