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A Better Measure.

MECHANICAL & TRIBOLOGICAL PROPERTIES OF CARBON FIBER COMPOSITE

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INTRO

Carbon fiber composite is composed of extremely strong carbon fibers bound by polymers, such as resin, epoxy or others. Its high strength-to-weight ratio and rigidity make carbon fiber composite an ideal material to be applied in a variety of industries, such as aerospace, automotive and civil engineering, sports goods and other consumer and technical applications. For example, its usage has been substantial increased in the most advanced aircrafts – The Airbus A350 XWB is built of 53% carbon fiber composite including wing and fuselage components, the Boeing 787 Dreamliner, 50%. It is also used extensively in high-end automobile racing and supercars.

Unlike isotropic materials such as steel and aluminum, carbon fiber composite often has directional strength properties due to the texture layouts of the carbon fibers. Therefore, the local mechanical and tribological properties of the carbon fiber composite vary depending on the direction of the carbon fibers and the proportion of the carbon fibers relative to the polymer matrix at the test location. A reliable and accurate evaluation protocol of the local mechanical and tribological properties becomes vital for developing and comparing the carbon fiber composite in Quality Control and Research and Development.

MEASUREMENT OBJECTIVE

Combined with the wear test by Tribometer and surface analysis by Optical 3D Profilometer, we showcase the versatility and accuracy of the Nanovea instruments in testing composite materials with directional mechanical properties.

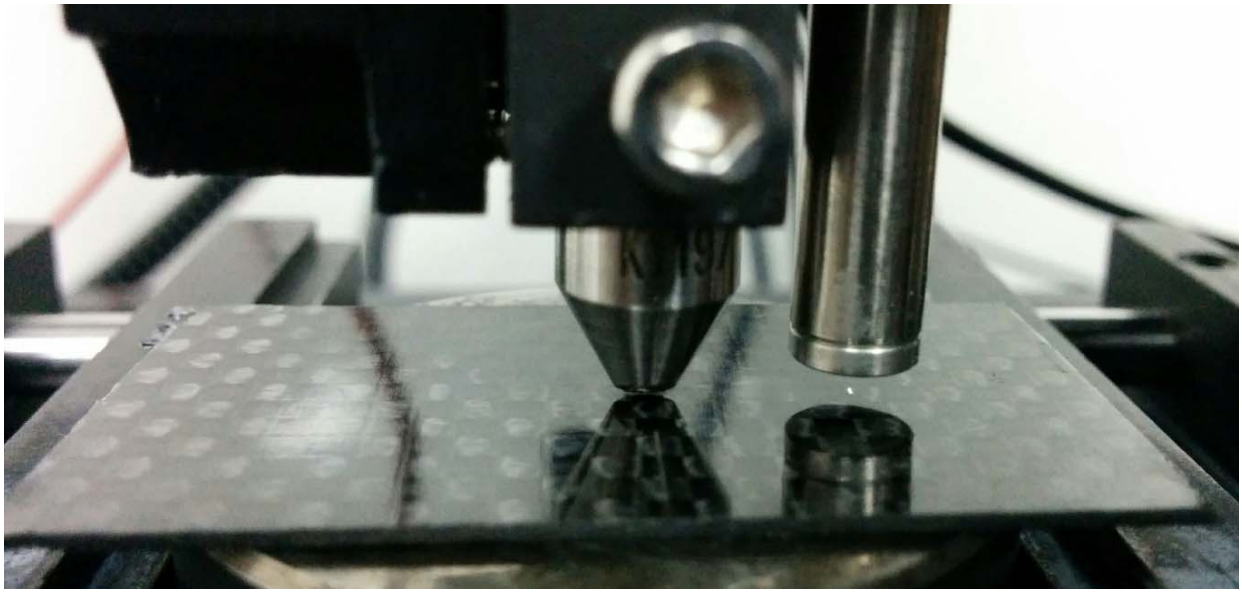


Fig. 1: Indentation of the carbon fiber sheet.

MEASUREMENT PRINCIPLE

Principle of instrumented indentation test

The indentation test is based on the standards for instrumented indentation, ASTM E2546 and ISO 14577. It uses an established method where an indenter tip with a known geometry is driven into a specific site of the material to be tested, by applying an increasing normal load. When reaching a pre-set maximum value, the normal load is reduced until complete relaxation occurs. During the experiment the position of the indenter relative to the sample surface is precisely monitored.

The resulting load/displacement curves provide data specific to the mechanical nature of the material under examination. Established models are used to calculate quantitative hardness and modulus values for such data.

Analysis of Indentation Curve

Following the ASTM E2546 (ISO 14577), hardness and elastic modulus are determined through load/displacement curve as for the example below.

