(REFEREED RESEARCH)

# HARDNESS PROPERTIES OF PLASTIC OPTICAL FIBERS BY NANOINDENTATION

## NANOİZ TESTİ İLE PLASTİK OPTİK LİFLERİN ÖZELLİKLERİNİN İNCELENMESİ

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**ABSTRACT**

Mechanical properties are significant for plastic optical fibers in weaving or knitting processes in textile applications. Both core and cladding in plastic optical fiber contribute to the mechanical properties. Nanoindentation is a promising method to investigate the mechanical properties (hardness, stiffness and Young's modulus) in nanoscale displacement and small load range. Nanoindentation creep as the highly time-dependent deformation has a significant effect on nanoindentation properties of polymeric materials. In present work, core and cladding in both latitudinal and longitudinal cross sections of plastic optical fibers with four diameters were investigated in different conditions (loading rates and holding times) under the maximum load of 0.3 mN. The results show that cladding is softer than core and both strong loading rate and holding time sensitivities on nanoindentation creep no matter in latitudinal or longitudinal cross sections. It is also found that the greater the fiber diameter, the higher the hardness and modulus.

**Keywords:** Nanoindentation, Creep, Plastic optical fiber, Cross section, Surface roughness, Hardness, Modulus

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### 1. INTRODUCTION

Nanoindentation testing is eviendently utilized to investigate the mechanical properties of materials in nanometer-scale displacement and smaller load range than other testing methods (e.g. DMA and Instron). With the increasing use of very small structures, functionally graded materials, nanocomposites, and other heterogeneous materials in various engineering areas such as electronic, mechanical and biomedical engineering, a critical evaluation for both stress and deformation is needed to predict the reliability and failure behavior of such structures.

Measurements on mechanical properties of polymer surface can provide improved understanding of molecular structure of the material itself, manufacturing and polymerization processes. Nanoindentation is widely used to measure the mechanical properties, such as hardness, Young's modulus and stiffness, which can be obtained from the load-displacement behavior carried out by applying the increasing load and decreasing load to the surface with a shaped indenter. However, it is necessary to consider the creep effect during the measurement of polymeric materials. This "nose" phenomenon, which may be found in view of the highly time-dependent deformation nature of polymers, may influence the evaluation of contact displacement of the surface markedly, resulting in effects on the evaluation of indentation hardness.

The Oliver and Pharr method was developed to measure the hardness and elastic modulus of a material from load-displacement curve during one cycle of loading and unloading. Although this method was originally developed for applications with sharp and geometrically self-similar indenters (e.g. the Berkovich triangular pyramid), lots of experiments and experience have been proved that Oliver and Pharr can also be used with a variety of axisymmetric indenter geometries including the sphere, even better (1).

When nanoindenter contacts the polymeric surface and penetrates into the polymer, additional difficulties for the indenter to go inside the polymer are caused by the complicated viscoelastic-plastic response that is typical character of such kind of material. Polymer has highly strain-dependent and strain rate-dependent properties and shows substantially different behaviors when the indentations are preceded under different contact conditions. The viscoelastic-plastic response of such material, therefore, can provide the values of hardness and elastic modulus, which are usually a function of the imposed contact conditions, such as the geometry of the contact and the penetration depth (i.e. the strain), the loading rate (or strain rate) (2, 3) and the ambient temperature (4, 5).

Plastic optical fiber (POF) is a media that can transfer the dielectric waveguide light or infrared radiation from one place to another place based on the theory of total internal reflection according to the different refractive indices of fiber core and cladding (6). POF was firstly introduced as the substitude of glass optical fiber in the short distance communications in 1960s, nowadays, POF has already widely used in textiles, there are two main areas (7), one is utilized in textile tructures (such as knits and jacuquard wovens) activily emitting light in clothing densign (8) and active visible protective textiles (9), another is sensor-optical fibers (10) in textile structures (e.g. geo-textiles).

When POF is woven or knitted into textile structure, the mechanical characterizations of the whole fiber are supported from both fiber core and cladding which are separated from each other, the mechanical deformation occurs in weaving or knitting processes, the different properties of fiber core and cladding would lead to quick mechanical failure or more optical loss. So the mechanical properties of both core and cladding should be taken into account.

