NANOINDENTATION PROPERTIES OF PLASTIC OPTICAL FIBER INTERPHASE

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Abstract

Plastic optical fiber (POF) is a medium that can transfer light from one place to another place based on the total reflection theory according to the difference of refraction indices between fiber core and fiber cladding. When POF is utilized as the lighting element in textile structure (knits and jacquard wovens) in soft shining textiles which can active emit light, the mechanical properties should be taken into account since the bends in macro or micro leads to optical attenuation. Both POF core and POF cladding are made from polymeric materials and devoted to mechanical properties and illumination performance, which make the interphase properties between fiber core and fiber cladding critical in mechanical deformation, strength distribution and fracture mechanism. The interphase properties in POF were investigated by nanoindentation testing with various nanoindentation depths and spacings. 10-15 nanoindents in a line through the interphase zone were carried out from fiber cladding to fiber core. The hardness and elastic modulus in the transition region were measured and analysed. The results indicate that POF cladding is evidently softer than POF core due to the lower hardness. There is a gradient in the values of hardness and elastic modulus of cladding and core. The interphase width is estimated effectively with the minimal nanoindentation depth and sensitive spacing value, and is in the range of 800-1600 nm when the nanoindentation depth is 40 nm and spacing is 400 nm.

Keywords: Plastic optical fibers, nanoindentation, interphase, hardness, elastic modulus

1. INTRODUCTION

Traditional optical fiber (OF) is generally made of fiber core and cladding and served as the medium for light transmission from one place to another place [1]. The logical theory of light propagation in optical fiber is based on total internal reflection which takes place when the core refraction index is greater than the cladding refraction index and the incident angle is bigger than then critical angle in Snell’s law [2]. Plastic optical fiber (POF) was introduced in 1960s as the substitute of glass optical fibers in short distance communications links due to the requirement of a lot of connections [3] and is made from polymeric materials [4]. POF has been catching more attention gradually due to its advantages, such as light weight, cheap price, flexibility, large numerical aperture and easy connection. There are two main parts of its application in textile field, one is lighting textiles (like clothes design), and another is sensing textiles (like geo-textiles) [5]. The first developed textile product based on plastic optical fiber was introduced by SÄCHSISCHES TEXTIL PORSCHUNGS INSTITUTE in 2000 [5]. Nowadays, a lot of efforts have been made to explore its applications in textiles, for instant, the pattern design in soft lighting fabrics [6] and table cloth for decoration [7]. When POF is knitted or woven into textile structures, the mechanical deformation occurs and leads to the increase of optical loss which influences the lighting effect subsequently. POF core and POF cladding are made from different polymers. The properties of fiber core and fiber cladding, as well as the interphase between them in POF, which not only decide the light propagation in optical fiber, but also affect the mechanical properties of the whole fiber, hasn't reported in detail. Most of the studies of mechanical behavior of POF have focused on the attenuation induced by bends and tensile or torsion stresses [8, 9, 10]. In the studies on fracture mechanism and strength distribution, the contributions of core and cladding of POF is still obscure, which makes the interphase properties between core and cladding urgent to be investigated.

Nanoindentation technique is a promising method to investigate the mechanical properties (e.g. hardness, Young's modulus and stiffness) of materials in nanometer-scale displacement and smaller load range than other testing methods (e.g. DMA and Instron). Nanoindentation can be also utilized to figure out the interphase characteristics in reinforced composites [11, 12, 13]. The interphase between reinforced phase and matrix is a transition region, which usually ranges from nanometers to micros. Unlike the interphase of normal reinforced composites, the interphase of POF mainly contributes to the interval refraction of light inside the core. While the interphase properties have an effect on the mechanical and physical properties of the whole fiber more or less.

In this contribution, the interphase properties between POF core and cladding were investigated by nanoindentation. The latitudinal cross sections of specimens were tested with different nanoindentation depths and spacings between adjacent indents related to the surface roughness and the interaction between created stress field and plastic zones, respectively. The distinct gradients of results were analyzed to estimate the interphase width between fiber core and cladding.

2. EXPERIMENTAL

2.1 Material

The plastic optical fibers (POFs) with 0.5 mm diameter were purchased from Grace POF Co., Ltd., China. The basic properties of POFs were given in Table 1.

Table 1 Basic properties of plastic optical fibers

Basic Properties	Values	Basic Properties	Values
Product model	GDOF-S-020R	Numerical aperture	0.44
Core material	PMMA	Acceptance angle [°]	52.2
Cladding material	PMMA/Teflon	Storage temperature [°C]	-20 ~ +70
Diameter [mm]	0.5	Specific gravity [g/cm^3]	1.19
Core refraction index	1.49	Wavelength [nm]	400 ~ 780
Cladding refraction index	1.42	Bending radius limit	8 × fiber diameter

2.2 Preparation of specimens

The specimens for evaluation of interphase properties were prepared as follows:

A bundle of POFs were inserted into suitable cable which was put into appropriate holes of button for normal clothes. Super glue was used to fix the above three units together.

