GROWTH OF ZINC OXIDE NANO-RODS ON OPTICAL FIBERS FOR OPTICAL INTERCONNECTS AND SIDE COUPLING APPLICATIONS

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ABSTRACT

The thesis introduces a novel concept that utilizes the scattering properties of Zinc Oxide (ZnO) nanorods to control light guidance and leakage inside optical fibers. ZnO rods scatter the light at larger angle than the critical angle inside the fiber. Hence, part of the incident light is coupled into fiber modes. A dense and highly ordered array of ZnO NR's is grown onto the cladding surface of the fiber using a simple low temperature hydrothermal technique, which involves two processes: seeding nanoparticles and growth of the nanorods. Nanorods length and density are found to be the most crucial parameters for the optimum side coupling. These parameters are controlled through the seeding and growth time to maximize the light side coupling to the cladding modes. Optimums seeding of nanoparticles concentrations are 1 mM zinc acetate, 10 mM of zinc nitrate and 10 mM of hexamine. The optimum rods growth time was 8 hour in oven. Maximum excitation of the cladding mode by side coupling of light was obtained with nanorods of length $\sim 2.2 \mu m$, demonstrating average coupling efficiency of $\sim 2.65\%$. To increase coupling efficiency though core modes excitation, the fiber cladding is chemically etched to allow light to reach the core region. Growth of ZnO NR's on the etched region allows the collection of light incident angles as large as 90 degrees to scatter inside the core region. This combination of nanostructures and fiber systems is used to demonstrate a simple wide field of view (FOV) optical receiver. The angular response of the receiver is tested using an in-house built nephelometer. Light coupling efficiency is extracted by de-convolving the finite beam extinction from the measured power. The results were compared to the first order analytical model where the phase function is assumed to linearly shift with the incident angle. The trend of the experimental measurements agrees with the model. 180° FOV is verified and maximum coupling efficiency of around 2.5% for a single fiber is reported. Excitation of core

modes through side coupling allows for application of these devices towards wide FOV optical receivers.

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ABSTRAK

Kerja yang dibentangkan di sini memperkenalkan satu konsep baru yang menggunakan ciri-ciri penyerakan nanorod zink oksida untuk mengawal pemanduan cahaya dan kebocoran di dalam gentian optik yang disalut dengan nanorod.Rod zink oksida menyerak cahaya pada sudut yang besar daripada sudut genting di dalam gentian.Oleh sebab itu, sebahagian pelanggaran cahaya digandingkan ke dalam mod gentian. Satu kepadatan dan susunan yang amat teratur nanorod zink oksida ditumbuhkan pada gentian menggunakan proses hidroterma bersuhu rendah. Pertumbuhan hidroterma adalah kaedah yang ringkas dan kos rendah yang mana tidak memerlukan vakum dan sistem yang rumit. Proses tersebut mempunyai dua bahagian: pembenihan nanopartikal dan pertumbuhan nanorod. Apabila penyerakan cahaya di dalam gentian, panjang nanorod dan kepadatan didapati sebagai parameter yang penting untuk mengoptimum gandingan sisi.Dua parameters tersebut dikawal melalui pembenihan dan masa pertumbuhan.Kesan pertumbuhan nanorod pada penyerakan cahaya dan gandingan kepada gentian optik diselidiki secara eksperimen.Pertama sekali, process tersebut dioptimumkan untuk memaksimumkan gandingan kepada mod pelapisan.Optimum pembenihan nanopartikal dengan kepekatan 1 mM zink asetat, 10 mM zink nitrat dan 10 mM hexamine dilaporkan. Masa pertumbuhan rod tersebut juga dioptimumkan, di mana 8 jam pertumbuhan di dalam oven didapati mencapai kuasa gandingan yang tinggi kepada pelapisan mod. Pengujaan maksimum mod pelapisan oleh gandingan sisi cahaya ditentukan dengan nanorod ZnO dengan panjang ~2.2 µm, menunjukan purata kecekapan gandingan ialah ~2.65%.Untuk meningkatkan kecekapan melalui pengujaan mod teras, pelapisan gentian tersebut ditanggalkan untuk membenarkan cahaya sampai ke bahagian teras.Pertumbuhan nanorod ZnO pada gentian yang dinipiskan secara kimia membenarkan pengumpulan sudut-sudut tuju cahaya pada 90 darjah untuk menyerak di dalam bahagian teras. Kombinasi nanostruktur dan system-sistem gentian digunakan untuk menunjukan satu penerima gentian pandangan medan luas. Sambutan sudut penerima diuji menggunakan satu nephelometer yang dibina di dalam makmal.Kecekapan gandingan cahaya diekstrak oleh penggabungan semula kepupusan rasuk terhingga daripada kuasa yang diukur. Keputusan-keputusan dibandingkan dengan model analisis peringkat pertama di mana fungsi fasa dianggap beranjak secara linear dengan sudut tuju.Trend pengukuran eksperimentasi bersetuju dengan model tersebut. 180° FOV disahkan dan maksimum kecekapan gandingan adalah sekitar 2.5% untuk satu gentian tunggal dilaporkan. Pengujaan mod teras melalui gandingan sisi membolehkan penggunaan alat-alat ini ke arah penerima-penerima gentian pandangan medan luas.

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DEDICATION

This thesis dedicate

To my parents

And well-wishers

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LIST OF SYMBOLS AND ABBREVIATIONS

0D	Zero dimension
1D	One dimension
2D	Two dimension
3D	Three dimension
ZnO NR's	Zinc oxide nanorods
UV	Ultraviolet
ZAH	Zinc acetate hydrate
NaOH	Sodium Hydroxide
PVD	Physical vapor deposition
CVD	Chemical vapor deposition
Hexamin (HMT)	Hexamethylenetetramine
SMF	Single mode fiber
MMF	Multimode mode fiber
POF	Plastic fiber
РММА	Poly methyl methacrylate
СҮТОР	Cyclized transparent optical polymer cytop
LED	Light emitting diode
GaAs	Gallium arsenide
SEM	Scanning electron microscopy
OWC	Optical wireless communication

XRD	x-ray diffraction spectroscopy
C_2H_6O	Ethanol
$Zn(NO_3)_2, 6H_2O$	Zinc nitrate hexahydrate
Thiol, C ₁₂ H ₂₆ S	Dodecanethiol 908%
HF	Hydrofluoric acid
Tween 80	Polyoxyethylene (20) sorbitan monooleate
OH-	Hydroxyl
Н	Hour
μΙ	Microliter
Ml	Milliliter
min	Minute
CCD	Charge-coupled device
Eq	Equation
CE	Coupling efficiency
BER	Bite error rate
DAC	Digital to Analogue Converter
ADC	Analog to digital converter

CHAPTER 1

INTRODUCTION

1.1 Motivation

Nanotechnology is one of the most growing technologies due to its' potential applications in different fields such as electronics, mechanical device, optical and magnetic component, biotechnology and tissue engineering (Sohaebuddin et al., 2010; Hayat et al., 2014). Nanomaterial size are reduced into the nanoscale, while their surface area, surface roughness and surface area to volume ratios increased (Zhang and, Webster, 2009; Pajnič et al., 2015; Li et al., 2009). This technology involves the development of nanostructured materials, which basically can be classified into four types based on the number of dimensions; zero-dimensional (0-D), one-dimensional (1-D), two-dimensional (2-D), and three-dimensional (3-D). 0-D nanomaterials are defined as materials wherein all the dimensions are measured within the nanoscale. The most common representations of zero-dimensional nanomaterial are nanoparticles, which include gold, silver and quantum-dot metallic nanoparticles (Cao, 2004). 1-D nanomaterials have needle like-shaped where one dimension is outside the nanoscale. Carbon nanotube (Meagan et al., 2008), nanowire and ZnO nanorods (ZnO NR's) are examples of the one dimension nanomaterial's (Dingand, Wang, 2009). 2-D nanomaterials have two of the dimensions that are not confined to the nanoscale. Therefore they exhibit plate-like shape. Nanofilms, nanolayers, and nanocoatings are examples of 2-D nanomaterials (Hang et al., 2010; Rao et al., 2009).3-D nanomaterials are materials that are not confined to the nano scale in any dimension. These materials

are referred to dispersions of nanoparticles, bundles of nanowires, and nanotubes as well as multi nanolayers (Schodek et al., 2009; Liu et al., 2013).

One dimension (1D) nanostructures are primary material for electronic and photonic devices due to their small size and improve change carrier mobility (He et al., 2007;Weintraub et al., 2010). ZnO is one of the most important nanomaterials that have been widely studied in recent years. Due to the lack of center of symmetry in wurtzite structure of ZnO, this material has strong piezoelectric and pyroelectric properties when a stress force is applied (Gandh et al., 2010; Mazingue et al., 2005; Baruah et al., 2008). Furthermore, ZnO is an important semiconductor with a large direct band gap of 3.37 eV, high electron mobility of 200 cm² V ⁻¹ S⁻¹, and high excitation binding energy of 60 meV. Therefore, ZnO nanostructures have many applications in optoelectronic, sensors, transducer, catalysts, optical coating, and electrical devices (Chen et al., 2012; Wang, 2007). An efficient excitation emission at room temperature is due to high binding energy (60 meV) in ZnO crystal while wide band energy makes this material suitable for short wavelength optoelectronic devices (Özgür et al., 2005; Baruah & Dutta, 2009).

Many studied have been reported about different morphology's of ZnO nanostructure such as, nanowire, nanorods, nanocomb, nanorings, nanobows, nanobelts, nanohelics, and nanocage (Schmidt-Mende and, MacManus-Driscoll, 2007 ;Hughes & Wang, 2005; Zhuo et al., 2008; Yu et Al., 2008; Leschkies et al., 2007). ZnO nanostructures attracted a lot of attention and play an important role in many recent devices because of their wide range of properties (Desai and, Haque, 2007). Among these nanostructures of ZnO, particularly nanorods and nanowires are widely used in electronic and optical devices due to their high surface to volume ratio (Guo et al., 2002; Wei et al., 2005) Growth of nanorods has been reported on different flat surfaces such as glass (Umar et al., 2009), silicon (Choy et al., 2003) and sapphire (Hong-Yuan et al.,

2007). There was however less attention given to curved surfaces; optical fiber and waveguides (Zhou et al. ,2013).Utilizing the growth of ZnO NR's on round optical fiber is a new field of interest due to their optical quality and diversity of fabrication scheme (Chen et al. , 2004 ;Thankappan et al. , 2013)

1.2 Background theory

Typical fiber structure comprises of a higher refractive index core surrounded by a lower index cladding (Keiser, 2010). Thus, the existence of nanorod structures on the fiber surface allows the light to scatter from the fiber as well as to couple inside the guiding region. ZnO high refractive index (~2) and high surface to volume make it proper material to enhance light coupling between the device and ambient environment (Zhou et al., 2013). When growing ZnO on the cladding region, scattering by nanorods excites cladding modes which are sensitive to the changes of the surrounding environment. This effect is illustrated in Figure 1.1 below. This scattering of light by the nanorods can be implemented using the current fiber optics technology for potential applications in many areas such as optical communication, fiber laser and interferometry (Goodman, 2005).



Multimode optical microfiber

Figure 1.1: Scattering light with ZnO NR's on cladding and core modes of silica multimode optical fiber

For proper utilization of the scattering properties of ZnO NR's on fiber one needs to understand the light mechanism inside optical fiber .A fundamental optical parameter of any material is the refractive index. In free space light wave travels at a speed of (C =3 ×10⁸ m/s). The speed of light is related to the frequency (v) and wavelength (λ) through equation (1.1):

$$c = v\lambda$$
 (1.1)

Then the refractive index (n) can express by this equation:

$$n = \frac{c}{v} \tag{1.2}$$

Where *v* is the speed of light inside the material. Hence, the value of the refractive index changes depends on the material density. For example, the refractive index for air is almost unity $(n \sim 1)$, while for water it is as high as n = 1.33. For a solid-state material, refractive index increased due to higher density such as for the case of glass, n = 1.5.

The light propagation inside an optical fiber can be explained based on the concept of reflection and refraction .The light transmitted is trapped inside the core of the optical fiber due to the total internal reflection phenomenon. This total reflection can happen when the light ray incident at the interface between the core and cladding regions inside the optical fiber at angles larger than the critical angle. When a plane wave of light propagates through an interface between two dielectric media, it refracts according to the following Snell's law:

$$n_1 \sin \theta_1 = n_2 \sin \theta_2 \tag{1.3}$$

If the incident region has lower refractive index than the transmission region, the transmitted and reflected light are as shown in Figure 1.2 and described by the following equations (1.4) and (1.5).



Figure 1.2: Light is incident from a lower refractive index medium (Fresnel reflection).

$$reflection = |r|^2 = \left|\frac{n_1 cos\theta_1 - n_2 cos\theta_2}{n_1 cos\theta_1 + n_2 cos\theta_2}\right|^2$$
(1.4)

$$transmission = |t|^2 = 1 - |r|^2$$
(1.5)

Consider the case of a waveguide formed by two interfaces similar to Figure 1.2. For light incident from the top region (lower index region), light will transmit and reflect at each interface as indicated in Figure 1.3. Hence, light leaks at each bounce and guidance become impossible (Bahaa et al., 2007).



Figure 1.3: Leakage of the light inside the high index region when incident from the side of the waveguide.

When light passes from a denser medium to a lower one, at a large enough angle light can be transmitted at 90° degrees as shown by the green arrow in Figure1.4. The angle that corresponds to this behavior is commonly referred to as critical angle and can be expressed using the following equations:

$$\sin\theta_1 = 1 = n_c \cdot \sin\theta_c \tag{1.6a}$$

$$\sin\theta \ critical = \frac{1}{nc} = \frac{1}{1.5 \ galss} = \frac{2}{3} = 0.7$$
 (1.6b)

where θ_c is a critical angle, and n_c is defined as the medium index.



Figure 1.4: Total internal reflection when light propagates from a denser to lower density medium at angles larger than the critical angle.

At larger angles, light cannot penetrate through lower index region resulting in total internal reflection to the incident medium as shown by the red arrow in Figure1.4. For guidance to be achieved, the angles of the rays inside the waveguide Figure1.4 should be larger than the critical angle (θ_c). To achieve this, an extra element needs to be induced at the interface to scatter the incident light at large angles as illustrated in Figure1.5.



Figure 1.5: Allowing light to guide inside the high index region when incident from the side utilizing an extra element (diffraction or scattering for example).

This concept of light propagation between two different media can be used for coupling light inside optical microfiber. In order to reach this goal, the light incident angle should be larger than critical angle between glass and air. This can only be done through structuring the surface or placing another material on the fiber.Based on this concept, a new approach utilizing ZnO NR's on optical fiber to control light guidance and leakage inside the fiber is introduced. This work aims to grow ZnO NR's on cladding surface of the fiber to allow the side coupling of light via a scattering effect. The ZnO is also grown onto the tapered fiber surface to increase the coupling efficiency by exciting both cladding and core modes inside the fiber. This approach is low cost (no lithography or vacuum systems involved) and controllable for free space to fiber coupling. Due to the polar nature of the (001) crystal plane of ZnO, anisotropic growth of ZnO to obtain nanorods structure can be easily achieved by using solution phase or gas phase synthesis techniques (Wang, 2009;Yi et al.,2005). Low temperature hydrothermal growth is one of the most widely used technique because of its simplicity,

low cost and potential to produce highly ordered arrays of ZnO NR's in large scales (Baruah, & Dutta, 2009). The alignment and uniformity of ZnO NR's have a strong effect on the scattering properties of the structure. During the growth of ZnO NR's, chemical concentration and time for growth are two important main factors which can effect on length and alignment of ZnO NR's. Hence, these two factors are investigated during the growth process.

University

1.3 Research objective

This work aims to optimize of the growth of Zinc Oxide nanorods on different type of optical fiber substrates, and to demonstrate light side coupling onto the optical fibers for optical antenna application in telecommunication system. To achieve this, the following objectives are outlined to guide this research work.

- To obtain a uniform growth of ZnO on both silica and plastic optical fiber substrates. Uniform coating of ZnO NR's on these substrates is an important factor to have maximum light coupling inside the fiber. The optimization is done through tuning the growth factors such as length and density of nanorods.
- To optimize the light coupling to cladding modes in both single and multimode glass optical fiber. Scattering properties of ZnO NR's which are grown on the cladding region of the silica fiber directly allows the incident light to couple into the cladding region and excite cladding modes.
- 3. To optimize light coupling to core modes in multimode optical fiber (MMF) by etching the cladding region of the fiber with chemical compound.
- To optimize the light side coupling into a plastic optical fiber. Plastic fibers have a large core region and thus ZnO NR's are grown directly on the plastic optical fiber by hydrothermal technique.
- 5. To design wide field of view (up to 90°) optical receiver for optical wireless communication system by growing ZnO NR's onto optical fiber surface.

