Fabrication and characterisation of polymer optical fibers for smart sensing and optical amplification

M Rajesh
International School of Photonics
Cochin University of Science and Technology
Cochin-682022, India

PhD Thesis submitted to Cochin University of Science and Technology in partial fulfillment of the requirements for the award of the Degree of Doctor of Philosophy

July 2006

Fabrication and characterisation of polymer optical fibers for smart sensing and optical amplification

Ph D Thesis in the field of Photonics

Author
M Rajesh
Research Fellow
International School of Photonics
Cochin University of Science & Technology
Cochin – 682 022, India
Email: rajeshm@cusat.ac.in / rajnair.gvr@gmail.com

Research Advisor
Dr. V P N Nampoori
Professor, International School of Photonics
Cochin University of Science & Technology
Cochin – 682 022, India
Email: vpnnampoori@cusat.ac.in

International School of Photonics,
Cochin University of Science & Technology
Cochin – 682 022, INDIA

www.photonics.cusat.edu

July 2006

Front cover: A bunch of dye doped polymer optical fibers.

CERTIFICATE

Certified that the research work presented in the thesis entitled "***Fabrication and characterisation of polymer optical fibers for smart sensing and optical amplification***" is based on the original work done by Mr. **M Rajesh** under my guidance and supervision at the International School of Photonics, Cochin University of Science and Technology, Cochin – 22, India and has not been included in any other thesis submitted previously for the award of any degree.

Cochin 682 022
15 July, 2006

Dr. V P N Nampoori
(Supervising Guide)

DECLARATION

Certified that the work presented in the thesis entitled "***Fabrication and characterisation of polymer optical fibers for smart sensing and optical amplification***" is based on the original work done by me under the guidance and supervision of Dr. V P N Nampoori, Professor, International School of Photonics, Cochin University of Science and Technology, Cochin – 22, India and has not been included in any other thesis submitted previously for the award of any degree.

Cochin 682 022
15 July, 2006

M Rajesh

Preface

The global proliferation of optical fiber-based communication networks have been the enabling factor in the shift of modern Information technology era from the Electronics Age to the Photonics Age. Even though silica glass fibers have enabled this Information Superhighway, the plastic optical fibers (POFs) are of great commercial interest because they can maintain flexibility at thicker fiber sizes making them more easy to handle and install when the communication systems make their way from the local loop (premise networks, local area networks) closer towards, and into, the home. POF may also be an important building block for devices used in future ultra fast all-optical communication systems because it has many attractive properties that silica optical fiber lacks. Polymer materials have a much lower Young's modulus than inorganic glass and therefore, can possess much larger diameter and still retain their flexibility. Polymer optical fibers also possess a higher numerical aperture (NA). As a result, the acceptance angle, or the light gathering capacity, is large compared to glass optical fibers.

Availability of inexpensive sources in the visible region has increased the utilization of POF in data communication. Implementation of optical communication in the visible region necessitates the development of suitable optical amplifiers working in this region. POF doped with dyes or rare earth elements are potential candidates for this purpose. Laser dyes, which act as highly efficient media for lasing and amplification have a wide range of tunability in the visible region. The range of tunability of laser dyes like Rhodamine B and Rhodamine 6G usually comes between 570nm to 640nm. The advantage of incorporating laser dyes in solid matrices such as POF is that it is

easier and safer to handle them than when they are in liquid form. From recent studies it is found that the dye doped polymer materials haves better efficiency, beam quality and superior optical homogeneity when compared to other solid matrices.

Although many of the applications of optical fibers are based on their capacity to transmit optical signals with low losses, it can also be desirable for the optical fiber to be strongly affected by certain physical parameters of the environment. In this way, it can be used as a sensor of such a parameter. There are many strong arguements for the use of POF as sensors. In addition to their easiness to handle and economically competitive, they present the advantages common to all multimode optical fibers. Specifically, the flexibility and small size of optical fibers enable a great sensitivity to be achieved without having to occupy big volume. Moreover, it has been proved that POF can be employed to detect a great variety of parameters, including temperature, humidity, pressure, and presence of organic and inorganic compounds, wind speed and refractive index.

The proposed thesis presented in six chapters deals with the work carried out on polymer optical fibre smart sensor and dye doped polymer optical fiber amplifier.

Chapter 1: Polymer fiber optics has become a very interesting area as it has a very profound application in short distance communication such as LAN, DTH, Automotive communication and in both industrial and domestic lighting. Since the use of conventional energy is a buzz word of the present energy thirsty world, fiber optic lighting has become a boon. During day time when there is surplus of natural light source, the sun, it can be very efficiently utilized for illumination purposes in large industrial areas by guiding light through large core polymer fibers, which will cut the energy bills to virtually zero. Due to its ease of

fabrication and large numerical apertures polymer optical fiber based devices like couplers, routers, mode scramblers, mode filters can be easily and cost effectively manufactured. Polymer integrated devices like waveguides has suddenly emerged to limelight due to its ease of fabrication and its versatility. Interestingly the waveguide and allied component fabrication can be done by following the conventional lithographical techniques. These factors are discussed in detail in the introductory chapter.

Chapter 2: The second chapter discusses in detail about the fabrication of a polymer optical drawing system designed and fabricated indigenously. The POF drawing system used a novel idea for preform heating using three IR lamps which was found to be more temperature stable than conventional coil wound furnaces. Precise fiber diameter control was achieved by using computer and microcontroller controlled driver motors whose rotational speed can be fixed to a predetermined value. The chapter also discusses in detail about the conventional techniques used for drawing optical fibers with necessary emphasis given to polymer optical fibers.

Chapter 3: As the saying goes "If you haven't measured it, you haven't made it" by Wayne Knox, it would be unfair to this thesis if necessary characterisation of fibers, depending on application, is not done. The third chapter deals with the characterisation of both dye doped and undoped POF for amplification and sensing applications.

Chapter 4: Optical amplification in the visible region has become a hot subject area since polymer fiber optics has come to the short communication scenario.

As it is evident, the band width of a channel is not only dependent on material characteristics, but also on the wavelength used for optical communication purpose. So, as one shift to visible communication regime there is a considerable increase in the communication bandwidth. A very simple and efficient technique is to use organic laser dyes with fluorescence in the visible spectrum. Incorporating laser dyes like Rhodamine and coumarin, in polymer matrix relatively simple, makes it a very useful device for optical amplifiers. The fourth chapter discusses in detail about the fabrication of polymer optical fiber amplifier using the above technique.

Chapter 5: Fiber optic sensors have changed the way we measure various parameters which cannot be efficiently measured by using conventional sensors. For example fiber optic chemical sensors have been developed which can measure traces of chemical in the range of parts / billon. Earlier this sensitivity which can only be dreamed of has now become a reality with the emergence of fiber optic sensors. This is due to the fact that the measurements involving light is very sensitive since the transmission of light through an optical fiber can be varied and measured by employing various techniques. With the emergence of polymer fiber optics, the sensitivity of fiber optic sensors has increased to a greater extent since POF are very sensitive to variations in temperature, pressure, strain and stress than glass fibers due to its material properties. More over speciality fibers like microstructured fiber can readily be fabricated using polymer optical fibers which can be effectively utilized for biosensors. Fiber optic sensors are widely used in the field of civil engineering to monitor cracks and fatigue of buildings and bridges. In the last chapter one such sensor has been discussed which is used to study the setting characteristics of various grades of

cement used for civil engineering. Studies were carried out on both glass and polymer optical fibers.

Summary and conclusions of the work carried out are given in **Chapter 6.** Future prospects are also discussed in this chapter.

List of publications

International Journals:

1. "A fiber optic smart sensor for studying the setting characteristics of various grades of cement" **M Rajesh**, K Geetha, M Sheeba, P Radhakrishnan P G Vallabhan & V P N Nampoori, ***Optics and Lasers in Engineering, Volume 44, Issue 5, May 2006, Pages 486-493.***

2. "Characterization of Rhodamine 6G doped polymer optical fiber by side illumination fluorescence" **M Rajesh**, K Geetha, M Sheeba, P Radhakrishnan P G Vallabhan & V P N Nampoori, ***Optical Engineering*** *In press*

3. "Fabrication and characterisation of dye doped polymer optical fiber as light amplifier", **M Rajesh**, M Sheeba, K Geetha, C P G Vallabhan, P Radhakrishnan and V P N Nampoori, (Communicated to Applied optics)

4. "Design and fabrication of dye-doped polymer optical fiber for optical amplification." **M Rajesh**, M Sheeba, K Geetha, C P G Vallabhan, P Radhakrishnan and V P N Nampoori, SPIE Proceedings: Symposium on Optics & Photonics, August 2006, San Diego, California USA.

5. "Loss characterization in Rhodamine 6G doped polymer film waveguide by side illumination fluorescence" K Geetha, **M Rajesh**, V P N Nampoori, C P G Vallabhan and P Radhakrishnan, ***J. Opt. A: Pure Appl. Opt. 6 (2004) 379–383***

6. "Laser emission from transversely pumped dye-doped free-standing polymer film", K. Geetha, **M. Rajesh**, V.P.N. Nampoori, C.P.G. Vallabhan and P.Radhakrishnan, ***J.Opt. A: Pure Appl. Opt. 8 , 189-193 (2006),***

7. "Fiber optic sensor for the detection of adulterant traces in coconut oil", M Sheeba, **M Rajesh**, K Geetha, , C P G Vallabhan ,V P N Nampoori, P Radhakrishnan, ***Measurement science and Technology*** *16(2005)2247-2250*

8. "Propagation characteristics and wavelength tuning of amplified spontaneous emission from dye doped polymer film waveguide", K. Geetha, **M. Rajesh,** V.P.N. Nampoori, C.P.G. Vallabhan and P.Radhakrishnan ***Appl. Opt. In press,***

9. "A modified design of refractometer using side polished polymer fibre". , K. Geetha, **M. Rajesh**, V. P. N. Nampoori and P. Radhakrishnan, (Communicated to Optical and Quantum Electronics)

International conferences

1. 'Fiber optic respiration sensor" **M Rajesh**, Jibu Kumar, C P G Vallabhan, V P N Nampoori, P Radhakrishnan.ICLAOM-03,Dec. 1 - 4, 2003, Indian Institute of Technology Delhi, India.

2. "A Fiber optic distributed sensor to characterize the properties of concrete mix" **M Rajesh**, M Sheeba, K Geetha, P Radhakrishnan, C P G Vallabhan & V P N Nampoori, Photonics 2004 ,9-11 December, cochin India

3. "Fiber optic sensor for the detection of paraffin oil traces in coconut oil" **M Rajesh** , M Sheeba , K Geetha , P Radhakrishnan, C P G Vallabhan & V P N Nampoori, Photonics 2004 ,9-11 December, cochin India

4. "Design of a refractometer with wide dynamic range using side polished polymer optical fibre" K. Geetha, **M. Rajesh**, C.P.G. Vallabhan , V.P.N. Nampoori and P.Radhakrishnan, Photonics 2004 ,9-11 December, cochin India

5. "Design of a refractometer with wide dynamic range using side polished polymer optical fibre", K. Geetha, **M. Rajesh,** C.P.G. Vallabhan , V.P.N. Nampoori and P.Radhakrishnan, Proceedings of Seventh International Conference on Optoelectronics, Fiber Optics and Photonics (PHOTONICS 2004) Dec 9-11, Kochi (2004)

6. "Fabrication of Polymer Optical Fiber based amplifiers" ,**M Rajesh**, M Sheeba, K Geetha, C P G Vallabhan, P Radhakrishnan and V P N Nampoori, ICOL-2005,12-15 December, IRDE -Dehradun, India

7. "Polymer optical fiber based temperature sensor", M Sheeba, **M Rajesh**, C P G Vallabhan, V P N Nampoori, and P Radhakrishnan, ICOL-2005,12-15 December, IRDE -Dehradun, India

National Conferences

1. "An Optical fiber sensor for the measurement of concrete setting, **M Rajesh**, M G Jibu Kumar, C P Girijavallabhan, V P N Nampoori, P Radhakrishnan. 'National Laser Symposium, IIT Khargpur Dec 2003

2. "Side Illumination Fluorescence Technique for the characterization of optical loss in Dye doped planar waveguides", K. Geetha, **M. Rajesh**, V. P. N. Nampoori, C.P.G. Vallabhan and P. Radhakrishnan, National Laser Symposium 2003, Kharagpur.

3. "Side Illumination Fluorescence Technique for Loss Characterization of Dye Doped POF", **M Rajesh**, K Geetha, M Sheeba, C P G Vallabhan, P Radhakrishnan and V P N Nampoori, NLS-2005, Vellore Institute of Technology-Vellore, Tamilnadu, India.

Acknowledgments

I don't have words to thank my research supervisor Prof. VPN Nampoori, without him, I believe my research work will be like a stroke of line drawn in water. He gave me all the support and courage to start a new field of research at ISP. When he gave me the idea of drawing polymer optical fibers and to make it operate as an amplifier in the visible communication regime as my research topic, to be frank, at the back of my mind I was a little bit skeptical. Due to his constant encouragement and support the idea was conceived and today I feel proud that I have made his dream come true. His ability to conceive things in totality and his tremendous vision of future and ideologies which motivates people around him always inspired me. Moreover I believe I am the luckiest researcher to get a supervisor like "Nampoori sir" in the whole world. His magical smile and love will remain immortal in me.

I always have been an ardent believer of hard work and if I am given a chance to quote an example of an industrious person, the first name that comes to my mind is Prof. P Radhakrishan. He is always a role model to me. His way of meticulously planning things and to implement them accordingly dazed me always. I always had the liberty to approach him at will with my doubts and paper corrections which he deals with great care and affection. I am indebted to him for giving me all the support and guidance through out my research career.

I don't know how to express my gratitude to Prof.C P Girijavallabhan in giving me all the support, love and care during my research days at ISP. I believe that his aura is enough to revitalize ones research attitude. His tremendous knowledge both in science and current affairs is really fascinating. My research work would have been incomplete without the timely advice which I have received from him.

I sincerely thank Prof. V M Nandakumaran for his help and support. His valuable suggestions have helped me a lot in my research work. I am equally thankful to Mr. M. Kailasnath for timely help and encouragement. I greatly acknowledge the fruitful discussions with Dr. Rani Joseph and Dr. K.E. George of PS&RT department.

The financial assistance from CELOS, CUSAT, funded by UGC India is greatly acknowledged.

It would be unfair if I do not mention the help and assistance given to me by the non-teaching staff of ISP and CELOS. I remember them with gratitude.

Thanking my colleagues, I feel is the most difficult part. I know that there are many people who helped me morally through out my research days. Aneesh and Prashanth,

without their help I couldn't have materialized my dream of drawing polymer fibers. It was due to their timely help in raising fund for the fabrication of the fiber drawing tower saved my research sinking to abyss.

My seniors Unnikrishnan, Pramod, Benoy paul, Deepthy, Thomas Lee, Achama Kurian and Sajan, I remember them with gratitude. They are the people who helped me in taking my first steps to research. Unnikrishnan, with his quick-wit attitude always tried to comfort me whenever I get stuck in between my research. Sajan on the other hand was always helpful in resolving many experimental problems which I encountered. Thomas Lee who was my lab senior helped me to set up my experiments and encouraged me to get things done. Benoy Paul and Achama Kurian helped me in getting my polymerization experiments in track. If you have a sister to guide you in the right track, that's the greatest blessing. Deepthy, I used to call her "Chechi", whose valuable advice and care helped me to orient my research in the right direction.

To mention about my colleagues, Geetha was very supportive through out my research days at ISP. Through her sharp reasoning ability, she helped me to resolve many problems related to my research. Shanthi, and Sr. Ritty helped and encouraged me with fruitful suggestions.

I feel getting very good juniors is really a boon. Thomas is such a person whom I owe a lot. He helped me in relieving a lot of responsibility from me in managing the lab. This has in turn constructively affected my research career. Dann, Sajeev, Litty, parvathy and Lyjo need a special mention since they always were very supportive. I remember the wonderful moments with Abraham, Pravitha, Jyotsna, Bindu, Rekha, Santhi, Dilna, Sreeja, Jayasree and Suresh.

A friend in need is a friend indeed. I have quite a few friends who helped and supported me through out my research days. Jijo, Vinu, Rajesh, and Manu, are my beloved friends. I remember with gratitude the help and support given by Thomas of PS&RT.

Sheeba, I don't have words to describe her support and care which has changed my research days to one of the colorful and fruitful episodes of my life.

Family is the most important supporting element in my research life. My Achan, Amma, Chechi, Chettan, Malu and Ammu, without their support I would not have even dreamt of achieving my goal.

Rajesh

Contents

2. Fabrication of Polymer optical fibers

The whole of science is nothing more than a refinement of everyday thinking-
Albert Einstein

Polymer Photonics: An Overview

Fiber optics has emerged as a strong backbone for communication systems. Glass fibers paved the way for efficient data traffic between continents with very high data rates, which was thought to be impossible a few years before. The path of glass fibers is being followed by polymer optical fibers. Polymer optical wave-guide is still in its infancy and a lot of research work has been taking place for the fabrication of low loss, efficient polymers for fiber and integrated optics.