In this paper, the effects of loading rate sensitivity and holding time sensitivity on mechanical properties of POF were investigated by nanoindentation testing. The surface of the cross section of each sample should be polished and smooth enough for the nanoindentation tests which were performed with various loading time (5, 10, 15, 20 and 30 s) and holding time (5, 10, 15, 20 and 30 s) under the maximum load of 0.3 mN to discuss the dependence of the indentation creep behavior on loading time and holding time. Meanwhile, the effects of fiber diameter and cross sections in different directions were also studied to investigate the mechanical properties of plastic optical fibers (POFs) by nanoindentation.

## 2. MATERIALS AND METHODS

### Materials

The plastic optical fibers (POFs) with different diameters were prepared by Grace POF Co., Ltd., Taiwan. The basic properties of POFs are shown in Table 1.

**Table 1.** Basic characterizations of POFs.

| Basic characterizations | Values |
|---|---|
| Core material | PMMA |
| Cladding material | PMMA/Telfon |
| Diameter (mm) | 0.25/0.5/0.75/1.0 |
| Core refraction index | 1.49 |
| Cladding refraction index | 1.42 |
| Numerical aperture | 0.44 |
| Acceptance angle (°) | 52.2 |
| Specific gravity (g/cm$^3$) | 1.19 |
| Wavelength (nm) | 400 ~ 780 |
| Limit of bending radius | 8× fiber diameter |

### Preparation of specimens

The specimens for nanoindentation testing were prepared as follows:

(1) For preparation of latitudinal cross sections, a bundle of POFs were put into suitable cables which were inserted into appropriate holes of button for normal clothes. Super glue was used to fix all parts as an unmovable unit. The cable with fibers inside was cut in both sides of the button.

(2) For preparation of longitudinal cross sections, the fibers were arranged straightly one by one on the glass slides (1 cm × 1 cm) by using the super glue.

(3) The fibers both in buttons and on the glass slides were polished by the polishing papers with different sizes. The smallest particle diameter of polishing papers used was 1 micro. Then the samples were fine-polished with W0.5 water-based diamond polishing paste until the surface roughness was small enough for nanoindentation tests. The specimens were polished in the clockwise direction by hand with the speed of 50 ~ 60 times per minute. The specimens in latitudinal cross section and longitudinal cross section were prepared at last.

### Nanoindentation tests

The nanoindentation tests were carried out by Hysitron with a three-side pyramidal Berkovich diamond indenter. The nanoindentation creep for POFs was conducted by two ways: when the holding time was 10 s, the loading time was changed from 5 s to 30 s (Figure 1a); when the loading time was 10 s, the holding time was changed from 5 s to 30 s (Figure 1b). For both ways, the unloading time was always the same as the loading time. The fibers with different diameters prepared in latitudinal and longitudinal cross sections were measured to investigate the diameter effect on nanoindentation properties at 10 s loading time and 10 s holding time under 0.3 mN maximum load.

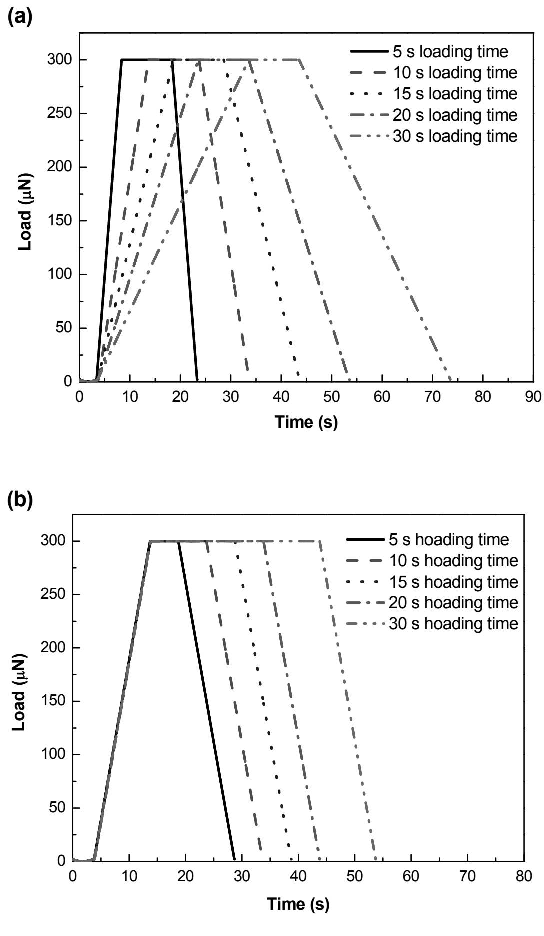


**Figure 1.** Experimental designs of nanoindentation creep tests of POFs under 0.3 mN maximum load: (a) loading rate sensitivity; (b) holding time sensitivity.