1.4 Organization of dissertation

This thesis comprises of six main chapters where the first and last chapters are dedicated to introduction and conclusion, respectively. The first chapter explains the motivation, objective and outline of this thesis. The introductions of Zinc Oxide nanomaterial and background theory of the side coupling are also discussed in this chapter towards application in optical telecommunication. The second chapter reviews on the literatures regarding the growth of ZnO NR's, optical fibers and previous works on ZnO nanostructures applications and wide field of view (FOV) antenna. The growth of ZnO NR's on the optical fiber and its controlling parameters to achieve well alignment of nanorods are presented and discussed in Chapter three. The forth chapter discusses ZnO NR's scattering properties on the optical fibers. An analytical model is developed to demonstrate the effect of the nano-rods parameters on the scattering pattern as well as the coupling efficiency. In chapter five, the side coupling of light into the core mode of optical fiber by etching the cladding region of optical fiber and its application in wide field of view optical reception is explained. The findings of this work are concluded in Chapter 6. Some suggestions for future works are also given in this final chapter.

CHAPTER 2

LITERATURE REVIEW

2.1 ZnO NR's growth

ZnO has a Wrutize structure with no center that makes it a strong piezoelectric and pyroelectric property (Yang et al., 2002). The structure of ZnO Crystal is a hexagonal structure with lattice parameter a=0.3226, c = 0.52065 nm as shown in Figure 2.1.This structure has alternating planes implicated of tetrahedral coordinated Zn⁺² and O^{-2} ions attached along c- axis , where the ends are terminated with either polar or nonpolar surfaces (Baruah & Dutta, 2009; Gandhi et al., 2010). The most common polar surface is (0001). In ZnO crystal the highest growth is along c- axis (Gandhi et al., 2010), which results in anisotropic structures. Many polar surfaces show large surface reconstruction, when ZnO surface is stable, flat and does not suffer of reconstruction (Yanget al., 2002). Other non-polar surface are {011⁻⁰} and {21⁻¹⁰} which have low energy in compare to ±(0001) surfaces, as result the dipole moment will disappear and the piezoelectricity properties is reduced. Hence, the most favorable morphology to increase the piezoelectricity effect is to fabricate nanostructure that keep large area of ±(0001) polar surface(Wu & Xue,2010).



Figure 2.1: The Wurtzite structure model of ZnO.

Figure 2.2 shows various growth morphology of ZnO nanostructures as shown in the figure, there are three types of fast growth directions: $<2\overline{110}>$ ($\pm [\overline{12}\ \overline{10}],\pm [2\overline{110}],\pm [1\overline{12}\ \overline{10}],\pm [1\overline{12}\ \overline{10}\ \overline{10}],\pm [1\overline{12}\ \overline{10}\ \overline{10}],\pm [1\overline{12}\ \overline{10}],\pm [1\overline{12}\ \overline{10}\ \overline{10}],\pm [1\overline{12$



Figure 2.2: Different growth morphology of ZnO nanostructure.

Synthesis of ZnO nanostructure is possible in both solution and gas phases (Weintraub et al., 2010). Zinc Acetate hydrate (ZAH) in alcoholic solution with Sodium Hydrate (NaOH) or Tetraethyl Ammonium Hydrate (Kohls et al., 2002), spray pyrolysis (Krunks & Mellikov, 1995; Andrade & Miki-Yoshida, 1999), Zinc Acetate Hydrate (ZAH) derived nano-collodial sol-gel route (Spanhel, 2006) and electrophoresis (Xu et. al. 2005; Pradhan, & Leung, 2008) are some examples of solution phase synthesis approaches. Gas phase synthesis examples are vapor liquid solid (Park et al., 2003), physical vapor deposition (PVD) (Dalal et al., 2006, Wang et. al., 2005) chemical vapor deposition (CVD) (Satoh et al., 2005; Wu & Liu, 2002), microwave assisted thermal decomposition (Lagashetty et al., 2007; Liu et al., 2007), and thermal oxidation of pure Zn and condensation (Li & Gao, 2007; Yao et al., 2002). Gas phase methods are more expensive complicated and usually need to have vacuum environment and high temperature. Among solution phase techniques, hydrothermal method is one of the most interesting and beneficial due to its simplicity. Hydrothermal technique does not need high temperature, vacuum environment, its substrate independent (Baruah & Dutta, 2009; Ko et al., 2012; Liu & Zeng, 2003).

Hydrothermal method was first reported by (Vergés et al., 1990). It was successfully used to synthesis ZnO NR's on glass and silicon substrates by using the Methylenamin or Hexamine (HMT) and Zinc Nitrate solutions. HMT is highly water soluble compound. Hence, thermal degradation of HMT releases hydroxyl ions in the solution which react with Zn⁺²ions to form ZnO. The following reactions are involved in forming ZnO. The reaction to form ZnO is presented in following equations:

$$(CH_2)_6 N_4 + 6H_2 O \rightarrow 6HCHO + 4NH_3$$
 (2.1)
 $NH_3 + 6H_2 O \rightarrow NH^{+4} + OH^-$ (2.2)
 $2OH^- + Zn^{+2} \rightarrow ZnO + H_2 O$ (2.3)

There are several explanations for the role of HMT in the chemical reaction. Typically, HTM prepares OH⁻ ions for the reaction with Zn^{+2} . Some believes that it acts as a buffer since the rate of its hydrolysis decreases with the increase of the PH. Sungunan et al. (2006) provided a very different statement, which HTM attaches to the non – polar facet of ZnO and allows ZnO epitaxial growth on the polar facet (0001). This process helps to guarantee a shape of this mechanism as shown in Figure 2.3 (Baruah & Dutta, 2009).



Figure 2.3: (a) Attachment of HTM to the non - polar facet of ZnO crystal, Hexagonal ZnO crystal, (b) possible attachment of HTM to the non-polar facet of ZnO and allow polar surface to growth at c-axis.

2.2 Optical fiber

Optical fiber is a cylindrical waveguide that is widely used for light transmission in communication systems. It typically consists of two dielectric regions: core and cladding as shown in Figure 2.4. The core is made of a high index material (generally doped silica) while the cladding layer surrounded the core has a lower refractive index. The polymer buffer is coated surrounding the cladding for protection and robustness. The transmission properties of any optical waveguide are dictated by its structural and material characteristics. In optical communication, those properties affect the transmission bandwidth, the power budget of the link as well as the response of the waveguide to environmental disturbance (Katsunari Okamoto,2006).In optical fiber, the propagating light is guided through two main mechanisms: total internal reflection and constructive interference between multiple reflections. These two mechanisms set specific ways of light propagation (or angles) inside the guiding region typically referred to as modes. Each mode is defined by its propagation angle (or propagation constant) and amplitude profile.



Figure 2.4: Schematic of an optical fiber structure: a circular solid core with refractive index n₁ surrounded by cladding region with refractive index n₂<n₁ and plastic buffer covers the fiber for mechanical support.

Optical fibers are classified into single mode and multimode based on the size of the core as shown in Figure 2.5. The single mode fiber (SMF) allows only one mode of propagation while multimode fiber (MMF) allows more than one modes to propagate inside the core.



Single- mode fiber

Figure 2.5: Comparison of the single mode and multimode optical fibers and the light guidance inside both fibers.

The number of modes depends on the dimension of the structure as well as the refractive index contrast between the core and the cladding. The number of modes that an optical fiber can support is determined by the V-number of the fiber defined as:

$$V = \frac{2\pi a}{\lambda} (n_1^2 - n_2^2)^{1/2} = \frac{2\pi a}{\lambda} NA$$
 (2.4.a)

where *a* is the core radius, λ is the wavelength in vacuum, n_1 is the maximum refractive index of the core, n_2 is the refractive index of the homogeneous cladding, and applying

the usual definition of the numerical aperture *NA*. For a value less than 2.405, the fiber supports only one mode. It is then called single mode fiber (SMF). Multimode optical fiber (MMF) has higher value of V number. For a very large value of the V number, the approximate total number of modes is (Keiser,2010):

$$M \approx \frac{1}{2} \left(\frac{2\pi a}{\lambda}\right)^2 (n_1^2 - n_2^2) = \frac{v^2}{2}$$
(2.4.b)

Smaller core radius and lower index contrast reduce the number of modes and pushes the fiber towards single mode case. MMF has a larger core radius, which makes it easier to launch optical power into the fiber. The large mode profile of the MMF reduces light intensity inside the fiber and hence allows high power transmission due to the reduction of the material non-linearity. The larger core size also allows an easier coupling from light emitting diode (LED) source. The coupling is usually difficult for SMF since the core diameter is very small. However, most of the commercial laser diode sources are typically pigtailed with SMF (Azadeh, 2009).

There are several applications for both SMF and MMF. SMF is widely used in optical telecommunication, networking and sensing devices (Lim et al., 2012). Having a single mode operation makes SMF as a suitable medium to form optical resonators, where a phase matching condition is necessary. Resonators are very sensitive to the surrounding changes, and hence they are very suitable for sensing applications (Wang et al.,1994).Sustaining a single mode of propagation also minimizes fiber dispersion and allows for long distance optical communication (Lim et al., 2012). SMF can also be integrated with other optical technology to make new devices. However, this type of device is more difficult to be manufactured as well as to properly work because of the reduced allowable mechanical tolerance (Skoog et al., 2003). On the other hand, MMF gives rise to modal dispersion. This restrains its application to short distance communication (less than 500 m), which typically referred to as the last mile. However,
MMF are relatively easier to be fabricated as compared to SMF. Because of the larger core diameter, it is easier to work especially for optical antenna application in this present work.

2.2.1 Polymer optical fiber(POF)

Beside silica fibers, there is also polymer or plastic optical fiber (POF), which was introduced in the past few decades. However, it was not received much attention in communication and scientific applications because of its high attenuation. It was not until 1990 when graded – index plastic fiber was developed and in 1996 low attenuation perfluorinated fiber was achieved (Zubia, & Arrue, 2001). The first and the most popular POF with poly methyl methacrylate (PMMA) core was developed at 1960, which then became very well-known material with wide range of applications (Nihei et al., 1997; Grattan & Sun, 2000). Recently POF is replacing progressively copper cables. They are also used as complement for glass MMF in short-line communication link (Zubia& Arrue, 2001). The POF are recognized for their large core diameter which makes them easy to handle, highly flexible, resistant to vibration, low in cost and have higher coupling light from light source to fiber (Zubia, & Arrue, 2001). Installation and alignment of POFs are easier compared to glass MMF (Beres et. al., 2011). It can be used at a wide wavelength range from 650-nm to 1300 nm (Golnabi, & Azimi,2008). Figure 2.6 shows the schematic diagram of typical POF with PMMA core. As shown in the figure, the POF has a core region covered by a plastic buffer which acts as a cladding.



Figure 2.6: Plastic optical fiber structure.

POF can be used only in short-distance communications because of its higher attenuation compared to silica fiber and its high dispersion due to the large number of modes it supports. Using POF with PMMA core can achieve optical transmission at 156 MB/s over a distance up to 100 m and 500 MB/s over 50 m (Numata et al., 1999). Another kind of POF is CYTOP which is made from an amorphous fluorinated polymer. This new type of POF has lower attenuation and the transmission distance can reach up to 1 km (Koike et al., 1995; Naritomi, 1996). POF has also many sensor applications where it is widely used in sensing for chemical and physical properties (Beres et al., 2011). For instance, it can be used for detecting structural deformation and corrosion of metallic surface through the collection of the back scattered light reflected by the surface imperfections (Mohanty & Kuang, 2011). POF was used for detecting different shapes of several material based on the change of the reflected light intensity.

Early endoscope developed for medical and industrial applications used shortlength low transparency fiber and microwave predecessor of laser (Bilro et al., 2012). POF was demonstrated as well for measuring light leakages from one fiber fabricated (Golnabi & Azimi, 2008).Tapered POF was also coated with ZnO nanostructure to use in measuring different concentration of uric acid (Batumalay et al., 2014).A plastic fiber can also be used as aerosol chemical sensor where it is coated with a thin film of tetraethylorthosilan (TEOS) by using sol-gel technique to increase its sensitivity. The sensing mechanism depends on the change of the refractive index of the medium surrounding the POF and the thin film coating (Kulkarni et. al., 2010).

2.3 Recent progress on the application of ZnO nanostructure

The rapid development of optoelectronic components and fibers industries has helped the expansion of guided wave technology for sensing applications (Grassini et al., 2015). For instance, tapered glass MMF is well known in applications for chemical sensing such as gas detection sensor and spectroscopy (Beres et al 2011). This is due to the large extension of the evanescent wave into the sensing region. Structuring the top surface of the waveguide allows more light to leak to the sensing region and hence it enhances the device sensitivity to the changes of the environment. ZnO nanostructure especially nanorods are also good candidates for sensor applications due to their attractive properties. Near cylindrical geometry and large refractive index (~2) allows ZnO nanowires /nanorods to be used as optical waveguides by themselves (Zhiyong & Jia, 2005). High sensitivity of ZnO to chemical environment due to its oxygen vacancies on the surface that effect the electronics properties of ZnO gives a wide range of applications in sensing devices as well. Also, it enhances the adsorption and dissociation of small molecule such as methanol and water (Danwittayakul et al., 2014).

In literature, scattering of light due to ZnO NR's grown on the side of optical fibers have been used for humidity sensing (Liu et al., 2012). Photo current gas sensor made of Ru-sensitized ZnO nanoparticles, has also been shown to be highly influenced by the gas molecules adsorbed at the surface (Schidt-Mende & MacManus –Driscoll, 2007).

Bao et al, (2006) fabricated single nanowire light emitting diode which used Si as substrate and ZnO nano-wires spread on the substrate and then PMMA thin film, which was spin coated on the substrate. ZnO homojunction light emitting diode (LED) in the crystal GaAs substrate by ultrasonic assisted spray pyrolysis is another examples of the application of ZnO nanostructures(Schidt-Mende, & MacManus –Driscoll,2007; Du et al., 2006).

Mechanically polished D-shaped fiber was used to enhance the interaction between the evanescent beam and the nanorods grown on flat surfaces for ammonia detection (Dikovska et al., 2010). Combining the side polishing technique and long period gratings, ethanol vapor sensor were fabricated utilizing ZnO NR's grown on optical fibers (Konstantaki et al., 2012). Presence of gas alters the modal properties and hence the spectral resonances. Mechanical polishing was avoided by thinning the optical fiber through thermal tapering prior to ZnO nanorod growth, which allowed for larger evanescent tail in the ambient (Batumalay et al., 2014). Enhancing the interaction between the fiber core and external medium to increase the sensitivity of LPG technique for biosensor because of lower refractive index, small size of molecule and thickness of cladding, cladding mode reduced by using side polished methods (Jang et al., 2009).

In all the systems explained above, light was launched from one end of the fiber while signal was collected at the other end. And the efficiency of this process depend on evanescent field coupling between the cladding mode and surrounding refractive index (Chen et al.,2007In this work the main focus is to use ZnO nano-rods to scatter light, which is launched from the side of the fiber inside the guiding region.

2.4 Recent progress on wide field of View (FOV) antenna for communication application

Achieving wide field of view optical communication system is challenging. In optical wireless communication (OWC) system free space optical link is used to send information to single or multiple detectors. The orientation of the detector should have minimal effect on the quality of the received signal in ideal situation. There are some studies to overcome this problem by utilizing bulk lens system such as fish eye lens system (Deng, et al., 2012), and hemispherical based imaging receiver (Wang et al., 2012). These systems are bulky and provide a field of view up to 120°, but still at large angles the response degrades dramatically. In order to solve this issue angle diversity design of detector was proposed and used (Carruther & Kahn, 2000; Jeong et al., 2001). Although, using angle diversity FOV up to 180° can be achieved, this approach needs precise arrangement of multiple small FOV detectors. Figure 2.7 shows the use of bulky and the improved angle diversity designed in optical communication system to improve wide FOV.



Figure 2.7: Utilizing bulky hemispherical lens and angle diversity in optical wireless communication system to improve wide field of view.

There is also a report on the use of bundle optical fibers to improve the FOV in an optical detection system (Jeong et al., 2001). However, the achievable detection angle was still limited compared to angle diversity. Recently, optical antenna utilizes nano-resonators to covert optical signal into localized energy is also proposed for application in OWC (Bharadwaj et al., 2009). The proposed Optical antenna was mainly used for sub-diffraction limit microscopy. The antenna is which are wavelength selective and can also provide efficient coupling. However, the fabrication of this approach needs expensive lithography techniques.

CHAPTER 3

GROWTH OF ZINC OXIDE NANORODS ON OPTICAL FIBER

3.1 Introduction

Zinc oxide (ZnO) nanostructures such as nanowires and nanorods have been recognized as extremely important materials in a broad range of high technology applications (Chen et al., 2004; Thankappan et al., 2011; Tena-Zaera et al., 2008). For instance, ZnO has a higher refractive index compared to silica fiber and, ZnO NR's directly grown on cladding of optical fibers allow light coupling into nanorod waveguides (Voss et al., 2007). Nanorod arrays are also very favorable for sensing applications as they have a larger surface to volume ratio. Utilizing ZnO grown on long period gratings (Konstantaki et al., 2012) and side polished fibers (Dikovska et al., 2007; Dikovska et al., 2010) are common examples of its sensor applications. Optical coupling between the optical fibers and nanorods grown on them are potentially promising for various novel optical sensing applications such as humidity (Liu et al., 2012).