1.1 Introduction

Optical polymers are clear plastics that provide excellent light transmission [1-3]. In photonic applications, they offer advantages over optical glass; they weigh less, and they can be molded into spherical, aspheric and symmetric shapes. Many polymers have valuable optical properties, but few are in widespread use or have the potential for such use. Typical uses for these optical polymers include lenses [4-6] for video and still cameras; projection televisions; compact disc drives; light-emitting diodes [7-10]; printers and bar-code readers; ophthalmic lenses; light-guides; optical films; high-density optical storage media; diffractive optics; flat panel displays[11-13]; metallised reflectors; and optical fibers, fiber couplers and connectors. The key transparent polymers for optical and photonic applications include acrylic, usually polymethylmethacrylate (PMMA) or a modification of this material, polycarbonate (PC), polystyrene (PS), allyl diglycol carbonate (ADC) and cyclic olefin copolymer (COC). These materials are thermoplastics, except for ADC, which is available only as a thermoset resin.

Manufacturers use most of these materials in traditional molding, extrusion and other processes. Many other polymers have some optical applications. For example, styrene-acrylonitrile copolymer is used in reflectors; polysulfone is useful in bubble lenses on circuit boards because it can withstand potting temperatures; and polyetherimide offers high thermal stability in infrared laser lenses. Those who use optical polymers in photonic applications face many choices including acrylic, PC, PS, ADC and COC. Speciality material grades containing a variety of additives often have properties that differ greatly from those discussed below.

1.2 Optical properties of polymers

Optical properties of a polymer are light transmittance, yellowness, haze, refractive index and Abbe number [14-16]. The polymers mentioned here have light transmittance values exceeding 88 percent within the wavelength range 380 to 1000 nm. PMMA and COC typically have 92 percent transmittance over this range, while others fall somewhat lower. The high refractive index of PC and PS limits their transmittance above 1000 nm. Fiber optic systems can use higher transmittance bands between about 1200 to 1350 nm, and 1500 to 1600 nm. Some processing and end-use conditions can cause optical polymers to acquire a yellow tint. High temperature (for example, heat from lighting) and UV (from natural and artificial light) can cause yellowing when in use. PS and PC tend to turn yellow more than PMMA with UV exposure. Optics for collimating diode lasers and light-emitting diodes are among the potential applications of optical polymers. Choosing the right plastic will determine the ultimate success of the application.

The environmental stability of optical polymers (i.e. the stability of their optical and mechanical characteristics with temperature and humidity) is an important issue because most of the polymers do not have properties that are appropriate for operation in communication environments. A key characteristic for practical applications is the thermal stability of the optical properties since organic materials may subject to yellowing upon thermal induced oxidation. The presence of hydrogen in a polymer allows the formation of Hydrogen-Halogen elimination products, which results in carbon double bonds which are subjected to oxidation.

Refractive index normally decreases as temperature or wavelength increases (unlike in glass, where index increases with temperature). Component designers can compensate for changes in refractive index by carefully matching the polymer to the wavelength and by using composite glass-and-plastic lenses that compensate for higher temperatures [17-19].

Birefringence, the effect of variations in the index of refraction of a material when measured in different directions, can occur in plastic components when polymer chains align during molding. Careful management of molding conditions can minimize birefringence and other effects of optical anisotropy. Typically, lower injection speed and longer holding time contribute to reducing birefringence. Physical forces on a component can also affect birefringence. The proportionality between birefringence and applied stress is the material's stress-optic coefficient. This is important in laser optics, compact discs, polarized-light applications and precision optics, which must minimize directional variations in refractive index.

The Abbe number is a measure of dispersion and indicates how refractive index varies with wavelength. Dispersion is important in optoelectronic systems that use relatively wide bandwidths. Correlating Abbe number and refractive index shows that PMMA, ADC and COC fall within the realm normally occupied by crown optical glasses and that of PS and PC are more like flint glass. Optical properties may also change with wavelength, temperature and moisture. For instance, the thermal effect of a high-power laser beam passing through a polymer can influence refractive index change that causes the beam to bloom or spread. Even in low- gaze plastics; this can occur at energy densities higher than 100 to 200 W/cm^2.

Other factors, such as surface roughness, gloss, internal contamination and color, may also be of concern in developing photonic devices.

1.2.1. Other properties

Specifications for photonic applications usually extend beyond optical properties to include a range of physical, mechanical, thermal and other characteristics. Density, for instance, is significant because lower density means lower optical component weight and material cost. Moisture absorption can change a component's optical geometry, increases refractive index slightly and causes optical inhomogeneities in acrylics and other moisture-sensitive materials. Components also must meet a variety of mechanical criteria: tensile and impact strength, hardness, elongation at break, scratch resistance and flexural modulus (stiffness). Heat deflection temperature, the temperature at which a polymer deflects a set distance under a set load, determines the extent of deformation that may occur when optical components are close to high-intensity bulbs or other heat sources. The coefficient of linear thermal expansion affects the ability of plastic components to hold dimensions precisely as temperature changes.

All of these polymers will replicate fine surface features under the right circumstances. PC and PMMA are more difficult to force into fine features during molding than are COC and PS. ADC fills fine surface features because it is liquid when cast. Optical polymers differ greatly in their chemical resistance. Methanol, for instance, attacks both PC and PMMA, but not COC, while the opposite is true for hexane. In developing a photonic component, we have to determine the chemicals which the component will likely encounter during its life cycle.

1.2.2. Processing considerations

Another design consideration is ease of processing. The thermoplastic resins - PMMA, PS, PC and COC - can be injection-molded, but ADC must be cast. Casting (i.e. reacting in a mold) typically takes about 17 hours at 140 °c. PMMA, PS, PC and COC generally flow well during molding, fill complex molds well and have similar cycle times. Except for COC they should be dried before molding to minimize the possibility of splay and yellowing. Shear and cooling stresses during injection molding can cause inhomogeneities in polymers. But care in processing can minimize these variations.

PMMA and PS tend to process similarly at comparable temperatures. PC and COC also have similar processing temperatures, which are about 100°C higher than PMMA and PS. Processing temperature is in line with the end-use temperature capability for these materials. Optical thermoplastics can also be cast, extruded onto formed rollers or machined. Cast optical parts made from ADC and PMMA can have lower haze than molded parts because the manufacturer can filter the reagents before casting. In addition, diamond turning of cast or molded and annealed blanks can produce prototype components and small production runs without the need for polishing. Cost is not a hard-and-fast issue but depends on the volumes and grades purchased. The cost of all of these materials is nearly insignificant for small components such as fiber optic coupler lenses. Material costs naturally become higher as components grow larger, but the processing time for large and thick lenses usually is a more significant part of overall component cost.

1.3 An overview of optical fibers and applications

Communication using an optical carrier wave guided along a glass fiber has a number of extremely attractive features, several of which were apparent when the technique was originally conceived. Furthermore, advances in the technology to date have surpassed even more optimistic predictions, creating additional advantages. Hence it is useful to consider the merits and special features offered by optical fiber communications over more conventional electrical communications.

The information carrying capacity of a transmission system is directly proportional to the carrier frequency of the transmitted signals. The optical fiber yields greater transmission bandwidth than the conventional communication systems and the data rate or number of bits per second is increased to a greater extent in the optical fiber communication system. Further, the wavelength division multiplexing operation enhances the data rate or information carrying capacity of optical fibers to many orders of magnitude.

Due to the usage of the ultra low loss fibers and the erbium doped silica fibers as optical amplifiers [20-23], one can achieve almost lossless transmission. In the modern optical fiber telecommunication systems, the fibers having a transmission loss of 0.2 dB/km are used. Further, using erbium doped silica fibers over a short length in the transmission path at selective points; appropriate optical amplification can be achieved. Thus the repeater spacing is more than 100 km. Since the amplification is done in the optical domain itself, the distortion produced during the strengthening of the signal is almost negligible.

Since photons are considered to be chargeless particles they have a high immunity to internal noise, cross talk, electrical noise, ringing, echoes or electromagnetic interferences. It is also very immune to lightning, and thus immune from lightning caused hazards. The transmitted signal through the fibers does not radiate. Further the signal cannot be tapped from a fiber in an easy manner. Therefore optical fiber communication provides hundred percent signal security. Fiber optic cables are developed with small radii, and they are flexible, compact and lightweight. The fiber cables can be bent or twisted without damage. Further, the optical fiber cables are superior to the copper cables in terms of storage, handling, installation and transportation, maintaining comparable strength and durability, low risk of fire, explosion, and ignition.

Due to the recent advancement in optical fiber technology, the fabrication costs of optical fibers are coming down drastically. Even though the initial cost of installation is very high when compared to electrical communication cables, it has very low maintenance cost.

1.3.1 Materials used in the fabrication of optical fibers

There are a number of materials used for the fabrication of optical fibers. All these materials are selected in such a manner that it satisfies certain conditions, such as a) it should have good optical transmission quality for efficient optical transmission b) it must be flexible and must be able to be draw into long fibers and c) the cladding and the core materials should have slightly different refractive indices to satisfy total internal reflection.

Glass fibers

Glass fibers are mostly made from silica or silicates. Glass fibers are very low loss fibers when compared to plastic fibers. Glass fibers are widely used in longer distance communication purposes. There are a number of glass fibers available, some among them are:

a) Silica glass fibers

They are made by fusing metal oxide, sulphides and selenides. Oxide glasses are mostly used for making optically transparent glasses. The most common is silica (SiO_2), which has refractive index of 1.458 at 850nm. To produce two similar materials that have slightly different refractive indices for core and cladding, various oxides are added to silica.

b) Fluoride glass fibers

Fluoride glass fibers are used for optical fibers that have extremely low transmission losses at mid IR wavelength region with the minimum loss being around 2.55μm. Heavy metal fluoride glasses use ZrF. The constituents of fluoride glass are mainly ZrF, BaF, LaF, AlF and NaF referred to as ZBLAN. The material forms the core of a glass fiber. To make a lower refractive index fiber, ZrF is partially replaced by HaF to get a ZHBLAN cladding.

c) Active glass fibers

Active glass incorporates rare-earth elements into a normally passive glass to give the resulting material which has new optical and magnetic properties. These new properties allow the material to perform amplification, attenuation and phase retardation of light passing through it.

d) Chalcogenide glass fibers

These contain at least one chalcogen element (S, Se, orTe) and typically one other element such as P, I, Cl. It has high optical non-linearity and long interaction length. Typically losses are in the range of 1dB/m.

Polymer fibers:

In 1966, Dupont introduced the first type of Polymer Optical Fiber (POF) product named Crofon with PMMA core to the market. The development activities in the past 40 years were aimed at removing some major disadvantages of POF, such as high transmission loss, low thermal resistance and narrow bandwidth. Now various types of POF products including GI type POF, single mode POF, fluorescent POF, non-linear POF, etc. have been developed, which are widely used in the fields of light and image transmitting, sensing, and information transmission in short distance. POF is also largely used in illuminating, advertising, decorating, and art and craft making, and low cost POF with Polystyrene (PS) core is mostly used in such applications.

1.4 Polymer fiber optics

The global proliferation of optical fiber-based communication networks has been the enabling factor in the shift from the Electronics Age to the Information (i.e., Photonics) Age; as is supported by the exponential growth in Internet subscription and video/multimedia conferencing. Silica glass fibers have enabled this Information Superhighway, but plastic optical fibers (POFs) are of great commercial interest because they can maintain flexibility at thicker fiber sizes making them more easy to handle and install when the communication systems make their way from the local loop (premise

networks, local area networks) closer towards, and into, the home. POF may also be an important building block for devices used in future ultrafast all-optical communication systems because it has many attractive properties that silica optical fiber lacks. An important one is its low processing temperature (typically, less than 200^{o}C). This allows organic nonlinear optical materials to be incorporated into the POF, which is otherwise impossible in silica-based fiber because of its high process temperature (typically 1800^{o}C to 2000^{o}C). Many useful devices, such as optical switches, may be constructed based on fibers with fast response and high nonlinearity.

In general the polymers have certain advantages which make it a suitable candidate for optical fiber fabrication. They are as follows:

- Easy mold and form into any shape
- High-impact applications(Inorganic materials such as glass , would shatter in such situations)

But, some of the problems are:

- Poor dimensional stability
- Poor scratch resistance

So far, highly purified inorganic glass core materials are still unsurpassed in their low transmission loss or low attenuation. However, fibers made of glass need to have very small diameters to obtain flexibility due to its high Young's modulus. Furthermore, glass fibers are very brittle and very sensitive to damage to the surface. This implies that connecting glass optical fibers is a time consuming process that require high precision tools and skilled workers. Polymer materials have a much lower modulus than inorganic glass and therefore, can possess much larger diameter and still retain their flexibility. Polymer optical fibers also possess a higher numerical aperture (NA). As a result, the acceptance angle, or the light gathering capacity, is large compared to glass optical fibers.

		Core	Cladding	Attenuation (dB/km)	Application
All silica optical fibers		Silica	Silica	0.5	Long distance data communication
Polymer clad optical fibers	PCF	Silica	Silicone	10	Short-Mid distance data communication
	HPCF	Silica	Fluorinated polymer	5	Mid distance data communication
Compound glass optical fibers		Glass	Glass	15	Short-Mid distance data communication Industrial lighting
Plastic optical fibers		PMMA	Fluorinated polymer	140	Short distance data communication Industrial lighting, decoration

Table 1.1: Comparison of glass fibers with polymer fibers

The combination of large core diameter and high numerical aperture facilitates fiber installation, which is a dominant cost factor. Accordingly, the installation costs for POF system are much lower than for an inorganic glass fiber network. For instance, cheap injection molded type of connectors are used to establish POF links instead of the high precision ceramic ferrules required for GOF links. What is more, no expensive lens systems are required to couple the light into the fiber, due to the high numerical aperture. This makes POFs a suitable candidate in short distance data communication applications. Plastic optical fiber systems are found in local area network, fiber to home applications, fiber optic sensor and the automotive [24] or aviation [25] industry.

1.4.1. Advantages of POF over silica fibers

- POF is very elastic in contrast to silica fiber, which is very brittle. This property is important for the fiber interface within the opto-electronic systems where space is usually limited.
- Commercial POF has a typical diameter of 1mm with no cladding or only a thin cladding, making it a multimode fiber. The large core diameter and flexibility allow ease of handling, large alignment tolerance and connection cost.
- Low processing temperature (200°C to 250°C) of POF allows organic nonlinear optical materials to be incorporated into the POF which is otherwise impossible in silica fibers because of its high processing temperature (1000°C to 1500°C).
- The transmission windows of POF are distributed throughout the visible wavelength range. The utilization of these windows will allow very convenient and low cost deployment of POF systems.
- Silica fibers have very small non-linear coefficient requiring a large amount of optical power or very long fiber length to induce sufficient non-linear phase change. Polymer fibers can be made non linear by appropriate doping material.

1.4.2 Application of polymer optical fibers

1. In Telecommunications:

Optical telecommunications using POF (Plastic Optical Fibers) have not reached their potential for a number of reasons, the foremost being the rapid growth of glass optical fiber technology and because plastic optical fibers have been relegated to low speed, short distance applications. As a result of recent technical developments of graded index POF with bandwidths

of 3GHz/100m, single-mode POF, optical amplification in plastic fibers, new POF materials with low loss at 1550nm and higher power and faster sources have been developed. Applications such as FDDI, ATM, Escon, Fiber channel, SONET, and FTTH are now within the realm of plastic optical fibers. Present interest is concerned with high-technology Local Area Network applications. The real growth market is now, and will continue to be simple point-to-point links in a wide range of applications across all industries. Data Links for PCs and workstations; Local Area Networks; industrial data links; consumer digital data links; optical computing; optical interconnects; automobile networks and links are some of the other applications [26-30].

Polymers offer optical data transmission rates of 10 billion bits per second. Optical fibers are being developed from polymer plastics (optically active polymers), which would enhance the optical data transmission and optical signal. Data is being transmitted increasingly over the public networks by means of light traveling through optical fibers. Fiber-optic data transmission also has significant advantages for the private sector. Among other things, it permits the simultaneous transmission of several television programs and facilitates rapid access to image and video documents via the Internet. To do this, a computer, television set or telephone must be equipped with a so-called transceiver for connection to the fiber- optic cable. Normally the transceiver comprises of a photodiode for receiving and a laser diode for transmitting the optical signal.

2. Fiber amplifiers

Availability of inexpensive sources in the visible region has increased the utilization of POF in data communication. Implementation of optical communication in the visible region necessitates the development of

suitable optical amplifiers working in this region [31-36]. POF doped with dyes or rare earth elements are potential candidates for this purpose. Laser dyes, which act as highly efficient media for lasing and amplification have a wide range of tunability in the visible region. The range of tunability of laser dyes like Rhodamine B and Rhodamine 6G usually comes between 570nm to 640nm. The advantage of incorporating laser dyes in solid matrices such as POF is that it is easier and safer to handle them than when they are in liquid form. From recent studies it is found that the dye doped polymer materials have better efficiency, beam quality and superior optical homogeneity when compared to other solid matrices.