Contact stiffness can be calculated from the slope of initial unloading curve. During the unloading period, only elastic deformation is recovered (Figure 2). The dependence between stiffness and elastic modulus of specimen can be expressed by Equation 1, where *S* is the experimentally measured contact stiffness from unloading data, *A* is the contact projected area between indenter and specimen (for an ideally sharp Berkovich indenter, the cross-sectional area in terms of contact depth equals to 24.5 $h_c^2$ ), *P* is the load on the indenter. $E_r$ is the reduced modulus related to both the elastic modulus ($E_i$, $E_s$) and Poisson's ratio ($v_i$, $v_s$) of indenter and specimen, as shown in Equation 2. *β* is a constant which is used to account for the triangular and square cross sections of indenters in nanoindentation (e.g., *β*=1.034 for a triangular punch).

$$S = \frac{dP}{dh} = \frac{2\beta}{\sqrt{\pi}} \cdot \sqrt{A} \cdot E_r \tag{1}$$

$$\frac{1}{E_r} = \frac{(1-v_s^2)}{E_s} + \frac{(1-v_i^2)}{E_i} \tag{2}$$

Hardness, *H*, is calculated from the indent produced by ideally sharp Berkovich tip,

$$H = \frac{P_{\max}}{A} = \frac{P_{\max}}{24.5h_c^2} \tag{3}$$

where $P_{max}$ is the maximum load applied during the indentation.

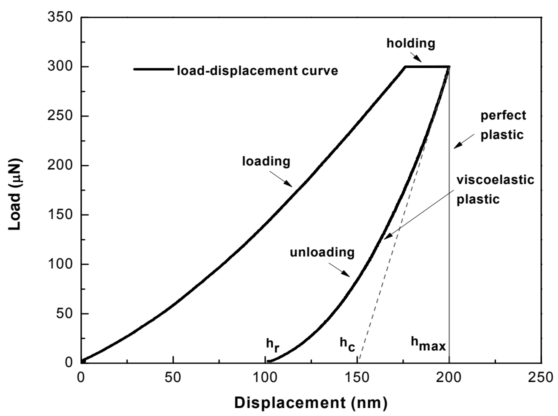


**Figure 2.** Typical load-displacement curve of POFs.

## 3. RESULTS AND DISCUSSION

### Surface roughness of specimens

For the investigation of topography of specimens, the scan size of all images is set as 5 μm. One example is given in Figure 3 and Table 2. The cladding is on the left-hand side in 2-D image, which is the right corner in 3-D image. There are some small peaks and valleys on the surface of specimen due to man-hand polishing processes. The values of nanoindentation depth should be greater than the root mean square surface roughness in order to minimize the influence of surface roughness on testing results (11). The root mean square surface roughness for each specimen is less than 70 nm in this contribution.

**Table 2.** Whole image statistics of latitudinal cross section

| Parameters | Values |
|---|---|
| Project area (μm$^2$) | 25 |
| RMS roughness ($R_q$) (nm) | 33.4153 |
| Average roughness ($R_a$) (nm) | 21.6935 |
| Mean height (nm) | 29.0533 |
| Max height (nm) | 94.1807 |
| Min height (nm) | -392.754 |
| Peak-to-valley (nm) | 486.934 |

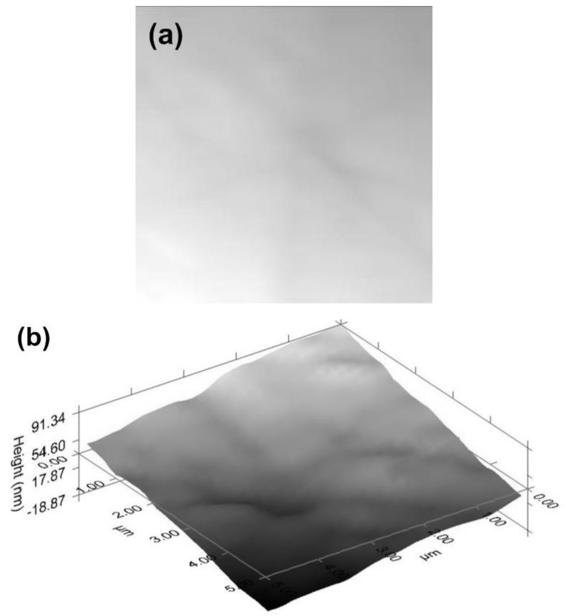


**Figure 3.** 2-D height image (a) and 3-D height image (b) of the latitudinal cross section of 0.5 mm diameter specimen.