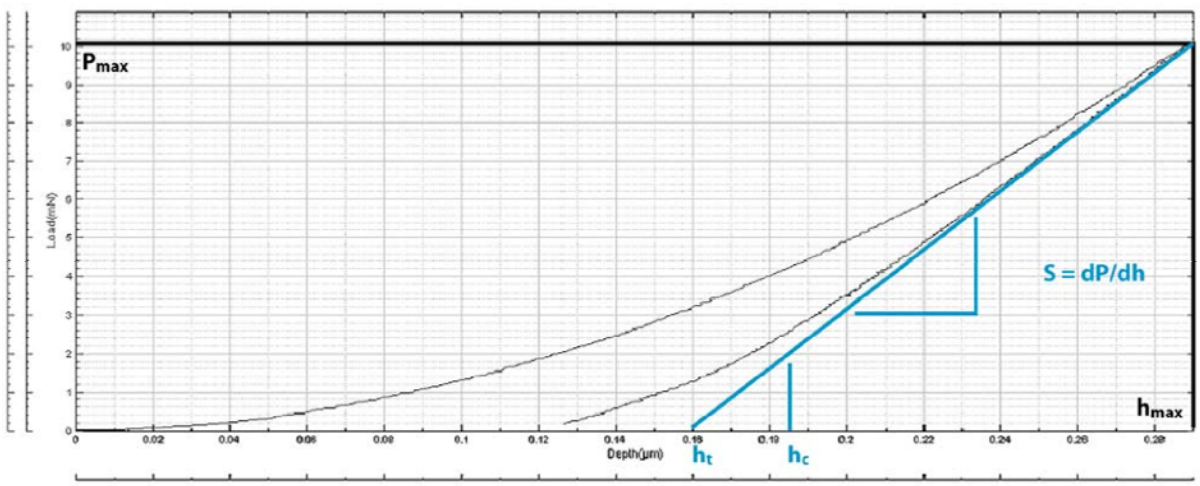


Fig. 2: Load-displacement curve of indentation.

Hardness

The hardness is determined from the maximum load, P_{max}, divided by the projected contact area, A_c:

$$H = \frac{P_{max}}{A_c}$$

Young's Modulus

The reduced modulus, E_r, is given by:

$$E_r = \frac{\sqrt{\pi}}{2}\frac{S}{\sqrt{A_c}}$$

Which can be calculated having derived S and A_C from the indentation curve using the area function, A_C being the projected contact area. The Young's modulus, E, can then be obtained from:

$$\frac{1}{E_r} = \frac{1-\nu^2}{E} + \frac{1-\nu_i^2}{E_i}$$

Where E_i and ν_i are the Young's modulus and Poisson's ratio of the indenter and ν the Poisson's ratio of the tested sample.

How are these calculated?

A power-law fit through the upper 1/3 to1/2 of the unloading data intersects the depth axis at h_t. The stiffness, S, is given by the slope of this line. The contact depth, h_c, is then calculated as:

$$h_c = h_{\max} - {3P_{\max}}/{4S}$$

The contact Area A_c is calculated by evaluating the indenter area function. This function will depend on the diamond geometry and at low loads by an area correction.

For a perfect Berkovich and Vickers indenters, the area function is A_c=24.5h_c^2. For Cube Corner indenter, the area function is A_c=2.60h_c^2. For Spherical indenter, the area function is A_c=2πRh_c , where R is the radius of the indenter. The elastic components, as previously mentioned, can be modeled as springs of elastic constant E, given the formula: $\sigma = E\varepsilon$ where σ is the stress, E is the elastic modulus of the material, and ε is the strain that occurs under the given stress, similar to Hooke's Law. The viscous components can be modeled as dashpots such that the stress-strain rate relationship can be given as $\sigma = \eta \frac{d\varepsilon}{dt}$, where σ is the stress, η is the viscosity of the material, and dε/dt is the time derivative of strain.

Since the analysis is very dependent on the model that is chosen, Nanovea provides the tool to gather the data of displacement versus depth during the creep time. The maximum creep displacement versus the maximum depth of indent and the average speed of creep in nm/s is given by the software. Creep may be best studied when loading is quicker. Spherical tip is a better choice.

Principle of scratch test

The scratch testing method is a very reproducible quantitative technique. Critical loads at which failures appear are used to compare the cohesive or adhesive properties of coatings or bulk materials. During the test, scratches are made on the sample with a sphero-conical stylus (tip radius ranging from 1 to 200 μm) which is drawn at a constant speed across the sample, under a constant load, or, more commonly, a progressive load with a fixed loading rate. Sphero-conical stylus is available with different radii (which describes the "sharpness" of the stylus). Common radii are from 20 to 200 μm for micro/macro scratch tests, and 1 to 20 μm for nano scratch tests.

When performing a progressive load test, the critical load is defined as the smallest load at which a recognizable failure occurs. In the case of a constant load test, the critical load corresponds to the load at which a regular occurrence of such failure along the track is observed.

In the case of bulk materials, the critical loads observed are cohesive failures, such as cracking, or plastic deformation or the material. In the case of coated samples, the lower load regime results in conformal or tensile cracking of the coating which still remains fully adherent (which usually defines the first critical load). In the higher load regime, further damage usually comes from coating detachment from the substrate by spalling, buckling or chipping. Fig. 3 illustrates the principle of scratch testing.

