Evaluation of light backscattering by zinc oxide nanorods on fiber end of silica optical fiber

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Light backscattering by Zinc Oxide (ZnO) nanorods at different growth duration is investigated. Synthesis of ZnO nanorods was done using hydrothermal techniques on the end of silica optical fiber (SOF), the growth duration of nanorods is varied at 4, 6 and 8 hrs. Optical and physical characterization was accomplished to validate the light backscattering and morphology of ZnO. Optical characterization was performed by measuring the output power of the backscattered light, the result indicates that 8 hrs growth duration is optimum to achieve the highest backscattering. The sample with the optimum growth time was analyzed using the field emission scanning electron microscopy (FESEM) image and electron dispersive spectrum (EDS) result. Both results confirmed the presence of ZnO on the end of silica fiber.

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Keywords: ZnO nanorods, Hydrothermal technique, Light backscattering

1. Introduction

Research in light scattering has started in the late 19th century to determine the size of particles. Now, the rapid development of light scattering extended the application widely especially in the astronomy, oceanography, photographic science, meteorology and coatings technology [1]. Light scattering is a phenomenon where light are randomly change its direction by one or more path due to the irregularities in the medium through which they pass. Light scattering comprises of two magnitude direction which are forward scattering and backscattering. In forward scattering, the direction of light scattering radiation are same as the light is pass through the medium (diffraction). In other hand, backscattering is a reflection of scattering radiation from the medium to the direction which they came. Light backscattering is important in some applications especially in optical communication to characterize irregularities and imperfections in the optical fiber [2] by measuring the reflection of light corresponding to the position along the fiber once the light is hit the irregularities surface.

Zinc Oxide (ZnO) have attracted the increasing attention to study the creation of ZnO nanostructures, growth mechanism and the investigation of their structural, optical and electronics properties [3-4]. ZnO has a wide bandgap (3.37 eV), high excitation energy (60 meV) and high thermal stability make it attractive for various applications especially in optoelectronics and optical sensor [5-7]. ZnO can be synthesized using two techniques either in gas phase or chemical phase [8]. Gas phase techniques normally carried out in high temperature from 500 °C to 1500 °C using gaseous environment in a closed chambers. Chemical phase techniques are carried using a chemical solution for example sol-gel, hydrothermal and controlled precipitation [9]. But, hydrothermal synthesis is the simplest, less hazardous, low temperature process and easy to control the morphology. To control the morphology of ZnO, several parameter need to adjusted including concentration of seeding solution, growth duration, synthesis temperature, pH of seeding solution and how the seeding layer deposited [10].

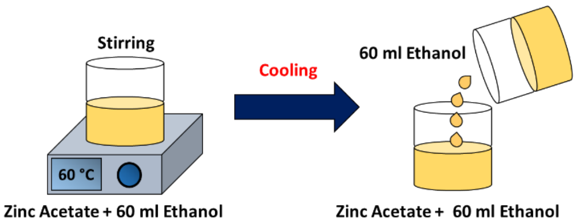
Study on light backscattering by ZnO has started using the colloidal suspension to study the optical bahaviour of ZnO. Krishnan et al reported that, concentration and size particles in the ZnO colloidal suspension can affected the backscattering of light by means, backscattering can be increased by increasing the concentration and size of particles [11]. In ZnO nanorods, the amount of light backscattered are depends on the several factor: diameter, length and number of nanorods. This factor can serve to increase the multiple scattering of ZnO nanorods and enhanced the backscattering where the factor need to be controlled especially in the concentration of seeding solution, annealing time and growth duration [12]

The present study focuses on the hydrothermal synthesis of ZnO nanorods on the tip of silica optical fiber (SOF) and characterization of ZnO nanorods to study the light backscattering of different growth duration. Two characterization was conducted: optical and physical characterization. In optical characterization, measurement system was develop to measure the light backscattering. For physical characterization, field emission scanning electron microscopy (FESEM) and electron dispersive spectrum was performed to study the morphology and confirm the presence of ZnO.