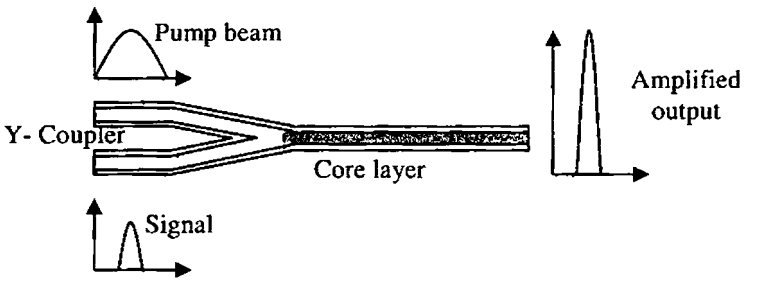


Figure 1.1.A general dye doped fiber amplifier configuration

In dye doped optical fiber amplifiers the pump power can be limited to a very low level and utilized in a most efficient manner since the power is well confined to the core region and is propagated with less diffraction. The most important aspect of reducing the pump power is that it reduces the bleaching effects thereby increasing the stability of the medium. A general dye doped amplifier configuration is shown in the figure 1.1.

The first step in the fabrication of dye doped POF is to develop doped polymer preforms which can be fabricated following the standard fabrication techniques, suitable for fiber drawing. The base material used for the

fabrication of polymer preform is methyl methacrylate (MMA) monomer. Methyl methacrylate is a suitable candidate for the fabrication of polymer preforms since it has good optical quality and compatible with most of the organic dopants.

Properties of laser dyes

Since the beginning, the development of the dye laser has been closely tied up with the discovery of new and better laser dyes. The Pthalocyanine solution employed for the original dye laser (Sorokin and Lankard 1966) is hardly used today, but the compound rhodamine 6G, found soon afterwards (Sorokin et al. 1967), is probably the most widely employed laser dye at present. In the years following the discovery of the dye laser, various other compounds were reported for this purpose [37-41]. Almost all were found by screening commercially available chemicals, but this source of new laser dyes will soon become exhausted. Considering the large number of available chemicals, it is perhaps surprising that so few good laser dyes have been found so far.

Organic dyes are characterized by a strong absorption band in the visible region of the electromagnetic spectrum. Such a property is found only in organic compounds which contain an extended system of conjugated bonds, i.e. alternating single and double bonds. The long wavelength absorption band of dyes is attributed to the transition from electronic ground state S_0 to the first excited singlet state S_1. The reverse process S_1 to S_0 is responsible for the spontaneous emission known as fluorescence and for the stimulated emission in dye lasers (figure 1.2). Because of the large transition moment, the rate of spontaneous emission is rather high (radiative lifetime of the order of nanoseconds) and the gain of a dye laser may exceed that of solid-state lasers by several orders of magnitude.

When the dye is pumped with an intense light source(flash lamp or laser), the dye molecules are excited typically to some higher level in the singlet manifold, from where they relax within picoseconds to the lowest vibronic level of S1 i.e. upper lasing level. For optimal lasing efficiency it would be desirable for the dye molecules to remain in this level until they are called on for stimulated emission. However, there are many nonradiative processes that can compete effectively with the light emission and thus reduce the fluorescence efficiency.

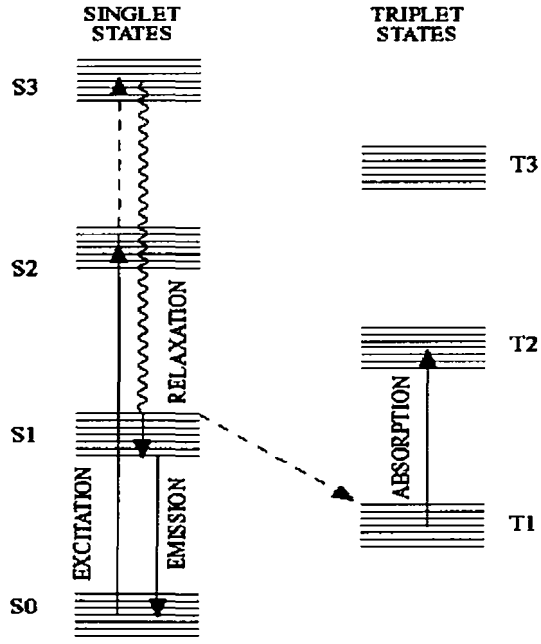


Figure 1.2: Schematic energy levels of a dye molecule.

These nonradiative processes can be grouped into those that cause a direct relaxation to the ground state S_o (internal conversion) and those that are responsible for intersystem crossing to the triplet manifold. Because of the relatively long lifetime of the triplet molecules (of the order of microseconds) the dye accumulates during the pumping process in the triplet state T_1, which

often has considerable absorption for laser light. In addition to these general requirements, an efficient laser dye in its first excited state should have negligible absorption at the wavelength of the pump light and the laser emission as well. Otherwise losses would occur, as in triplet absorption, because the decay to the first excited singlet or triplet level is nonradiative. Along with these properties, a laser dye should have an absorption spectrum which matches the spectral distribution of the pump source.

3. Fiber optic sensors

Although many of the applications of optical fibers are based on their capacity to transmit optical signals with low losses, it can also be desirable for the optical fiber to be strongly affected by certain physical parameters of the environment. In this way, it can be used as a sensor of such a parameter. There are many strong arguments for the use of POF as sensors [42-46]. In addition to their easiness to handle and low price, they present the advantages common to all multimode optical fibers. Specifically, the flexibility and small size of optical fibers enable a great sensitivity to be achieved without having to occupy big volume. Moreover, it has been proved that POF can be employed to detect a great variety of parameters, including temperature, humidity, pressure, and presence of organic and inorganic compounds, wind speed and refractive index. On the other hand, POF based optical sensors eliminate the risk of electric sparks in explosive environments, and they can be read from remote positions.

As an example to polymer optical fiber based sensor, several chemical sensors have been developed using plastic optical fibers. The sensing segment is made of porous polymer fiber, combined with selective chemical indication systems. By careful selection of polymer systems and indicators, the chemical reagents can be covalently bonded to the porous plastic fiber.

These sensors can be used to detect a variety of chemical species and to measure various chemical parameters, both in vapor and solution. They provide high sensitivity and stability. Sensor characteristics, including dynamic range, linearity, and response time, can be tailored to meet specific applications by altering the polymer composition and polymerization procedure.

4. Polymer optical fiber passive devices

In telecommunication systems many passive optical fiber devices like couplers, filters, scramblers, tapers and lenses are necessary. Their uses depend on low insertion loss, mass production reliability, low-cost effective production and thermal and mechanical stability. A general structure of a simple fiber optic mode filter is shown in figure 1.3.

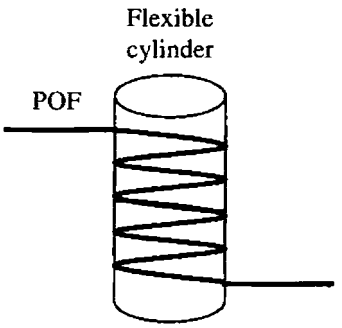


Figure 1.3: POF mode filter. The technique to make a mode filter is simple. Simply wrap a fiber around a cylinder.

Optical couplers are used not only in communication systems but also in signalization, lighting, and decoration systems, where it is necessary to distribute or redirect light to other outgoing fibers. Automotive, entertainment and sensor industries represent other areas of potential application. The most common techniques used for the fabrication of couplers are twisting and fusion, side polishing, chemical etching, cutting, molding and gluing and thermal deformation [47-48].

Large core optical fiber exhibits a large number of propagating modes. The presence of such modes can cause modal noise in telecommunication systems. This problem is significant for high-speed telecommunication system. The technique to obtain a POF modal filter is rather simple: merely wrap a POF segment around a small diameter cylinder.

In some applications, most of which are related to sensors, it is not only necessary to filter higher order modes, but to scramble them. This treatment is done to guarantee that all propagation modes carry the same power. The device used for this application is called a scrambler. Mode scramblers [49] have been designed and assembled that consist of polyester plate that has induced microbends and a POF segment that is positioned to compress against the microbends (figure 1.4).

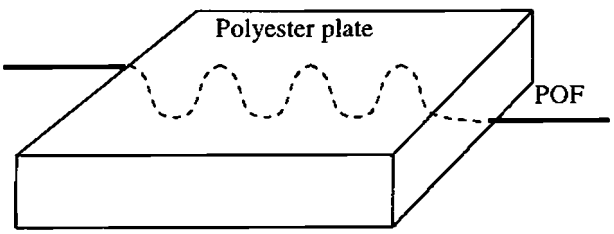
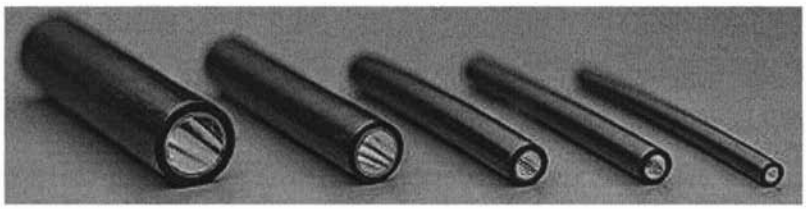


Figure 1.4: POF mode scrambler

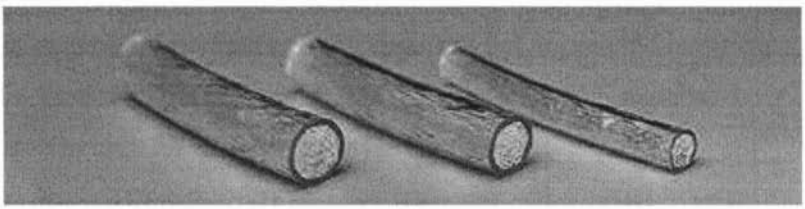
5. Fiber-optic lighting

Optical wave-guides are useful in lighting and illumination applications. Especially light guides prepared from polymeric materials possess a low weight, but high strength-to-weight ratio. Furthermore, polymers are easily processed and hence result in a flexible layout design using standard processing techniques such as compression molding, extrusion and injection molding. The use of polymeric materials in lighting applications

opens up a large number of applications in lightweight mobile devices and also in the field of signage and displays as a replacement for high voltage neon devices, which are rigid, fragile and limited in length.



Solid Core Plastic End Emitting Fiber



Stranded Plastic Side Emitting Fiber

Figure 1.5: Speciality plastic optical fibers which are used for illuminating purpose

Optical wave-guides in lighting or illumination applications are split up in two distinct categories, the end or point source lighting and the side or line lighting [50]. The former involves transmission of light from the proximal to the distinct end, analogous to optical fibers in data communication. The latter term is used to describe light guides that combine the properties of light transmission and light emission, in which the desired emission direction of the light is often perpendicular to its propagation. Commercially available step index polymer optical fibers are suitable for most end lighting applications, since only transmission of light is required. Because the technology is advancing rapidly, in a couple of years it will make sense to use fibers even for general lighting. Some commercially available polymer optical fibers suitable for lighting is shown in the figure 1.5.

1.4.3 Materials used for POF

PMMA

The material most frequently used for the fabrication of POF is the thermoplastics PMMA (Polymethylmethacrylate), better known as Plexiglas. From the beginning of 80's the available POF were found to have an attenuation of around 150dB/km. PMMA-SI-POF has a theoretical minimum attenuation of 106dB/km at 650nm [51-52] which is due to the Rayleigh scattering and absorption of C-H bonds. In addition there are the losses resulting from waveguide structure, particularly when taking into account the attenuation resulting from cladding.

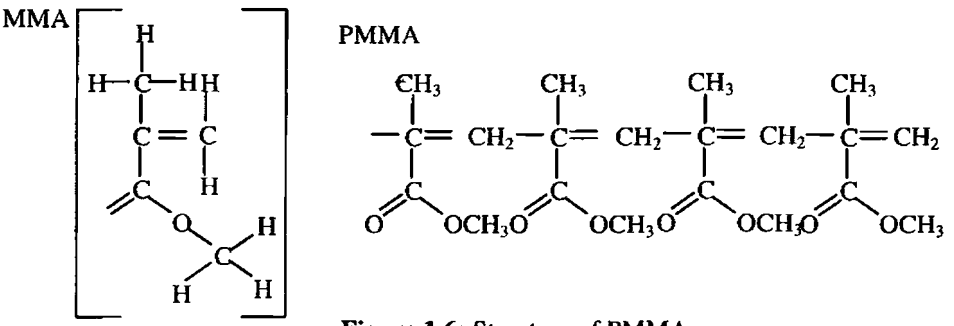


Figure 1.6: Structure of PMMA

PMMA [53-55] is produced from ethylene, hydrocyanic acid and methyl alcohol. It is resistant to water, lyes, diluted acids, petrol, mineral oil and turpentine oil. PMMA is an organic compound forming long chains with typical molecular weight around 10^5. PMMA is amorphous in nature when polymerized and has a very good optical transparency. The density of PMMA is 1.18g/cm^2. Its tensile strength is approximately 7-8kN/cm^2 [57]. The refractive index of PMMA is 1.492 and glass transition temperature T_g lies between +95°Cand +125°C. At room temperature and 50% relative humidity the material can absorb upto 1.5% water, which also affects the attenuation characteristics. The bond structure of PMMA is as shown in the figure 1.6.

MMA monomer has eight C-H bonds. The vibrations of this compound or more precisely its harmonic waves are a main cause for the losses encountered in PMMA polymer fibers. [56, 57]. In particular the harmonic waves at 627nm (6^{th} harmonic wave) and 736nm (5^{th} harmonic wave) essentially determine the level of attenuation within the application range of PMMA-POF because these are not narrow absorption lines but relatively wide bands. There are many ways to reduce the absorption losses of polymer fibers using different materials in which less or no C-H vibrations are present such as deuterium, fluorine and chlorine. The list of possible materials which can be used either for forming the core or cladding material with their refractive indices for GI fibers are as given below.

MMA	methylmethacrylate	n=1.492
VPAc	vinyl-phenylacetate	n=1.567
VB	vinyl benzoate	n=1.576
PhMA	phenyl methacrylate	n=1.570
BzMA	benzylmethacrylate	n=1.562
BB	bromobenzene	n=1.56
BBP	benzyl n butyl-phtalate	n=1.54
DPS	diphenyl-sulfide	n=1.49

POF for higher temperatures

Fiber with higher temperature resistance is required primarily for use in certain areas of automotive technology (engine compartment) and automation technology. A typical characteristics for all these materials used at temperatures over +100 °C is the higher attenuation compared to PMMA fiber. The lowest attenuation values here are in the range between 650nm and 800nm. Due to the larger refractive index difference between, for example, Polycarbonate (PC) as the core material (n=1.51) and special polymers serving as cladding (Teflon-AF), the NA of these fibers may reach up to 0.90. Partially fluorinated PC fiber has a feature of showing good temperature resistance up to 145 °C with a bandwidth at 200MHz-100m [56].

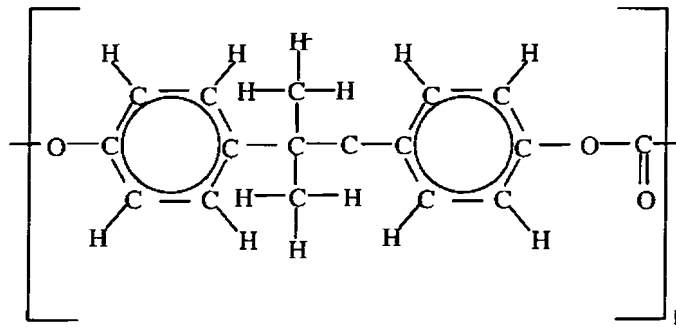


Figure 1.7: Structure of Polycarbonate

Polystyrene polymer fibers

Polystyrene is another suitable candidate for making POFs. Its molecular structure is as shown in the figure 1.7. The initial fibers had an attenuation of over 1,000dB/km, later it was possible to reduce this to 114dB/km at 670 nm. The NA of these fibers which can be used at temperatures up to 70 °C is 0.56 i.e. a little higher than that for standard PMMA-POF. The refractive index of PS is n=1.59 so that it is possible to use PMMA as the cladding material (n=1.49), as is possible for PC (n=1.48).

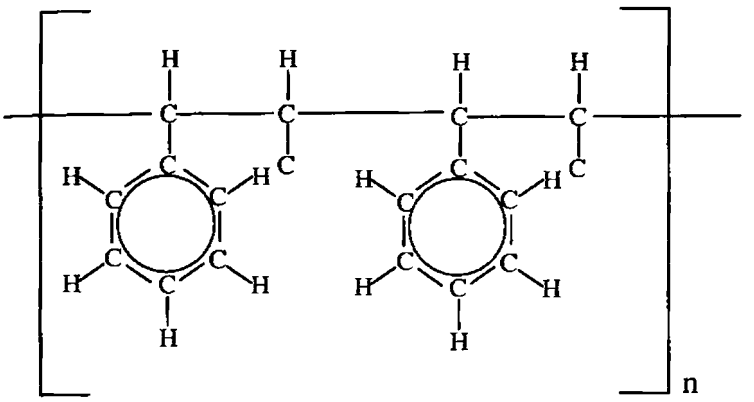


Figure 1.7: Structure of Polystyrene

Deuteriated polymers

For achieving a significant reduction in the absorption loss of polymers one has to substitute the hydrogen with heavy atoms. One method is by replacing it with deuterium. This isotope has twice the atomic mass compared to hydrogen. Chemically, deuterium behaves the same way as hydrogen so that heavy water (D_2O) can be used as a base material for synthesis. The first Deuteriated SI-POF was produced by Dupont in 1977[56, 57]. In 1983, NTT produced a SI-POF in deuteriated material has a minimum attenuation of 20dB/km at 680nm. Deuteriated polymers have a number of advantages associated with them. Chemically these materials behave identically to the substances made from normal hydrogen. The attenuation is approximately one order magnitude less than in the values achieved by PMMA fibers. The decisive disadvantage of deuteriated POF when compared to PMMA-POF is that there is always water vapor present in the atmosphere which will be absorbed by the fibers. This will lead to cores in which protons slowly replace the deuterium so that the absorption loss will increase again.