The cable with fibers inside was cut in both sides of the button and then polished with polishing papers until smooth enough. The smallest particle diameter of polishing papers was 1 micro. After that, the samples were fine-polished with W0.5 water-based diamond polishing paste. All polish processes were carried out in the clockwise direction by hand with the speed of 50 ~ 60 times per minute. The specimens of latitudinal cross section were prepared at last.

2.3 Nanoindentation tests

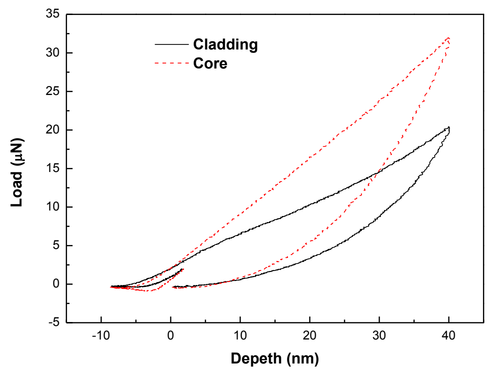
The nanoindentation tests were preceded by Hysitron with a three-side pyramidal Berkovich diamond indenter. The maximum nanoindentation depths were 120, 80, 40 nm and related spacings were set as 1900, 1300, 700 and 400 nm to avoid overlapping of plastic deformation zones between adjacent indents. Alma Hodzic et al. have reported that the plastic deformation zone resulted from the stress forms when the indenter goes inside the specimens [13]. The width of nanoindentation on specimens w is dependent on the nanoindentation depth h due to the geometric shape of Berkovich indenter [14],

$$w = 2h(\tan 65.3°)/(\tan 30°) = 7.532h \quad (1)$$

$$s = 2w \quad (2)$$

where 65.3° and 30° are the constant parameters of Berkovich indenter. *s* is the safe spacing that the adjacent plastic deformation zones will not be overlapped by each other. For instant, when the nanoindentation is 80 nm, the calculated nanoindentation width is 0.603 μm, the spacing is 1.205 μm, which means 1.3 μm is enough to avoid the overlapping of the plastic deformation zones.

Both loading time and unloading time were 10 s, as shown in Fig. 1. POFs were tested from cladding to core almost in the straight lines through the center of fiber by nanoindentation.



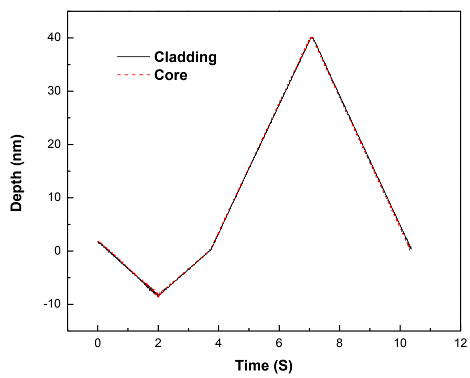


Fig. 1 Experimental design of POF core and cladding under 40 nm maximum depth, 400 nm spacing, 10 s loading time and 10 s holding time: typical load-depth curve (left) and depth-time curve (right).

Contact stiffness can be calculated from the slope of initial unloading curve. During the unloading period, only elastic deformation is recovered (Fig. 1). The dependence because stiffness and elastic modulus of specimen can be expressed by Equation (3),

$$S = \frac{dP}{dh} = \frac{2\beta}{\sqrt{\pi}} \cdot \sqrt{A} \cdot E_r \quad (3)$$

$$\frac{1}{E_r} = \frac{(1-v_s^2)}{E_s} + \frac{(1-v_i^2)}{E_i} \quad (4)$$

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 $24.5h_c^2$), *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 (4). *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).

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

$$H = \frac{P_{max}}{A} = \frac{P_{max}}{24.5h_c^2} \quad (5)$$

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

3. EXPERIMENTAL

3.1 Surface roughness

The Topography of one specimen is displayed in Fig. 2, the corresponding image statistics with 5 μm scan size is shown in Table 1. There are visible small hills and valleys on the surface in especially 3-D image, which could be explained by the intrinsic material properties. As we know, compared with metal, the polymeric material is relatively hard to be polished due to its visco-elastic properties. The polishing processes were finished by hand and might lead to the unevenness of sample surface.