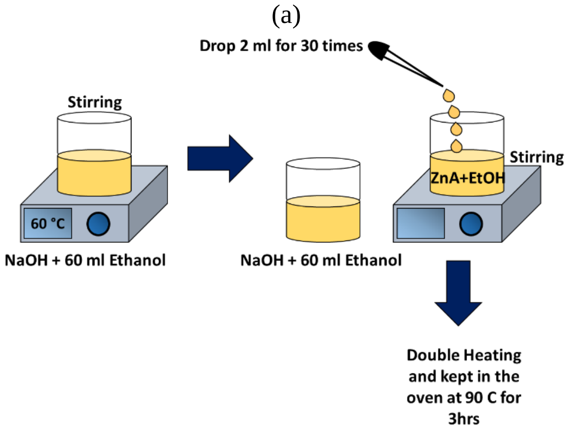
2. Synthesis of ZnO Nanorods

Fiber preparation: In the experiment, standard single mode silica fiber of length 40 cm with core and cladding diameter of 9 µm and 125 µm, respectively used. The small part of fiber tip was stripped and then cleaned by dry tissue and isopropanol, followed by steady flow of DI water. The area outside of 5 cm away from the end of the fiber was covered with electrical tape to ensure the ZnO only growth on the tip. The tape was removed at the end of the synthesis.

ZnO Seeding Procedure: ZnO seeding solution was prepared by dissolved 0.0132 g of zinc acetate dihydrate [$Zn(O_2CCH_3)_2 \cdot 2H_2O$, Merck] into 60 ml of ethanol to form 1 mM solution under stirring condition at 60 °C, and then the solution was left cooled at ambient for 5 min. After cooling, 60 ml of pure ethanol was added into the zinc acetate solution. 0.0016 g of sodium hydroxide [NaOH, Merck] in 60 ml ethanol was then added to zinc acetate solution using pipette, 2 ml in every minutes for 30 times with slow stirring condition. Then, the seeding solution was kept in a water bath at 60 °C for 3 hrs. The pH condition of the seeding solution are in range pH 8 was checked using electronic pH meter. White precipitate is formed after 3 hrs in the water bath show the existence of ZnO nanoparticles in the seeding solution. In order to provide the nucleation site for growth process, ZnO seeding solution was dropped on the tip of SOF using pipette and dry for 2 min at 60 °C. The process are repeated for 10 times. After the drop and dry process, the SOF was left for annealing process at temperature 90 °C for 3 hrs. Fig. 1 depicts the preparation of seeding solution.



(a)



(b)

Fig. 1. Preparation of seeding solution (a) Dissolving zinc acetate dihydrate under slow stirring condition (b) Adding $Zn(O_2CCH_3)_2$ solution into sodium hydroxide in 60 ml of ethanol using pipette with slow stirring

ZnO Growth Procedure: 10 mM growth solution of 1.4019 g of hexamethylenetetramine [$(CH_2)N_4$, Sigma-Aldrich] and 2.9747 g zinc nitrate hexahydrate [$Zn(NO_3)_2 \cdot 6H_2O$, Ajax Finechem Pty Ltd] was dissolved in 1000 ml of deionized water. Then, the SOF was dipped into 300 ml of growth solution and heated in the oven at 90 °C as shown in Fig. 2. The growth duration was varied: 4, 6 and 8 hrs to study the light backscattering by ZnO nanorods. The solution was replenished every 5hrs to maintain a constant growth rate.

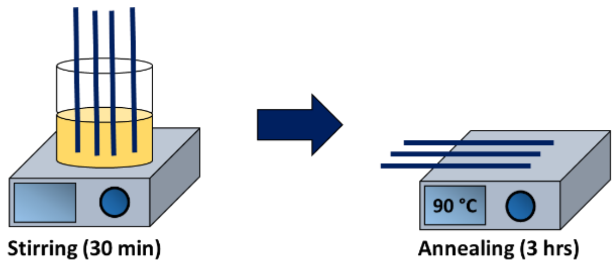


Fig. 2. Dipping with slow stirring to provide nucleation site on the end of SOF

3. Characterization of ZnO nanorods

The optical characterization was performed by measuring the light reflection output power from the SOF coated with ZnO nanorods with three different growth duration: 4, 6 and 8 hrs. Fig. 3 shows the optical system for the measurement of light backscattering by ZnO nanorods. 1550 nm ASE light was launched into the silica fiber and measuring the backscattered light using an optical spectrum analyzer (OSA). The ASE light was obtained by pumping and EDF using 980 nm laser diode. The backscattered light by ZnO nanorods on the fiber end was circulated into the OSA using an optical circulator, which is located in between the light source and fiber sample.