### Loading rate effect on nanoindentation creep of POFs

It is visible that the cladding is softer than the core of 0.5 mm plastic optical fiber, as shown in Figure 4. The values of elastic modulus and hardness are decreased with increasing in the loading time. When the loading time is lower, the loading rate is higher, smaller indent is created in the end of holding period, which leads to greater hardness due to the smaller contact area according to Equation 3. With the higher loading rate, less creep happens during the loading period, and the creep phenomenon could remain in the subsequent unloading time when the elastic modulus is measured.

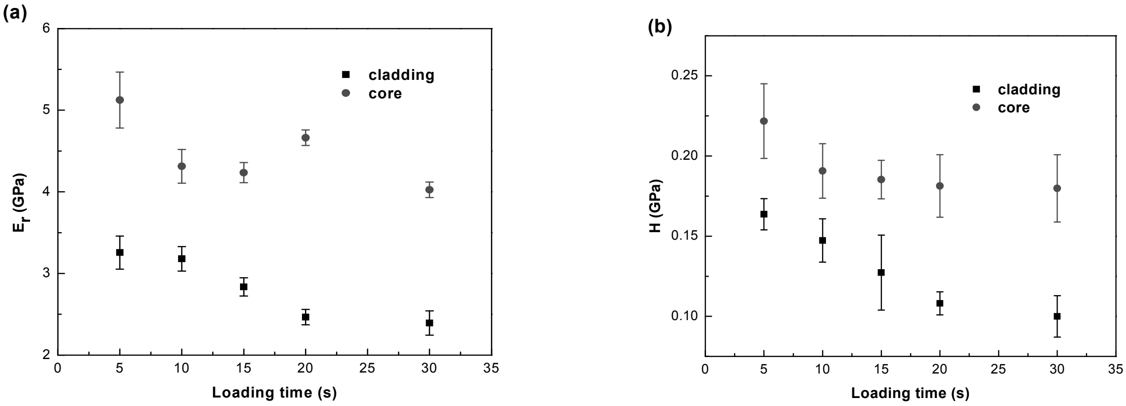


**Figure 4.** Elastic modulus (a) and hardness (b) of 0.5 mm POFs with 10 s holding time.

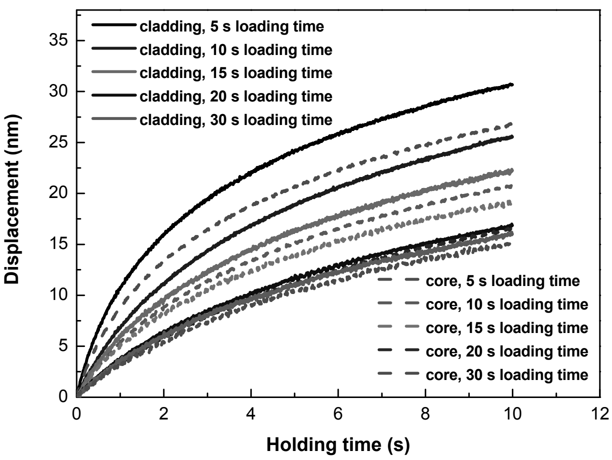


**Figure 5.** Displacement during holding period of 0.5 mm POF.

At the beginning stage of holding period corresponding to the transient creep in creep deformation curves during holding time in Figure 5, it is found that the displacement is increased faster than that of the following stage which shows a relatively stable displacement. Moreover, the higher the loading rate (lower loading time), the more the creep displacement. The reason behind it may be the strain rate with lower loading rate is lower, longer time is required to reach the maximum load, resulting in the creep deformation during the loading time (12). It could be also explained by the dislocation substructure formed beneath the indenter due to different indentation stress with various strain rates, while this substructure definitely plays a significant role in subsequent creep behavior (13).

