

# EFFECT OF ZnO NANOMATERIALS COATED GLASS GROWTH DURATION TO THE ADJACENT MICROFIBER LIGHT TRANSMISSION AT ROOM TEMPERATURE

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## Abstract

This paper reported the effect of ZnO nanorods growth time to the light transmission of the silica optical fiber. It was conducted by placing silica microfiber on a glass surface coated with Zinc Oxide (ZnO) nanorods. The microfiber was initially tapered into waist diameters of 10  $\mu\text{m}$  using flame brushing technique. Then, the glass substrates were coated with ZnO nanorods for 6 hours, 9 hours, 12 hours, 15 hours, 18 hours and 21 hours of growth time using hydrothermal synthesis technique. Amplified Spontaneous Emission was launched at one end of the microfiber and the other end was connected to the Optical Power Meter to observe the output light transmission behaviour. The samples were tested at room temperature to observe several performance items such as transmitted power, transmission loss and scattering coefficient. Based on the experimental results, it was found that 15-hours growth sample produce the best results as compared to the results as compared to the other growth time samples in term of transmitted power, transmission loss and normalize scattering coefficient. The behaviour of the light transmission changes in different growth time samples. This work could be benefitted in design consideration for various sensing application at room temperature.

## Keywords

Microfiber, ZnO Nanorods, Hydrothermal

## 1. Introduction

Silica optical fiber become more responsive to the outside environment when the waist diameter reduces below the core diameter. This is due to the guided light produced large fraction evanescent wave outside the microfiber and varies with the refractive index change at the surrounding medium. It would change the transmitted light behaviour inside the microfiber in term of optical phase and intensity [1]. Microfiber also exhibit unique characteristics such as low manufacturing cost, tight optical confinement [2], large evanescent field [3] and field enhancement [4].

The light coupled into the cladding modes could be modified by changing the refractive index of the coating material. Zinc oxide (ZnO) is one of favourite nanomaterial used as coating material. It is an n-type semiconductor with band gap energy is 3.37 eV and have special attribute such as room temperature ferromagnetism, piezoelectric behaviour and huge magneto-optic [5, 6]. It also changes the electrical conductivity which modifies the complex refractive index of the ZnO nanostructures [7]. Thus affecting the optical scattering of the light penetrate into the ZnO nanorods [8]. Growth duration is the most important factor effecting the optical characteristic of the ZnO nanorods because it change the nanorods dimension such as diameter, density and length [9]. These parameters would change the light scattering inside the ZnO nanorods. Microfiber exhibit good evanescent coupling capability with other waveguides such as glass substrates. The purpose of the new structure is to solve issue of striving to handle the directly coated microfiber during the synthesis and sensing application. This would lead to variation in transmitted light inside the microfiber when laid on the nanorods coated glass. This report investigated the effect of nanorods coated glass growth duration to the light transmission

through the adjacent microfiber according to the specific performance parameters. The performance parameters employed in this work are transmitted light, transmission loss and the scattering coefficient. These parameters are essential in sensing applications because higher transmission loss and scattering coefficient would translate to a better sensing response.

## 2. Theory

The behaviour of the light transmission when travelling through the sensitive material is accordance to the Lambert-Beer law. It states that the attenuation of light is relied to the properties of the material through which the light is travelling. The light intensity inside the microfiber is depended on the portion of total fraction power of the evanescent field and absorption coefficient of the sensitive material. The formulation of the law mechanism is shown in the following Equation 1 [10]:

$$I = I_0 e^{-\alpha L} \quad (\text{Eq. 1})$$

Where  $I$  is the amount of light leaving after the interaction with the sensitive material,  $I_0$  is the amount of light entering before interaction with the sensitive material,  $\alpha$  is the scattering coefficient and  $L$  is the length of the sensing region on the glass substrates (for this work is 7.6 cm). When light transmits through materials, the strength of the light transmitted would be reduced by absorption and scattering phenomenon which is also called extinction. Scattering occurs when incident light interacts with the atom of the nanomaterial. The atom will be excited to a higher energy level and instantly drops to its original level and emits a photon at the same frequency as the one it absorbed. It occurs normally when there are refractive index mismatches at boundaries [11]. The scattering coefficient ( $\alpha$ ) can be derived from Equation 1 to become as:

$$\alpha = \frac{-\ln\left(\frac{I}{I_0}\right)}{L} \quad (\text{Eq. 2})$$

## 3. Fabrication and Experimental Setup

Diameter of single-mode fiber (Corning SMF-28, USA) was reduced into a waist diameter of  $\sim 10 \mu\text{m}$  using flame brushing technique as shown in Figure 1. The hydrothermal synthesis process of the ZnO nanorods was conducted onto microscope glass surface (Heathrow Scientific LLC, USA) according to our previous report [12]. It starts with ultrasonic cleaning process and water bath procedure for 15 minutes. Then, it was placed in an oven at  $90^\circ\text{C}$  for 1 hour to remove organic material. Then, the seeding process took place to form a 1 mM solution at temperature of  $60^\circ\text{C}$ . In order to increase pH in alkaline, aliquots of sodium hydroxide pellets [NaOH] (Friendemann Schmidt Chemical, Germany) was added to the solution [13]. Subsequently, ZnO growth solution was prepared where zinc nitrate hexahydrate [ $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ] (Sigma-Aldrich) and hexamethyleneteramine or HMT [ $(\text{CH}_2)_6\text{N}_4$ ] (Sigma-Aldrich) were dissolved in 500 mL of deionized (DI) water to form 10 mM aqueous solution. The seeded glass substrates were then placed in the solution and heated in an oven at temperature of  $90^\circ\text{C}$ . In the experiment, the ZnO nanorods were grown for 6 hours, 9 hours, 12 hours, 15 hours, 18 hours and 21 hours.

After the synthesis process completed, Field Emission Scanning electron microscopy (FESEM) was performed to observe the structure of ZnO nanorods growth on the glass substrates. It was conducted together with Energy Dispersive X-ray (EDX) to identify the chemical elements of the samples. Figure 2 illustrated the experimental setup of the humidity sensing. The sample was connected to Amplified Spontaneous Emission (ASE) from an erbium doped fiber amplifier (EDFA) at one end of the microfiber and the other end was connected to the Optical Power Meter (Thorlab) to collect the output data. It is placed in a constant room temperature condition (27°C) where the nanorods coated surface was directed upwards. The temperature was maintained at a constant room temperature to avoid variation of the light transmission. Based on the literature, the amount of absorbed water molecules from the surrounding reduces when temperature raised due to entropy increase of water molecules [14]. This would slightly change the refractive index and effect the light transmission. In order to monitor the temperature level around the sample's surface, the thermocouple (Hygrometer RS 1365, Sensitivity: 1%) was placed as close as possible to the proposed sensor [15].