Up to date, many works have been reported on growing nanorods structure on different flat surfaces such as glass (Umar et al., 2009), silicon (Choy et al., 2003), and sapphire (Hong-Yuan et al., 2007). However, there was less attention given to grown ZnO structure on curved surfaces such as optical fiber and waveguides (Wang, 2004; Liu et al., 2012). The growth of ZnO structure on round optical fiber is expected to find new applications due to their optical quality and diversity of fabrication scheme (Konstantaki et al., 2012; Batumalay et al., 2014). In this chapter, the growth of ZnO

NR's on both silica and plastic optical fibers are demonstrated. ZnO NR's are chosen in this work to grow on the optical fiber due to their unique properties compared to other nanostructures. For instance, scattering properties of ZnO NR's cause light to couple to the cladding and core regions. High alignment of ZnO NR's is expected to maximize the coupling efficiency. Hydrothermal method is selected in this work since it is a well-known which can provide a highly uniform growth. The growth technique is optimized to improve the rod's length and increase density. In Figure 3.1 is shown methodology of hydrothermal growth of ZnO NR's on optical fibers.



Figure 3.1: Hydrothermal growth of ZnO NR's on optical fibers.

3.2 Growth of ZnO NR's on silica multimode fiber

In this section, we discuss on the growth of ZnO NR's on silica multimode optical fiber as a substrate. In the experiment, the fiber used is a standard multimode optical fiber (MMF, Thorlab FG105LCA) with core and cladding diameters of 105 and 125 μ m, respectively. The MMF has a low loss transmission in a wide wavelength region ranging from visible to IR (400 - 2400 nm). A maximum attenuation of 12 dB/km is obtained at 850nm. At first, the plastic buffer of the MMF was stripped using acetone in an ultrasonic bath. In this process the fiber was dipped in acetone for 15 minutes before it was cleansed with ethanol and washed with deionized water. ZnO NR's were grown using a modified method suggested by (Sugunan et al., 2006). That started with seeding a substrate with ZnO nanocrystals, followed by a controlled hydrothermal growth process (Baruah & Dutta , 2009).

A zinc acetate (ZnC₄H₆O₄, Merk, 99% purity) solution prepared in ethanol (C₂H₆O, Carlo Erba, 99.7% purity) was used to grow the seeding layer of ZnO nanoparticles. This layer serves as the nucleation site for the hydrothermal growth of the ZnO NR's. Zinc acetate dehydrate concentration was varied from 1mM to 6 mM in order to study the effect of the seeding layer on the nanorods formation on the fiber. In order to deposit the seeding layer, the cleansed fiber was placed on a hot plate while maintaining the temperature at 60 °C. Then 100 μ l of the zinc acetate solution was dropped on the fiber before the solvent was slowly dried at atmosphere. The dropping process was repeated 7 times and at the end of process the fiber was annealed at 350°C for 1 hour under the hood.

In this work, the ZnO NR's' growth is investigated for two different hydrothermal processes; using microwave and oven. ZnO NR's were grown on a seeded

silica fiber substrate by using hydrothermal process where an aqueous solution containing 10 mM zinc nitrate hexahydrate (Zn (NO₃)₂, 6H₂O, Aldrich, 99%), and 10 mM hexamethylenetetramine or HMT (CH₁₂N₄, Carlo Erba, 99.5%) was used as precursor solution in the beaker and heated up at 95°C for 3 h in a microwave oven and later heated to 90°C in normal oven. The hydrothermal reaction time was varied from 5 h to 20 h and in order to maintain a constant growth rate of the nanorods the old precursor solution was replenished with new solution every 5 h untill the end of hydrothermal process (Baruah & Dutta, 2009). Finally the ZnO NR's coated fiber was retracted from the precursor solution and rinsed thoroughly with DI water several times, followed by drying in oven at 90 °C. The obtained ZnO nanorords coated substrates were then characterized by scanning electron microscopy (SEM; model: JEOLJSM-6301F) and x-ray diffraction spectroscopy (XRD; model: JEOLJDX-3530 with Cu K α radiation).

Figures 3.2 and 3.3shows the SEM micrographs of ZnO NR's grown on the surface of silica optical fiber by microwave and oven respectively. It is observed in Figure 3.2 that the growth of nanorods by microwave is really not uniform. However, the uniformity is significantly improved by using a normal oven as shown in Figure 3.3. Therefore, for rest of experiments in this work and also for plastic optical fiber substrate normal oven was used to grow ZnO NR's.



Figure 3.2: (a) Low magnification scanning electron microscope (SEM) images of the fiber coated with ZnO NR's by microwave technique, (b) Higher magnification of the nanorods.



Figure 3.3: (a) Top and (b) Cross sectional SEM images of ZnO NR's grown on the surface of silica MMF using oven.

As shown on Figure 3.3 (a), the diameters of the nanorods were found to be in the range 100-130 nm. The average length of the nanorods was found to be around 1.8 μ m determined from cross sectional SEM micrograph of the nanorods coated fiber as shown in Figure 3.3 (b). The XRD pattern of Figure 3.4 shows Wurtzite structure verified from the power diffraction standards (JCPDS) no. 36-1451. The maximum XRD peak intensity was found at twice of the diffraction angle(20) of 34.42° corresponding to the

(001) plane of ZnO, indicating that the grown ZnO nanorords are well oriented in their c-axis and the preferential growth of the ZnO NR's are along the [0001] direction (Bora et al., 2014)



Figure 3.4: XRD pattern of ZnO NR's grown on silica MMF.

3.3 Growth of ZnO NR's on an etched silica multimode fiber

In this work, the cladding region is etched to couple light through the core region of the MMF in our proposed optical antenna device application. After removing the plastic buffer during cleaning process by using acetone solution in an ultrasonic bath for 5 min and cleaving the tip of MMF, the glass fibers were dipped in a hydrofluoric acid (HF) for different time durations (5 min – 60 min) in order to ensure the complete removal of the cladding region (Kbashi, 2012). The etched samples were cleaned thoroughly with deionized water and dried in air for 15 min. Due to aggressive properties of the HF, after 45 minutes of dipping the fiber tips dissolved completely. Figure 3.5 shows the measured fiber diameters with respect to the etching time in hydrofluoric acid. It is thus clearly seen in the figure that increasing time of dipping successfully reduced the diameter of the fiber down to the core region. After 8 min of etching the diameter of fiber reaches the core region and the cladding is totally dissolved. Upon prolonged etching, the core region is thinned following an almost quadratic reduction.



Figure 3.5: Fiber diameter versus the etching time in hydrofluoric acid.

After etching of the cladding region of MMF, the hydrothermal growth of ZnO NR's on core region of MMF is carried out using the same procedure as explained in section 3.2. Figure 3.6 shows the SEM images of ZnO NR's, which were grown on the etched silica MMF. As shown in the figure, highly uniform growth of ZnO NR's was achieved with an average diameter of 120 ± 20 nm and length of 1.74 ± 0.05 µm at 13 µm⁻²densities.



Figure 3.6: SEM images of (a) Uniform growth of ZnO NR's on the core region of silica MMF, (b) Cross sectional view of the MMF showing the growth thickness, (c)High magnification top view of the rods showing the profile and density,(d) Side view of the rods on the optical fiber showing the growth directionality.

3.4 Growth of ZnO NR's on a plastic fiber

The deposition of the seeding layer on the POF substrate is different to silica optical fibers and glass substrates. After cleaning POF samples, they were treated with several different methods in order to achieve optimum results. In growing ZnO NR's on the POF fiber, at first, we followed the optimum growth condition for silica MMF (1mM Zinc acetate and 8 h growth time). However, the POF substrate is made of Poly (methyl) methacrylate (PMMA). Therefore, we cannot achieve a proper nanorods growth on POF with the optimum growth condition of silica fiber. This is attributed to the fact that attachment of rods on the POF was not strong. Figure 3.7 shows the SEM image of the POF coated with ZnO NR's with same condition as the previous silica

MMF. Although, nanoparticles are seeded uniformly on the POF surface, the nanorods did not grow thoroughly.



Figure 3.7: SEM image of not successful growth of ZnO NR's on the POF surface

In an attempt to solve this issue, the POF was first treated by 1% Dodecanehiol 98% (thiol) solution before the seeding process. The POF sample was dipped into the Thiol solution for 30 min before it is heated at 100° C for 15 min. The seeding and growth of nanorods then follows the same process of silica MMF. The SEM image of the sample treated with Thiol is shown in Figure 3.8, which indicates a slight improvement where separate patches of ZnO rods grew on the fiber. The growth was not uniform.



Figure 3.8: SEM image of the POF, which was treated with dodecane thiol to have stronger attachment between NR's and fiber surface.

In order to further improve the growth condition, 1mM Sodium hydroxide (NaOH) was dissolved at 20 ml ethanol at 60°C under stirring condition and added during the preparation of zinc acetate solution in ethanol. This solution was kept inside water bath at 60°C for 3h, after that it was used for the seeding process. This technique provides more hydroxyl ion (OH⁻) (Baruah, et al., 2008), which is useful for hydrothermal process. Figure 3.9 depicts the SEM image of ZnO NR's, which was grown on the POF with assistance of NaOH. It is observed that the nanorods are successfully grown on the POF surface by adding NaOH, but with no proper orientation.



Figure 3.9: SEM image of the nanorods grown on the POF, which was treated with NaOH during seeding process



Figure 3.10: SEM image of nanorods grown on the POF, which was treated with thiol and NaOH during the seeding process.

The growth of ZnO NR's is also attempted by using a combination of both NaOH and Thiol treatments. The result is shown in Figure 3.10. As seen in the figure, the rods have a stronger attachment with the surface but they are not distributed properly on the

POF.Finally, Polyoxyethylene (20) sorbitan monooleate ((Tween 80, C_{32} H₆₀ O_{10}), which is nonionic surfactant that contains hydrophilic group to treat the POF is used. The structure of tween 80 is depicted in Figure 3.11.



Figure 3.11: Struture of polysorbate 80 (tween 80) with hydrophilic group.

As is shown, the (OH) group can help to provide more OHion during hydrothermal process. In the experiment, 1 ml of Tween 80 is dissolved in 10 ml deionized (DI) water, and then POF sample is dipped into this solution for 10 min at 45° C. The sample was dried at air. Figure 3.12 shows the SEM image of the POF, which was treated with Tween 80. Although the rods are grown on POF with strong attachment, there are not as homogenous as desired. Patches of larger rods arepresented among the nanorods.



Figure 3.12 :SEM images of ZnO NR's on the POF, which was tracted with tween

80.



Figure 3.13: (a) Cross sectional SEM of the aligned ZnO NR's growth on the POF, which was treated with tween 80 and NaOH , (b) Top view.

Based on the previous SEM images, NaOH was added into Zinc acetate solution after using Tween 80 during the seeding process. The SEM image of this combination is shown in Figure 3.13. The images show that the nanorods are grown with more uniformity and better radius distribution on the POF surface.

The results for POF treatment before hydrothermal growth of ZnO NR's depicted in table 3.1.

				11			
Treatment solution	Time			Temperature			Result
	Dipping	Annealing	growth	seeding	Annealing	Growth	
Thiol 98%	30min	1h	8 h	60 °C	100° C	95 °C	NR's did not growth
NaOH	NA	3h	8h	60°C	60°C	95°C	Not strong attachment
Thiol +NaOH	30 min in thiol	3h	8h	60 °C	60°C	95°C	Not NR's /flower shape
Tween 80	10 min	10 min	8 h	60C	45 °C	95°C	Not uniform growth
Tween 80+ NaOH	10min in tween 80	3h	8h	60°C	60°C	95°C	Uniform& alignment

Table 3.1: Results of POF treatment to have optimum growth of ZnO NR's

3.5 Summary

Hydrothermal growth of ZnO NR'swas applied on two different kinds of optical fibers; silica and plastic MMF. Compared to the conventional approach with microwave, the use of oven at growth temperature of 95° C produced more uniform and highly aligned ZnO rods. It is found that the 8 hours growth time and the use of 1 mM Zinc acetate are the optimum condition for the hydrothermal process to produce ZnO NR's with highly uniform, dense and excellent directivity on the fiber surface. The same conditions were used to grow ZnO NR's on cladding surface of both fibers. The ZnO NR'sgrowth was also successfully demonstrated onto the core surface of the silica fiber, which was etched using HF acid. It is also found that the ZnO hydrothermal growth on PMMA surface is more complicated compared to silica surface. Therefore, some pre-treatments have to be carried out on the POF to obtain uniform and well-oriented nanorods.

CHAPTER 4

OPTIMIZATION OF GROWTH CONDITION FOR MAXIMUM SIDE COUPLING

4.1 Introduction

Scattering properties of ZnO NR's coated on multimode fiber controls light guidance inside the fiber from outside leakage. Light scattering of the nanorods is governed by rods' shape, density and refractive index contrast between the rods and the environment. The effect of the hydrothermal growth condition of the nanorods on these factors and their influence on light scattering and guidance are experimentally investigated. The optimum results for coupling light to cladding and core modes are obtained through sample characterization. Characterization and growth optimization are demonstrated in this chapter.

4.2 Scattering properties of ZnO nanorods on optical fiber

The mechanism for side coupling of the input light to the guided modes inside the fiber through the scattering from the nanorods is illustrated in Figure 4.1. Scattering by nanorods excites cladding or core modes. Cladding modes are excited when the nanorods are coated on the cladding region. Exposing the core region through etching allows for direct core modes excitation. The nanorods also cause leakage from those modes allowing two possible light coupling collection schemes as shown in Figure 4.1(a). Light exits from the side and is collected from the fiber end or side fiber probe. Scattering is controlled by the shape of the nanorods, density and uniformity that can be

designed by optimizing growth conditions (Baruah & Dutta, 2009). ZnO NR's scatter the incident light inside the fiber at angles greater than the critical angle. That excites the guided modes of the fiber and light is coupled.



Figure 4.1: Schematic representation of two possible configuration of side coupling (collection from the end of fiber or collection of light from the leakage from the side of the fiber) to cladding modes with guided and leakage intensity responses of light paths in the side-coupling configuration The total power of the coupled light inside the fiber reduces exponentially while propagation due to leakage from the nanorods themselves. The effect is graphically illustrated in Figure 4.1(b). When neglecting absorption by ZnO, the output light can be expressed as:

$$P_{guide}(z) = P_o \exp\{-2\alpha_s \times (z - z_o)\} + P_{\infty}$$
(4.1)

 P_o and P_{∞} are the coupled light power at $(z = z_0)$ and background $(z \rightarrow \infty)$. The constant (α_s) is the nano-rods scattering coefficient. In order to relate the scattering coefficient to the rods growth conditions we define the scattering cross section, C_{sc} . Scattering cross section is a statistical value of an area per unit area within which light falling on one rod will scatter. Light falling outside this area does not scatter. This concept is illustrated in Figure 4.2 and defined in equation(4.2).



Figure 4.2: Illustration of light scattering from one rod.

$$P_0 = P_{source} \times \frac{C_{SC}}{1 m^2}$$
(4.2)

This is only the case for one rod. For multiple rods (depicted in Fig. 4.3), the effect is assumed to be a linear summation of the scattering from each rod, $(\Sigma P_{source} \times C_{sc}/_{1 m^2})$.



Figure 4.3: Illustration of light scattering from several rods.

In this assumption the multiple scattering (or cross talk) is neglected. The total scattered power due to multiple rods is expressed in equation (4.3).

$$P_{total} = P_{source} \times N_{Rods} \times {C_{sc}} / {1 m^2}$$
(4.3)

Where P_{source} is the power of the incident light. The number of rods, N_{Rods} , per unit area can be defined through the multiplication of the rods density, ρ_a , by a 1 square meter.

$$N_{rods} = \rho_a \times 1m^2 \tag{4.4}$$

The total light scattered inside the fiber is expressed in the following equation.

$$P_0 = P_{source} \times \rho_a \times 1m^2 \times \frac{C_{SC}}{1m^2}$$
(4.5)

The equation (4.5) can be reduced to the following equation (4.6).

$$P_0 = P_{source} \times \rho_a \times C_{sc} \tag{4.6}$$

The expression in equation (4.6) represents the total power scatters inside the fiber. However, not all light scattering inside is guided (coupled). Only light scattered with angles larger than the critical angle contributes to the coupling. This can be defined through a reduction ration.

$$P_0 = P_{source} \times \rho_a \times C_{SC} \times \dot{\eta} \tag{4.7}$$

In order to derive an expression for η' , one needs to calculate the probability distribution function of the light scattering from one rod versus radial and azimuthal angles (θ, ϕ) . This is typically referred to as the phase function, $p(\theta, \phi)$ and it is illustrated in Figure 4.4



Figure 4.4: (a) Definition of the polar coordinated and (b) Measured polar plot for the ZnO NR'S grown on coated fibers, considering symmetry along the θ direction

As $p(\theta, \phi)$ is a probability distribution function, the integration over all angles should be unity.

$$\int_0^{2\pi} \int_0^{2\pi} p(\theta, \phi) \sin\theta \, d\theta \, d\phi = 1 \tag{4.8}$$

The pattern is assumed to be symmetric along the azimuthal angle, the expression in equation (4.8) is reduced to:.

$$\int_{0}^{2\pi} p(\theta) \sin\theta d\theta = 1 \tag{4.9}$$

Only light scattered at angles large than critical angles contributes to the guidance inside fiber as demonstrated in Figure 4.2. The fraction of scattered light that is guided can be written as:

$$\dot{\eta} = 2 \times 2\pi \int_{\theta c}^{n} p(\theta) \sin\theta d\theta \tag{4.10}$$

Hence, P_0 can be expressed:

$$P_0 = 2 \times P_{source} \times 2\pi \times \rho_a \times C_{SC} \int_{\theta}^{\pi} p(\theta) \sin\theta d\theta$$
(4.11)

It is worth mentioning, that P_0 is the total power coupled into the fiber assuming an excitation from one point. The effect of finite beam extension will be explained in the next chapter.