Fluorinated Polymers

The atomic mass of fluorine is many times greater than that of hydrogen so that absorption bands are moved significantly further into the infra-red zone. The theoretical minimum values are less than 0.2dB/km [56, 57]. i.e. comparable to silica fibers in the wavelength range of about 1,500nm. But in practice it is observed that it is very difficult to achieve those results. Another important aspect is that it is very difficult to process a fluorinated polymer processed in its amorphous state. Teflon material tends to crystallize, which accounts for scattering losses within the fiber. An interesting fact is that, PF- SI-POF has not produced yet simply due to the fact that there are no suitable cladding materials available for this purpose. Dye doped POF are expected to play a vital role in future communication systems based on solitons as they can replace EDFA's which can operate only in the IR region and thus allow for potential visible light communication systems.

1.5 Polymer integrated optics

Polymer waveguide technology has a great potential for economic mass production of complex planar photonic circuits that comply with the severe requirements imposed by applications in communication systems [58-61]. The low-cost prospect arises from the availability of a wide range of cheap optical polymers and the simplicity of fabricating waveguides from them. A significant subset of optical polymer materials has shown excellent optical, chemical and mechanical characteristics that are very attractive for applications in integrated optical devices as discussed previously. Polymers can also be used for active routing, switching and even high speed modulation of optical data, because all polymers have large thermo-optic coefficients and , in addition , a variety of polymers have shown good electro-optic properties. A large number of optical polymers have proven to

be thermally, chemically and mechanically stable, thus fulfilling the heavy requirements needed for operation under harsh environmental conditions.

The most appealing characteristic of polymer waveguide technology is the simplicity and flexibility of waveguide fabrication methods. Polymer thin films can be deposited in a wide thickness range by spin or dip coating using relatively simple equipment. A variety of channel waveguide fabrication methods exist, ranging from existing micro-technology techniques such as etching , to mass production methods developed especially for polymers, including molding and laser delineation.

1.5.1 Polymer material properties for integrated optics

For a polymer to be selected as a material for wave guide technology, it has to satisfy the following set of requirements: [62]

- Low optical losses (not more than 0.1dB/cm) in the communication spectral windows around 800,1300or 1500nm.
- Low wavelength dispersion
- Low birefringence
- Low polarization dependent losses
- Thermally stable optical and mechanical properties
- Resistant to humidity
- Good mechanical properties such as flexibility and roughness
- Low cost

1.5.2 Fabrication of polymer optical waveguides

Fabrication methods used to produce polymer waveguides have to comply with the following set of general demands

- Optical losses introduced by fabrication, such as that caused by scattering on rough side walls, have to be kept a minimum.
- The method has to be simple and reproducible.
- The fabrication process should be suitable for low-cost mass production
- High precision, sometimes well below 1μm is required.

There are at least three fundamental categories of channel waveguide fabrication techniques, namely:

1. Optical lithography combined with wet etching.
2. molding
3. Photolithographic delineation

1.5.2.1 Optical lithography combined with wet etching

In optical lithography technique, first a polymer layer stack is deposited on a polymer substrate. The first problem is the addition of the under-cladding to the substrate and addition between subsequent polymer layers. A detailed sketch is as shown in the figure1.8. After the above mentioned process is completed a deposition process is initiated by spin coating which should result in crack-free homogeneous layer which covers the full chip area. Between subsequent stages, the layers should be cured to avoid partial dissolution of existing layers by freshly deposited layers [63].

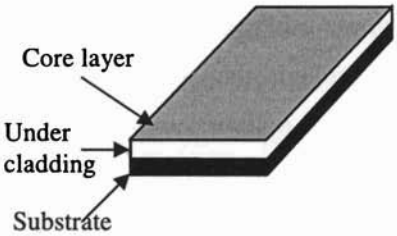
The structure of the waveguide core can be defined by removing unwanted parts of the core layer using an etching process. In such a process, first a core layer is deposited by spin or dip coating on a lower index cladding layer.

After photolithography process using a photo resist, a waveguide pattern is formed. The channel waveguide is then realized by etching. Dry or wet etching can be used to structure polymer waveguides. Among dry etching reactive ion etching (RIE) is the most efficient and widely used because of good selectivity, little undercut and high productivity [64].

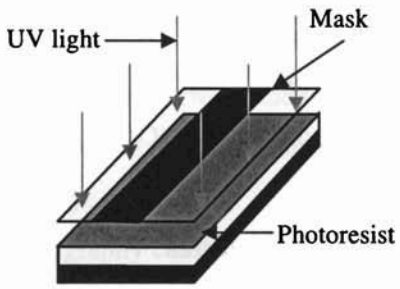
Pure oxygen plasma is most commonly used to etch polymers. Certain gases can be added to oxygen in the gas feed to enhance the concentration of highly reactive atomic oxygen free radicals that increase the etching rate. One common additive is tetrafluoromethane (CF_4).

1.5.2.2 Molding techniques

The term "molding" categorizes a number of low-cost replication techniques, including casting, compression molding, injection molding, soft embossing, hot embossing and LIGA (Lithographic Galvanoformung und Abformung)[59,65,66]. The choice of the method is chiefly determined by the polymer being used. Compression molding, like all other molding techniques, requires a mold. A mold has to be created in the form of the required structure in a mechanically polished brass substrate using a diamond turning machine and replicating this structure in nickel using electroforming. The compression molding process starts by depositing a film of polymer, from which the structure is going to be made, on to a substrate. The thickness of the deposited film should be equal to the height of the intended structure. Then both the mold and the polymer film are heated to polymer T_g temperature. After heating, the two parts are pressed together. The polymer film, which is softened by heat, takes exactly the shape of the mold. The final step involves hardening the created structure by heat treatment or UV curing.



Step1: Deposition of waveguiding layer stack with undercladding and core layer

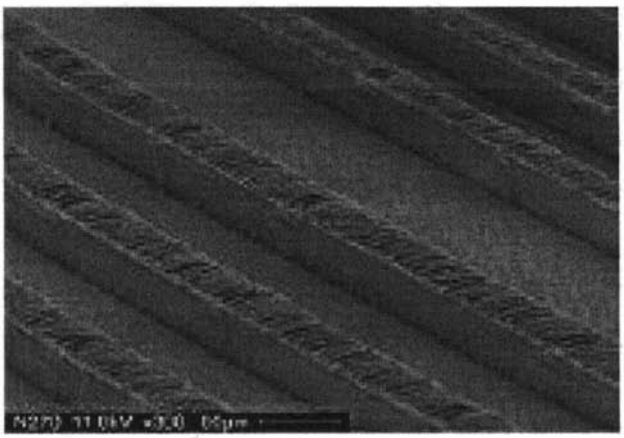


Step2: Photolithography process of the resist layer

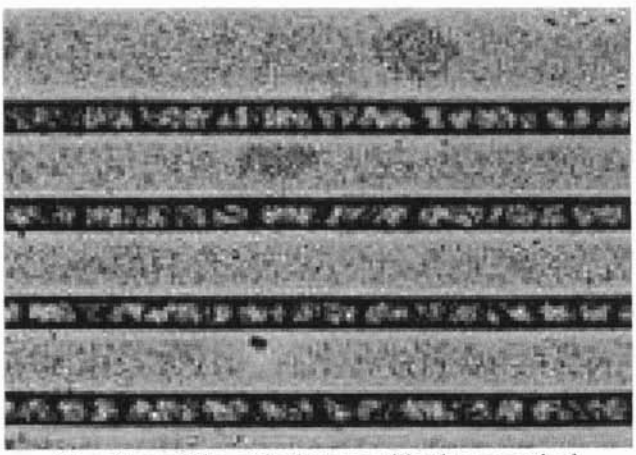
Step3: Formation of waveguide pattern by photolithography

Step4: Realization of the channel waveguide by etching.

Figure 1.8: Fabrication of polymer waveguide using lithography and etching



SEM picture of polymeric multimode channel waveguides



Top view of the etched waveguides by an optical microscope

Figure 1.9: Channel waveguides made by molding

1.5.2.3 Photolithographic delineation

Polymer waveguides can be realized by externally induced dopant diffusion. There are two types, namely, photolocking and selective polymerization. In both, a monomer is doped with a certain photosensitive dopant. The doped monomer is spun onto a substrate and the layer is exposed under a mask for UV light. The dopants are locked into the host polymer during photo-polymerization reaction [66]. When the polymer material is heated after the removal of the mask, the exposed regions are polymerized, whereas out-diffusion of the dopants can occur in the unexposed regions. In photolocking, the core regions are exposed and the dopants in the cladding are out diffused, which lower the refractive index because the dopants have higher refractive index than that of the host polymer.

1.6 Polymer based integrated waveguide devices

Devices which are of great interest in optical communication and integrated optics using polymers are power splitters, switches, modulators, wavelength multiplexers/demultiplexers, optical cross–connects, dispersion compensators, gain equalizers, light sources and optical amplifiers.

Due to relatively large thermo-optic coefficient of polymers, it is possible to realize active devices that require low switching power. The main structure used for thermo-optic devices are the Mach-Zehnder interferometers (MZI), switched directional couplers (SDC) and digital optical switches (DOS) [62, 63]. In a system context the most important properties of a switch is its isolation: the attenuation of power at an output port in the "off" state with respect to the power at an output port in the "on "state. The isolation provided by thermo optic polymer switches is not perfect. For MZI and SDC-type switches even small errors in the waveguide dimensions or refractive index can significantly reduce the attainable isolation below 20dB. Conversely, a

DOS has relatively liberal tolerances of birefringence, fabrication accuracy and driving conditions. On the other hand switches can be combined to form an $n \times m$ matrix that connects n inputs to m possible outputs.

Wavelength-division multiplexing (WDM) is currently the most important technology in optical communications. Several polymer-based devices for WDM applications have been demonstrated, such as optical filters, demultiplexers and add-drop (de) multiplexers which involve electrooptic polymer devices. Figure 1.10 shows the different polymer integrated optic devices along with their transfer functions.

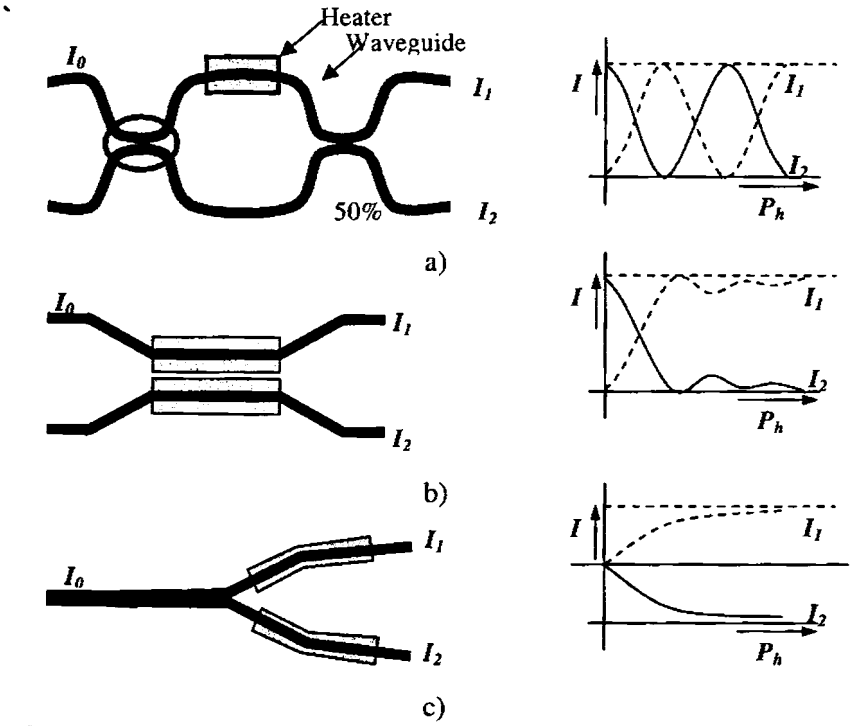


Figure1.10: Important thermo-optic switching structures: a) Mach-Zehnder interferometer; b) Switched directional coupler and c) digital optical switch. The graphs show their corresponding idealized transfer functions

1.7 Conclusion

In this chapter we discussed in detail the emergence of polymer photonics as a potential field. Different types of polymers for application in fiber optics and integrated optics were also discussed.

References

1. Spangler, C. W., Suo, Z., Drobizhev, M., Karotki, A. and Rebane, A., "New Organic Dendrimers with Greatly Enhanced Multi-photon Absorption for Photonics Applications", Organic Nanophotonics, NATO Science Series, F. Charra, V. M. Agranovich and F. Kajzar, Eds. Kluwer Academic Pub., Doordrect, 1398 (2003).
2. L Eldada and L.W Shacklette," Advances in polymer integrated optics" , IEE.J. Select.Top.Quantum Electron,6,54,2000
3. L Eldada," Advances in polymer integrated optical componemetry", Integrated Photonics Research Conference, Proc, IPR.ITUH1,2001
4. B. Ward, A. C. Baker, and V. F. Humphrey "Nonlinear propagation applied to the improvement of resolution in diagnostic medical ultrasound" *J Acoust Soc of America* 101, 143-154, 1997.
5. Paul C. Nicolson, Jurgen Vogt, "Soft contact lens polymers: an evolution" *Biomaterials* 22, 3273–3283, 2001
6. "Surface chemical structure for soft contact lenses as a function of polymer processing", *J Biomed Mat Res*, 32, 45 – 54,1998
7. J. H. Burroughes, D. D. C. Bradley, A. R. Brown, R. N. Marks, K. Mackay, R. H. Friend, P. L. Burns and A. B. Holmes" Light-emitting diodes based on conjugated polymers" *Nature* **347**, 539 - 541 1990
8. N. C. Greenham, S. C. Moratti, D. D. C. Bradley, R. H. Friend and A. B. Holmes, "Efficient light-emitting diodes based on polymers with high electron affinities", *Nature* **365**, 628 – 630, 1993.
9. V. L. Colvin, M. C. Schlamp & A. P. Alivisatos, "Light-emitting diodes made from cadmium selenide nanocrystals and a semiconducting polymer", *Nature* **370**, 354 – 357, 1994.
10. Y. Yang and A. J. Heeger, "Polyaniline as a transparent electrode for polymer light-emitting diodes: Lower operating voltage and higher efficiency", App Phy Lett, 64, 1245-1247, 1994.
11. P. E. Burrows, G. Gu, V. Bulovi´c, Z. Shen, S. R. Forrest, and M. E. Thompson "Achieving Full-Color Organic Light-Emitting Devices for Lightweight, Flat-Panel Displays", *IEEE Trans Elect Dev*, 44,118,1997
12. G. Gu and Stephen R. Forrest, "Design of Flat-Panel Displays Based on Organic Light-Emitting Devices", *IEEE J. Sel Top Quant Elect*, 4, 83,1998
13. Park S.K., Han J.I, Kim W.K., Kwak M.G, "Deposition of indium-tin-oxide films on polymer substrates for application in plastic-based flat panel displays", *Thin Solid Films*, 397, 49-55(7), 2001.
14. Prediction of polymer properties,3rd ed, Jozef Bicerano, Marcell Dekker,2002
15. Handbook of polymer science and technology, vol2, "Nicholas P Chremisinoff", Marcell Dekker,1989
16. Photonic polymer systems, Fundamentals, methods and application, Donald L Wise, Wise L Wise, Marcel Dekker, 1998