### Holding time effect on nanoindentation creep of POFs

Compared with loading rate, holding time displays less sensitivity, as shown in Figure 6. Theoretically, hardness should be decreased with the increase of holding time due to the large contact area. The results with cladding at 15 s holding time one obviously imperfect, which might be explained by the manual polishing processes. As we know, it is more difficult to polish soft materials than hard ones (like metal). In present work, it is relatively easy for core to produce a smooth surface with less time, during which the cladding is uneven. It would lead to the unexpected data in final results.

Figure 7 shows that the creep deformation is increased according to the increment of holiday time for both cladding and core of 0,5 mm for. The cladding represent higher value due to the less stiffness of material.

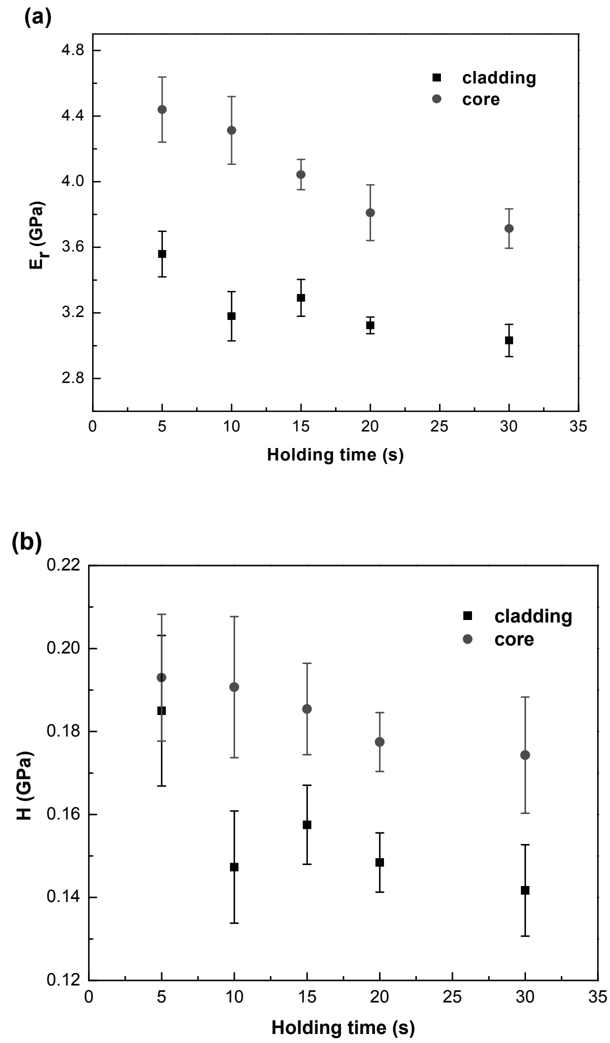


**Figure 6.** Elastic modulus (a) and hardness (b) of 0.5 mm POFs with 10s loading time.

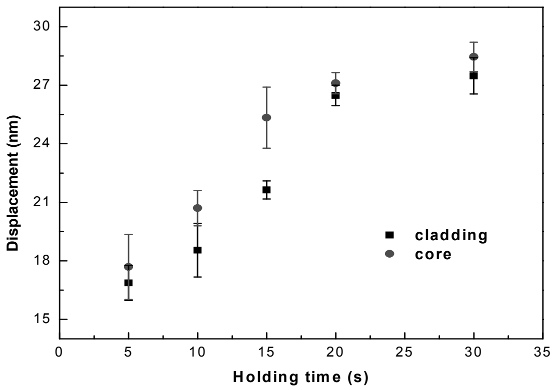


**Figure 7.** Displacement during holding period of 0.5 mm POF.

### Fiber diameter effect of plastic optical fiber

Figure 8 indicates that the fiber diameter plays a negative role in nanoindentation tests. Most of experimental data are higher with thinner fiber diameter. It is implied that the coarse fiber is much stiffer than fine one. The values in longitudinal cross section are greater than those in latitudinal direction. The reason behind it might be explained by the different surface roughness of longitudinal and latitudinal cross sections. The surfaces of fiber specimens are not absolutely smooth, as shown in Figure 9, which might affect the contact areas. The intrinsic properties in different cross sections and the limited testing times for each condition may also influence the results.