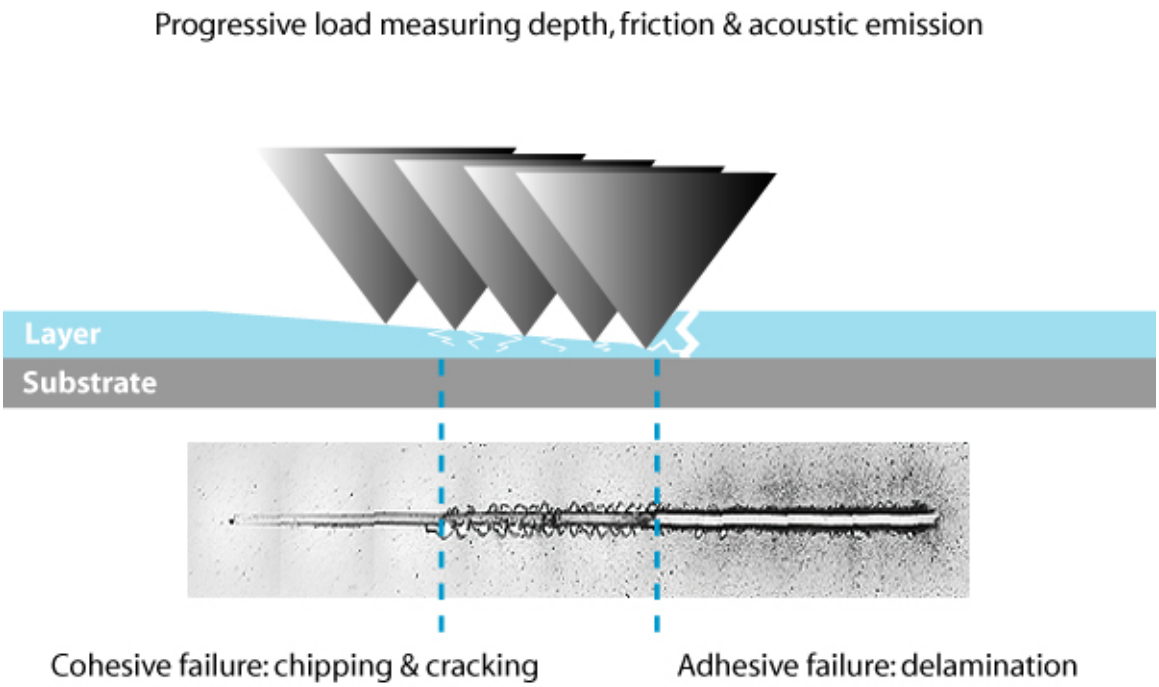


Fig. 3: Principle of scratch testing.

Principle of linear wear test

The sample is mounted on a moving stage, while a known force is applied on a pin, or ball, in contact with the sample surface to create the wear. As the sample moves in a linear reciprocating (see Fig. 4) motion, the resulting frictional forces between the pin and the sample are measured using a strain gage sensor on the arm. The wear test is generally used as a comparative test to study the tribological properties of the materials. The, coefficient of friction, COF, is recorded in situ. The volume loss allows calculating the wear rate of the material. Since the action performed on all samples is identical, the wear rate can be used as a quantitative comparative value for wear resistance. This simple method facilitates the determination and study of friction and wear behavior of almost every solid state material combination, with varying time, contact pressure, velocity, temperature, humidity, lubrication, etc.

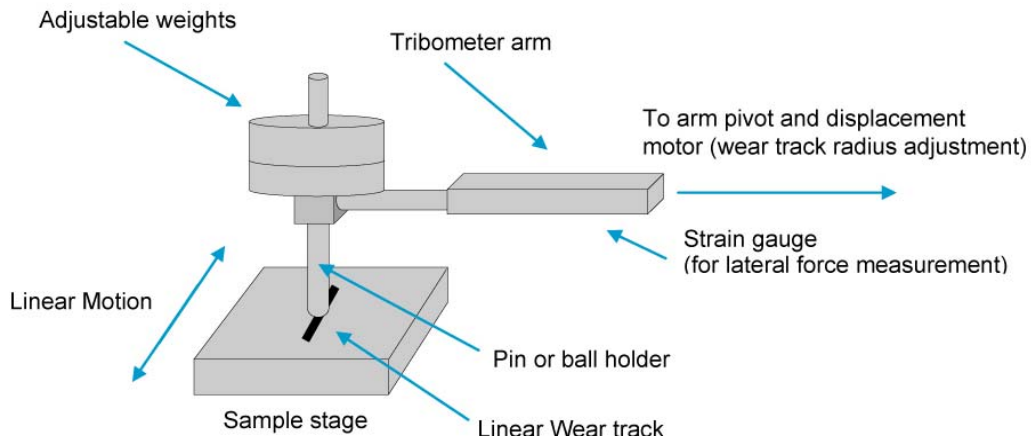


Fig. 4: Schematics of linear reciprocating wear test.

Principle of profilometer measurement

The axial chromatism technique uses a white light source, where light passes through an objective lens with a high degree of chromatic aberration. The refractive index of the objective lens will vary in relation to the wavelength of the light. In effect, each separate wavelength of the incident white light will re-focus at a different distance from the lens (different height). When the measured sample is within the range of possible heights, a single monochromatic point will be focalized to form the image. Due to the confocal configuration of the system, only the focused wavelength will pass through the spatial filter with high efficiency, thus causing all other wavelengths to be out of focus. The spectral analysis is done using a diffraction grating. This technique deviates each wavelength at a different position, intercepting a line of CCD, which in turn indicates the position of the maximum intensity and allows direct correspondence to the Z height position.