17. Ruth H. Pater, "Interpenetrating polymer network approach to tough and microcracking resistant high temperature polymers. Part II. LaRC-RP41", *Poly Eng and Sci.*3,20–27,2004.
18. G. Fischbeck, R. Moosburger, C. Kostrzewa, A. Achen, and K. Petermann,"Singlemode optical waveguides using a high temperature stable poylmer with low losses in the 1.55pm range", *Eec Lett*, 33,518. 1997
19. AH Frazer, "High Temperature Resistant Polymers", New York, Interscience Publishers, 1968.
20. Philippe M. Becker, Anders A. Olsson, Jay R. Simpson, "Erbium-Doped Fiber Amplifiers: Fundamentals and Technology", Lucent Technologies, Academic press,1999
21. E. Desurvire J. R. Simpson, P. C. Becke ,"High-gain erbium-doped traveling-wave fiber amplifier", Opt. lett.,12, 888,1987
22. Mori, A. Ohishi, Y. Sudo, S, "Erbium-doped tellurite glass fibre laser and amplifier", Elec. Lett., 33, 863-864, 1997
23. Miniscalco, W.J. ,"Ewrbium-doped glasses for fiber amplifiers at 1500 nm", J. Light Wave Technology, 9, 234-250, 1991
24. Kibler, T. Poferl, S. Bock, G. Huber, H.-P. Zeeb, E., "Optical data buses for automotive applications", *J Lightwave.Tech*, 22, 2004
25. Cooper, K.R. Elster, J. Jones, M. Kelly, R.G ,"Optical fiber-based corrosion sensor systems for health monitoringof aging aircraft", *AUTOTESTCON Proceedings IEEE Systems Readiness Technology Conference*, 2001
26. Anthony Ng'oma, Ton Koonen, Idelfonso Tafur Monroy, Henrie van den Boom, Peter Smulders, Giok-Djan Khoe,"Low Cost Polymer Optical Fibre based Transmission System for Feeding Integrated Broadband Wireless In-House LANs", *Proceedings Symposium IEEE/LEOS Benelux Chapter*, Amsterdam,2002
27. H. P. A. van den Boom, W. Li, P. K. van Bennekom, Tafur Monroy, and Giok-Djan Khoe, "High-Capacity Transmission Over Polymer Optical Fiber", *IEEE J SEL TOP QUAN ELEC*, 7, 2001
28. Takaaki Ishigure, Yasuhiro Koike, and James W. Fleming ,"Optimum Index Profile of the Perfluorinated Polymer-Based GI Polymer Optical Fiber and Its Dispersion Properties", *J Lightwave.Tech*, 18, 2000
29. T. Ishigure, E. Nihei, and Y. Koike, "Optimum refractive-index profile of the graded-index polymer optical fiber, toward gigabit data links," *Appl. Opt.* 35, 2048, 1996
30. Andreas Neyer, Bj¨orn Wittmann, and Matthias J¨ohnck ,"Plastic-Optical-Fiber-Based Parallel Optical Interconnects", *IEEE J. SEL TOP QUAN ELEC*, 5, 393,1999
31. A. Tagaya, S. Teramoto, E. Nihei, K. Sasaki, and Y. Koike, "High-power and high-gain organic dye-doped polymer optical fiber amplifiers: novel techniques for preparation and spectral investigation ," *Appl. Opt.* 36, 572-578 ,1997
32. A. Tagaya, Y. Koike, T. Kinoshita, E. Nihei, T. Yamamoto, and K. Sasaki," Polymer optical fiber amplifier", *App Phy Lett*, 63, 883-884,1993

33. Tagaya, A. Teramoto, S. Yamamoto, T. Fujii, K. Nihei, E. Koike, Y. Sasaki, K., "Theoretical and experimental investigation of rhodamine B-dopedpolymer optical fiber amplifiers", *IEEE J. QUAN ELEC*, 31, 2215-2220, 1995.
34. C. Koeppen, S. Yamada, G. Jiang, A. F. Garito, and L. R. Dalton, "Rare-earth organic complexes for amplification in polymer optical fibers and waveguides," *J. Opt. Soc. Am. B* 14, 155, 1997.
35. Tsuyoshi Yamamoto, Kazuhito Fujii, Shigehiro Teramoto, Akihiro Tagaya, Eisuke Nihei, Takeshi Kinoshita, Yasuhiro Koike, and Keisuke Sasaki, "High-power polymer optical fiber amplifiers and their applications", *Proceedings of SPIE, Doped Fiber Devices and Systems*, 2289, 142-152, 1994.
36. Gang Ding Peng Chu, P.K. Zhengjun Xiong Whitbread, T.W. Chaplin, R.P. ,"Dye-doped step-index polymer optical fiber for broadband opticalamplification", *J LIGHTWAVE TECH*, 14, 2215-2223,1996
37. P.P. Sorokin, J.R. Lankard and V.L Moruzzi,"Frequency- locking of organic dye lasers to atomic resonance lines", Appl.Phy.Lett,15,179-181,1969
38. Snavely, B.B. ,"Flashlamp-excited organic dye lasers", *Proceedings of the IEEE*, 57, 1374- 1390,1969
39. P. P. Sorokin and J. R. Lankard, "Infrared Laser Action in Sr Vapor Resulting from Optical Pumping and Inelastic Collisions", *Phys. Rev.* 186, 342–343,1969
40. C. V. Shank,"Physics of dye lasers", *Rev. Mod. Phys.* 47, 649–657,1975
41. P. P. Sorokin, J. R. Lankard, E. C. Hammond, V. L. Moruzzi, "Laser-pumped Stimulated Emission from Organic Dyes: Experimental Studies and Analytical Comparisons", *IBM J.Res.Dev*, 11, 130, 1967
42. J. Zubia, G. Garitaonainda, and J. Arrue, "Passive Device Based on Plastic Optical Fibers to Determine the Indices of Refraction of Liquids ," *Appl. Opt.* 39, 941-946, 2000.
43. Bartlett R.J. Philip-Chandy R. Eldridge P. Merchant D.F. Morgan R., Scully P.J., "Plastic optical fibre sensors and devices", *Transactions of the Institute of Measurement and Control*, 22, 431-457(27), 2000.
44. Philip-Chandy, R. Scully, P.J. Eldridge, P. Kadim, H.J. Grapin, M.G. Jonca, M.G. Dapos Ambrosio, M.G. Colin, F. , "An optical fiber sensor for biofilm measurement using intensitymodulation and image analysis" , I*EEE J. SEL TOP QUAN ELEC*, 6,, 764-772, 2000
45. Masayuki Morisawa, Kohji Uchiyama, Toshiki Hosaka, Hideaki Inoue, and Shinzo Muto, Akira Namazue, Eisuke Nihei, and Yasuhiro Koike, "New cladding polymer for optical oxygen sensor using fluorescent plastic fiber", *Proceedings of SPIE, Chemical, Biochemical, and Environmental Fiber Sensors VIII*, 2836, 336-340, 1996.
46. Zubia, J. Aresti, O. Arrue, J. Lopez-Amo, M ,"Barrier sensor based on plastic optical fiber to determine the windspeed at a wind generator", I*EEE J. SEL TOP QUAN ELEC*, 6, 773-779, 2000
47. M.Naritomi," Proceedings of the Asia-Pecific Polymer optical Fiber workshop", Sydney,38-59,2001

48. C.Ernst,R.Hohmann, M. Loddoch,C. Marheine and H Kargl," Proceedings of the international polymer optical fiber workshop", Sydney,95-96,2001
49. R.Attia and Marcou, "Mode scrambler for polymer optical fibers", *Opt.eng*, 39, 299, 2000.
50. Andrew Bierman, Nadarajah Narendran, and Nishantha Maliyagoda , "How to report light loss values for optical fibers used in fiber optic lighting applications", *Proceedings of SPIE Illumination and Source Engineering*,- 3428, 62-72, 1998.
51. J. Brandrup, E.H. Immergut, D.R. Bloch and E.A. Grulke, Polymer Handbook (The), 2 Volume ,Wiley, 2003
52. A Ravve , Principles of Polymer Chemistry , Springer, 2000
53. Bruno Vollmert ,Polymer Chemistry, Springer-Verlag, 1975
54. Marvin J Weber, Weber J Weber ,Handbook of Optical Materials, CRC Press, 2003
55. Marc J Madou , Fundamentals of Microfabrication, CRC Press, 2002
56. Werner Daum, Jrgen Krauser, Peter E Zamzow, Olaf Ziemann, Pof - Polymer Optical Fibers for Data Communication, Springer, 2002.
57. Olagoke Olabisi, Handbook of Thermoplastics, Marcel Dekker, 1997
58. Y. Shi, C. Zhang, H. Zhang, J. H. Bechtel, L R. Dalton, B H. Robinson, W. H. Steier, "Low (Sub D1-Volt) Halfwave Voltage Polymeric Electro-optic Modulators Achieved by Controlling Chromophore Shape" *SCIENCE* ,288, 2000
59. Eldada, L. Shacklette, L.W. , "Advances in polymer integrated optics", *IEEE J. SEL TOP QUAN ELEC*, 6, 54-68, 2000
60. Eldada, L. Chengzeng Xu Stengel, K.M.T. Shacklette, L.W. Yardley, J.T. , "Laser-fabricated low-loss single-mode raised-rib waveguiding devices in polymers", *J Lightwave.Tech*, 14, 1704-1713 ,1996
61. Louay Eldada, Robert Blomquist, Lawrence W. Shacklette, and Michael J. McFarland' "High-performance polymeric componentry for telecom and datacom applications", *Optical Engineering*, 39, 596-609, 2000.
62. Hari Singh Nalwa, Polymer Optical Fibers, American Scientific Publishers, CA,2004
63. Sulur, Polymer Waveguides for GIPOF applications, Master Thesis, University of Twente, The Netherlands, 2001.
64. D. Elliot, "Microlithography Process: Technology for IC Fabrication" McGraw-Hill, New York, 1986.
65. L Wu, F.Li, S. Tang, B. Bihari and R.T. Chen, *IEEE Photon. Technol.Lett*, 8, 1647, 1996.
66. Fischer, D. Voges, E, "Multimode polymeric waveguide devices fabricated by two-componentinjection molding", *Elect.Lett*, 33, 1626-1627, 1997.

There is a single light of science, and to brighten it anywhere is to brighten it everywhere. **Isaac Asimov**

Fabrication of Polymer optical fibers

For drawing polymer optical fibers (POF), an indigenously developed optical fiber drawing tower has been used. This chapter discusses in detail about the fabrication and working of the POF drawing tower.

2.1 Introduction:

Polymer optical fibers (POFs) [1] were introduced by Dupont in the mid 1960s at approximately the same time when glass optical fiber (GOF) was suggested as a transmission medium for communication. Dupont's fiber was having a polymethylmethacrylate (PMMA) core and fluoropolymer cladding, with a step index refractive index profile. Even though POF has the fascinating ability to guide light, it had limited applicability due to large attenuation (~dB/m) and hence negligible commercial value. At the same time GOFs had attenuation in excess of 1000dB/Km, which made its use in optical communication impractical [2]. In 1976, Mitsubishi rayon introduced Eska™ by further developing DuPont's extrusion technology. It had an attenuation of 300dB/km which limited its application to lighting only (illumination). The core was PMMA and polyfluoroalkyl methacrylate was the cladding for Eska™.[3-5]

By 1970s the attenuation of POF came down to 160dB/km at a wavelength of 650nm and 20dB/km at 680nm using deuterated PMMA (PMMA-d8) [6, 7]. All these were SI fibers. Only by 1976 the first graded index (GI) POF came into existence. It was Ohtsuka and Hatanaka who made GIPOF by heat drawing graded index plastic rod [8]. Earlier, graded index plastic rods were prepared by two different methods, namely two-step copolymerization and photo-copolymerization [9-15]. But the attenuation was found to be fairly large for the fibers drawn from preforms made by the above methods. Soon interfacial gel polymerization technique came into existence and fibers with fairly negligible attenuation were fabricated [16-22].

With great strides made in the field of GIPOFs in reducing loss parameters, many applications like high bandwidth data communication for LAN and home network were realized [23,24]. Possibility of realizing larger diameter

of POFs made splicing more easy and allowed the use of low cost light sources and connectors.

2.2 Fabrication of step index and graded index plastic optical fibers

2.2.1 Step-index fibers

A step index fiber has a simple structure owing to its simpler fabrication techniques. The most common methods used are a) continuous extrusion method and b) preform method.

2.2.1.1 Continuous extrusion method:

This method was successfully developed by Mitsubishi rayon [3, 4], and is one of the well-developed methods for developing step index polymer optical fibers (SIPOF). In this method, a purified monomer (methylmethacrylate), an initiator and a chain transfer agent are fed into a polymerization reactor, where polymerization takes place. The polymer thus formed is then fed into an extruder by a gear pump. This is the core material for the SIPOF. The core material and the cladding material, which are fed by separate extruders, proceed into a co-extrusion die or spinning block, where concentric core –cladding structure of SIPOF is formed.

The most important advantage of continuous extrusion is high production rate. The drawback is that this cannot be operated continuously since the materials used for extruding SIPOF gets thermally degraded as time progresses. This will, in turn, have adverse effect on the optical properties of the fiber.

2.2.1.2 Preform method:

Fabrication of POFs by preform method involves two stages. In the first stage a cylindrical preform of 1-5 cm in diameter and up to 1m in length is made. In the second stage, the preform is drawn into fiber by the heat-drawing process.

Preform –making process:

In the fabrication of glass optical fibers, fabrication techniques like MCVD (modified chemical vapour deposition), OVD (outside vapour deposition) and VAD (Vapour axial deposition) methods are used. One of the familiar methods is applied here to make a preform for POFs. A cylindrical tube, which serves as the cladding layer is made by polymerizing a cladding material inside a rotating cylindrical reactor. Materials that can be polymerized by the radical polymerization reaction are used and the reaction is induced thermally or by UV radiation using photo initiator. Due to the fast rotational speed of the reactor about its axis, a tube of uniform thickness is formed once the reaction is complete. This tube is then removed from the reactor and filled with a core material mixed with an initiator and a chain transfer agent. The core material is then polymerized to make a preform that is drawn to a fiber by heat-drawing process.

There are two other commonly used methods for preform fabrication. First is the rod-in-tube method, in which a pre-fabricated polymer core rod is inserted into a pre-fabricated polymer cladding tube with a tight-fitting [25]. The disadvantage of this method is that the rod diameter is usually too large for the drawn fiber to achieve a single –mode operation. Also bubbles may be trapped at the core-cladding interface. The second method is the hole-in-rod technique [26] where a hole is drilled into a cladding polymer rod followed by pouring in core monomer. A polymerization process is then initated to

obtain a composite core-cladding polymer preform. The core-cladding interface of this preform may not be smooth because of drilling. This could lead to excessive loss.

"Teflon technique" [27] is the most successful technique for the fabrication of the preform. In this technique a thin teflon string is properly fixed in the center of a glass tube. The thermal polymerization of the filled tube is carried out in a temperature controlled oil bath. After the monomers are fully polymerized and heat treated, the teflon string is removed and we obtain a polymer tube (polymer rod with a small hole in its center). The bottom side of the core is sealed and the hole is then filled with the initiated monomers for the core. Again it is kept in an oil bath for further polymerization.

The advantages of using the Teflon technique in the fabrication of polymer preform are a) its nonsticking property allows the string to be easily removed b) it has a very good chemical and thermal stability c) it gives smooth finish for the inner surface of the polymer tube. d) it gives good core-cladding interface and hence reduces the losses due to scattering.

Heat drawing process:

The heat drawing process is shown schematically in figure 2.1. The preform is positioned vertically in the middle of the furnace (or oven) where its lower portion is heated locally to the drawing temperature [28-30]. Both convective and radiative heat transfer mechanisms are important in heating the preform. When the lower part of the preform reaches a temperature beyond its softening point, it necks downward by its own weight due to gravity. Once this initiation of the drawing process is achieved, tension is applied to the fiber by drawing rollers and the fiber is drawn continuously while the preform is fed at a pre-determined rate.

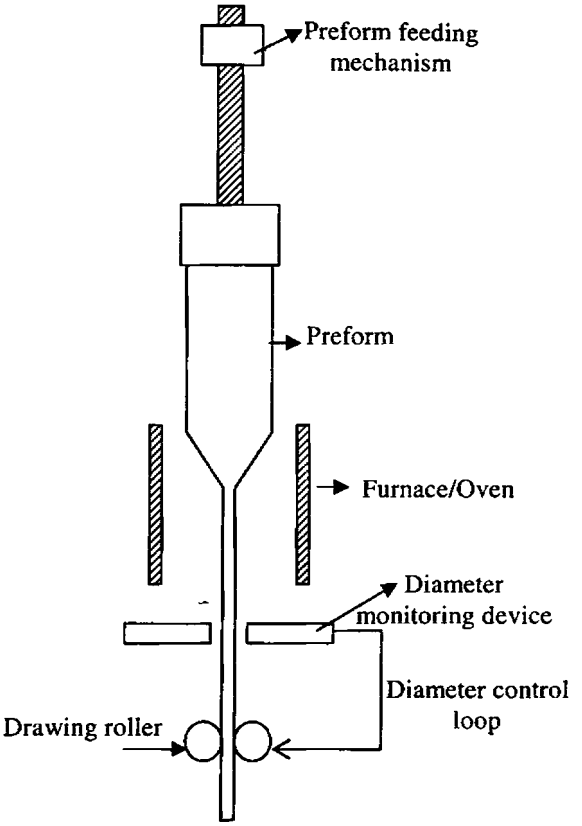


Figure 2.1 Schematic diagram of heat drawing process

The fiber diameter is continuously measured and the desired value is maintained by controlling the speed of the roller. Another design of the fiber drawing system is the horizontal drawing rig. The only difference in this case from the conventional drawing system is that the whole drawing procedure is horizontal. GD Peng etal [27] has widely used this type of configuration and has observed no fiber sagging during drawing and the fiber diameter was kept at an acceptable tolerance. The horizontal machine offers an additional advantage of convenience of saving the need of climbing up and down the ladder during the draw initiation phase.

2.2.2 Graded index Polymer optical fibers

To date, several methods have been suggested for the manufacture of GI-POFs. Although continuous extrusion is the dominant method for the manufacture of GIPOF as in the case of SIPOF, the preform method appears to be the preferred approach due to its versatility.

2.2.2.1 Preform method

For the fabrication of graded index polymer optical fibers the most common approach is the preform method. Both photocopolymerization and interfacial gel polymerization techniques are widely used for this purpose.