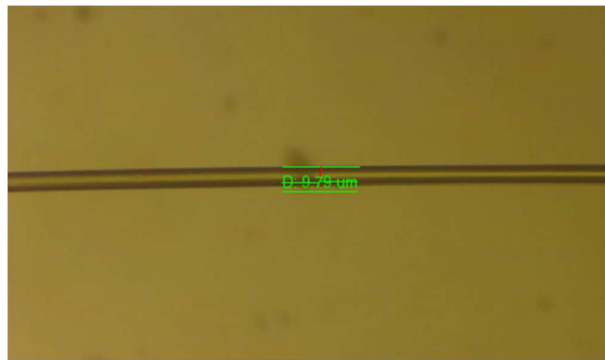


Figure 1. Microfiber waist diameter of 10μm

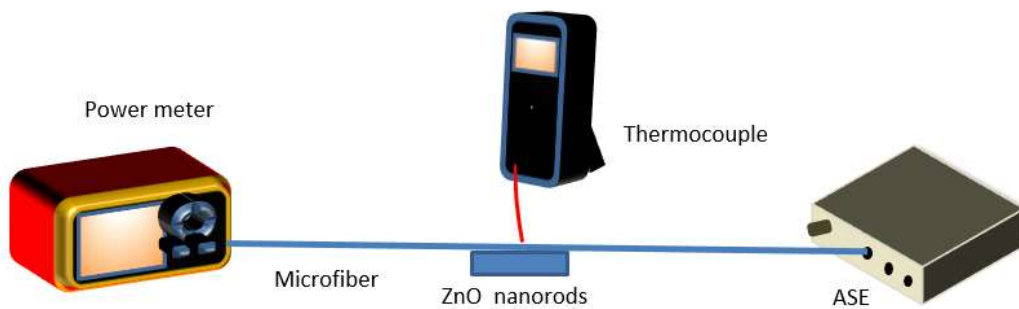


Figure 2. Experimental setup

**4. Results and Discussion**

Figure 3 illustrates the FESEM image of 15 hours growth time sample at 10.00 kX magnification and the EDX elemental analysis of the coating layers. It shows that the element on the glass consist of only zinc and oxygen. The transmitted power after light travel through

the coating material at different growth time is shown in Figure 4. It is noteworthy to mention that ZnO nanorods acted as a changeable refractive index layer resulting light absorption inside the coating layer. Consequently, the transmitted light decreased steadily due to high light refraction into ZnO nanorods coating layer [16]. Based on [17], forward scattering would dominate with longer and higher density nanorods while backward scattering dominates with shorter and lower density nanorods. The forward scattering would decrease the light transmission through the microfiber while backward scattering increase the light transmission. Therefore, the best trade-off between these nanostructures need to be determine. Based on [11], nanorods density reduced when the growth time increase and nanorods lengths increased when the growth time increase. Therefore, based on results in Figure 4, the 15 hours growth time sample has the best trade-off between the rods length and density which produce the lowest transmitted light value.

Figure 5 shows the transmission loss of all samples. It shows that the highest power losses occur for 15 hours growth time sample. As aforementioned, this is because 15 hours growth time times has the best trade-off between rods density and length which cause lesser barrier of light absorption inside the nanorods as reported in [18]. It was optimal in limiting backscattering and enhancing forward scattering. Besides, ZnO has higher thermal expansion coefficient with  $4.75 \times 10^{-6} \text{ K}^{-1}$  as compared to silica with  $0.55 \times 10^{-6} \text{ K}^{-1}$  which absorbed more evanescent wave from the microfiber into the nanorods [17]. Thus the sample produce the highest light absorption inside ZnO nanorods which translate into highest transmission loss.

Scattering coefficient ( $\alpha$ ) was calculated based on Equation 2. Figure 6 shows that the 15 hours growth time sample produce the highest normalize  $\alpha$ . When the structure exposed to the air, the water molecules chemisorbed on the ZnO nanorods surface due to weak hydrogen bonding [19]. Evanescent waves absorbance relied to the surrounding analyte if other parameters are known [20]. Normalize  $\alpha$  occur due to the combined effect of forward scattering and surface absorption into the ZnO nanorods. As for 15 hours growth sample, it is the optimal sample that encourage more evanescent field appearance around sensitive region and resulting in more light coupling into the ZnO nanorods waveguides. Thus, the sample exhibit the highest normalize  $\alpha$  due to its optimum nanorods physical dimension that allow more light absorption inside the nanorods [17].