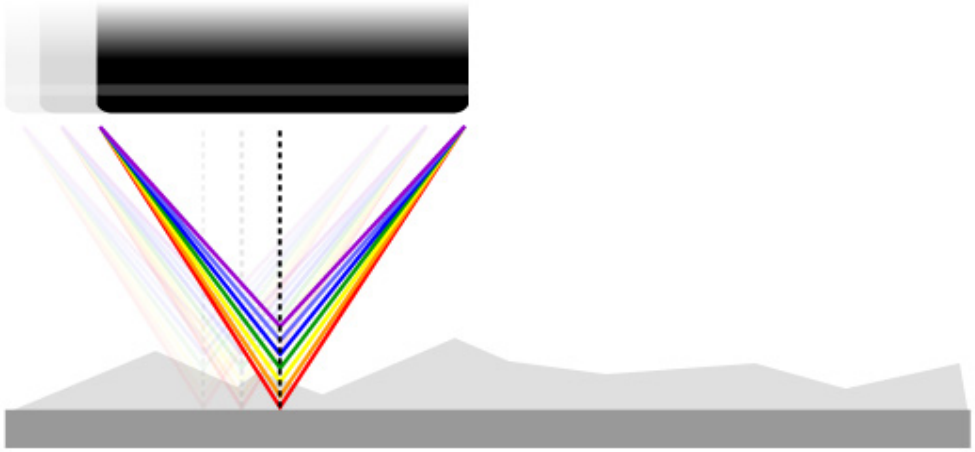


Fig. 5: Schematic of axial chromatism technique.

Nanovea optical pens have zero influence from sample reflectivity. Variations require no sample preparation and have advanced ability to measure high surface angles. Capable of large Z measurement ranges. Measure any material: transparent or opaque, specular or diffusive, polished or rough.

TEST CONDITIONS & PROCEDURE

SAMPLE DESCRIPTION

A high-strength lightweight carbon fiber sheet is used for the comprehensive mechanical and tribological evaluation.

HARDNESS AND YOUNG'S MODULUS

The local hardness and Young's modulus were measured using Micro Mode of Nanovea Mechanical Tester. The test conditions are shown in Table 1.

Applied Force (N)	3
Loading rate (N/min)	6
Unloading rate (N/min)	6
Indenter type	Knoop & Vickers

Table 1: Test conditions of hardness and Young's modulus.

SCRATCH RESISTANCE

The scratch resistance of the carbon fiber sheet were tested using Micro Scratch Mode with a diamond stylus (200 µm radius). Images of the whole scratch (panorama) were automatically generated and the different areas of the scratch were correlated with the friction coefficient by the system software. The test parameters are listed in Table 2.

Load type	Constant
Load	10 N
Scratch Length	10 mm
Indenter	Rockwell C
Indenter material (tip)	Diamond
Indenter tip radius	200 µm

Table 2: Scratch test parameters.

WEAR RESISTANCE

The wear behavior of the carbon fiber sheet was evaluated using linear reciprocating setup of Nanovea Tribometer. The test conditions are listed in Table 3.

Test parameters	Value
Normal force	20 N
Wear track length	12 mm
Speed	200 cycles/min
Revolutions	6000
Pin geometry	Spherical 6 mm dia.
Pin material	Al_2O_3

Table 3: Test conditions of the linear reciprocating wear measurement.

RESULTS AND DISCUSSION

Hardness and Young's Modulus measured using Knoop and Vickers indenters

The hardness, *H*, and Young's Modulus, *E*, of the high-strength carbon fiber sheet were tested at different locations using a Knoop indenter in order to investigate the effect of directional carbon fiber on the strength enhancement as shown in Fig. 6.