Photocopolymerization

The first GI POF was fabricated by Ohtsuka and Hatanaka in [8, 9] 1976 by heat drawing graded index plastic rods. At that time, graded index plastic rods were made by two- step co-polymerization and photo- copolymerization techniques [10-15]. In two step co-polymerization, a polymerized rod is prepared and immersed into a co-monomer that has a lower refractive index. The co-monomer then diffuses into the rod to form a concentration gradient in the radial direction. This is subsequently fixed by further polymerization. To prevent the immersed rod from dissolving too much while allowing the co-monomer to diffuse inward, the pre-polymerised rod has a cross linked network structure. Conversely, a GI rod made by photo-copolymerization does not have a network structure, and can be heat drawn into POF.

Figure 2.2 shows schematic of the photo-co-polymerization apparatus by Ohtsuka etal [13]. A 2.9mm diameter glass tube reactor was positioned vertically in a constant temperature chamber and rotated about its vertical axis while being irradiated by UV radiation. The UV radiation quickly induces polymerization reaction forming a gel phase. Since the UV intensity

was higher near the wall of the glass tube, the gel phase formed first on the inner wall of the glass tube and grew inward towards the center of the tube.

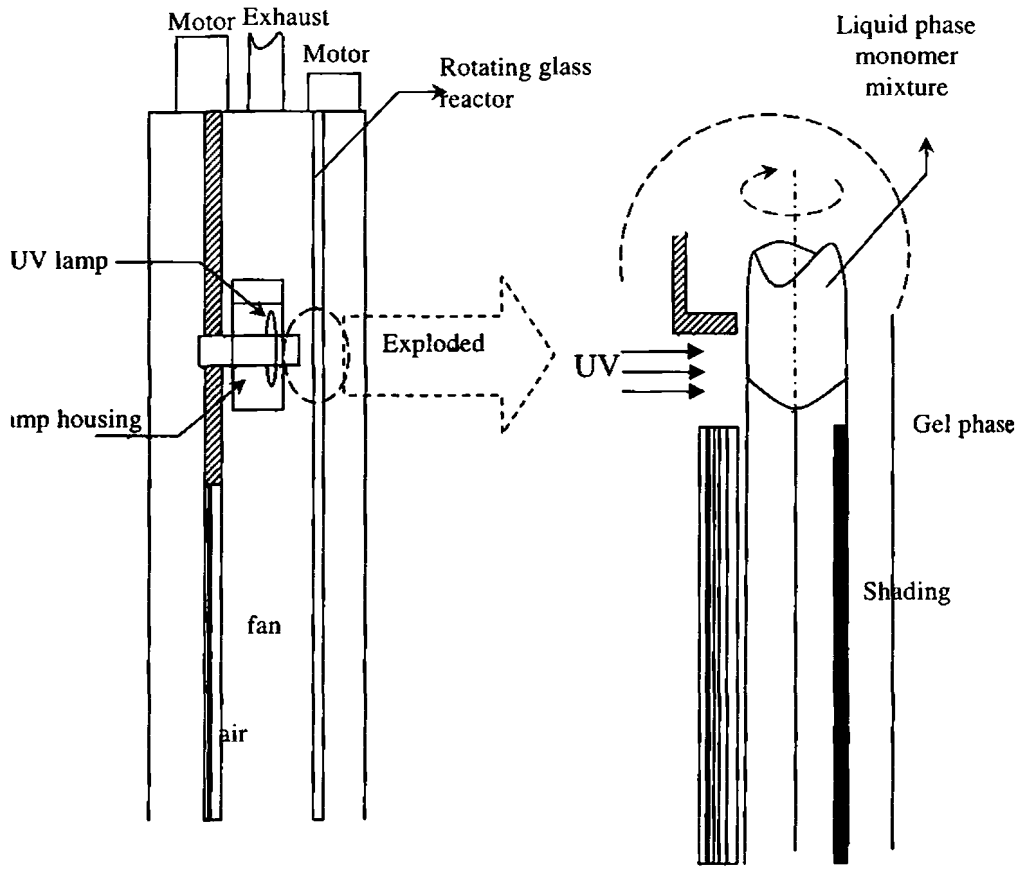


Figure 2.2 Schematic of photo-co-polymerization

Interfacial gel polymerization

The best known method for the fabrication of GIPOF is the interfacial gel polymerization method which was pioneered by Koike and his co workers [16, 17]. At first, this method was applied to the co-polymerization of the monomer mixture with different reactivity ratios and different refractive indices. Later it was applied to mixtures of a monomer and a non reacting organic dopant. In this method a transparent polymeric tube (eg: PMMA) is prepared and this tube is then filled with a mixture of two monomers (eg: Methylmethacrylate and vinyl benzoate) that is polymerized thermally while the tube is rotated. The inner wall of the tube is swollen by the monomer mixture as it forms a thin gel phase. Due to "gel effect" the polymerization reaction is faster inside the gel phase than in the monomer bulk phase. Consequently, the reaction occurs preferentially on the inner surface of the tube and the co-polymer phase extends inward towards the center of the tube as reaction proceeds. Due to the difference in the reactivity ratios the composition of the copolymer changes gradually in the radial direction. If the monomer with higher reactivity ratio has a smaller refractive index than the other monomer, the preform will have a gradually increasing refractive index towards the center. The refractive index profile obtained by this method depends on the relative ratios of monomer mixture.

2.2.2.2 Drawing GI POF

There are many methods for drawing GIPOF. Some of these methods are a) internal diffusion and surface evaporation b) closed extrusion method and c) co-extrusion method.

The internal diffusion and surface evaporation (IDSE) process was developed by Mitsubishi rayon [31-33]. Here the polymer solution is charged into a

cylinder and pushed by a piston through a nozzle to form a strand fiber. In closed extrusion method which was developed by Ho and coworkers [34-36], a polymer solution for the core is placed in a material supply tank, and another polymer solution for cladding is put in another supply tank. These solutions are heated to a moderate temperature ($60^{o}C$) and fed into a concentric (or co extrusion) die by separate gear pumps. A bilayer concentric fiber is then extruded from the die and proceeds into an enclosed (or diffusion) zone that is maintained at a constant temperature.

The co-extrusion method was proposed by many workers like Koike and Nehei, Sohn and Park and Park Walker [37, 38]. In all these methods both the core and the cladding material are extruded with a die and feeder mechanism which feeds the core and the cladding material.

2.3 Polymerization process of PMMA:

This section provides the details of PMMA fiber drawing techniques. Polymethylmethacrylate (PMMA) is the widely used polymer to make polymer optical fibers. Polymerization is a process which allows simple low molecular weight compounds to combine and form a complex high molecular weight compound [39]. For this, each molecule of the compound should have the compatibility to react at least with two other molecules of the same or some other compound. In other words, they have a functionality of two. Low molecular weight compounds having a functionality of two or more are called monomers. For them to polymerize, we have only to induce suitable chemical reaction between them. Then these monomer molecules combine to form fewer but higher molecular weight molecules.

Chemical reaction which takes place during polymerization is a) chain polymerization and b) step polymerization.

Chain polymerization is characterised by a self addition of the monomer molecules, to each other, very rapidly through a chain reaction. No byproduct is formed and the product has the same elemental composition as that of the monomer. The bifunctionality is provided by the double bonds present in the monomer. Compounds containing reactive double bonds can therefore, undergo a chain polymerization reaction. Chain polymerization consists of three major steps, namely, initiation, propagation and termination and the process can be brought about by a free radical, ionic or coordination mechanism. Here, we concentrate on free radical polymerization.

2.3.1 Free radical polymerization

The initiation of the polymer chain growth is brought about by free radicals produced by decomposition of compounds called initiators. The term" chain growth" represents a process involving a continuous and very rapid addition of the monomer units to form polymer molecules or polymer chains. As more and more monomer units are added, the length of the polymer chains increases rapidly and the chain grows in length.

2.3.1. 1 *Initiator*

Initiators are thermally unstable compounds and decompose into products called free radicals. If R-R is an initiator, and the pair of electrons forming the bond between the two R's, can be represented by colon, the initiator can be written as R: R. When energy is supplied to this compound in the form of heat, the molecule splits into two symmetrical components. Each carries with it one of the electrons from the electron pair. This type of decomposition, where the molecule is split into two identical fragments is called 'homolytic

decomposition'. The two fragments, each carrying one unpaired (lone) electron with it, are called "free radicals".

i.e. $R - R \longrightarrow R^{\circ}$ ---------- (2.1)

The decomposition of the initiator to form free radicals can be induced by heat energy, light energy or catalysts. A host of low molecular weight compounds comprising mainly of azo compounds, peroxides, hydroperoxides and pre-esters are used as initiators.

2.3.1.2 *Initiation*

We know that a free radical contains a lone (unpaired) electron which is always looking for another lone electron to couple with it and get stabilized. A free radical, is therefore, highly reactive and can attack any molecule which either has a lone electron or is prepared to part with one of its electron. This is what happens in the process of initiation. The free radical R° attacks the double bond in the monomer molecule resulting in the following chemical change.

$$R^{\circ} + \underset{\underset{X}{|}}{CH_2} = CH \rightarrow R - CH_2 - \underset{\underset{X}{|}}{C^{\circ}H} \qquad \text{----------} \qquad (2.2)$$

2.2.1. 3 *Propagation*

After "initiation", comes, the step called "propagation". In the propagation step, the radical site at the first monomer unit attacks the double bond of a fresh monomer molecule. This results in the building up of the second monomer unit to the first and the transfer of the radical site from the first monomer unit to the second, by the unpaired electron transfer process.

$$R - CH_2 - \underset{\underset{X}{|}}{C^{\circ}H} + CH_2 = \underset{\underset{X}{|}}{CH} \rightarrow R - CH_2 - \underset{\underset{X}{|}}{CH} - CH_2 - \underset{\underset{X}{|}}{C^{\circ}H} \qquad \text{---} \qquad (2.3)$$

It should be noted that this chain still contains a radical site (indicated by a dot) at its end-carbon atom and can, therefore, attack yet another monomer molecule with a simultaneous transfer of the radical site to the monomer unit added.

2.3.1.4 *Termination*

The process "termination" is the final step. Here, any further addition of the monomer units to the growing chain is stopped, and the growth of the polymer chain is arrested. Since the decomposition of the initiator produces many free radicals at the same time, each one of them can initiate and propagate the chain growth simultaneously and, hence, at any given time, there may be quite a few growing chains present in the system. Depending on factors such as temperature, there exists a statistical probability of the two growing chains coming close to and colliding with each other. Such a collision results in the arrest of the chain growth.

2.4 Fabrication of the fiber preform

The base material used for the fabrication of polymer preform is methylmethacrylate (MMA) monomer. Methylmethacrylate is a suitable candidate for the fabrication of polymer preforms since it has good optical quality and compatible with most of the organic dopants. The refractive index of pure methylmethacrylate is about 1.41 and it will increase up to 1.48 to 1.49 due to volume reduction during phase transition from liquid to solid. We here concentrated primarily on only core fibers (air cladding), neglecting the scattering losses associated with it due to the absence of cladding.

Commercially available methylmethacrylate will contain inhibitors like hydroquinons. Inhibitors are used for transporting MMA with out polymerizing. Inhibitors are removed by repeatedly washing the monomer

with 5% NaOH solution followed by flushing with distilled water. The remaining water is removed by adding suitable drying agents like $CaCl_2$. The monomers are purified by distillation under reduced atmosphere.

Suitable initiators like benzoyl peroxide or azobisisobutyronitrile (AIBN) are used to start the polymerization. We used benzoyl peroxide for the present work since there is no gas such as nitrogen which gets released during polymerization as in the case of AIBN. This reduces the possibility of air bubble formation in the polymer preforms. Along with the initiator, n-butyl mercaptan is used as the chain transfer agent to regulate and terminate the polymerization process. Adding appropriate quantity of chain transfer agents and initiator controls the molecular weight of the monomer. The molecular weight regulation is an important factor that governs the drawability of the polymer preform. An optimum molecular weight (typically between 60,000 and 1,00,000) is fixed by numerous trial and error methods. To make dye doped fibers we have used Rhodamine 6G dye since it has a relatively good photostability and a high fluorescence efficiency. Rhodamine 6G at 460ppm concentration is chosen for this study and it is added along with the initiator and chain transfer agent into the monomer. The resulting mixture is stirred well so as to avoid aggregate formation.

The monomer mixed with initiator, dye and chain transfer agent is poured into a glass tube of required diameter and length. This is then kept in constant temperature bath at 70^{o}C for 48hours, at 90 ^{o}C for 18hours and at 105 ^{o}C in air furnace for 8 hours. These steps lead to a high quality polymer preform which can be used for drawing the fiber.

2.5 Polymer Optical Fiber Drawing Tower: Technical details

The polymer optical fiber drawing tower which we used for the fabrication of POF is designed and fabricated indigenously (figure 2.3).

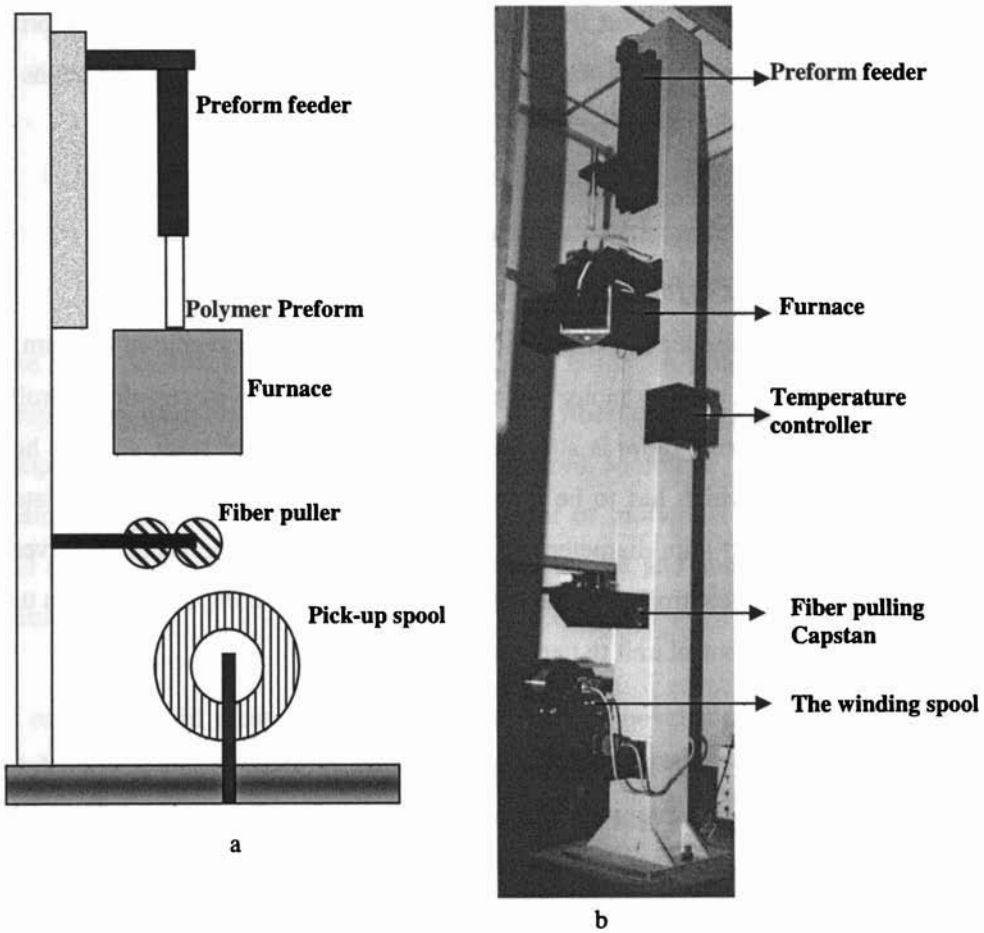


Figure 2.3 a) Polymer optical fiber drawing system b) actual photograph of the polymer optical fiber drawing station developed at ISP

The total height of the tower is 2.5 meter and weighs around 900kgs. It has five important stages namely preform feeder, furnace, temperature controller, fiber puller, and winding spool. These five stages are individually controlled by an electronic controller unit. The stages are designed in such a fashion that any alteration or repair can be done individually by removing each stage. The distance between each stage can also be optimized by making small variations. In short the system design is very flexible and appropriate alterations can be easily made to improve the system. Brief descriptions of each stage are as follows.

2.5.1 Brief description of the parts

2.5.1.1 The Preform feeder

The preform feeder is a translation stage with a total traverse of 800mm. It is driven by a stepper motor which is controlled by the electronic controller unit. A preform holder is attached to the preform feeder stage so as to hold the preform which has to be drawn to optical fibers. It can accommodate a preform upto 30mm diameter and a length of 1 meter. The speed of traverse of the stage is controlled by the data programmed to a computer which in turn instructs the control unit to perform the operation.

2.5.1.2 The furnace

The polymer preform is melted using a furnace to draw optical fibers. An ideal furnace should have very good temperature stability and temperature profile suitable for drawing the fiber. There are several methods which can be used to manufacture good temperature stability furnaces. The most commonly used furnaces employ inductive heating. In inductive furnaces, usually a heating coil (normally nichrome) is wounded over a ceramic tube which is then fired by passing current through the coil.