To characterize the scattering properties of the NR's, and hence the coupling efficiency, two approaches were explored in this work. In the first approach, the incident light is scanned along the longitudinal axes of the fibers coated with ZnO NR's and the output power is measured. The measurements are then fitted to equation(4.1) to extract the coupling efficiency and P_0 . In the second approach, ZnO NR's were grown on flat glass substrate and the phase function is measured using a nephelometer. P_0 was estimated from equation (4.11) where the rods density and shape were extracted from the SEM images of the grown rods.

The next section explains the longitudinal scanning approach and shows the optimization results. In section 4.3, phase function measurement is explained. In the first approach microwave hydrothermal method was used, while oven was used in the second one. Microwave was originally used due to its faster growth rate. However, as mentioned in the previous section, it resulted in non-uniform growth and hence in the second approach (and for the rest of the thesis work) oven was used.

4.3 Extraction of scattering coefficient using longitudinal scanning approach

In this approach the fiber coated with NR's is considered as a black box, where the output power is measured versus different input locations along the fiber axes. To prove the concept introduced in the previous section, the growth time was fixed while controlling the molarity of the seeding layer. Different concentrations of zinc acetate were utilized during the seeding process: 1, 2, 3, 4, 5 and 6 mM, and for each concentration several samples were prepared and tested.

Optical characterization was performed by measuring the output power while scanning a focused white LED source along the fiber. The output is recorded using a charge-coupled device (CCD camera) with 10x objective lens imaging the fiber end. Still images captured by the camera were processed to extract the tip region and the pixel values of that region were averaged and recorded with respect to the z location. The optical set up which was used to characterize the samples is depicted in Figure 4.5. The setup is designed to measure the coupled light versus incident location along the fiber axes. It comprises of CCD camera, detector, sample holder, automatic translation stage, beam splitter and LED light source. The end of the sample is placed. at the focal point of the output objective lens. The light collected into the objective is divided through a beam splitter to be partially read by a detector and for an image of the tip to be recorded in the camera. The output of the camera and the detector are synchronized using a computer unit which records a sequence of images and power readings while moving the stage parallel to the fiber. The scattering coefficient can then be directly extracted from measurements.



Figure 4.5: Optical characterization setup for the side coupling in wet etched fibers coated with ZnO NR's.

The obtained results are shown in Figure 4.6. This graph demonstrates the displacement of samples to light source versa coupling intensity. The optical microscope images in Figure 4.6 clearly show the excitation in the cladding modes where the

difference in the recorded intensity can be visualized. In this plot, it is clear that 4 mM of zinc acetate concentration for seeding shows the highest intensity. This is confirmed by the optical images of the tip of the fiber for different samples. Figure 4.7 illustrates the measured coupling intensity (left axis) and average scattering coefficient (right axis) versus the concentration of Zinc acetate used for growing ZnO seed crystals.



Figure 4.6 : Different concentration of zinc acetate (2-6 mM concentartion) used for growing seed crystal. Images of the output tip of the fiber show relative coupling intensities and coupling to cladding modes..



Figure 4.7: Measured coupling intesnity (left axis) and average scattering coefficient (right axis) versusthe concentration of zinc acetate for growing ZnO seed crystals.

By increasing the concentration of the Zinc acetate, scattering coefficient, α_s increases in almost a linear fashion that can be due to the directionality of the nanorods when ZnO NR's were grown from seeds formed with higher molar concentrations. Higher zinc ion concentration for the seed growth could lead to the randomness in directionality of the nanorods due to the fast seeding process. That can explain the large standard deviation in the measured scattering coefficient in the plots.

The solid line in Figure 4.7 shows the average coupling intensity versus the zinc acetate concentration used for the ZnO crystallite growth (seeding). The intensity response shows a different trend where a peak value is observed in samples where the seed growth was done with 4 mM zinc acetate. The coupling intensity is directly proportional to the coupled power, P_o , through the area of the excitation source. As

shown in equation (4.11), P_o does not only depend on the scattering strength but it also relates to the directionality of the nanorods defined by the phase function p.

In Figure 4.7,the scattering coefficient follows a linear trend with the concentration, while the coupling intensity peaks at 4 mM. These two plots can be combined in one graph where the coupling intensity is demonstrated versus the scattering coefficient. This is depicted in Figure 4.8. In this demonstration, maximum coupling efficiency is obtained when the scattering coefficient due to the nanorods reaches a value near 15 cm⁻¹. Though, seeding concentration was the main control parameter in this analysis, the graph in Figure 4.8 gives a wider scope of control. In order to achieve maximum coupling, the scattering coefficient has to be controlled to the optimum value of 15 cm⁻¹. This can be done through different control parameters.



Figure 4.8: Coupling intensity versus scattering coefficient.

4.4 Scattering parameters extraction using nephelometer

In this section, a nephelometry method is used to characterize the scattering properties of the nanorods scattering parameters such as cross section and maximum power coupling are directly extracted from the direct measurement of the phase function and rods geometry (dimensions and density). Phase function is measured using a homemade nephelometer, which required the ZnO NR's to be grown on flat surface in order to reach optimum conditions. In this work, the nanorods were grown on flat glass substrate based on hydrothermal technique using an oven to ensure a uniform growth. This technique also allows a better control of ZnO NR's properties.

Direct measuring of the phase function for ZnO NR's coated on optical fiber is a challenging process due to the geometrical effect of the fiber cross section. That works as a cylindrical lens and hence affects the angular spectrum of the scattering process. Also, light interacts with the rods layer at multiple locations due to the roundness of the fiber. That makes it extremely difficult to extract the effect of one layer of the coated rods. One way to avoid this difficulty is to measure the scattering properties of ZnO NR's grown on a flat surface. Then using the optical condition obtained as initial conditions for the fiber growth. Fine tuning is then applied on the fiber case for maximum coupling efficiency.

In the following section, direct measurements of the phase function and growth optimization on flat surface are studied. This is followed by optimization on the fiber surface.

4.4.1 Direct angular spectrum measurement

The presence of the ZnO NR's on flat substrate or optical fiber causes the incident light to scatter angles larger than the incident one. The angular spectrum distribution of the scattered light depends strongly on the rods shape and density. Optical scattering is characterized through scattering cross section, C_{sc} , and phase function $p(\phi,\theta)$. Cross section is the ratio of the total scattered field to the incident light represented as an area normalized to unit area. Phase function is a probability distribution function of the light scattering at radial and azimuthal angles θ and ϕ . To evaluate C_{sc} and p of the grown ZnO NR's on glass substrate, an optical nephalometer was used. Figure 4.9 shows the schematic setup for the homemade nephelometer.

In the experiment, a collimated fiber coupled white light LED source is used to shine light on the sample. An optical detector is placed at the end of a rotating arm to measure the optical power as a function of the azimuthal angle θ (radial symmetry is assumes as the direction of the rods are along the direction of light incidence.) The average scattering cross section for one rod, C_{sc} , is estimated from the measured ratio of the total scattered power to the incidence divided by the rods density (number of rods per unit area, $\rho_{a.}$) This density is measured from the SEM images. Knowing C_{sc} and ρ_{a} , the scattering coefficient (defined as the scattering loss per unit length of propagation) as $\alpha_s = C_{sc} \times \rho_a / L$, where L is the average length of the rods measured from the SEM images. This quantity is important especially when dealing with optical fiber which will be explained in the results and analysis section.



Figure 4.9: Optical nephelometer setup for testing scattering properties of ZnO grown on glass substrate.

The total power (P_0) that scatters inside the fiber is expressed by the following equation:

$$P_0 = P_{source} \times \frac{c_{sc}}{1m \times 1m} \times \dot{\eta} \times N_{rods}$$
(4.12)

The coupling efficiency (CE) is expressed as a ratio of the total light power to the source power defined by the following equation:

$$CE = \frac{P_0}{P_{source}} = C_{sc} \times \rho_a \times \dot{\eta}$$
(4.13)

In equation(4.12) the density of rods is calculated from the SEM images. Using the expression for the reduction ratio in terms of the phase function, equation (4.13) can be written as:

$$C.E. = 2 \times 2\pi \times \rho_a \times C_{SC} \int_{\theta_c}^{\pi} p(\theta) \sin\theta d\theta$$
(4.14)
Re-arranging equation (4.14):

$$C.E. = 2 \times \int_{\theta_c}^{\pi} 2\pi \times \rho_a \times C_{SC} \times p(\theta) \sin\theta d\theta$$
(4.15)

The term inside the integral can be defined as $\Psi(\theta)$:

$$\Psi(\theta) = 2\pi \times \rho_a \times C_{SC} \times p(\theta) \tag{4.16}$$

It is worth mentioning that in the nephlometry experiment the measured power angular spectrum is due to all the excited rods. Hence, $\Psi(\theta)$ is directly measured by dividing the angular spectrum by the source power. It can then be referred to as the normalized power angular spectrum. The cross section can be estimated by the integration of equation (4.16) over all the scattering angles.

$$2\int_0^{\pi} \Psi(\theta) \sin\theta d\theta = 2\int_0^{\pi} 2\pi \times \rho_a \times C_{SC} \times p(\theta) \sin\theta d\theta$$
(4.16)

Using the relation in Eq. 4.9, Eq. 4.16 can be simplified to:

$$2\int_0^{\pi} \Psi(\theta) \sin\theta d\theta = \rho_a \times C_{SC}$$
(4.17)

The rods density is calculated from the SEM image. The left hand side is directly measured from the Nephelometer when integrating over all the angles. Hence, the scattering cross section is extracted as:

$$C_{SC} = 2 \frac{\int_0^{\pi} \Psi(\theta) \sin\theta d\theta}{\rho_a}$$
(4.18)

The reduction ratio is then directly measured.

4.4.2 Growth optimization on flat surface

The measured normalized angular power spectra for different seeding concentrations are depicted in Figure 4.12. The plots show that the lowest concentration (1 mM of Zinc Acetate shown in dark blue color) has high normalized power at angles larger than the critical angle, θ_c . Red, magenta, green colors represents 2, 4 and 6mM respectively. It is clear from the graphs that higher concentrations have lower power compared to 1mM. The coupling efficiency for the different concentrations is estimated from the plots in Figure 4.12 using equation (4.15) and they are depicted in Figure 4.10.



Figure 4.10: The measured normalized angular power spectrum for different seeding concentration of zinc acetate, lowest concentration showed higher power at larger angles.



Figure 4.11:Variation of the coupling efficiency (CE) versus the concentration of zinc acetate used for preparing the ZnO seed layer on glass substrates.

As shown in Figure 4.11, that maximum coupling efficiency of 3.68±0.13% was obtained at zinc acetate concentration of 1 mM. The coupling efficiency, CE As defined by equation (4.13) comprises of the multiplication of three components: scattering cross section, rods density and the reduction ratio. The effect of each component is separately discussed in the following sub-section.

4.4.3 Effect of C_{sc} , ρ_{a} and $(\dot{\eta})$ on the coupling efficiency

The rods density can be measured directly from the SEM images of the NR's. A specific area with known dimensions is selected and the number of rods is counted inside the area. The density is then calculated by dividing the number of rods by the area selected. This process can be repeated several times on different images of the same

sample to provide statistical information. The results of hydrothermal growth of NR's on glass slides as flat substrate ,obtained from SEM images for different concentration are depict in Figure 4.12. The number of rods per unit area is almost constant and it is estimated around 11 - 12 μ m⁻². Increasing the concentration did not seem to have significant effect as depicted in Figure 4.12 .Furthermore, it was observed that changing the zinc acetate concentration from 1 to 6 mM did not affect the length and diameter of the NR's on statistical basis .The average length of NR's was about 4.5 μ m and the diameter was around 150nm.



Figure 4.12: Variation in density pa versus different concentrations of Zinc acetate used for preparing the ZnO seed layer on galss subtartes.

The scattering cross section is calculated by integrating the normalized angular spectrum in Figure 4.10 and dividing by the rods density as defined by equation (4.18). The estimated C_{sc} for each concentration of zinc acetate used for preparing the ZnO

seed layer on the glass substrates is depicted in Figure 4.13. The figure shows that C_{sc} decreases with increase of zinc acetate concentration from 1 to 4 mM. The minimum value of C_{sc} is around 5×10^{-11} cm², which was obtained at 4 mM. The maximum cross section is obtained for seeding solution with 1mM concentration. The maximum obtained cross section was 6.2×10^{-11} cm²



Figure 4.13: Variation of Csc versus different concentrations of zinc acetate used for preparing the ZnO seed layer on glass substrates.

Knowing the coupling efficiency, scattering cross section and rods density, the reduction ratio can be directly calculated from equation (4.13) and it is illustrated in Figure 4.14. The graph in Figure 4.14 shows that there is almost no change in the reduction ratio with concentration. The reduction ratio is around 0.405 within the zinc acetate concentration from 1 to 6 mM. The measured values of C_{sc} , ρ_a and η ' show that

with respect to the concentration of zinc acetate, scattering cross section plays the most significant role in controlling the coupling efficiency. Best coupling was obtained at low concentration, which is the opposite of the microwave growth explained in section 4.3.



Figure 4.14: Variation reduction ratio (ή) versus different concentrations of Zinc acetate used for preparing the ZnO seedlayer on glass substartes.

4.5 Growth optimization on optical fiber

Based on the analysis in the previous section, the seeding molarity is fixed to 1mM in all the following growth conditions. When controlling the molarity on a flat substrate, the growth time was fixed to 5 hours. In this section, we describe the optimization of ZnO NR's growth on optical fiber in term of the growth time. To further improve the side coupling efficiency due to ZnO NR's on the optical fiber, the growth time is varied over the range from 5 to 20 hours. It is observed that different growth

time produces different nanorods length. The SEM micrographs of ZnO NR which were grown hydrothermally on optical fiber at different growth times are shown in Figure 4.15. The variations of length and diameter of the nanorods against the growth time is plotted in Figure 4.16. By increasing time of growth from 5 to 20 hours the length of nanorods are increased from 1.7 to $6.7\mu m$ while the diameter of nanorods is observed to increase from 70 nm to 150 nm.



Figure 4.15: SEM microgarphs of ZnO NR's grown hydrothermally on optical fibers for (a) 5 h , (b) 8 h, (c) 10 h, (d) 15 h, and (e) 20 h.

ZnO crystal shows partial polar feature with the (001) plane exhibition maximum polarity due to the presence of the partially charged Zn^{+2} and O^{2-} lattice points in an

alternating fashion (Wang,2009). Because of the polar nature, the (001) crystal plane of ZnO exhibits highest relative energy compared to the other non-polar crystal planes, allowing the crystal to grow faster in [0001] direction under favorable growth conditions. In addition to that, the hexamine (HTM) molecule, used as the source of OH⁻ ions during the hydrothermal process, also play an important role on the final morphology if the NR's (Sugunan et al., 2006). Being a non- polar chelating agent.HTM preferably adsorbs on the lower energetic non- polar facets of the ZnO crystal which eventually reduces the Zn⁺² ion supply on these sites, leaving only the high energetic (001) polar face available for the epitaxial growth. This also explains the observed slow lateral growth of the ZnO NR's, i.e. the diameter of the NR's grow slower, compared to the growth along the length (Bora etal., 2014).



Figure 4.16: Variation in the (a) length and (b) Diameter of the nanorods as a function of the growth time.

Figures 4.17 (a) and (b) show the estimated scattering cross section and scattering coefficient respectively, as a function of the length of the nanorods. The scattering cross section (C_{cs}) was observed to increase with the increasing length of the nanorods approaching slowly to saturation as shown in Figure 4.17 (a). As the length of the nanorods increases, multiple scattering from the surface of the nanorods also increases, resulting in a broader light scattering pattern. This, therefore, enhances the scattering cross section of the longer nanorods. As seen in Figure 4.17(b), the scattering coefficient, α_s , however peaks at the nanorods length between 2 µm and 3 µm. However, in case of ZnO NR's longer than ~3.5 m, the scattering strength indicated by the forward scattering coefficient (α_s) was observed to reduce gradually in Figure 4.17(b) due to the increase in multiple scattering effects in the nanorods.



Figure 4.17: (a)Scattering cross section (Csc) and (b) Scattering coefficient (αs) versus the length of the ZnO NR's.

Figure 4.18 shows the relation between the coupling efficiency (CE) and nanorods length. When the length of the nanorods was increased from $\sim 1.7 \mu m$ to 2.2 μm , CE was also observed to increase, reaching a maximum of $\sim 2.65\%$ due to the

increase of the forward scattering. However, a steady drop in CE was observed with further increase in the length of the ZnO NR's, despite the improvement in scattering cross section. A minimum CE of 0.98% was obtained for the longest ZnO NR's (~6.7 μ m), where the scattering cross section was maximum (2.53 x 10⁻³ cm²). The coupling response of the scattering coefficient and correspondingly the CE seem to conflict with the original assumption. Longer rods generally correlate to stronger scattering and hence more CE. To better understand this effect one needs to investigate other factors that affect α_s .



Figure 4.18: Change in CE versus the lenght of the NR's.