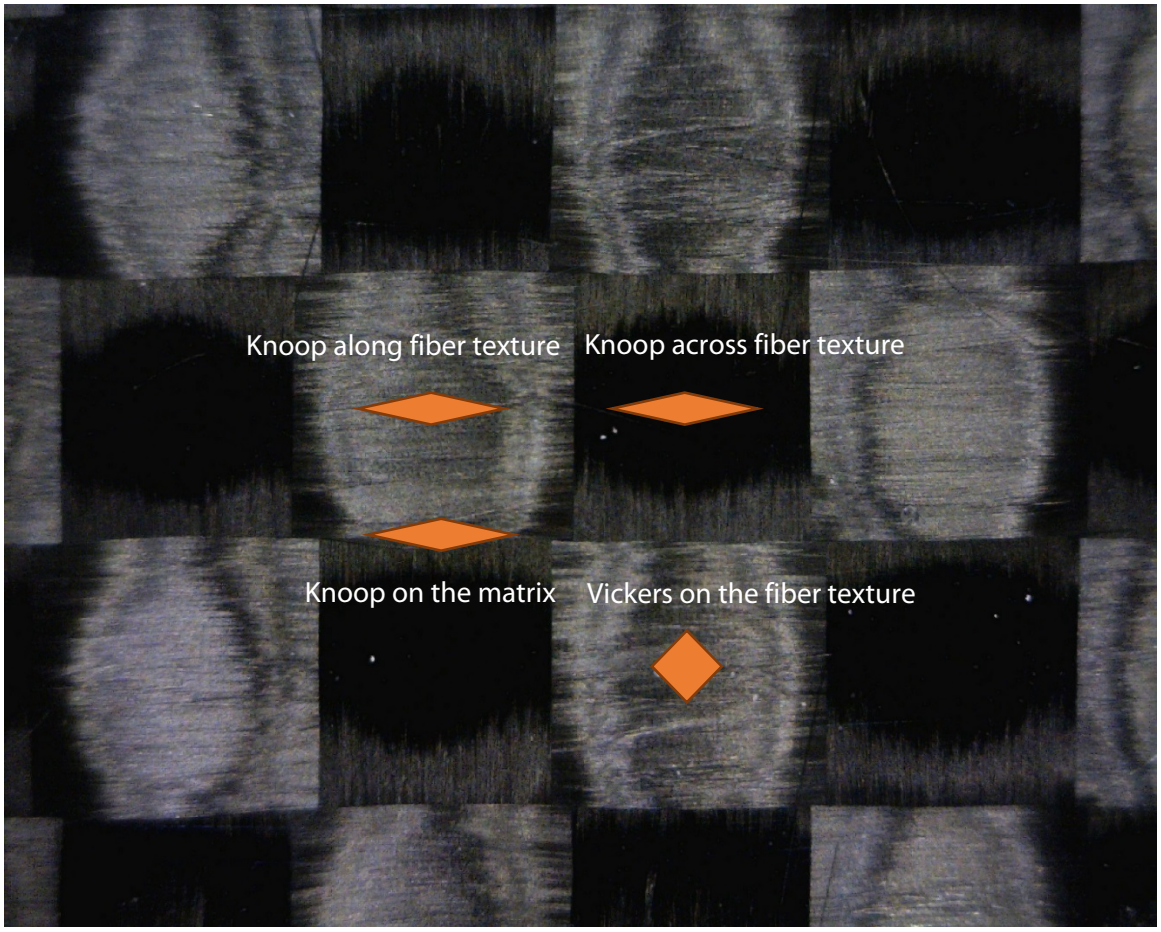


Fig. 6: Micrograph of the carbon fiber sheet with illustration of indentation locations.

The load-displacement curves are shown in Fig. 7 and the test results are listed in Table 4. It can be observed that the carbon fiber composite exhibits different *H* and *E* values at different locations and carbon fiber orientations. The sharper Vickers tip created a deeper penetration at the same load compared to the Knoop tip. As shown in Fig. 8, the Knoop indenter across the fiber texture left a smaller indentation mark on the sample, primarily attributed to the strength and rigidity reinforcement of the carbon fiber underneath the indent. The resulted *H* and *E* values are 0.68 and 12.2 GPa, respectively. In comparison, the Knoop tip with the sharper edge along the carbon fiber texture penetrated deeper into the gap between fibers, leading to substantially decreased *H* and *E* values at 0.22 and 6.5 GPa, respectively. The carbon fibers are patterned and combined by epoxy or resin matrix. The indentation on the matrix at the border of the pattern shows a large mark. The

Vickers tip has a symmetric shape and thus less impact from the direction of the carbon fiber pattern, resulting in measured *H* and *E* values of 0.46 GPa and 7.5 GPa.

In this indentation study we show that Nanovea Mechanical Tester equipped with a Knoop tip is an ideal tool for analyzing the local mechanical properties of the composite materials with directional strength reinforcement.