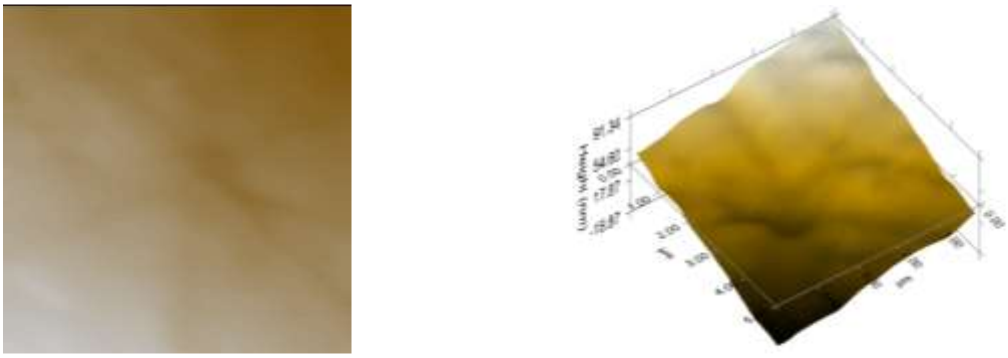


Fig. 2 2-D height image (left) and 3-D height image of sample surface(right)

In this investigation, all values of nanoindentation depth should be greater than the root mean square surface roughness which is 33.4153 nm in Table 2, in order to minimize the influence of surface roughness on testing results [12].

Table 1 Whole image statistics of latitudinal cross section

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

3.2 Local mechanical properties within interphase

According to the RMS surface roughness, 120 nm nanoindentation depth (approximately three times of RMS value) is completely sufficient to investigate the nanoindentation properties of samples. It is found from the results of the testing with 120 nm depth and 1900 nm spacing distance that there is no points connecting the lower values and higher values, which means, the interphase thickness between core and cladding is less than 1900 nm. Meanwhile, high position displays great values for both hardness and modulus that is indicated the fiber cladding is softer than the fiber core according to the testing position from cladding to core.

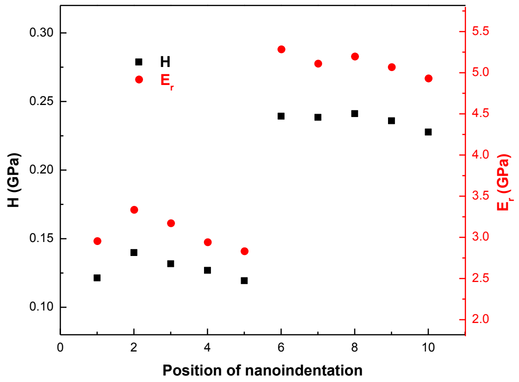
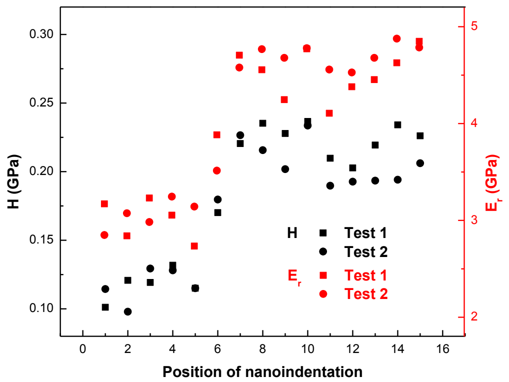


Fig. 3 Hardness and elastic modulus with 120 nm depth and 1900 nm spacing

When the nanoindentation depth is declined to 80 nm (approximately two times of RMS value), the spacing of any two adjacent indenters is about 1300 nm, calculated by Equations (1) and (2), there is only one evident point which builds the bridge between two different parts, as shown in Fig. 4 (left). Two possibilities for testing position are considered in the interphase region, firstly, only one indent is conducted probably near to the middle place of interphase region, that is to say, the interphase width is in the range of 0 ~ 2600 nm. Secondly, the indent might be near to the border of cladding or core, which implies the interphase width could be in the range of 0 ~ 1300 nm. However, it is still unable to predict the exact interphase width in this case. When the nanoindentation is tested near to the border of core, the plastic zone would be restricted due to the greater values of elastic modulus and hardness of core than those of cladding. To obtain relatively effective interphase width, the nanoindentation depth should be smaller than 80 nm.



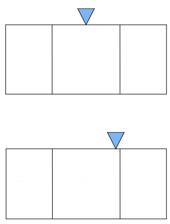


Fig. 4 Hardness and elastic modulus with 80 nm depth and 1300 nm spacing (left); Possibilities of one indent in the interphase region (right)

When the nanoindentation depth is set as 40 nm (near to the RMS value), the spacing between each two adjacent nanoindentation is about 700 nm, two indents in transition zone could be found in Fig. 5 (left). Apparently, there are three possibilities for testing position described in Fig. 5 (right). According the above results, it is estimated that the interphase width is in the range of 700 ~ 1900 nm, which is still not satisfied enough with the minimum nanoindentation depth.