As mentioned in section 4.4.1, the scattering coefficient can be expressed as:

$$\alpha_s = C_{sc} \times \rho_a / L \tag{4.19}$$

As shown in equation (4.19) rods density, ρ_a , is another important factor in defining the scattering coefficient. Figure 4.19 shows the variation in ρ_a with respect to the length of the ZnO NR's. As shown in the figure, ρ_a decreases significantly with increasing length of the ZnO NR's. For nanorods with length about 2.2 µm, ρ_a was

found ~2.91× 10⁹ cm⁻², which was reduced to ~1.22 x 10⁹ cm⁻² for nanorods with length ~6.7 μ m.



Figure 4.19: Rod density versus the lenght of the ZnO NR's.

We have observed that during the initial 10 hours of hydrothermal growth of the ZnO NR's, when the length of the nanorods approximately reaches 3.2 μ m, ρ_a decreases significantly from ~2.91x10⁹ cm⁻² to ~1.82x10⁹ cm⁻². However, during this growth period, the length of the nanorods was found to increase slowly in Figure 4.16(a), compared to the relative increase in the diameter of the nanorods in Figure 4.16 (b). This indicates that at the beginning of the hydrothermal growth of the ZnO NR's, smaller nanorods start to merge with each other similar to the Ostwald ripening forming fatter nanorods that leads to a decrease in the number of nanorods per unit area (ρ_a) (Liu et.al., 2007). Hence, in the initial 10 h of the hydrothermal growth, we observe rapid increment in the diameter of the nanorods from ~70 nm to ~150 nm, causing ~58% drop in ρ_a . On the other hand, when the hydrothermal growth proceeds for longer periods, the intermediate space between the ZnO NR's are reduced due to the formation of the fatter nanorods limiting the diffusion of the Zn^{2+} ions in the region and thereby restricting the lateral growth of the nanorods, as indicated by marginal change in the diameter of the nanorods beyond 10 hours of hydrothermal growth time Figure 4.16 (b). As a result, ρ_a was also found to vary at a relatively slower decay rate at the latter part of the hydrothermal process (from $1.82x10^9$ cm⁻² to $1.22x10^9$ cm⁻²). However, sufficient Zn^{2+} ions are still available for the polar (001) plane of the ZnO crystal, allowing the nanorods to grow continuously in the [0001] direction. The increase in the length of the nanorods was therefore found to be much faster at the latter part of the hydrothermal growth, i.e. after 10 hour, Figure 4.16(a), yielding maximum length of ~6.7 µm in 20 hours.



Figure 4.20: Decay of coupling efficiency (CE) along the fibers coated with ZnO NR's grown hydrothermally for different growth times.

Figure 4.20 shows the CE of 1mM zinc acetate sample along the fiber coated with ZnO NR's of different lengths obtained by hydrothermal growth for different time periods. In the experiment, the growth time varied from 5 to 20 hours. As shown in the figure, the CE is exponentially decaying with the distance. The inset of Figure 4.20 shows the optical microscope images in the tip of a nanorods coated optical fiber at two different distances, where the changes in the light intensity in the cladding mode can be clearly visualized. It is found that the sample with 8 h growth time has the highest CE, the optical images for this sample also proves this result. Hence, the 8 h growth time is chosen to be the optimum time for hydrothermal growth of ZnO NR's on the optical fiber.

4.6 Summary

The present study demonstrates controlled light scattering and efficient light coupling into the cladding mode of ZnO nanorod coated multimode silica fibers. The concentration of the zinc acetate solution used for the ZnO seeding process prior to the hydrothermal growth of the nanorods was found to play an important role in the light scattering from the nanorods. At higher zinc acetate concentrations, typically more than 1 mM, the randomness in the nanorods directionality was observed to increase due to the clustering of the seeds leading to higher multiple scattering and backscattering, which finally resulted in more than 10% reduction in coupled light. The hydrothermal growth time of the ZnO NR's were also found to be as important in the light coupling efficiency. Increase in the hydrothermal growth time from 5 to 20 h resulted in ZnO NR's with length from ~1.7 μ m to ~6.7 μ m respectively, consequently improving the scattering cross section (C_{sc}) of the nanorods. However, the number of nanorods per unit area (ρ_a) was observed to decrease almost exponentially from ~2.91 × 109 cm⁻²to

 $\sim 1.22 \times 109 \text{ cm}^{-2}$ with increasing growth time. Since the coupling efficiency is directly proportional to C_{sc} and ρ_a , the competition between the scattering cross-section and density of nanorods resulted in a maximum average coupling efficiency of $\sim 2.65\%$ for ZnO NR's of length $\sim 2.2 \mu \text{m}$ obtained by hydrothermal growth for 8 h.

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CHAPTER 5

EXCITATION OF CORE MODES THROUGH SIDE COUPLING TO MULTIMODE OPTICAL FIBER BY ZINC OXIDE NANORODS FOR WIDE ANGLE OPTICAL RECEPTION

5.1 Introduction

In optical wireless communication (OWC), where free space optical link is used to send information to single or multiple detectors, achieving high field of view (FOV) is always a major challenge. In an idealistic link, the orientation of the detector should have minimal effect on the quality of the received signal. Several approaches to improve the signal have been reported, like the utilization of bulk lens system such as fish eye lens system (Denget al., 2012) and the usage of hemispherical imaging receivers (Wanget al., 2012) Both systems are bulky but provide a field of view up to 120°. The response of the systems however degrades drastically at large angles. This drawback can be solved using angle diversity approach (Carrutheret al., 2000; Jeong et al., 2001) whereby FOV up to 180° can be achieved. This however requires precise arrangement of multiple numbers of small FOV detectors. In another approach, a bundle of optical fibers were used to improve the FOV of the optical detection system (Hahn et al., 2010), however the detection angles were limited, compared to angle diversity.

Several schemes for free-space to optical fiber side coupling, such as utilizing 45° mirror (Qiu et al., 2011) and using surface grating (Antelius et al., 2011) have been presented in the literature. Though the coupling efficiency of those schemes are higher,

both are however limited to small field of view. For instance, the 45° mirror incident angles are limited to incidence between 90° and 90°-θc, where θc is the critical angle. This advantage of large angle collection in the proposed system makes it suitable for large FOV optical detection in indoor OWC system applications. In indoor OWC systems, the illuminating light is used as a source for transmitting information. Typically, spot lights and LED light bulbs are fixed on the ceiling (average ceiling height is 3 meters). That sets a requirement on the distance from the source to the detector, which is generally a hand held device, to be within an approximate range of 1.5 meter. Also, as the detector is held or placed on a person who moves around transmission zone, having a wide angle of detection becomes a necessity for such applications (Ghassemlooy et al.,2013).

Optical communication systems typically require that the transmission link is not affected by the environment to minimize the noise. Excitation to cladding modes, as it was done in the previous chapter, does not satisfy this criteria. Cladding modes are very sensitive to the change of the environment (e.g. thermal fluctuation and refractive index change). This affect can only be minimized when core modes are excited. In this chapter, side coupling to core modes through Zinc Oxide (ZnO) nanorods grown around the fiber is demonstrated. The scheme utilizes wet etching to remove a cladding region of part of the fiber followed by hydrothermal growth of the nanorods on both tapered and untapered region. The combination of nanostructures and optical fiber system is then used to demonstrate a simple wide FOV optical receiver. Core modes are excited by the light scattered at the region where the fiber core is exposed. The performance of the FOV optical receiver is investigated for both multimode silica and plastic optical fiber system.

5.2 Fabrication of wide FOV optical receiver

In this work, the cladding region is etched to couple light through the core region of the multimode fiber (MMF) in our proposed optical antenna device. In the experiment, a standard MMF with core and cladding diameter of 105 µm and 125 µm, respectively, was used. The plastic buffer on the fiber was removed by dipping the fiber in acetone solution in an ultrasonic bath for 5 min. After removing plastic buffer and cleaving the tip of MMF, the silica MMF was dipped in a hydrofluoric acid (HF) for 8 min to ensure the complete removal of the cladding region. The fiber was then cleaned thoroughly with deionized water and dried in air for 15 min. Both etched and un-etched parts of the fiber were coated with ZnO seed crystals and then the hydrothermal growth of ZnO NR's was carried out based on the optimum conditions which were obtained in the previous chapters. 1mM zinc acetate was used for growing ZnO crystal seeds on the optical fiber. An equimolar solution of 10 mM Zinc Nitrate hexahydrate and Hexamine (Hexamethylenetetramine) was used for hydrothermal growth. The seeded fiber was dipped in 150 ml of the precursor solution in a beaker and heated to 95 °C in an oven for 8 hours. In order to maintain a constant growth rate of the nanorods, the old precursor solution was replenished with new solution every 5 hours till the end of the hydrothermal process (8 hours). Following the hydrothermal growth of nanorods, the fibers were taken out of the solution and rinsed thoroughly with DI water, and dried in air.

5.3 Concept of wide FOV optical receiver utilizing the ZnO NR's

The proposed wide FOV fiber based optical receiver system is illustrated in Figure 5.1(a) where non-line of sight transmission from a light source reaches the fiber receiver at large angles (close to 90°).



Figure 5.1: (a) Schematic view of the propsed optical antenna configuration (b) Schematic representation of scattering of light from the ZnO NR's through cladding and core modes of MMF and (c) Simulated response with the finite excitation beam effect.

To allow coupling to core modes, the fiber is wet etched to expose the core region. The wet etching process generates a transient region over which the fiber diameter gradually reduces from the un-etched part to the designated diameter as shown in Figure 5.1(b). ZnO NR's are then grown on the fiber starting from a location, Z_1 before the transient region to a location Z_2 as in Figure 5.1(c), and covers part of the un-etched fiber and the entire etched region. This configuration allows both the cladding mode and core mode excitation.

The excitation of cladding and core modes is achieved due to scattering of the incident light at large angles inside the optical fiber. Part of the scattered light has angles larger than the critical angle between air and the guiding region: cladding for cladding modes and core for core modes. This portion of the incident light couples inside the fiber. When the incident light hits the ZnO NR's covering the un-etched fiber, cladding modes are excited as scattered angles inside the core can never be larger than the critical angle between core and cladding. High confinement in the cladding region allows the guided light to leak with every bounce at the nanorods-fiber interface (shown by red arrows in Figure 5.1(b). The output power (P_{out}) is maximized at Z_1 and it decreases exponentially with the location of the light excitation as shown in Figure 5.1(c). Core modes are excited when light scatters from the nanorods at (and after) the transient region. Here, portion of the scattered light exceeds the critical angle between air and core region. The maximum coupling is hence obtained around Z_2 as shown in Figure 5.1(c). The scattered light at the etched region couples to the core modes with effective indices between air and the core cladding index. The transient region behaves as a tapered fiber where excited modes inside the etched fiber couples to the core modes inside the un-etched part of the fiber. Hence, light confined in the core region of the MMF travels to the detector without being scattered by the ZnO NR's on the cladding.

The output power as a function of the location of the source excitation can be expressed using Beer–Lambert law as (Skoog, West, Holler, Crouch, 2003):

$$P_{out}(z,\theta) = P_{0,cladding} Exp\left(-2\alpha_s(\theta)(z-z_1)\right) \times u(z-z_1) + P_{0,core} Exp\left(-2\alpha_s(\theta)(z-z_2)\right) \times u(z-z_2) + P_{\infty}$$
(5.1)

where the constants α_s , $P_{o,cladd}$ and $P_{o,core}$ are the nanorods induced scattering coefficient, maximum cladding coupled power and maximum coupled core power, respectively. The function u (z) is a step function. For instance, the step function, u (z z_o) is zero before $z = z_o$ and it is unity for $z > z_o$ as illustrated in Figure 5.2 (Fallah et al., 2014).



Figure 5.2: Definition of the step function u (Z-Z0)

Maximum coupled power to core or cladding mode is defined as the following (Fallah et. al., 2013):

$$P_0 = 2\pi P_{source} C_{sc} \rho_a \int_{\theta_c}^{\pi} P\left(\theta - \theta_{inc}\right) Sin\theta d\theta$$
(5.2)

Where P_{source} , C_{sc} , α_a and p are the source power, scattering cross section of one nanorod, density of nanorods per unit area and the phase function, respectively. The coefficient P_{∞} is the background power and θ_{inc} is the angle of incidence of the source light. The scattering constant \propto_s can be defined as:

$$a_s = C_{sc} \rho_a / L_{rod} \tag{5.3}$$

Where L_{rod} is the average length of one rod. In this simplified model, the difference between the core and cladding excitation is due to different critical angle between aircore interface and air-cladding interface. The core has higher refractive index and hence the critical angle (θ_c) is smaller than the cladding. According to equation(5.2), higher maximum power is expected for core excitation compared to cladding. This concept is depicted (dashed line plot) in Figure 5.1(c) where two exponential peaks are predicted at both locations Z₁ and Z₂ when scanning the excitation light along the side of the fiber.

It is important to mention here that the relation in equation (5.2) is based on the approximation that the MMF has a large number of modes such that any scattering angle larger than the critical angle is coupled back into the fiber cladding or core modes. This however can lead to error in the estimation since etched fiber results in smaller fiber diameter where less number of modes would be available. That could reduce the coupling efficiency.

5.3.1 Finite beam effect

The expression in equation (5.1) assumes that the incident light propagates in a form of Dirac-delta function (Luckhurst &Veracin,1989). In practice, light sources have

always a finite beam width and hence the equation needs to be modified to take this effect in consideration. Collimated light incident on the fiber can be assumed to be Gaussian with a finite beam waist of σ (Ding, & Liu, 1999)which causes the peaks to broaden. Mathematically, peak broadening can be represented as a convolution between a Gaussian beam and the expression in equation (5.1). Using this assumption, the total output power can then be expressed as:

$$P_{out}(z,\theta) = P_1 + P_2 + P_{\infty} \tag{5.4}$$

In equation (5.4), P1, P2 and P ∞ are the coupled power to the cladding, core and background respectively. This power can be expressed by a convolution between the finite beam effect and the ideal exponential decay:

$$P_i(z) = \tilde{P}_i(z) \circledast h(z), \quad i = 1,2$$
(5.5)

The ideal response, \tilde{P}_i , is defined for cladding and core regions as:

$$\tilde{P}_1 = P_{0,caldding} \cdot e^{-2\alpha_s}(z - z_1) \times u(z - z_1)$$
(5.6)

$$\tilde{P}_2 = P_{0,core} \cdot e^{-2\alpha_s}(z - z_2) \times u(z - z_2)$$
(5.7)

Where $P_{o,1}=P_{o,cladd}$ and $P_{o,2}=P_{o,core}$. The finite beam is assumed to be Gaussian and hence the finite response function, h(z), can be expressed as:

$$h(z) = \frac{1}{\sigma\sqrt{\pi}}e^{\frac{-z^2}{\sigma^2}}$$

Using the definition of convolution, the power coupled to the cladding is defined as:

$$P_1(z) = \int_{-\infty}^{\infty} \tilde{P}_1 \cdot e^{\frac{-(z-z')^2}{\sigma^2}} \cdot dz'$$
(5.8)

Using the expression in section 5.6 equation(5.8) can be written as:

$$P_1(z) = \frac{1}{\sigma\sqrt{\pi}} P_{0,cladding} \int_{Z_1}^{\infty} e^{-2\alpha_s(z'-z_1)} \cdot e^{\frac{-(z-z')^2}{\sigma^2}} \cdot dz'$$
(5.9)

$$P_1(z) = \frac{1}{\sigma \sqrt{\pi}} P_{0caldding} \cdot e^{2\alpha_s z_1} \int_{z_1}^{\infty} e^{-2\alpha_s z'} \cdot e^{-(z^2 - 2zz' + z'^2)/\sigma^2} \cdot dz'$$

Taking the terms which are not function of z'outside the integration sign:

$$P_{1}(z) = \frac{1}{\sigma\sqrt{\pi}} P_{0cladding} \cdot e^{-2\alpha_{s}z_{1}} \cdot e^{-\left(\frac{z}{\sigma}\right)^{2}} \int_{z_{1}}^{\infty} e^{-2(\alpha_{s} - \frac{z}{\sigma^{2}})z'} e^{-\left(\frac{z'}{\sigma}\right)^{2}} \cdot dz'$$
(5.10)

The exponent of the first term under the integration can be re-arranged as:

$$P_1(z) = \frac{1}{\sigma\sqrt{\pi}} P_{0cladding} \cdot e^{-2\alpha_s z_1} \cdot e^{-\left(\frac{z}{\sigma}\right)^2} \int_{z_1}^{\infty} e^{-2\frac{z'}{\sigma}\left(\sigma\alpha_s - \frac{z}{\sigma}\right)} \cdot e^{-\left(\frac{z'}{\sigma}\right)^2} \cdot dz'$$
(5.11)

From Eq5.6, one can define a new term

$$\gamma = \frac{z}{\sigma} - \sigma \alpha_s \tag{5.12}$$

Using the definition in the equation above equation (5.11) is reduced to:

$$P_1(z) = \frac{1}{\sigma\sqrt{\pi}} P_{0cladding} \cdot e^{-2\alpha_s z_1} \cdot e^{-\left(\frac{z}{\sigma}\right)^2} \cdot \int_{z_1}^{\infty} e^{\frac{2z'}{\sigma} \cdot \gamma} \cdot e^{\frac{-z^2}{\sigma^2}} \cdot dz'$$
(5.13)