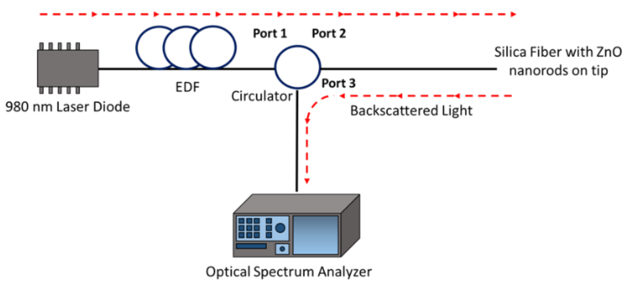
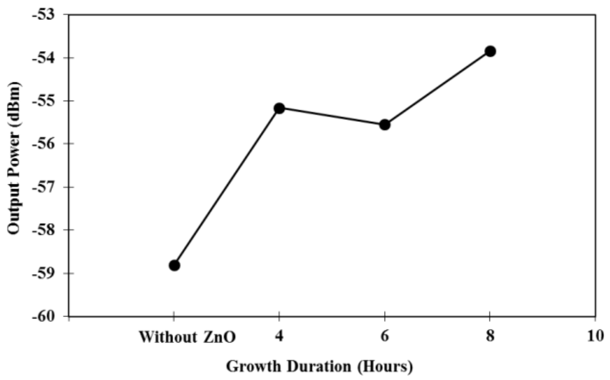
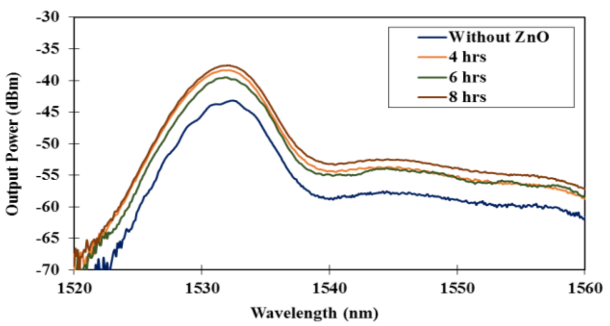


Fig. 3. Schematic configuration of light backscattering measurement for optical characterization

Growth duration during ZnO synthesis process is an important parameter to optimize light backscattering by ZnO nanorods in which affected by the length, diameter and number of nanorods. Fig. 5(a) shows the reflection output power at 1550 nm against different growth time and without presence of ZnO nanorods. It shows that the output power of backscattering light increases with the growth time. The highest output power can be achieved at 8 hrs growth time where the highest backscattering light was contributed by the increment in length, diameter and concentration of ZnO nanorods on the end SOF surface which can also enhances the multiple scattering. As the light is beamed through the ZnO nanorods structure, it encounters to produce a random scattering in many directions. A multiple scattering is taking place as the light hit the neighboring nanorods, which increases the intensity of light travelling through a certain scattering path. Some of the scattering light will be in backward direction path and at the same time most of the path are in forward direction. Concentration of nanorods are play important parameter influence the light backscattering. If the concentration of nanorods on tip of SOF increases, multiple scattering can be enhanced. Fig. 5(b) shows the backscattering phenomenon that happens in the ZnO nanorods. The light backscattering output power is expecting to be reduced as the growth duration increased, due to the increasing the diameter of nanorods and limits the concentration or number of nanorods on the tip of SOF.