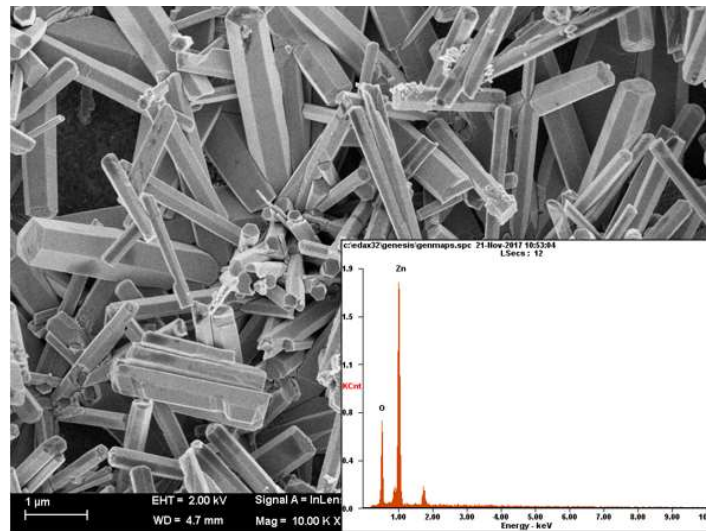


Figure 3. FESEM image and EDX graph of the 15 hours growth time sample

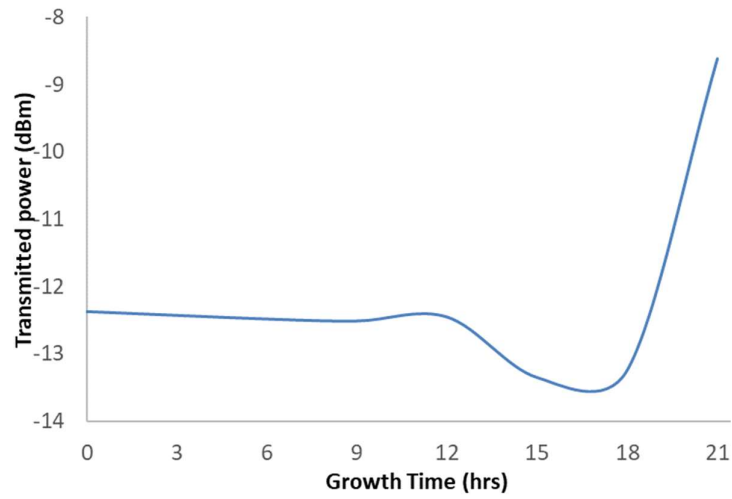


Figure 4. Transmitted power at the output of the structure

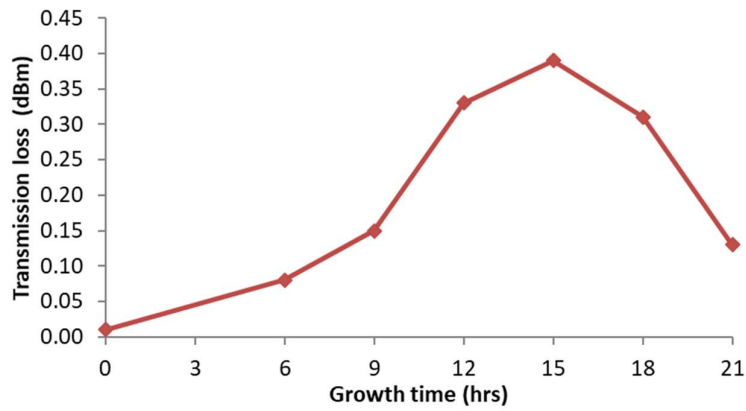


Figure 5. Transmission loss at the output of the structure

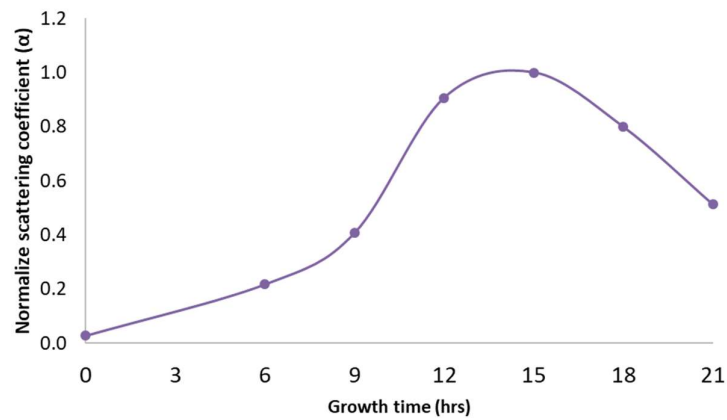


Figure 6. Normalize scattering coefficient of the structure

## 5. Conclusion

We have successfully analysed the effect of ZnO nanorods growth time to the light transmission of the proposed structure. The structure was designed by placing silica microfiber on a ZnO nanorods coated glass surface. The analysis was conducted by investigating several performance parameters such as transmitted power, transmission loss and scattering coefficient ( $\alpha$ ) which was tested at room temperature. Based on the experimental results, it was found that 15-hours growth sample produce an optimum result as compared to the other samples. The behaviour of the light transmission changes in different growth time samples. This is due to different morphological structures produced with different growth time. Thus modifies the light scattering and the light absorption characteristic inside the nanorods. This work could be considered in designing various sensing application at room temperature.

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