Completing the square for the exponents in the terms inside the integration, the above equation can be re-written as:

$$P_1(z) = \frac{1}{\sigma\sqrt{\pi}} P_{0cladding} \cdot e^{-2\alpha_s z_1} \cdot e^{-\left(\frac{z}{\sigma}\right)^2} \cdot \int_{z_1}^{\infty} e^{\left(\frac{(z')}{\sigma}\right) - 2\gamma \cdot \left(\frac{z'}{\sigma}\right) + \gamma^2 - \gamma^2\right)} \cdot dz' \qquad (5.14)$$

This can be further reduced to:

$$P_{1}(z) = \frac{1}{\sqrt{\pi\sigma}} P_{0cladding} \cdot e^{2\alpha_{s} z_{1}} \cdot e^{-\frac{z'}{\sigma^{2}}} \cdot e^{\gamma^{2}} \int e^{-(\frac{z'}{\sigma} - \gamma)^{2}} dz'$$
(5.15)

The integration in equation (5.8) can be solved by considering the definition of the complementary error function:

$$erfc(z) = \frac{2}{\sqrt{\pi}} \int_{t_1}^{\infty} e^{-t^2} dt$$
(5.16)

The integration term

$$\int_{t_1}^{\infty} e^{-\left(\frac{z'}{\sigma} - \gamma\right)^2} dz' \tag{5.17}$$

Can be re-arranged by defining the following:

$$t = \frac{z}{\sigma} - \gamma \tag{5.18}$$

$$dt = \frac{dz'}{\sigma} \tag{5.19}$$

$$t_1 = \frac{z_1}{\sigma} - \gamma \tag{5.20}$$

These results in

$$\frac{z}{\sqrt{\pi}} \int_{t_1}^{\infty} e^{-t^2} dt \times \sigma \times \frac{\sqrt{\pi}}{2}$$
(5.21)

Hence,

$$\int_{t_1}^{\infty} e^{-\left(\frac{z_1}{\sigma} - \gamma\right)^2} dz' = \frac{\sigma\sqrt{\pi}}{2} \cdot \operatorname{erfc}\left(\frac{z_1}{\sigma} - \gamma\right)$$
(5.22)

Using the result in equation (5.8), the expression for the power in the cladding region can be written as:

$$P_{1} = P_{0cladding} \ \frac{\sigma\sqrt{\pi}}{2} \cdot \frac{1}{\sigma\sqrt{\pi}} \cdot erfc \ \left(\frac{z_{1}}{\sigma} - \gamma\right) \cdot e^{-\gamma^{2}} e^{-\left(\frac{z}{\sigma}\right)^{2}} \cdot e^{2\alpha_{s} z_{1}}$$
(5.23)

Which is further reduced to:

$$P_{1} = \frac{1}{2} P_{0cladding} \ e^{\frac{-z^{2}}{\sigma^{2}}} e^{2\alpha_{s} z_{1}} \cdot e^{(\frac{z}{\sigma} - \alpha_{s} \sigma)^{2}} \ erfc\left(\frac{z_{1}}{\sigma} - \gamma\right)$$
(5.24)

Defining new constants ξ and μ as:

$$\xi = \frac{z}{\sigma}\mu = \alpha.\sigma \tag{5.25}$$

Using the constants defined above, the expression in equation (5.24) becomes:

$$P_{1} = \frac{1}{2} P_{o \ caldding} \cdot e^{2\mu \, (\xi - \xi_{1})} \cdot e^{\mu^{2}} \cdot erfc \ (\xi - \mu)$$
(5.26)

To illustrate the effect of the beam width, σ , and scattering constant, α_s , on the power coupling efficiency, the reduction of the peak power with the increase of the constant $\mu = \sigma \alpha_s$ is plotted in Figure 5.3. The graph shows an almost exponential decay of the peak value with larger values of μ . Hence, in order to eliminate the finite beam effect, one needs to divide the measured peak by the ratio in Figure 5.3 at the corresponding μ value.

In the calculations, the scattering coefficient of ZnO NR's was set to 15 cm⁻¹as previously measured and a beam waist of 300 μ m is considered. As shown in the plot, the coupling peak is reduced by a factor of 2.4 when compared to the idealistic case ($\sigma \rightarrow 0$). This reduction is estimated when varying the beam width scattering coefficient product (σ . α) from 0 to 1.5, as shown in Figure 5.3. This corresponds to a change of the beam width from 0 to 1 mm. When extracting the maximum coupling efficiency of the device, it becomes essential to eliminate the beam width effect through a deconvolution process.



Figure 5.3: Calculated reduction of the peak power with normalized beam width.

5.3.2 Effect of angle of incident on signal collection

In the proposed optical receiver, two main regions are significantly contributed to the signal collection: the beginning of ZnO NR's on the fiber (around Z_1) and the transient region (around Z_2). The effect of the angle of incidence can be estimated by using the first order approximation as described in equation(5.2), where the phase function can be extracted experimentally from the measured normalized angular power spectrum. The spectrum was obtained by using a nephelometer setup as described in Chapter 4 (Fallah et. al. 2013). The normalized coupled power angular spectrum(the ration of the total coupled power to the total power at normal incidence) are calculated based on equation(5.4) and the results are depicted in Figures. 5.4 and 5.5 in polar and linear forms, respectively. The calculation is based on the assumption that the growth was conducted at the optimum conditions; material concentrations of 1 mM zinc acetate, 10 mM zinc nitrate and hexamine and growth time of 8 hours in an oven at 95°C.



Figure 5.4: Calculated normalized power angular spectrum of the total coupled light inside the fiber at optimum growth condition.

At the optimum nanorod's growth parameters, the measured normalized angular power shows a broad spectrum which indicates that the coupling is maximum at the higher scattering angles. The calculations show higher coupling efficiency (3 dB) near the 0° incidence, compared to that at 90°. Practically, the results in Figure 5.5 might not be accurate near small incident angles, as on-axis coupling through the tip of the fiber could result in a higher power at smaller incident angles of light. Another source of error in the simple model built using equation (5.2) is due to the fact that at small incident angles the change in the scattering pattern is expected to be different than the proposed linear shift.



Figure 5.5: Calculated change of the coupled power with incident angle.

To realize the proposed wide FOV optical receiver, the optimum parameters obtained from previous chapter (section 4.5) was used to examine the excitation of core modes through hydrothermal growth of ZnO NR's on optical fiber. Core mode excitation is realized through the optimization of the removal of the cladding region (and part of the core) to allow the scattered light to guide into the core region to achieve maximum coupling efficiency. The following sections describe the optical characterization of the proposed antenna.

5.4 Optical characterization of the proposed wide FOV receiver on MMF

To characterize the fabricated samples, the coupling power as a function of the incident location was first investigated by scanning a fiber coupled laser source along

the fiber in a similar way explained in section 4.3. The graph in Figure 5.6 shows a typical plot of the measured power upon scanning along a sample comprised of a multimode fiber wet etched for 20 minutes followed by coating with ZnO NR's. The scanning region covers both locations z_1 and z_2 . The plot clearly shows two locations of high coupling power. The first area is at the beginning of the coating of the ZnO on the cladding region (location z_1) while the second maximum coupling is at the transition region (z_2) where light couples to the core mode through the nanorods. The graph shows that the power coupled to the core modes is higher than that coupled to the cladding modes. This is consistent with the assumption in section two, where lower critical angle between the core and the surrounding increases the linear integration and hence the total coupled power. The captured images of the tip of the fiber show high confinement in the cladding region for the first peak (z_1) while the core modes were fully excited as shown by the second peak (z_2). Moving away from the transient region, power exponentially decays due to leakage of the core mode through the NR's present on the etched region. This is shown in the plot and the images of the fiber tip Figure 5.6.



Figure 5.6: Light scattering through fiber from cladding mode to the core mode at different parts of the fiber; transition region has highst peak .The images taken at the tip of the fibers are also shown in the figure.The captured images show high confinement in the cladding and the core regions. The exponential decay of the power is due to leakage of the core mode.

To optimize the excitation of the core modes, different ZnO NR's coated samples prepared with different etching times. The measured coupled power for excitation around the transient region is shown in Figure 5.7. The peak power versus etching time is plotted in Figure 5.8. The results show that maximum coupling efficiency was achieved in samples etched for 20 minutes Fig 5.6 which corresponds to a core diameter of around 62 μ m. It is worth mentioning that the plots show a shift in peaks for different

samples. This is mainly due to inaccuracy in detecting the beginning of the etched region while scanning the fibers.



Figure 5.7: Measured power at the end of the fiber coated with ZnO NR's for different etching times versus the scanning distance when exiting the core modes.



Figure 5.8: Maximum core power versus etching time.

In these measurements, a fiber coupled laser source with output power of 11 μ W was used and the output of the fiber was not collimated. That resulted in a finite beam of approximately 540 μ m diameter (a value of $\alpha \sigma$ around 0.78) incident on the fiber sample. Hence, the portion of the light that excites the coupling to core and cladding modes can be expressed as:

$$P_{sourc} = P_{Laser} \frac{1}{2} erf(r/\sigma)$$
(5.27)

Where r is the radius of the fiber.

From equation (5.4), the incident source power can be estimated to be around 0.61 μ W. The idealistic coupling efficiency of the device is estimated by dividing the peak power in Figure 5.5 to the source power multiplied by the corresponding ratio from Figure 5.3 in order to eliminate the finite beam effect, as expressed in the following equation:.

$$\eta_{ideal} = \frac{P_{peak}}{P_{source} \times (\frac{P_{finite}}{P_{ideal}})}$$
(5.28)

Using equation (5.6), the ideal coupling efficiency (when the incident beam waist reaches zero) is 4.7%. Similarly, the coupling efficiency to the cladding mode is 2.7%. This is in agreement with the previously reported value of 5% total coupling considering both directions of propagation inside the fiber (Fallah et al., 2013).

It is worth mentioning that in order to ensure the proper alignment of the source parallel to the fiber, the magnified image formed by the divergence of the laser beam was observed on a screen placed at a distance of 6 cm away from the fiber groove. This provides a total magnification factor of 6.5. This image was used to ensure that the fiber is always centered with the laser beam through a rotation stage which holds the fiber sample. The light source was aligned along the center of rotation of the stage.

5.4.1 Wide Field of View

To measure the power coupling versus the incidence angle, an optical nephelometer is used as schematically represented in Figure 5.9. The setup consists of a rotating arm with the fiber placed along the center of the rotating stage and the source is placed at the end of the arm. The fiber is directly connected to an optical detector. The stage and power meter are synchronized through a power coupling unit where the power is recorded for each rotation angle. The source used here is a collimated red light emitting diode (LED) with a central wavelength of 650 nm. The source is rotated from 0 to 90 degrees and the ratio of the recorded power for different angles on incidence of source light is recorded to obtain the working angular spectrum and compared to the calculated values, shown in Figure 5.10. The plots show a good agreement between the measurements and the model following equation(5.2) for incident angles higher than 30° . At lower incidence angles, the measured power increases in an almost exponential form due to the extra butt coupling from the end of the fiber as well as the deviation of the scattered pattern from the approximated linear shift.



Figure 5.9: In house built nepholometer to measure the response of one fiber with ZnO NR's attached to an optical detector used as an optical reciever.

At lower incidence angles, the measured power increases in an almost exponential form due to the extra light received from the butt coupling from the end of the fiber as well as the deviation of the scattered pattern from the approximated linear shift. It is worth mentioning that butt coupling occurs due to the light incident on the tip of the fiber is at low angles which allows total internal reflection inside the fiber. This will strongly increase the amount light that reaching the detector and the butt coupling light may exceeds the received light due to scattering.



Figure 5.10: Measured normalized coupling power(to 90° incident) versus incident angle. Dashed line shows the theoretically calculated coupling power. The inner graph shows the polar plot of the normalized coupled power.

5.5 Optical characterization for FOV optical receiver on POF

In the previous section, excitation to core modes through etching the cladding region showed higher coupling efficiency due to the reduction of the critical angle inside the core (lower index contrast). Coupling efficiency can be further improved when increasing the exposed area through the use of larger core fiber. Large core glass fibers do exist however they do suffer of lack of flexibility and become very fragile. This increases the difficulty in handling them. The recent development in plastic optical fiber especially with the extensive reduction in their absorption made them a lower cost alternative for large core cladding less optical fiber. As explained in section 3.3.3, uniform growth of nanorods on POF was achieved through the use of 1mM zinc acetate in ethanol solution and 10 mM of zinc nitrate and Hexamine at 95°C inside oven for 8h.



Figure 5.11: Wide FOV optcial receiver based on plastic optcial fiber.
POF with optimal growth condition is then used for wide field of view optical reception as shown in Figure 5.12. In the setup, the incidence angle on the POF coated with ZnO NR's was fixed at 90°. The detector was placed at the end of the POF to record the signal in computer unit attached to the setup. The characterization process is divided in two parts: eye diagram analysis and Bit Error Rate (BER) estimation.

5.5.1 Eye diagram graph

To evaluate the POF samples for wide field of view reception at longer distances, eye diagram graphs are recorded. Figure 5.12 shows the optical set up used to measure the eye diagrams performance of the proposed optical antenna configured withthe POF.As shown in the figure, this optical set up comprises of a power supply, LED spot light source, a function generator, LED modulator circuit, an oscilloscope and a detector. The light is powered and modulated by a power supplier to provide a DC level. The function generator connected to a modulator that adds a modulation signal on top of the DC light. The spot light shines at an angle of 90° on the POF sample. The recorded optical intensity by the detector is displayed on the oscilloscope, which is connected to a PC unit to record the data. A software code keeps multiple tracks of the recorded ones and zeros to generate the eye diagrams.



Figure 5.12: Optical set up for POF wide FOV optical receiver for eye diagram measurement.

Here, two types of optical antenna samples are prepared; without and with pretreatment before seeding process for ZnO growth. Four samples have been prepared for optical antenna with different pretreatment; Thiol (98%), Thiol + NaOH, tween 80 and tween 80 + NaOH. Figs. 5.13.a.b.c.d show some examples of the eye diagrams, which were obtained for the antenna with treated POF samples at three different distances (from 10 cm to 90 cm). The samples are positioned at 90° from LED source in the experiment. As shown in Figure 5.13(d), the transmission performance is the best with the antenna sample treated with tween 80 and NaOH, which showing the largest eye diagram opening at distance as far as 90 cm.



Figure 5.13 :(a) Eye digaram garph for treated POF with thiol for different
distance , (b) Treated POF with Thiol and NaOH , (c) Treated POF with tween 80 ,
(d) Treated POF with tween 80 and NaOH (the x axis in all plots is time samples where the total width is the time period of one pulse).

The treated POF with other techniques are shown reasonable results at far as 90cm as shown in Figures 5.13 (a-d). The sample with pretreatment of tween 80 and NaOH in seeding solution shows the largest open eye diagram compared to other samples at the maximum distance of 90 cm. Figure 5.14 shows the detected peak to peak voltage at different distances when fixing the incident angle at 90° for various optical antennas with different treatments. As shown in the figure, the treated POF with NaOH and tween 80 showed the highest peak to peak voltage, which is in good agreement with the eye diagram results.



Figure 5.14: Voltage peak to peak versusthe distance (in cm) for POF treated with different compunds.

Our concern is to have wide FOV optical receivers. Figure 5.15 shows the voltage peak to peak against the light incident angles for both treated (with NaOH and tween 80) and untreated (bare) POFs. In the experiment, the light incident angle is varied from 0° to 90° and the distance from LED to optical antenna is fixed at 10 cm. Although bare POF showed a higher voltage at lower angles, the performance of the

optical antenna is improved at a larger angle of incidence for the proposed optical antenna with the treated POF.



Figure 5.15: Peak to peak voltage as a function of the angle for bare and tretaed POF.

In actual system Bit Error Rate (BER) is more reliable method to characterize the performance of the communication link. In the following section BER performance of the communication link is investigated for various POF samples as the optical antenna.

5.5.2 Bit Error Rate (BER) measurement

In telecommunication system, one of the important ways to test quality of transmitted signal is bit error rate (BER). BER is ratio between of number of bits that did not receive correctly (N $_{error}$) to the total number of transmitted bits (N $_{total bits}$).

$$BER = \frac{N_{error}}{N_{total \ bits}}$$
(5.29)

Without correction schemes, typical value for acceptable bit error in any communication link is in the order 10⁻³. That means for every 1000 bit which few error bits are received. This measured ratio is affected by many factors including: signal to noise and distortion. Figure 5.16 illustrates a simple BER test pattern.



Figure 5.16: Example of BER coding pattern tester.

The BER test scheme in figure 5.17 is used to characterize the performance of the different treatments of the POF samples when used as optical receiver. This is correlated to strength and pattern of the light scattered by the ZnO NR's on the fiber. As it is mentioned earlier in chapter 4, scattering properties of the nanorods are affected by the uniformity of growth. Better uniformity was observed from the SEM images when POF was treated with Tween 80 and NaOH (Chapter 3). The results from eye diagram supported this observation when the group of POF treated with tween 80 and NaOH, showed better results at longer distance and higher angles.

The BER measurements for all the samples were carried using the experimental setup in Figure 5.17. This set up comprises of bits pattern generator, transmitter, digital to analog converter (DAC), analog to digital converter (ADC) and receiver. A modulator circuit is used to modify the current supplied to the LED such that the desired bits pattern is induced on the light transmission. The modulated light intensity falls on the POF coated with ZnO NR's. The rods scatter the light inside the fiber core. Part of the scattered light is then guided to the end of fiber, which is connected to the detector.