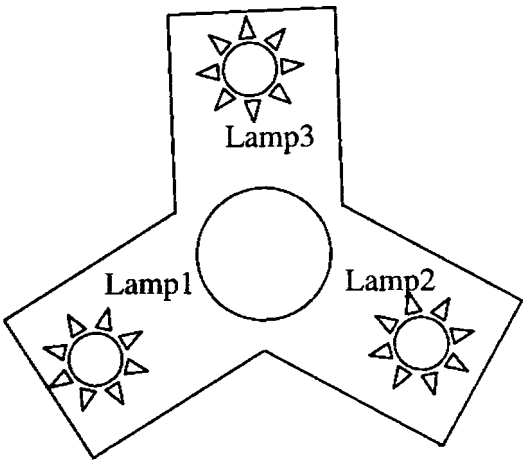


Figure 2.4 Cross sectional view of the furnace

The main drawback of these types of furnace is that in order to get good temperature stability, which is essential for fiber drawing, sophisticated temperature controller systems have to be utilized. Moreover, for attaining a desired temperature profiled furnace a number of individual coil windings and controllers are needed. This will also add weight and complexity to the furnace.

In order to avoid these difficulties we employed a novel technique for the fabrication of the furnace. Instead of using wire-wound furnace, we used IR radiation heated furnace. Three 75W IR lamps of Philips make were placed inside the furnace for heating the preform. They provide a maximum temperature of 250^{o}C which is more than enough for drawing POF. The profiling inside the furnace was adjusted by providing appropriate openings inside the furnace and controlling the amount of light inside the furnace. In order to change the furnace temperature profile, either a very slight tilt of the IR lamps or a slight change in the slit width will be sufficient.

To provide uniform heating of the preform the furnace can swing a maximum of 30^{o}. The swing period can be programmed using the computer and electronic controller unit.

Temperature distribution inside a furnace plays an important role in the fabrication of an optical fiber. An ideal temperature profile is as shown in the figure2.5. For temperature profiling of the furnace, a resistance temperature detector (RTD), PT100 is used. The detector is inserted into the furnace with the help of a stepper motor assembly. For profiling the furnace temperature, the RTD is inserted slowly into the furnace after it is kept at a temperature of $165^{o}C$ (which is the typical melting temperature of PMMA preforms).

Usually a multimeter capable of temperature measurement is connected to the temperature detector. The output from the RTD will be typically a change in the resistance in accordance with the applied temperature.

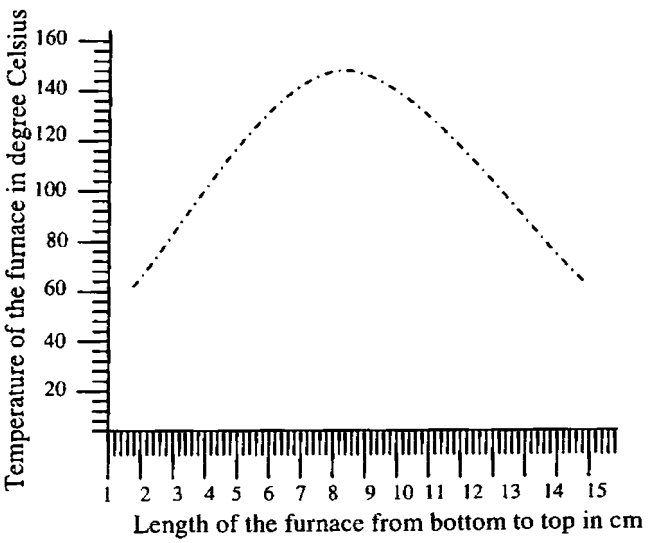


Figure 2.5 Ideal temperature profile of the furnace

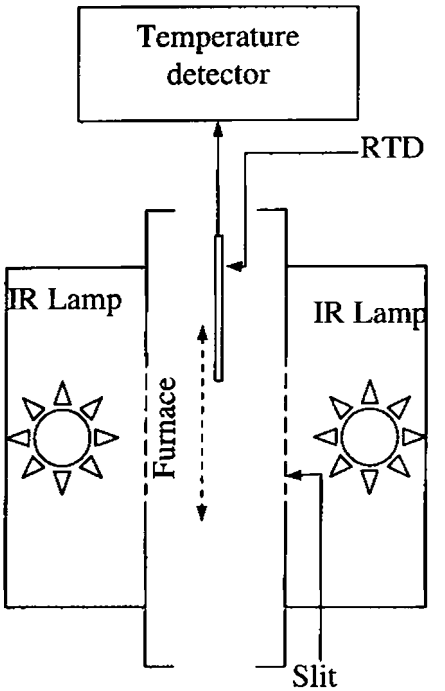


Figure 2.6 The set up for temperature profiling of the furnace

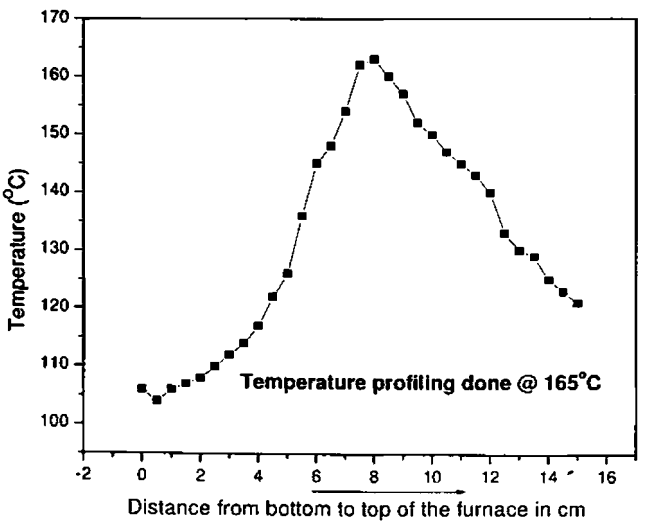


Figure 2.7 The temperature profile of the fabricated furnace along its length

The multimeter converts this variation in resistance into corresponding voltage change and a calibrated temperature reading is displayed at its output. The temperature at different points is measured and the profile obtained is as shown in the figure 2.7.

2.5.1.3 The Temperature controller

To control the temperature inside the furnace, the easiest way is to switch on and off the three IR lamps simultaneously. For that a thermocouple is attached to the central portion of the furnace where the temperature is maximum. We used a 'J type' thermocouple for serving this purpose. This is connected to a programable temperature controller for which the accuracy is +/- 1^{o}C. The required temperature can be programmed manually and the temperature controller constantly monitors whether the set temperature is achieved or not. When the set temperature is achieved it operates a relay which cuts off the power to the lamps. The controller then checks whether the temperature has come down or not. If it has come down it will activate the relay to switch the lamps to the ON condition and proceeds with the heating procedure. This can be easily represented by a flow chart as in figure 2.9.

2.5.1.4 The fiber puller

The fiber puller is another important unit of the drawing system. A schematic diagram is shown in the figure 2.8.

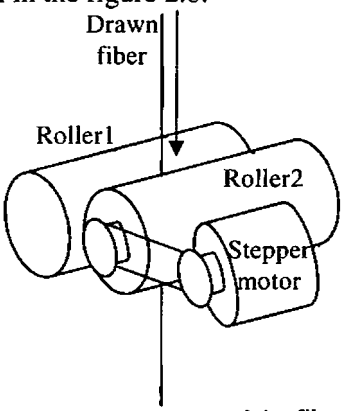


Figure 2.8 Schematic diagram of the fiber puller

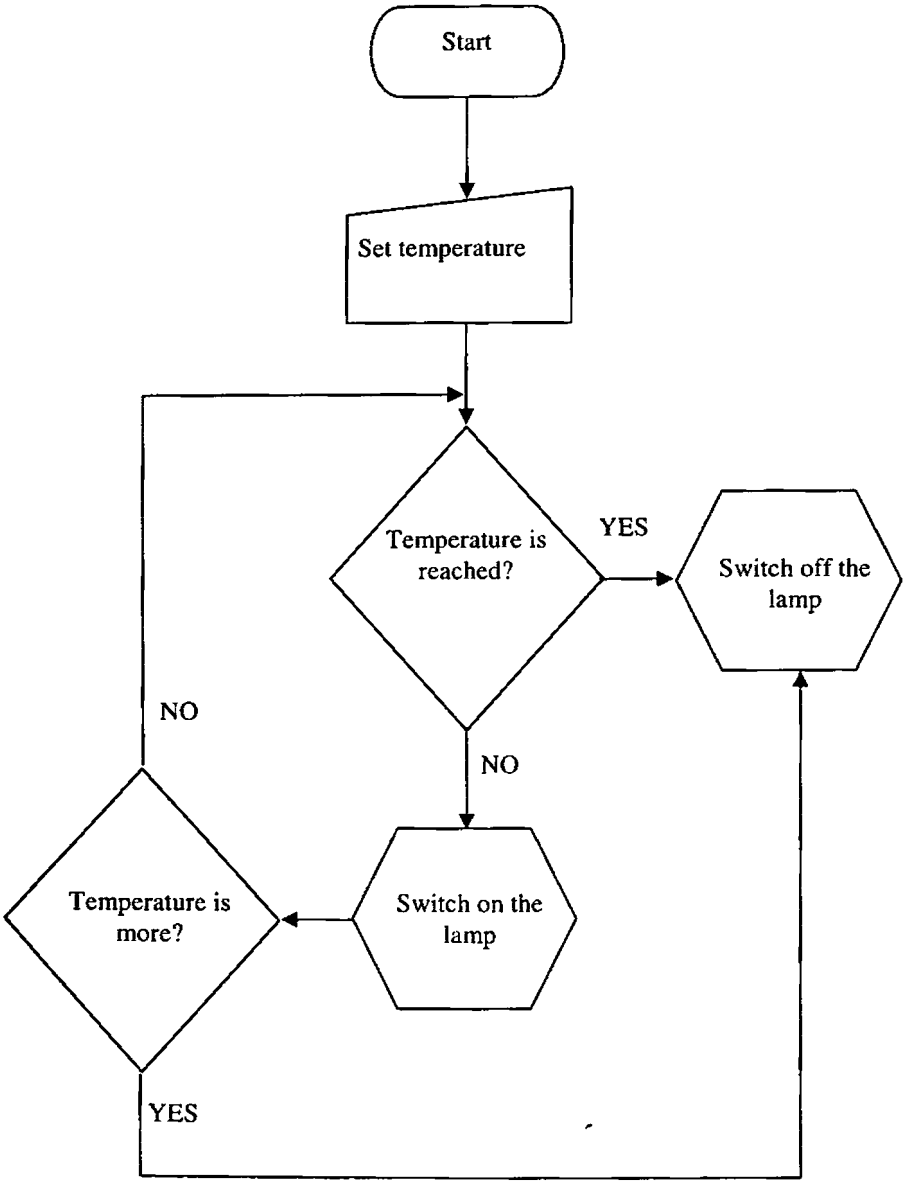


Figure 2.9 Flow chart representation of the temperature controller

The fiber puller has two rollers which are in close contact with each other. A stepper motor is connected to one of the rollers. The stepper motor is controlled by means of the computer and electronic controller unit. The required draw rate of the fiber can be programmed into the computer and the stepper motor drives the rollers at the specified draw rate. The draw rate along with the preform feed rate determines the diameter of the fiber.

$$\frac{D^2}{d^2} = \left[\frac{V_d}{V_f}\right]$$

Where D is the diameter of the preform, d is the diameter of the fiber drawn, V_f is the feed rate and V_d is the draw rate.

2.5.1.5 The winding spool

The winding spool (figure 2.10) has a two axes controller along with its rotation. The two axes controller helps the winding spool to precisely wind the drawn fiber so that the fiber will not get wound one over the other before one full spool traverse along the axis is completed. The traverse of the spool along the axis is controlled by a translation stage-stepper motor assembly over which the spool is attached. When the diameter of the fiber to be drawn is specified to the computer, the spool will move along its axis which corresponds to the fiber diameter exactly, along with the rotary motion. This will ensure that the fiber will not get wound one over the other and hence correct spacing is ensured. After one whole movement along the axis is completed, the spool will be moved perpendicular to its axis. The magnitude of the movement will be exactly in relation to the fiber diameter and the process continues.

The whole process is controlled by a limit switch on the preform feeder. If the preform feeder comes down and reaches its limit, the whole system will come to a halt.

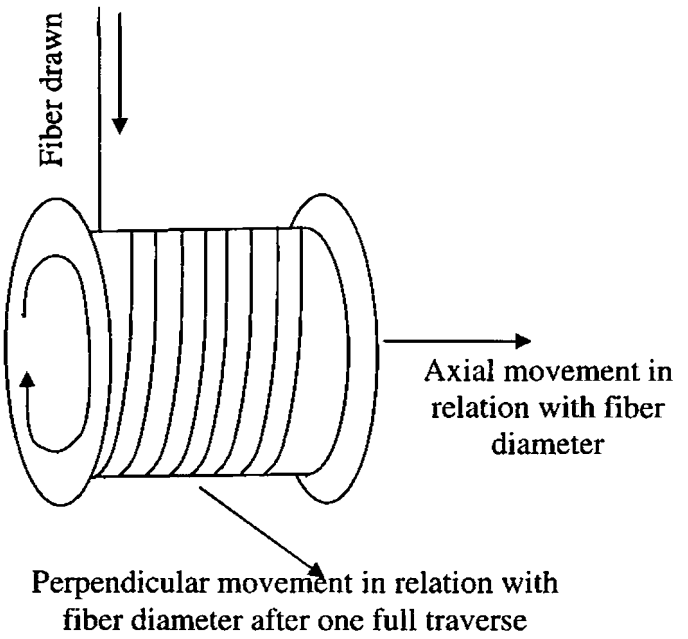
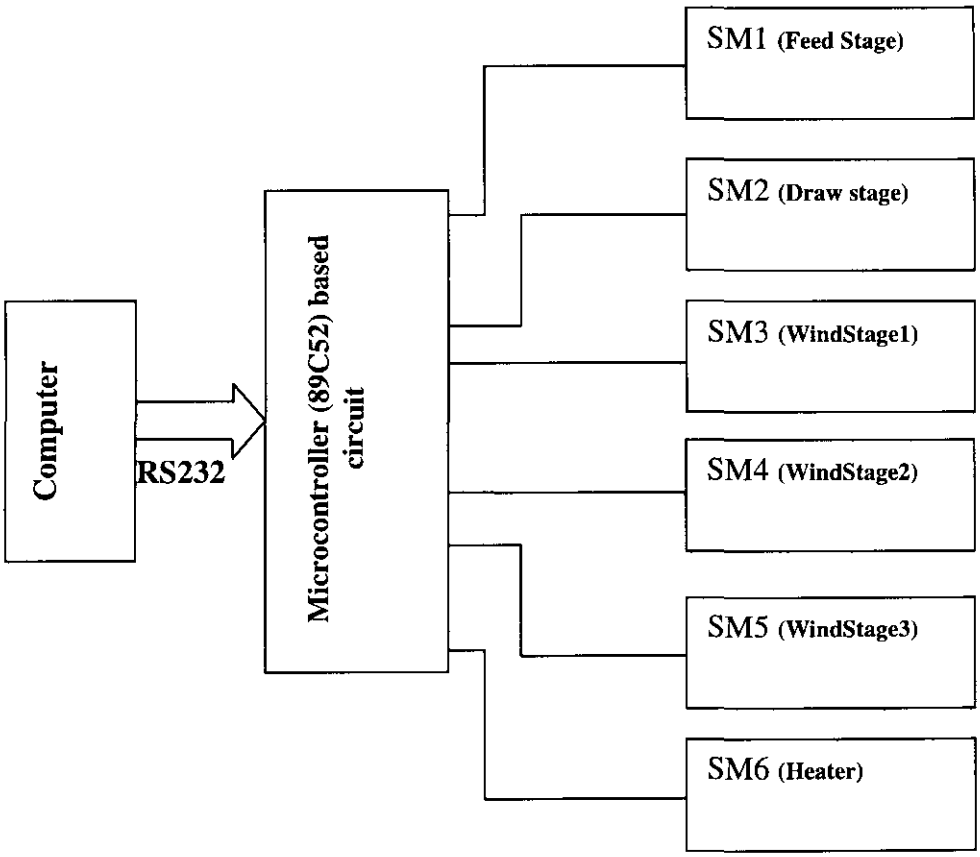


Figure 2.10 Schematic of the fiber winding spool

2.6 Electronic control unit

The electronic control unit is the most important element in the polymer optical fiber drawing system. It controls the whole fiber drawing system with respect to a set of preprogrammed values stored in its registers. A block diagram representation of the electronic control system which is interfaced to the computer is shown in the figure 2.11.