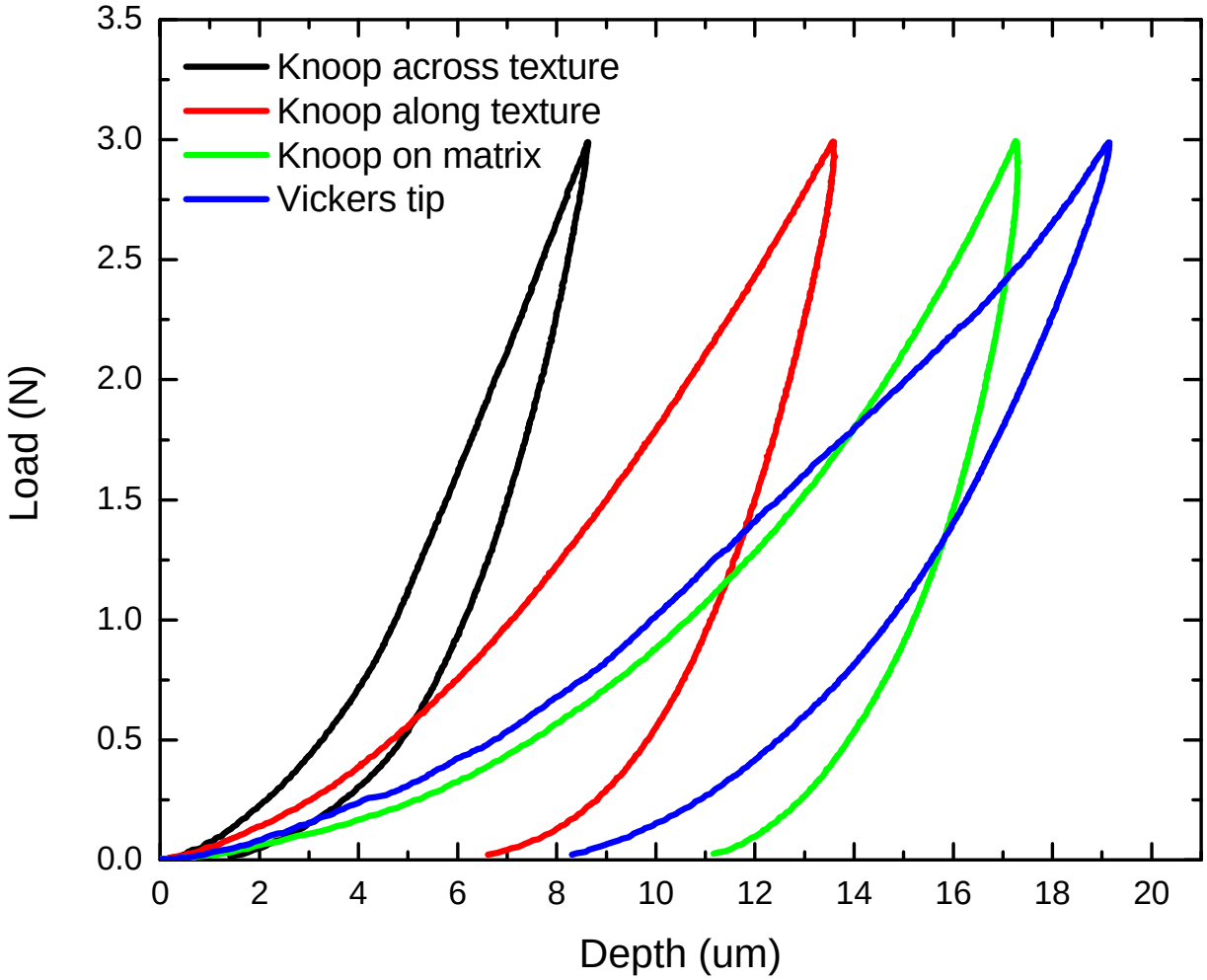
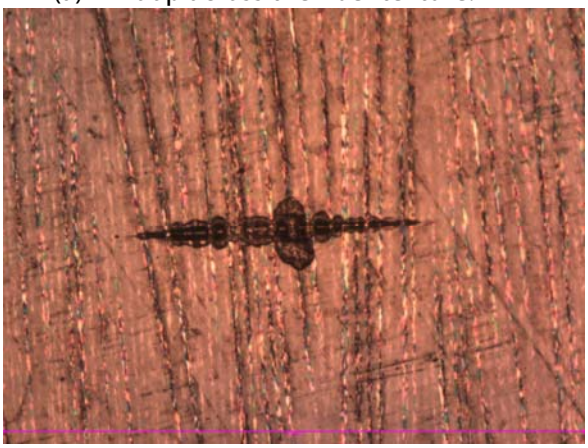


Fig. 7: Load-displacement curves of the indentations at different locations.

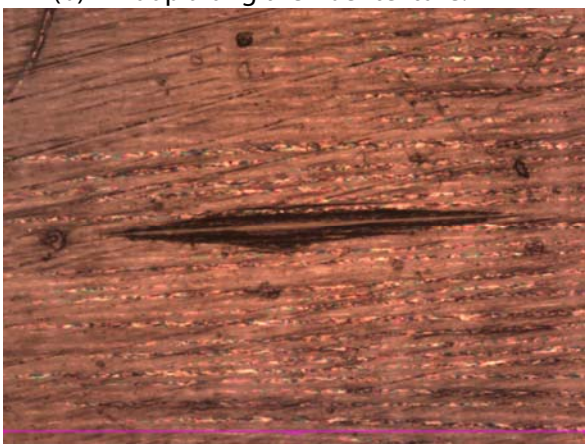
	Hardness (GPa)	Young's Modulus (GPa)
Knoop across the fiber texture	0.68	12.2
Knoop along the fiber texture	0.22	6.5
Knoop on the matrix	0.12	5.8
Vickers on the fiber texture	0.46	7.5

Table 4: Measured hardness, *H*, and Young's Modulus, *E*, at different locations.

(a) Knoop across the fiber texture:



(b) Knoop along the fiber texture:



(c) Knoop on the matrix:

(d) Vickers on the fiber texture:

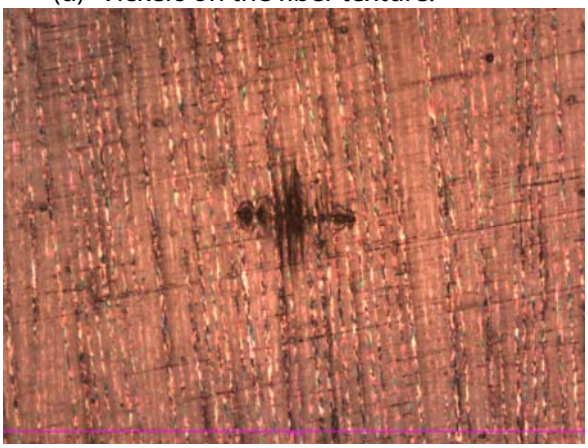
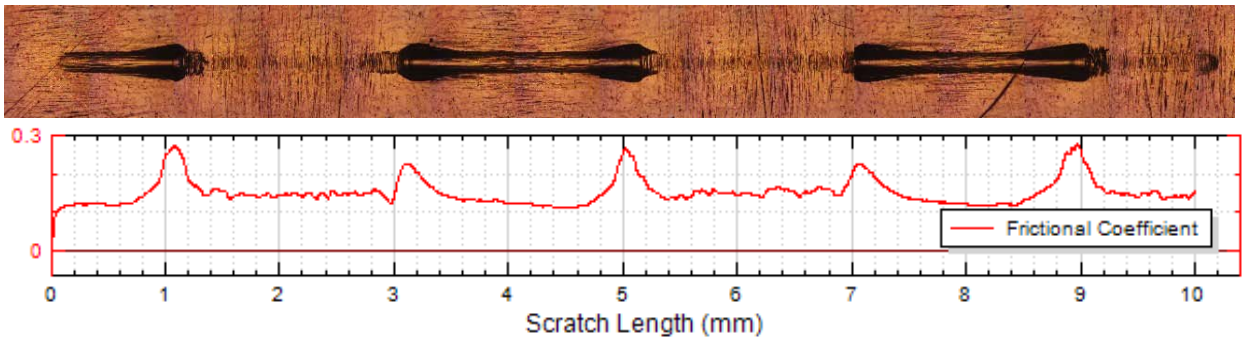