The optical signal is converted to the electrical signal by the detector. The converted signal is then shown on the oscilloscope. The detector is also connected to a DAC which sends the digital signal to the receiver module. The receiver module compares the received bits to the expected pattern. BER is the number of wrong bits divided by the total transmitted bits. Here, 10 bits pattern of 1100110011 are sent 1000 times and the bit error count is used to extract the BER. Counting the number of the error bit in the receiption over a period of time, the link bit error rate is estimated. In this case, the BER is calculated by counting the number of wrongly received bits and dividing it by 100000 (The total number of transmitted bits). In the setup, the receiver units counts the number of error and coversion to BER is done through division by 10000.Figure 5.18 shows bit error rate results, which were obtained using optical receiver based on POF coated with ZnO NR's. The performance of BERs is compared for different pre-treatment techniques that are used before growth. When no error correction schemes are applied, values less than 10⁻³ are assumed good transmission link.



Figure 5.17: BER set up to measure the quality of optical link when using coated POF as an optical receiver.

As it was expected and is cleared from the graph, the treated POF with tween 80and NaOH show the lowest BER as compared to POF with other treatments. The result is in good agreement with the eye diagram results of Figures. 5.13 and 5.14. Figure 5.19 shows the BER for the POF based receiver, which the ZnO growth was obtained by pretreating the fiber with NaOH and tween 80 at various high light incident angles(from 50° to 90°). In the experiment, the travelling distances are fixed at 40 and 50 cm. It is observed that the BER is almost zero at 40cm distance. The maximum BER of around 0.45 is obtained at 90° for 50 cm distance.



Figure 5.18 : BER for different treated POF groups at 90 °angle.



Figure 5.19: BER for treated POF with NaOH and tween 80 at 40 and 50 cm distance and high angles(50 ° to 90°).

It is worth to mention that in practice, OWC system is implemented where a spot light mounted on the ceiling is used for illumination as well as signal transmission. This sets of constrain on the distance between transmitted and receiver to about 1.30 m. From the presented experiment, it is shown that the longest distance was 50 cm. These are initial results and further improvement of the growth condition, increasing the number of sample and applying error correction schemes in the communication link can allow pushing the distance to the practical limits.

5.6 Summary

In this work we have presented a possible very wide FOV solution for free space optical wireless communication based on ZnO NR's grown on optical fiber. The fibers were thinned using HF acid wet etching process to ensure coupling to the core modes. In the proposed scheme, two excitation peaks are presented: one at the beginning of the ZnO NR's coated area, where cladding modes are excited, while the other located at the etching transient region where core modes are excited. At 90° light incidence, maximum coupling efficiencies of 4.7% and 2.7% were found for core and cladding modes, respectively. The amount of power coupled to the fiber slowly increases by 150% upon decreasing the angle of incidence from 90° to 30°. For lower angles of incidence, the power coupled to the fiber increases in almost exponential form due to the extra butt coupling from the end of the fiber as well as the deviation of the scattered. To increase the amount of power coupling to the core region, larger MMF diameter is needed. POF provides a flexible and low cost alternative to replace silica base MMF. ZnO NR's were coated on the POF using the same condition as MMF. The difference in the substrate material (PMM compared to fused silica) required an additional treatment with different chemical compound to have more uniformity on surface. Using POF with nanorods for

wide FOV optical reception, the eye diagram and BER were recorded for the different treatments at high angles and several distances.

CHAPTER 6

CONCLUSION AND FUTURE OUTLOOK

6.1 Introduction

In this work a new concept of side coupling to optical fiber utilizing the scattering properties of nano structures is introduced. Here ZnO NR's grown on the side of the fiber are used as scattering components due to their unique properties (high refractive index, shape controllability and ease of fabrication). ZnO rods scatter the incident light at angles larger than the critical angle allowing portion of the light to couple to the guided modes in the core and cladding regions. A highly ordered and dense ZnO NR's structure is grown on the cladding and core regions of silica glass optical fibers by hydrothermal technique. This technique is also used to grow ZnO NR's on plastic or polymer optical fiber (POF). Among solution and gas phase of growth techniques, hydrothermal method is used in this work as it does not require high temperature and complicated vacuum system. It is as well a low cost and simple process. The combination of nanostructures and fiber systems is used to demonstrate a simple wide field of view (FOV) optical receiver.

Growth of ZnO NR'S on optical fiber MMF to couple light into the cladding region

There was limited number of reported work dealing with growth of nanorods on rounded surfaces such as fiber. Hence, the effect of hydrothermal growth of ZnO NR's on the optical fiber to get optimum growth condition was investigated. The concentration of zinc acetate solution used for seeding was found to play important role in nanorods scattering. At higher zinc acetate concentration than 1 mM, the randomness in nanorods directionality increased due to the clustering of seeds. That caused higher multiple forward scattering and back scattering as well, as results; coupling efficiency reduces almost 10 %. Another important factor was growth time.

Increasing the growth time from 5 to 20 h increased the length of nanorods from 1.7 μ m to ~6.7 μ m respectively. Scattering cross section (C_{sc}) increased as a result. Although the increase in rods length caused higher scattering cross section, the density of nanorods decreased from ~2.91× 10⁹ cm ⁻² to ~ 1.22× 10⁹ cm ⁻². According to Eq 4.15, this coupling efficiency (CE) is directly related to density and scattering cross section. The optimum growth of nanorods is obtained when 1mM Zinc acetated dehydrate dissolved in 20 ml ethanol is used for seeding nanoparticle and then 10 mM zinc nitrate and hexamine are used for hydrothermal growth at 95° C inside oven for 8 hour. Maximum excitation of the cladding modes of silica multimode optical fiber (MMF) by side coupling of light is obtained with ZnO NR's of length ~2.2µm and average coupling efficiency was 2.65%.

Growth of ZnO NR'S on optical fiber MMF to couple light into core region by etching cladding

In order to grow nanorods on the core region of the optical fiber, wet etching process was used to remove the cladding region. Hydrofluoric acid (HF 50%) was applied to etch the cladding region of the standard MMF with core and cladding diameters of 105 μ m and 125 μ m respectively. The strong aggressive properties of HF caused silica to dissolve though in it. To get the optimum time for dipping MMF samples to reach the core diameter different dipping time were investigated. The results

showed that after 10 min the cladding region was removed. However, the optimum results were for the samples with 20 min etching time.

The optical characterization of the etched samples showed two excitation peaks, one at the beginning of the ZnO NR's coated area where cladding modes were excited and the other at the etching transition region where core modes were excited. Maximum coupling efficiencies of 4.7% and 2.7% happened at 90° of incident light for core and cladding modes respectively. The amount of power coupled to fiber could increase by 150 % upon decreasing the angle of incident from 90° to 30°. For lower angles of incident the power coupled to the fiber increase in almost exponential form. By utilizing a bundle of the fibers coated with ZnO NR's the total coupled power could be enhanced at higher angle.

Growth of ZnO NR'S POF

POF is another type of optical fiber that can be used to grow ZnO NR's. POF was used due to its larger core diameter which could increase the light coupling into the core modes. Using POF provides a lower cost alternative to large core glass fiber and it is also easier to handle. The growth of nanorods was initially applied with the optimum conditions obtained from glass MMF. Due to the difference in the substrate, PMMA in the case of POF, the attachment of nanorods was not stable and the rods could not grow uniformly. To achieve better uniformity, POF samples were treated by dipping method inside Tween 80 solution for 15 min, and ImM NaOH added into zinc acetate and ethanol seeding solution.

Wide FOV optical receiver

The combination of advantageous nanostructures and fiber system open a possible way to build a wide field of view optical receiver in optical communication system and applications. The angular response of the receiver is tested using an in-house built nephelometer. Light coupling efficiency is extracted by de-convolving the finite beam extinction from the measured power. The results were compared to a first order analytical model where the phase function is assumed to linearly shift with the incident angle. The trend of the experimental measurements agrees with the model. 180° FOV is verified and maximum coupling efficiency of around 2.5% for a single fiber is reported. POF samples were also used in practical test for wireless communication system.Eye diagram and bit error rate (BER) were measured to characterize the quality of the optical link when using wide FOV as an optical receiver. From these results, it is concluded that the excitation of core modes through side coupling allows for application of these devices towards wide field of view optical receivers.

6.2 Suggestion for future works

In this final demonstration of the use of ZnO on POF as a wide FOV receiver, the acceptable optical link was short (less than 50 cm). Though the main concept of utilization of the nanorods for wide field of view optical reception was demonstrated, more work is needed to improve the properties of the system to reach practical distances larger than 1 meter. To overcome this issue, having more accurate BER tester, detector, more powerful light emitting diode (LED) source as well as better control of the growth condition will be required. More fibers can be used to increase signal to noise ratio. Higher brightness LED sport light is to be used as well. With further enhancement of the

modulation and detector circuits a longer distance within the acceptable range of BER can be achieved.

REFERENCES

Andrade, E., & Miki-Yoshida, M. (1999). Growth, structure and optical characterization of high quality ZnO thin films obtained by spray pyrolysis. *Thin Solid Films*, *350* (1), 192-202.

Antelius, M., Gylfason, K. B., & Sohlström, H. (2011). An apodized SOI waveguide-tofiber surface grating coupler for single lithography silicon photonics. *Optics Express*, 19 (4), 3592-3598.

Azadeh, M. (2009). Fiber optics engineering (p. 379). Springer.

Bahaa E. A., Malvin C. T. (2007). Fundamental of photonics. Wiley Series in Pure and Applied Optics.

Bao, J., Zimmler, M. A., Capasso, F., Wang, X., & Ren, Z. F. (2006). Broadband ZnO single-nanowire light-emitting diode. *Nano letters*, 6 (8), 1719-1722.

Baruah, S., Thanachayanont, C., & Dutta, J. (2008). Growth of ZnO nanowires on nonwoven polyethylene fibers. *Science and Technology of Advanced Materials*, 9 (2), 025009.

Baruah, S., & Dutta, J. (2009). Hydrothermal growth of ZnO nanostructures. *Science and Technology of Advanced Materials*, *10*(1), *013001*.

Baruah, S., & Dutta, J. (2009). Effect of seeded substrates on hydrothermally grown ZnO NR's. *Journal of sol-gel science and technology*, *50 (3)*, *456-464*.

Baruah, S., & Dutta, J.(2009). pH-dependent growth of zinc oxide nanorods. *Crystal Growth*, 311 (8), 2549-2554.

Batumalay, M., Harith, Z., Rafaie, H. A., Ahmad, F., Khasanah, M., Harun, S. W., ... & Ahmad, H. (2014). Tapered plastic optical fiber coated with ZnO nanostructures for the measurement of uric acid concentrations and changes in relative humidity. *Sensors and Actuators A: Physical*, *210*, 190-196.

Beres, C., de Nazaré, F. V. B., de Souza, N. C. C., Miguel, M. A. L., & Werneck, M. M. (2011). Tapered plastic optical fiber-based biosensor–Tests and application. *Biosensors and Bioelectronics*, *30 (1)*, *328-332*.

Bharadwaj, P., Deutsch, B., & Novotny, L. (2009). Optical antennas. Advances in Optics and Photonics, 1 (3), 438-483.

Bilro, L., Alberto, N., Pinto, J. L., & Nogueira, R. (2012). Optical Sensors Based on Plastic Fibers. *Sensors (Basel, Switzerland), 12 (9), 12184–12207.*

Bora, T., Fallah, H., Chaudhari, M., Apiwattanadej, T., Harun, S. W., Mohammed, W. S., & Dutta, J. (2014). Controlled side coupling of light to cladding mode of ZnO nanorod coated optical fibers and its implications for chemical vapor sensing. *Sensors and Actuators B: Chemical*, 202, 543-550.

Cao, G. (2004). Synthesis, Properties and Applications. Imperial college press, London.

Carruther, J. B., & Kahn, J. M. (2000). Angle diversity for nondirected wireless infrared communication. *Communications, IEEE Transactions on, 48 (6), 960-969.*

Chen, B. J., Sun, X. W., Xu, C. X., & Tay, B. K. (2004). Growth and characterization of zinc oxide nano/micro-fibers by thermal chemical reactions and vapor transport deposition in air. *Physica E: Low-dimensional Systems and Nanostructures*, 21 (1), 103-107.

Chen, C. Y., Chen, Q. Z., Shi, M., Zhou, F., Hua, C., & Pang, H. (2012). Nonstoichiometric Zinc Oxide Nanorod Arrays Doped Co or Ni as Effective Antimicrobial Materials. *Advanced Materials Research*, *518*, 760-763.

Chen, N. K., Lin, C. L., & Chi, S. (2007). Wideband tunable wavelength-selective coupling in asymmetric side-polished fiber coupler with dispersive interlayer. *Optics Express*, *15* (26), *17747-17753*.

Choy, J. H., Jang, E. S., Won, J. H., Chung, J. H., Jang, D. J., & Kim, Y. W. (2003). Soft Solution Route to Directionally Grown ZnO Nanorod Arrays on Si Wafer; Room-Temperature Ultraviolet Laser. *Advanced Materials*, *15* (22), 1911-1914.

Dalal, S. H., Baptista, D. L., Teo, K. B. K., Lacerda, R. G., Jefferson, D. A., & Milne, W. I. (2006). Controllable growth of vertically aligned zinc oxide nanowires using vapour deposition. *Nanotechnology*, *17* (*19*), *4811*.

Danwittayakul, S., Lakshman, K., Al-Harthi, S., & Dutta, J. (2014). Enhanced hydrogen selectivity via photo-engineered surface defects for methanol steam reformation using zinc oxide–copper nanocomposite catalysts. *Applied Catalysis A: General*, 471, 63-69.

Deng, P., Yuan, X., Kavehrad, M., Zhao, M., & Zeng, Y. (2012). Off-axis catadioptric fisheye wide field-of-view optical receiver for free space optical communications. *Optical Engineering*, *51* (6), 063002-1.

Desai, A. V., & Haque, M. A. (2007). Mechanical properties of ZnO nanowires. *Sensors and Actuators A: Physical*, 134 (1), 169-176.

Dikovska, A. O., Atanasova, G. B., Nedyalkov, N. N., Stefanov, P. K., Atanasov, P. A., Karakoleva, E. I., & Andreev, A. T. (2010). Optical sensing of ammonia using ZnO

nanostructure grown on a side-polished optical-fiber. Sensors and Actuators B: Chemical, 146 (1), 331-336.

Dikovska, A. O., Atanasov, P. A., Andreev, A. T., Zafirova, B. S., Karakoleva, E. I., & Stoyanchov, T. R. (2007). ZnO thin film on side polished optical fiber for gas sensing applications. *Applied Surface Science*, 254 (4), 1087-1090.

Ding, D., & Liu, X. (1999). Approximate description for Bessel, Bessel–Gauss, and Gaussian beams with finite aperture. *JOSA A*, *16* (6), 1286-1293.

Ding, Y., & Wang, Z. L. (2009). Structures of planar defects in ZnO nanobelts and nanowires. *Micron*, 40 (3), 335-342.

Du, G. T., Liu, W. F., Bian, J. M., Hu, L. Z., Liang, H. W., Wang, X. S., & Yang, T. P. (2006). Room temperature defect related electroluminescence from ZnO homojunctions grown by ultrasonic spray pyrolysis. *Applied physics letters*, 89 (5), 052113-052113.

Fallah, H., Harun, S. W., Mohammed, W. S., & Dutta, J. (2014). Excitation of core modes through side coupling to multimode optical fiber by hydrothermal growth of ZnO NR's for wide angle optical reception. *JOSA B*?, *31*(9), 2232-2238.

Fallah, H., Chaudhori, M., Harun, S. W., Mohammed, W. S., & Dutta, J. (2013). Demonstration of side coupling to cladding modes through zinc oxide nanorods grown on multimode optical fiber. *Optics Letters, 38 (18), 3620-3622.*

Fan, H. J., Fleischer, F., Lee, W., Nielsch, K., Scholz, R., Zacharias, M., & Krost, A. (2004). Patterned growth of aligned ZnO nanowire arrays on sapphire and GaN layers. *Superlattices and Microstructures*, *36* (1), *95-105*.

Gandhi, A. C., Hung, H. J., Shih, P. H., Cheng, C. L., Ma, Y. R., & Wu, S. Y. (2010). In situ confocal Raman mapping study of a single Ti-assisted ZnO nanowire. *Nanoscale research letters*, 5 (3), 581-586.

Ghassemlooy, Z., Popoola, W., & Rajbhandari, S. (2012). *Optical wireless communications: system and channel modelling with Matlab*[®]. CRC Press.

Golnabi, H., & Azimi, P. (2008). Design and operation of a double-fiber displacement sensor. *Optics Communications*, 281 (4), 614-620.

Goodman, J. W. (2005). *Introduction to Fourier optics*. Roberts and Company Publishers.

Grassini, S., Ishtaiwi, M., Parvis, M., & Vallan, A. (2015). Design and Deployment of Low-Cost Plastic Optical Fiber Sensors for Gas Monitoring. *Sensors (Basel, Switzerland)*, 15 (1), 485–498.

Grattan, K. T. V., & Sun, T. (2000). Fiber optic sensor technology: an overview. *Sensors and Actuators A: Physical*, 82 (1), 40-61.