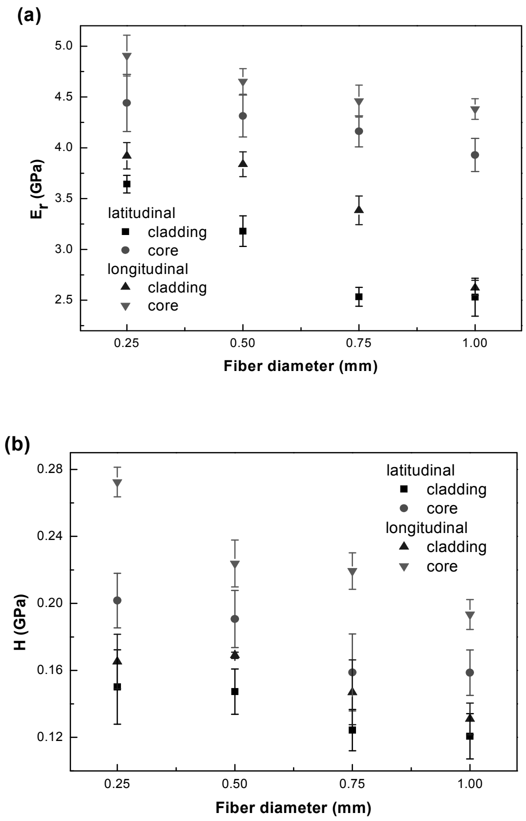


**Figure 8.** Comparison of elastic modulus (a) and hardness (b) of latitudinal and longitudinal cross sections of POFs with different diameters.

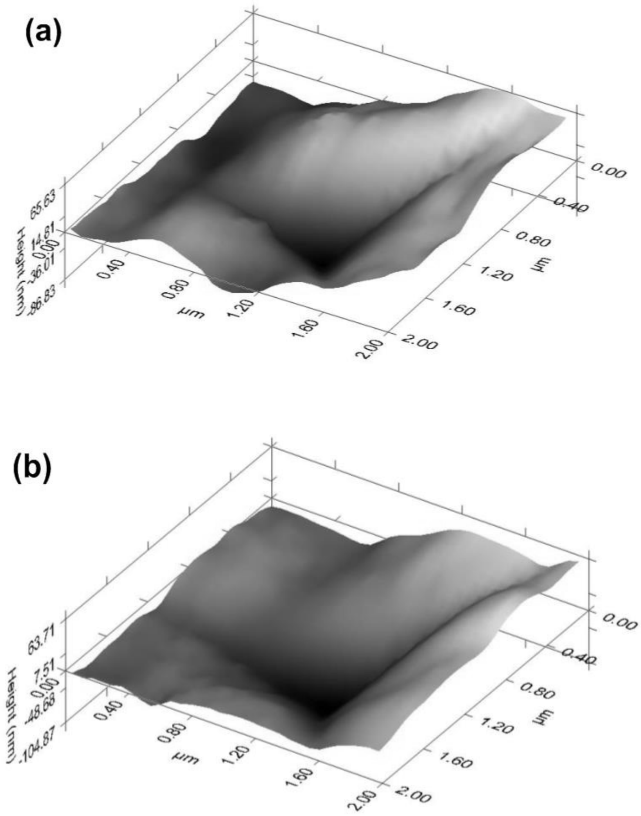


**Figure 9.** Indent images of longitudinal cross section of 0.5 mm POFs: (a) cladding and (b) core.

## 4. CONCLUSIONS

The mechanical properties of POFs applied in shining textiles should be taken into account in weaving, knitting and braiding processes. It is interesting to investigate the hardness properties of both core and cladding by nanoindentation.

This study presents the strong loading time sensitivity of nanoindentation performed upon the PMMA based POFs with different conditions. The holding time has less influence on nanoindentation creep than the holding time. It is also indicated that there is a normal tendency of the relationship between loading/holding condition and nanoindentation creep properties. It is found that the cladding of POF is softer than the core, which is particularly evidenced at various loading time. The fiber diameter plays a significant role in nanoindentation properties and declines as the hardness and modulus go up. Meanwhile, the longitudinal samples exhibit higher hardness and modulus than the latitudinal samples, which is probably resulted from the differences of surface roughness in latitudinal and longitudinal cross sections.

### Acknowledgements

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