Fig. 8: Indentations at different locations of the carbon fiber sheet (50X).

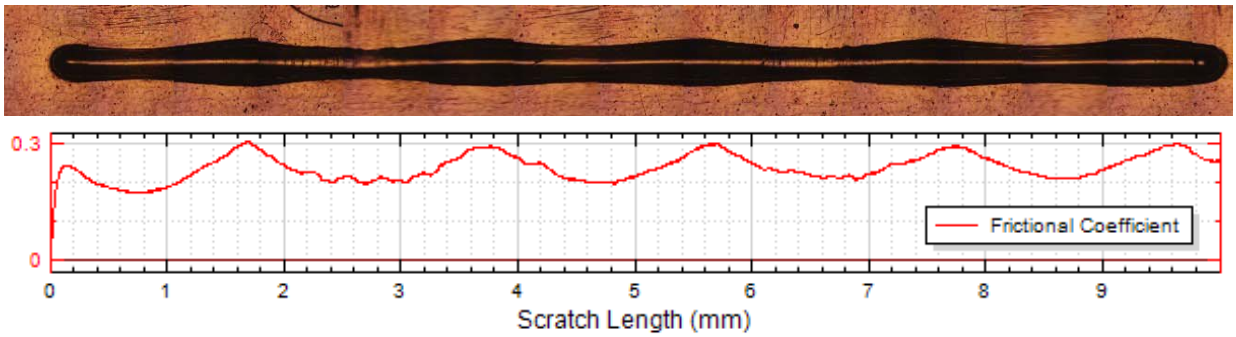
Scratch resistance measured using 200 µm diamond stylus

The scratch resistance of the carbon fiber composite on the texture and the matrix are compared using scratch tests at a constant load of 10 N as shown in Fig. 9. The penetration depth varies along the scratch track – the scratch along the carbon fiber texture shows a width of ~0.18 mm, while that across the carbon fiber texture barely shows a sign of shallow scratch. The scratch at the border of the pattern exhibit a deep groove, due to the relatively soft feature of the matrix. This demonstrates that the carbon fibers significantly enhance the scratch resistance of the test composite sample.

Such observation under the optical microscope is in agreement with the evolution of coefficient of friction (COF) recorded in situ during the scratch tests. The close loop control of the load with a separate precision load cell ensures the consistency of the applied constant normal load. The COF exhibits a relatively constant value of ~0.15 when the pin travels on the carbon fiber texture, and peak values above 0.25 as the pin digs into the groove at the border of the pattern. The scratch on the matrix along the pattern border shows a larger track and higher COF.



(a) Scratch on the texture:



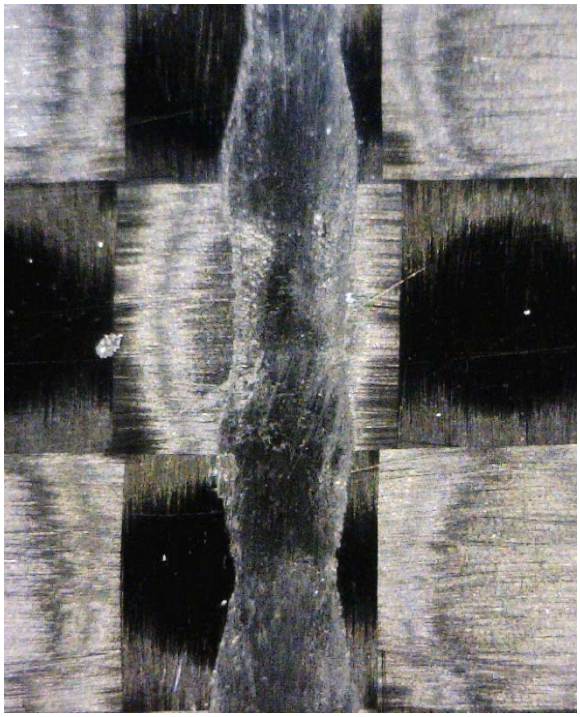
(b) Scratch on the matrix:

Fig. 9: Scratch tracks and evolution of COF during the scratch tests.

Wear resistance comparison using linear reciprocating tribometer

The wear tracks were compared using the microscope and 3D profilometer as shown in Fig. 10 and Fig. 11, respectively. Nanovea 3D profilometer allows visual examination of the 3D wear tracks and direct accurate determination of the wear volume and depth as summarized in Table 5. The wear test on the carbon fiber texture created a smaller wear track compared to the one on the matrix. Such difference in wear resistance is attributed to the strength enhancement of the carbon fiber texture, which gives rise to improved mechanical properties, including high *H&E* and scratch resistance. The enhanced mechanical properties by carbon fibers cut the wear rate by half compared to the polymer matrix.