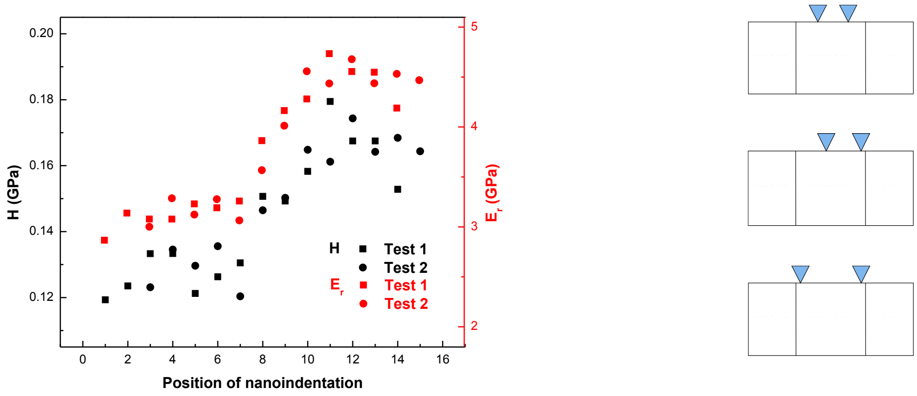


Fig. 5 Hardness and elastic modulus with 40 nm depth and 700 nm spacing (left); Possibilities of two indents in the interphase region (right)

When nanoindentation depth is 40 nm, the nanoindentation width is 302 nm from Equation (1), which means, the minimum safe spacing to avoid the overlapping of the plastic zones between each two adjacent indents is 608 nm (double of nanoindentation width). If the spacing is 302 nm, each two adjacent indents would be connected rather than overlapped, while the plastic zones would be definitely overlapped. In this case, the overlapped plastic zones might give inaccurate results. The spacing should be greater than 304 nm. To predict more effective interphase width, the minimum nanoindentation depth and sensitive spacing are considered.

The results with 40 nm nanoindentation depth and 400 nm nanoindentation spacing are summarized in Fig. 6 (left). There are three indents in the transition zone and two possibilities of testing position in the interphase region, as shown in Fig. 6 (right). If the second case happens, that is, two indents are carried out in the borders of cladding and core, the minimal interphase thickness is 800 nm. Based on the above results, the interphase width is estimated in the range of 800 ~ 1600 nm.

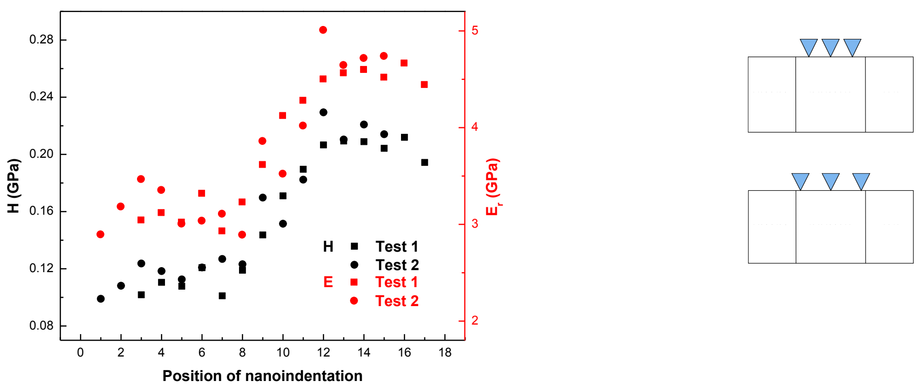


Fig. 6 Hardness and elastic modulus with 40 nm depth and 400 nm spacing (left); Possibilities of three indents in the interphase region (right)

CONCLUSION

Nanoindentation is a useful method to investigate the interphase properties of POFs, the nanoindentation testing displays that the POF core is relatively stiffer than POF cladding. The interphase thickness can be estimated with the minimum nanoindentation depth due to the value of surface roughness, and sensitive spacing between each two neighboring indents according to the overlap of plastic zones for adjacent indents. It is predicted that the interphase width might be in the range of 800 ~ 1600 nm with 40 nm nanoindentation depth and 400 nm spacing, which might benefit to the studies on mechanical deformation, strength distribution and fracture mechanism of POFs. Other techniques such as nanoscach testing could be also applied to measure the interphase properties of POFs [12, 13].

Textile structures based on POFs can be widely used in lighting decoration and technical textiles, the further study about the illumination and mechanical properties of POFs are necessary and urgent.

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