(a)



(b)

Fig. 4. (a) Output power against growth duration at 1550 nm wavelength, (b) Backscattered output spectra without presence of ZnO nanorods and presence of ZnO nanorods at growth duration 4, 6 and 8 hrs

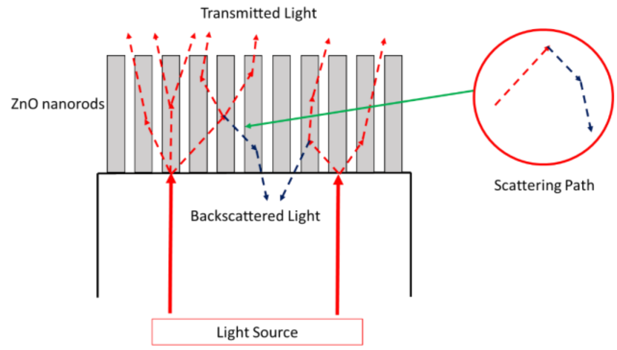


Fig. 5. Backscattering light generation with the presence of ZnO nanorods

The physical characterization was carried out for the sample with 8 hrs growth where the morphology image of ZnO was taken to confirm the presence of nanorods on the end of SOF. The morphology of the ZnO nanorods was observed by field emission scanning electron microscopy (FESEM) and the results are shown in Fig. 6. Figs. 6 (a) and (b) show the image with low and high magnifications, respectively. Both figures indicate that high concentration of the nanorods are successfully growth on the fiber end. Fig. 6 (c) depicts the side view of the nanorods with high magnification.

The material composition inside the nanorods structure was investigated by energy dispersive spectroscopy (EDS). Fig. 7 shows the EDS analysis result for the ZnO nanorods sample, which indicates the high peaks at 0.5 and 1KeV that correspond to high concentration of O and Zn elements. The EDS analysis shows the concentrations of Zn and O elements are 50.83 % and 23.12 %, respectively. The Si and C elements due to the material compositions of silica fiber show lower counts. The reasonably high C concentration is most probably attributed to the use of carbon tape during attachment of sample onto the specimen holder. The silica concentration comes from the SOF.

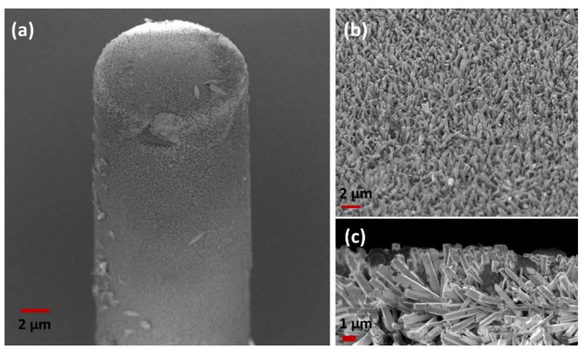


Fig. 6. (a) Low magnification view of ZnO nanorods, (b) and (c) view from to and side view at end of SOF surface with high magnification SEM

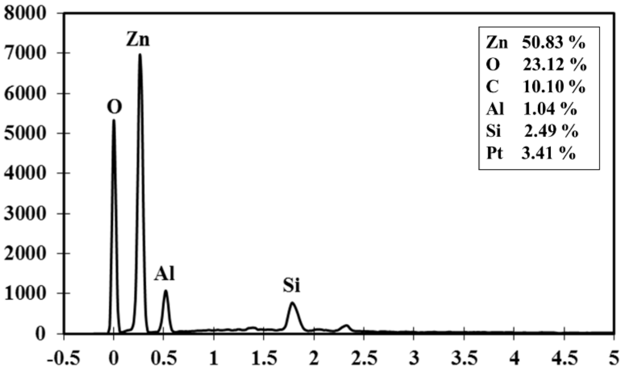


Fig. 7. EDS spectrum of ZnO nanorods on tip of silica fiber

4. Conclusion

The light backscattering by ZnO nanorods, which were coated onto a tip of silica optical fiber, was successfully demonstrated. ZnO nanorods was synthesized using the low temperature hydrothermal techniques which can promised the high concentration of nanorods on the fiber end. The ZnO sample was characterized to evaluate its optical and physical properties. In optical characterization, optical measurement was done to characterize and select the highest backscattering sample and the result shows that 8 hrs growth time can achieve the highest backscattering. The morphology of ZnO has been obtained for the sample with 8 hrs growth time and the presence of ZnO was confirmed from the FESEM and EDS results.

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