(a) Wear on Texture:



(b) Wear on Matrix:

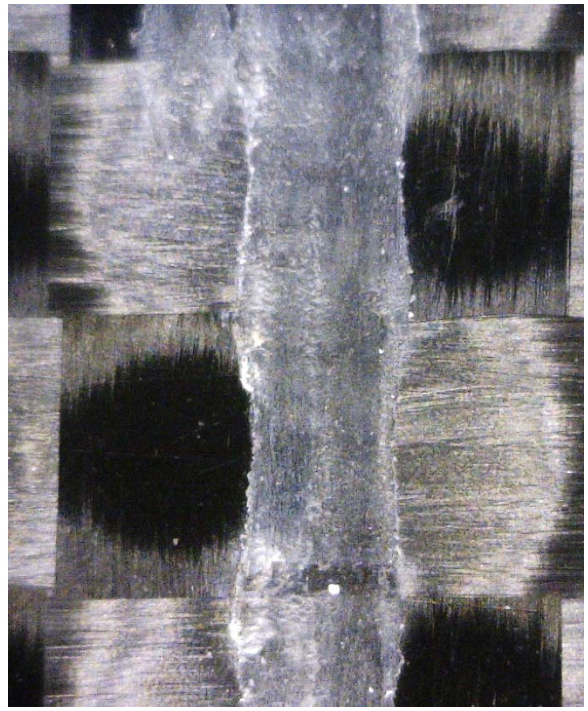
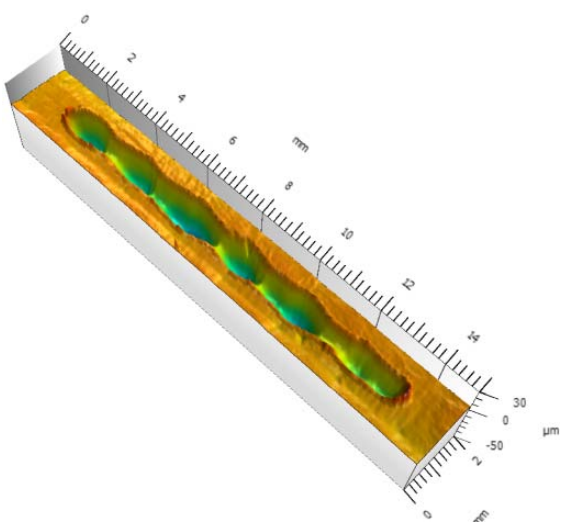


Fig. 10: **Wear tracks on the sample after the tests.**

(a) Wear on Texture:



(b) Wear on Matrix:

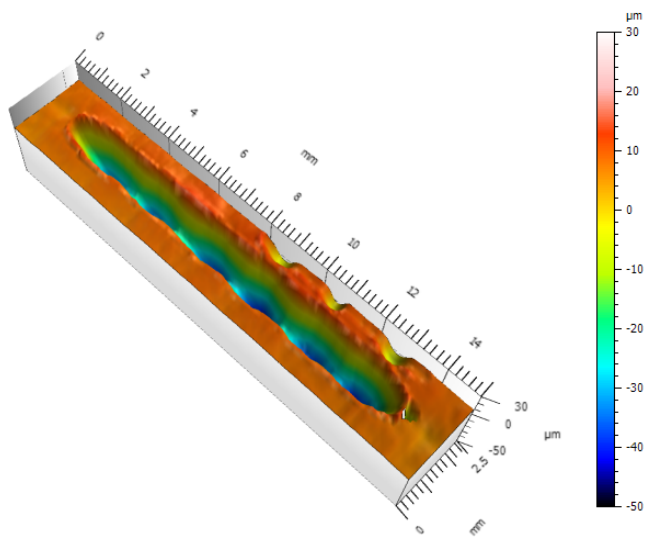


Fig. 11: 3D surface profile of the wear tracks.

	Volume (mm^3)	Depth (µm)
On the texture	0.126	38.5
On the matrix	0.258	55.6

Table 5: Summary of wear track volume and depth.

CONCLUSION

In summary, carbon fibers play a critical role in the enhancement of mechanical and tribological properties in the composite material. It is important to be aware that the values obtained in the indentation and wear tests are highly dependent on the location and fiber pattern orientation of the composite material.

In this study, we show that the Nanovea Mechanical Tester, Tribometer and 3D Profilometer complement each other by measuring different aspects of essential material properties. They can provide a complete mechanical & tribological evaluation of composite materials and allow users to get more insight into the physiomechanical behaviors of materials under different applications.

The Nano, Micro or Macro modules of the Nanovea Mechanical Tester all include ISO and ASTM compliant indentation, scratch and wear tester modes, providing the widest and most user friendly range of testing available in a single system. The Nanovea Tribometer offers precise and repeatable wear and friction testing using ISO and ASTM compliant rotative and linear modes, with optional high temperature wear, lubrication and tribo-corrosion modules available in one pre-integrated system. In addition, optional 3D non-contact profiler and AFM Module are available for high resolution 3D imaging of indentation, scratch and wear track in addition to other surface measurements such as roughness.

To learn more about Nanovea Mechanical Tester, Tribometer or Lab Services.