Guo, L., Ji, Y. L., Xu, H., Simon, P., & Wu, Z. (2002). Regularly shaped, singlecrystalline ZnO NR's with wurtzite structure. *Journal of the American Chemical Society*, 124 (50), 14864-14865.

Hahn, D. V., Brown, D. M., Rolander, N. W., Sluz, J. E., & Venkat, R. (2010). Fiber optic bundle array wide field-of-view optical receiver for free space optical communications. *Optics letters*, *35* (21), 3559-3561.

Hayat, A., Catanante, G., & Marty, J. L. (2014). Current Trends in Nanomaterial-Based Amperometric Biosensors. *Sensors*, *14* (12), 23439-23461.

He, J. H., Hsin, C. L., Liu, J., Chen, L. J., & Wang, Z. L. (2007). Piezoelectric gated diode of a single ZnO nanowire. *Advanced Materials*, *19* (6), 781-784.

Hong-Yuan, W., Wei-Guo, H., Pan-Feng, Z., Xiang-Lin, L., Qin-Sheng, Z., & Zhan-Guo, W. (2007). ZnO Nanostructures Grown on AlN/Sapphire Substrates by MOCVD. *Chinese Physics Letters*, 24 (6), 1738.

Hu, Y. H., Wang, H., & Hu, B. (2010). Thinnest Two-Dimensional Nanomaterial—Graphene for Solar Energy. *ChemSusChem*, *3* (7), 782-796.

Hughes, W. L., and Wang., Z. (2005).Controlled synthesis and manipulation of ZnO nanorings and nanobows. *Applied Physics Letters*, 86 (4), 043106.

Jang, H. S., Park, K. N., Kim, J. P., Sim, S. J., Kwon, O. J., Han, Y. G., & Lee, K. S. (2009). Sensitive DNA biosensor based on a long-period grating formed on the side-polished fiber surface. *Optics Express*, 17 (5), 3855-3860.

Jeong, W., Kavehrad, M., & Jivkova, S. (2001). Broadband infrared access with a multispot diffusing configuration: performance. *International Journal of Wireless Information Networks*, 8 (1), 27-36.

Kbashi, H. J. (2012). Fabrication of submicron-diameter and taper fibers using chemical etching. *Journal of Materials Science & Technology*, 28 (4), 308-312.

Keiser. G. (2010). Optical fiber communication -4th ed. Wiley Series in Pure and Applied Optics.

Kohls, M., Bonanni, M., Spanhel, L., Su, D., & Giersig, M. (2002). Green ErIII luminescence in fractal ZnO nanolattices. *Applied physics letters*, *81* (20), 3858-3860.

Ko, Y. H., Chung, K. S., & Yu, J. S. (2012). Fabrication and Optical Property of Vertically-Aligned ZnO/Si Double Nanostructures. *Journal of nanoscience and nanotechnology*, *12* (6), 4570-4576.

Koike, Y., Ishigure, T., & Nihei, E. (1995). High-bandwidth graded-index polymer optical fiber. *Lightwave Technology, Journal of*, *13* (7), 1475-1489.

Konstantaki, M., Klini, A., Anglos, D., & Pissadakis, S. (2012). An ethanol vapor detection probe based on a ZnO nanorod coated optical fiber long period grating. *Optics Express*, 20 (8), 8472-8484.

Krunks M. and Mellikov E. (1995). Zinc oxide thin films by spray pyrolysis method. *Thin Solid Films*, 270 (3), 33-36.

Kulkarni, A., Lee, J. H., Nam, J. D., & Kim, T. (2010). Thin film-coated plastic optical fiber probe for aerosol chemical sensing applications. *Sensors and Actuators B: Chemical*, 150 (1), 154-159.

Lagashetty, A., Havanoor, V., Basavaraja, S., Balaji, S. D., & Venkataraman, A. (2007). Microwave-assisted route for synthesis of nanosized metal oxides. *Science and Technology of Advanced Materials*, 8 (6), 484-493.

Leschkies, K. S., Divakar, R., Basu, J., Enache-Pommer, E., Boercker, J. E., Carter, C. B., & Aydil, E. S. (2007). Photosensitization of ZnO nanowires with CdSe quantum dots for photovoltaic devices. *Nano Letters*, 7 (6), 1793-1798.

Lim, K. S., Harun, S.W., Arof, H., Ahmad, H. (2012). Selected topic on optical fiber technology. Chapter 17, Fabrication and application of Microfiber. *ISBN 9788-953-51-0091-1, Page 680.*

Liu, B., & Zeng, H. C. (2003). Hydrothermal synthesis of ZnO NR's in the diameter regime of 50 nm. *Journal of the American Chemical Society*, 125 (15), 4430-4431.

Liu, J., Xie, C., Dai, X., Jin, L., Zhou, W., & Lieber, C. M. (2013). Multifunctional three-dimensional macroporous nanoelectronic networks for smart materials. *Proceedings of the National Academy of Sciences of the United States of America*, 110 (17), 6694–6699.

Liu, J. S., Cao, J. M., Li, Z. Q., Ji, G. B., & Zheng, M. B. (2007). A simple microwaveassisted decomposing route for synthesis of ZnO NR's in the presence of PEG400. *Materials Letters*, 61 (22), 4409-4411.

Liu, R., Zhang, T., Zhao, S. L., Xu, Z., Zhang, F. J., Yuan, G. C., & Xu, X. R. (2008). The preparation and characterization of 1-D orderly ZnO nanorod arrarys. *Guang pu xue yu guang pu fen xi= Guang pu*, 28 (10), 2249-2253.

Liu, Y., Zhang, Y., Lei, H., Song, J., Chen, H., & Li, B. (2012). Growth of well-arrayed ZnO NR's on thinned silica fiber and application for humidity sensing. *Optics Express*, 20 (17), 19404-19411.

Liu, Y., Kathan, K., Saad, W., & Prud'homme, R. K. (2007). Ostwald ripening of β -carotene nanoparticles. *Physical review letters*, 98 (3), 036102.

Li, X., Hu, D., Dang, Y., Chen, H., Roco, M. C., Larson, C. A., & Chan, J. (2009). Nano Mapper: an Internet knowledge mapping system for nanotechnology development. *Journal of Nanoparticle Research*, 11 (3), 529–552.

Li, Z. W., & Gao, W. (2007). Growth of zinc oxide thin films and nanostructures by wet oxidation. *Thin Solid Films*, 515 (7), 3323-3329.

Luckhurst, G.R., Veracin, C. A. (1989). The Molecular Dynamics of Liquid Crystals. *ISBN 0-7923-2809-4*.

Mauter, M. S., & Elimelech, M. (2008). Environmental applications of carbon-based nanomaterials. *Environmental Science & Technology*, *42* (16), 5843-5859.

Mazingue, T., Escoubas, L., Spalluto, L., Flory, F., Socol, G., Ristoscu, C., ... & Vainos, N. A. (2005). Nanostructured ZnO coatings grown by pulsed laser deposition for optical gas sensing of butane. *Journal of applied physics*, *98* (7), 074312-074312.

Mohanty, L., & Kuang, K. S. (2011). Surface structure monitoring with plastic optical fiber. *Optics and Lasers in Engineering*, 49 (7), 984-987.

Naritomi, M. (1996). CYTOP Amorphous Fluoropolymers for low loss POF. In *POF Asia Pacific Forum* (p. 23).

Nihei, E., Ishigure, T., Tanio, N., & Koiky, Y. (1997). Present prospect of graded-index plastic optical fiber in telecommunication. *IEICE transactions on electronics*, 80 (1), 117-122.

Numata, K., Furusawa, S., & Morikura, S. (1999). Transmission characteristics of 500 Mbps optical link using 650 nm RC-LED and POF. In *Proc. POF* (Vol. 99, pp. 74-77).

Okamoto, K. (2010). Fundamentals of optical waveguides. Academic press

Özgür, Ü., Alivov, Y. I., Liu, C., Teke, A., Reshchikov, M., Doğan, S., ... & Morkoc, H. (2005). A comprehensive review of ZnO materials and devices. *Journal of applied physics*, 98 (4), 041301.

Pajnič, M., Drašler, B., Šuštar, V., Krek, J. L., Štukelj, R., Šimundić, M., ... & Kralj-Iglič, V. (2015). Effect of carbon black nanomaterial on biological membranes revealed by shape of human erythrocytes, platelets and phospholipid vesicles. *Journal of nanobiotechnology*, *13* (1), 28.

Park, J., Choi, H. H., Siebein, K., & Singh, R. K. (2003). Two-step evaporation process for formation of aligned zinc oxide nanowires. *Journal of Crystal Growth*, 258 (3), 342-348.

Pradhan, D., & Leung, K. T. (2008). Controlled growth of two-dimensional and onedimensional ZnO nanostructures on indium tin oxide coated glass by direct electrodeposition. *Langmuir*, 24(17), 9707-9716.

Qiu, L., Goossen, K. W., Heider, D., O'Brien, D. J., & Wetzel, E. D. (2011). Free-space input and output coupling to an embedded fiber optic strain sensor: dual-ended interrogation via transmission. *Optical Engineering*, *50* (9), 094403-094403.

Rao.C.N.R., Prof. Dr. Sood, A.k., Prof. Dr., Subrahmanyam,K.S, and Govindaraj, A. (2009). Graphene: The New Two-Dimensional Nanomaterial. Angewandte Chemie International Edition. 48 (42), 7752–7777.

Satoh, Y., Ohshio, S., & Saitoh, H. (2005). Photoluminescence spectroscopy of highly oriented Y 2 O 3: Tb crystalline whiskers. *Science and Technology of Advanced Materials*, 6 (2), 215-218.

Schidt-Mende, S. and MacManus–Driscoll, J.L. (2007). ZnO nanostructure defects and devices. *Materials today*, 10 (5) 40-48.

Schodek, D. L., Ferreira, P., & Ashby, M. F. (2009). Nanomaterials, nanotechnologies and design: an introduction for engineers and architects. Butterworth-Heinemann.

Skoog, D.A., West, D.M., Holler, F.J., Crouch, S.R. (2003). Fundamentals of Analytical Chemistry- 8thed. Brooks Cole.

Sohaebuddin, S. K., Thevenot, P. T., Baker, D., Eaton, J. W., & Tang, L. (2010). Nanomaterial cytotoxicity is composition, size, and cell type dependent. *Particle and fibre toxicology*, 7 (1), 22.

Spanhel, L. (2006). Colloidal ZnO nanostructures and functional coatings: A survey. *Journal of sol-gel science and technology*, *39* (1), 7-24.

Sugunan, A., Warad, H. C., Boman, M., & Dutta, J. (2006). Zinc oxide nanowires in chemical bath on seeded substrates: role of hexamine. *Journal of Sol-Gel Science and Technology*, *39* (1), 49-56.

Tena-Zaera, R., Elias, J., & Lévy-Clément, C. (2008). ZnO nanowire arrays: optical scattering and sensitization to solar light. *Applied Physics Letters*, 93 (23), 233119.

Thankappan, A., Divya, S., Thomas, S., & Nampoori, V. P. N. (2013). Optical characterization of ZnO nanoplates embedded in polymeric matrices for optical limiting applications. *Optics & Laser Technology*, *52*, 37-42.

Umar, A., Ribeiro, C., Al-Hajry, A., Masuda, Y., & Hahn, Y. B. (2009). Growth of highly c-axis-oriented ZnO NR's on ZnO/glass substrate: growth mechanism, structural, and optical properties. *The Journal of Physical Chemistry C*, *113* (33), 14715-14720.

Vergés, M. A., Mifsud, A., & Serna, C. J. (1990). Formation of rod-like zinc oxide microcrystals in homogeneous solutions. *Journal of the Chemical Society, Faraday Transactions*, 86 (6), 959-963.

Voss, T., Svacha, G. T., Mazur, E., Müller, S., Ronning, C., Konjhodzic, D., & Marlow, F. (2007). High-order waveguide modes in ZnO nanowires. *Nano letters*, 7 (12), 3675-3680.

Wang, L., Zhang, X., Zhao, S., Zhou, G., Zhou, Y., & Qi, J. (2005). Synthesis of wellaligned ZnO nanowires by simple physical vapor deposition on c-oriented ZnO thin films without catalysts or additives. *Applied Physics Letters*, *86* (2), 024108-024108.

Wang, T. Q., Sekercioglu, Y. A., & Armstrong, J. (2012, December). Hemispherical lens based imaging receiver for MIMO optical wireless communications. In *Globecom Workshops (GC Wkshps), 2012 IEEE* (pp. 1239-1243). IEEE.

Wang, W. M., Grattan, K. T., Palmer, A. W., & Boyle, W. J. O. (1994). Self-mixing interference inside a single-mode diode laser for optical sensing applications. *Lightwave Technology, Journal of*, *12* (9), 1577-1587.

Wang, Z. L. (2004). Zinc oxide nanostructures:growth, properties and applications. *Journal of Physics: Condensed Matter*, *16* (25), R829.

Wang, Z. L. (2007). Novel nanostructures of ZnO for nanoscale photonics, optoelectronics, piezoelectricity, and sensing. *Applied Physics A*, 88 (1), 7-15.

Wang, Z. L. (2009). ZnO nanowire and nanobelt platform for nanotechnology. *Materials Science and Engineering: R: Reports*, 64 (3), 33-71.

Wei, M., Zhi, D., & MacManus-Driscoll, J. L. (2005). Self-catalysed growth of zinc oxide nanowires. *Nanotechnology*, *16* (8), 1364.

Weintraub, B., Zhou, Z., Li, Y., & Deng, Y. (2010). Solution synthesis of onedimensional ZnO nanomaterials and their applications. *Nanoscale*, *2* (9), 1573-1587.

Wu, J., & Xue, D. (2010). Controlled etching of hexagonal ZnO architectures in an alcohol thermal process. *Materials Research Bulletin*, 45 (3), 295-299.

Wu, J. J., & Liu, S. C. (2002). Low-temperature growth of well-aligned ZnO NR's by chemical vapor deposition. *Advanced Materials*, *14* (3), 215.

Xu, L., Guo, Y., Liao, Q., Zhang, J., & Xu, D. (2005). Morphological control of ZnO nanostructures by electrodeposition. *The Journal of Physical Chemistry B*, *109* (28), 13519-13522.

Yang, P., Yan, H., Mao, S., Russo, R., Johnson, J., Saykally, R & Choi, H. J. (2002). Controlled growth of ZnO nanowires and their optical properties. *Advanced Functional Materials*, *12* (5), 323.

Yao, B. D., Chan, Y. F., & Wang, N. (2002). Formation of ZnO nanostructures by a simple way of thermal evaporation. *Applied Physics Letters*, *81* (4), 757-759.

Yi, G. C., Wang, C., & Park, W. I. (2005). ZnO NR's: synthesis, characterization and applications. *Semiconductor Science and Technology*, 20 (4), S22.

Yu, K., Zhang, Q., Wu, J., Li, L., Xu, Y. E., Huang, S., & Zhu, Z. (2008). Growth and optical applications of centimeter-long ZnO nanocombs. *Nano Research*, *1* (3), 221-228.

Zhang, L., Webster, T.J (2009). Nanotechnology and nanomaterials: Promises for improved tissue regeneration. *Nano Today*, 4 (1), 66–80.

Zhang, Y., Yu, K., Ouyang, S., & Zhu, Z. (2006). Selective-area growth and field emission properties of Zinc oxide nanowire micropattern arrays. *Physica B: Condensed Matter*, 382 (1), 76-80.

Zhiyong Fan and Jia G. Lu.(2005). Zinc oxide nanostructure synthesis and properties. *Nanoscience and nanotechnology*, *5* (13), 1561-1573.

Zhou, Q., Chen, W., Xu, L., & Peng, S. (2013). Hydrothermal Synthesis of Various Hierarchical ZnO Nanostructures and Their Methane Sensing Properties. *Sensors* (*Basel, Switzerland*), 13 (5), 6171–6182.

Zhuo, R. F., Feng, H. T., Liang, Q., Liu, J. Z., Chen, J. T., Yan, D., ... & Yue, G. H. (2008). Morphology-controlled synthesis, growth mechanism, optical and microwave absorption properties of ZnO nanocombs. *Journal of Physics D: Applied Physics*, 41 (18), 185405.

Zubia, J., & Arrue, J. (2001). Plastic optical fibers: An introduction to their technological processes and applications. *Optical Fiber Technology*, 7 (2), 101-140.

LIST OF PUBLICATIONS

- Bora, T., Fallah, H., Chaudhari, M., Apiwattanadej, T., Harun, S. W., Mohammed, W. S., & Dutta, J. (2014). Controlled side coupling of light to cladding mode of ZnO nanorod coated optical fibers and its implications for chemical vapor sensing. *Sensors and Actuators B: Chemical*, 202, 543-550.
- 2. Fallah, H., Chaudhori, M., Harun, S. W., Mohammed, W. S., & Dutta, J. (2013). Demonstration of side coupling to cladding modes through zinc oxide nanorods grown on multimode optical fiber. *Optics Letters*, 38(18), 3620-3622.
- 3. Fallah, H., Harun, S. W., Mohammed, W. S., & Dutta, J. (2014). Excitation of core modes through side coupling to multimode optical fiber by hydrothermal growth of ZnO NR's for wide angle optical reception. *JOSA B*, *31*(9),2232-2238.