Fiber 2.11 Block diagram of the electronic controller module

A control software in visual basic acts as the user interface with the computer. The software window is as shown in the figure 2.12

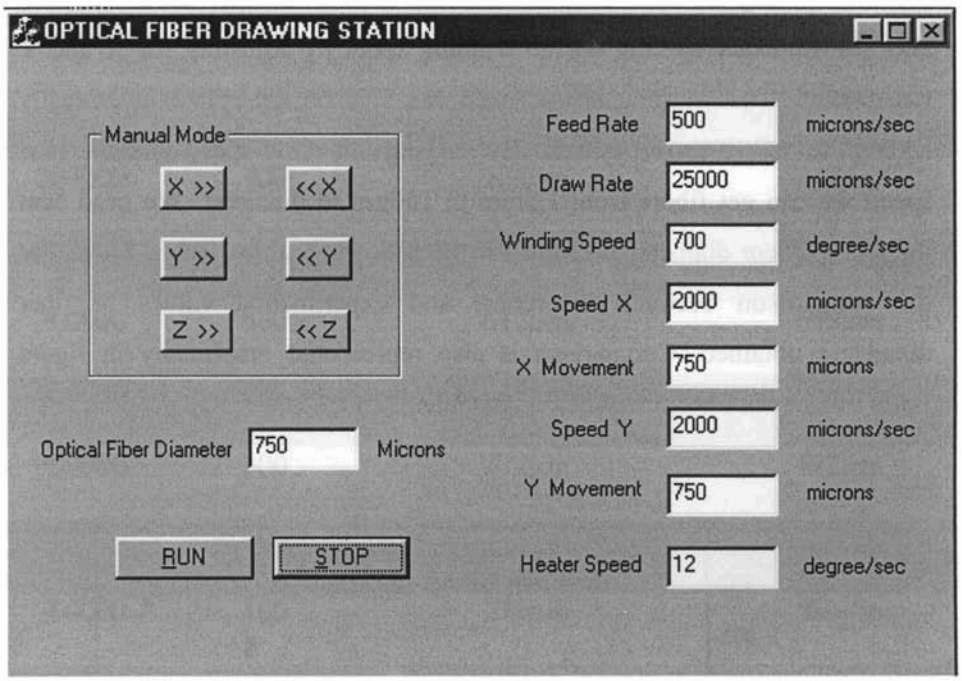


Figure 2.12 Software controller window

Values entered into this window are converted into hexadecimal values and are transferred to the microcontroller based electronic controller via RS232C interface. The microcontroller routes these data to the stepper motors (SM1 through SM6). Rotation sensors attached to the spooling and drawing stages feed back data to the control unit which inturn automatically corrects if it alters from its preset value.

A manual mode is provided to the preform feeder (Z) and to the winding spool (X and Y) to control the system manually if the system gets stuck in between due to some unwanted reasons.

We can specify the optical fiber diameter in the software, which will then allocate the exact spacing on the winding spool by adjusting the X and Y movements. The winding spool speed can also be controlled individually adding flexibility to the system. By varying the draw speed and the feed speed we can get fibers from 1.5mm to 150μm in diameter. We used 5cm long and 1.5cm diameter preform for the following study. Table 2.1 shows the comparison between theoretical and experimental values of fiber diameters obtained. Comparison is also represented graphically in figure 2.13. Values obtained are quite comparable.

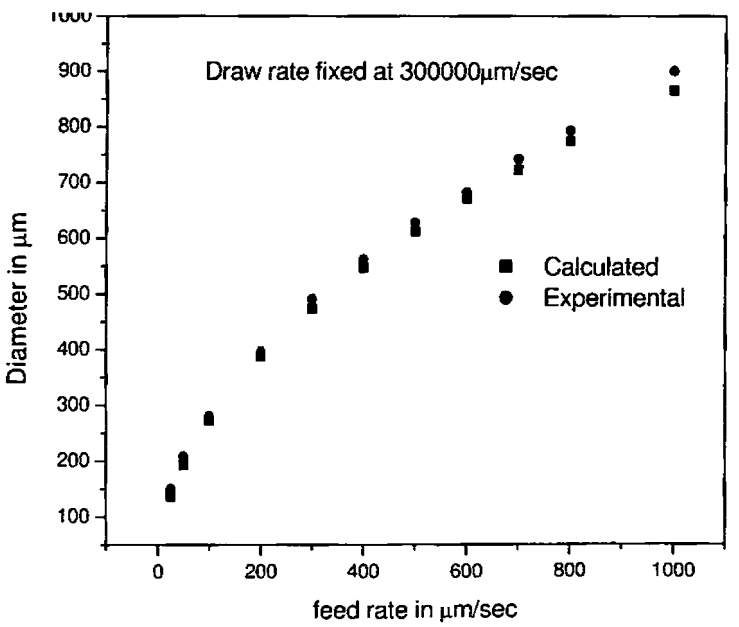


Figure 2.13: Comparison of experimental and calculated values of fiber diameter for various feed rates

µm/sec) rate	Vf (µm/sec) Feed rate	d- fiber diameter (Calculated)	d- fiber diameter (experimental values)
300000	1000	866µm	901µm
300000	800	774µm	793µm
300000	700	723µm	742µm
300000	600	671µm	682µm
300000	500	612µm	628µm
300000	400	547µm	562µm
300000	300	474µm	491µm
300000	200	387µm	396µm
300000	100	273µm	280µm
300000	50	193µm	208µm
300000	25	136µm	150µm

Table 2.1 Data showing the theoretical and experimental diameters obtained for various draw and feed rates

2.7 Conclusion:

Polymer optical fiber drawing station for drawing polymer optical fibers was indigenously designed and fabricated. The temperature profiling of the furnace was carried out and was found to be comparable with the ideal temperature profile. Polymer optical fibers using this facility were fabricated and were found to have satisfactory properties comparable with the theoretical values.

References:

1. Hari Singh Nalwa, " Polymer Optical Fibers ,American Scientific Publishers,2004
2. G.P.Agrawal, "Fiber-optic Communication Systems",2nd ed. Wiely-Interscience, New York, 1997
3. Hari Singh Nalwa, " Polymer Optical Fibers ,American Scientific Publishers,2004
4. Mitsubishi Rayon Co., U.K. Patent 1, 431, 147, 1974.
5. Mitsubishi Rayon Co., U.K. Patent 1, 449, 950, 1974.
6. C. Emsile, *J.Mater.Sci.*23, 2281(1998).
7. F.P Kapron, D.B. Keck and R.D.Maurer, *Appl.Phys.Lett.*17, 423(1970).
8. T. Kaino, K. Jingunji and S.Nara, *Appl.Phys.Lett.*42, 567(1983).
9. Y. Ohtsuka and Y. Hatanaka. *Appl.Phys.Lett.*29, 735(1976).
10. Nippon Selfoc Co., U.S. Patent 3, 995, 015, 1976.
11. Y. Ohtsuka, T. Senga and H. Yasuda. *Appl.Phys.Lett.*25, 659 (1974).
12. Y. Ohtsuka, T. Sugano and Y. Terao. *Appl.Opt.*20, 2319 (1981).
13. Y. Ohtsuka and I. Nakamoto. *Appl.Phys.Lett.*29, 559 (1976).
14. Y. Ohtsuka, Y. Koike and H. Yamasaki. *Appl.Opt.*20, 280 (1981).
15. Y. Koike, H. Hatanaka and Y. Ohtsuka, *Appl.Opt.*23, 1779 (1984).
16. Y. Ohtsuka and Y. Koike. *Appl.Opt.*24, 4316 (1985).
17. Y. Koike, Y. Takezawa and Y. Ohtsuka, *Appl.Opt.*27, 486 (1988).
18. Y. Koike, N. Tanio, E. Nihei and Y. Ohtsuka, *Polym.Eng.Sci.*29, 1200 (1989).
19. Y. Koike. *Polymer.*32, 1737(1991)
20. Y. Koike, E. Nihei (Kawasaki), U.S. Patent 5, 253, 323, 1993.
21. T. Ishugure, E. Nihei and Y. Koike, *Appl.Opt.*33, 4261 (1994).
22. Y. Koike, T. Ishugure and E. Nihei *J. Lightwave Technol.*13, 1475 (1995).
23. T. Ishugure, A. Horibe, E. Nihei and Y. Koike *J. Lightwave Technol.*13, 1686 (1995).
24. T. Ishugure, E. Nihei and Y. Koike, *Appl.Opt.*35, 2048 (1996).
25. D. Bosc and C. Tonien, "Fully polymer single-mode optical fiber". *IEEE Photon.Technol.Lett.*,4, 749-751 (1992).
26. M.G Kuzyk,U.K.Peak,and C.W.Dirk,"Guest-host polymer fiber for nonlinear optics", *Appl.Phys.Lett.*59, 902 (1991).
27. Gang Ding Peng Chu, P.K. Zhengjun Xiong Whitbread, T.W. Chaplin, R.P. "Dye-doped step-index polymer optical fiber for broadband optical amplification". *J. Lightwave Technol.*14, 2215 (1996).
28. T. Ishugure, M. Satoh, O Takanashi, E. Nihei, T. Nyu, S. Yamakashi and Y. Koike, *J. Lightwave Technol.*15, 2095 (1997).
29. J. Senior "Optical Fiber Communications", Prentice-Hall, London, 1985.
30. Toray Industry, Japanese Patent Publication 07-270629, 1995.
31. Mitsubishi Rayon Co., U.S. Patent 4, 822, 122, 1989.
32. Mitsubishi Rayon Co., U.S. Patent 4, 852, 982, 1989.
33. Mitsubishi Rayon Co., Japanese Patent.2-16505, 1990.

34. B.C. Ho , J.H Chen, W.C Chen, Y.H .Chang, S.Y. Yang, J.J .Chen and T.W. Tseng, *Polym J*, **27**,310(1995)
35. W.C Chen, J.H Chen, S.Y. Yang, J.Y .Cheng, Y.H .Chang and B.C. Ho, *J. Appl. Polym.sci*, **60**,1379(1996)
36. B.C. Ho, J.H Chen, S.Y. Yang, and Y.H .Chang, U.S. Patent 5, 555, 525, 1996.
37. C.W.Park, B.S Lee, J.K Walker and WI Choi.*Ind.Eng.Chem.Res.* 39,79(2000)
38. C.W.Park and J.K Walker U.S. Patent 6, 267, 915, 2001.
39. Kricheldorf R Kricheldorf, Hans R Kricheldorf "Handbook Of Polymer Synthesis: Second Edition" Marcel Dekker-New York, 2005

If you haven't measured it you haven't made it -- Wayne Knox

Characterization of dye doped and undoped polymer optical fiber

This chapter deals with the characterisation of both dye doped and undoped polymer optical fibers. Fluorescence studies were carried out on dye doped polymer optical fibers. Attenuation studies due to micro bends and sensitivity to temperature were also carried out for smart sensor applications.

3.1 Introduction

Polymer optical fibers (POF) have the potential to be used in optical logic-devices such as optical switches and amplifiers because of fiber waveguide geometry, which is compatible with silica glass fiber; low production cost and wide range of available materials that can be incorporated into the fiber core [1-5]. Properties like attenuation due to microbends, sensitivity to temperature and humidity of POFs can be effectively utilized for sensor applications [6-10]. Though these properties are not suitable for optical communication systems, these drawbacks are actually a boon for fiber optic sensors. For the smart sensor which will be discussed in chapter 5, attenuation due to microbends and temperature sensitivity are important parameters. The first part of this chapter deals with the characterisation of undoped polymer optical fiber for this specific application.

The length dependent optical attenuation in dye doped polymer optical fibers is an important parameter of interest. There are different techniques for measuring the propagation losses in fiber structures. Usually the propagation losses in fibers and planar waveguide structures are measured by the cut-back technique [11, 12] which consists in comparing the transmittance of several fibers with different lengths at a specific wavelength, or extrapolating the loss from a bulk measurement. The disadvantage of the cut-back technique is that it is a destructive method. Bulk measurements involve a broadband light source incident on a fixed length of material and a spectrometer to read the transmitted intensity. As an alternative to the above mentioned technique, a non-destructive side illumination fluorescence technique for measuring the optical attenuation in dye doped fibers has been developed by Kruhlak et al [13, 14]. Geetha et al have carried out similar studies in planar wave guides [15].

The second part of this chapter describes the use of this technique to characterize the loss mechanisms in Rhodamine 6G dye doped polymer optical fibers. This measurement technique requires a pump source to illuminate the fiber from the side. The fluorescence collected from one end of the fiber is used to characterize the attenuation mechanisms in the fiber.

3.2 Microbending and temperature characteristics of undoped POF for smart sensing applications

3.2.1 Microbending:

Radiative losses will occur when an optical fiber undergoes a bend of finite radius of curvature. Microscopic meandering of the fiber core axis, known as microbending, can be generated at any stages during manufacturing process or during fiber optic cable installation purposefully for sensor applications [16]. Usually microbends occur due to environmental effects, particularly variations causing differential expansion or concentration. Microbends are repetitive small scale fluctuations in the radius of curvature of the fiber axis, as is illustrated in the figure 3.1

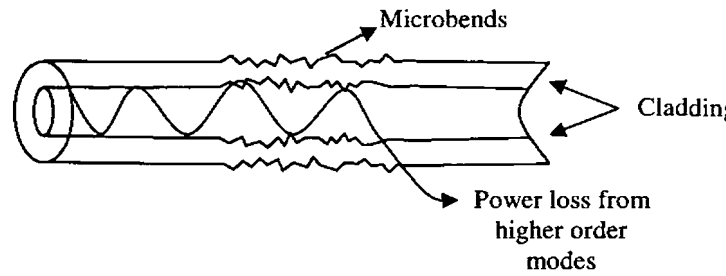


Figure 3.1 Small scale fluctuations in the radius of curvature of the fiber axis lead to microbending losses.

Microbending introduces slight surface imperfections which can cause mode coupling between adjacent modes, which in turn create radiative loss which is dependent on the amount of applied fiber deformation, the length of the fiber, and the exact distribution of power among different modes.

Radiation losses caused by microbends in optical fibers can be utilized for sensor applications to study stress, strain, and temperature by embedding optical fibers in the materials under study. When we compare the loss effects due to microbending in silica and polymer optical fibers, polymer has more microbend induced attenuation due to its material property. Studies were carried out on polymer optical fiber for its sensitivity to mechanically induced microbends for application as smart sensors.

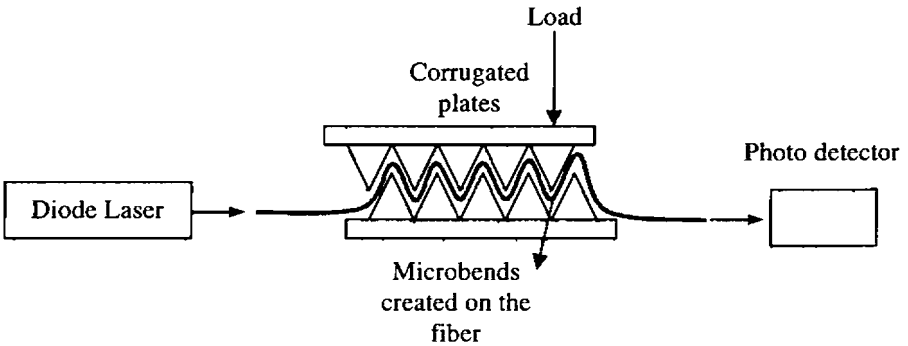


Figure3. 2 Experimental setup: making microbends on the fiber

The experimental setup for making microbends on the fiber is as shown in the figure3.2. An undoped polymer optical fiber was placed in between two corrugated plates (of 2.5mm pitch) and a load is applied on it. The power which is coupled through the fiber changes in accordance with the amount of load placed on the corrugated plates. A photodiode is used to monitor the intensity of the light output from the fiber. The experiment was repeated with glass fibers in order to study the difference in sensitivity. The graph thus

obtained is as shown in the figure 3.3.As can be clearly seen from the graph there is a definite increase in sensitivity for polymer optical fibers for microbend induced attenuation than for glass fibers. This accounts for its advantage over glass fibers for smart sensor applications.

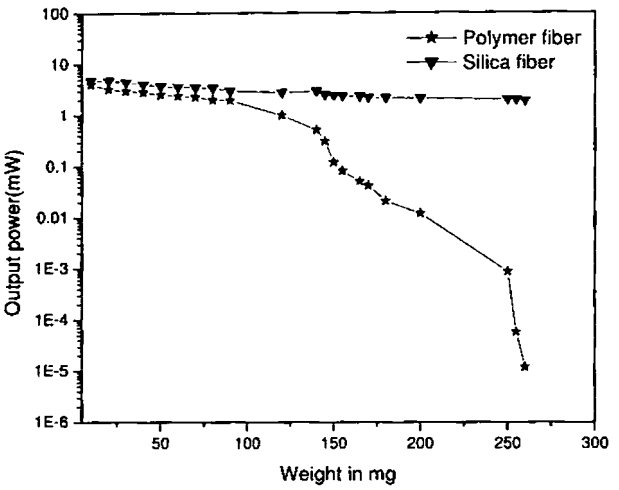


Figure 3.3 Graph showing the sensitivity of both glass and polymer optical fibers to applied weight

3.2.2 Temperature:

Polymer optical fibers are very sensitive to temperature variations. This property is made use in many temperature sensing applications. The power carried out by bound rays (bound ray power) is essentially confined to the fiber core and for a given fiber excitation, this power depends on the core-cladding refractive index difference. Any variation of this parameter along the fiber causes power coupling between bound and leaky rays which results in a change of the propagating bound ray power.

In the present studies, a POF is used with only core and air acts as the cladding. As temperature increases, refractive index of the POF decreases

thereby reducing the local numerical aperture of the fiber. This causes the leaking of higher order modes out of the fiber which results in the decrease of transmitted intensity of laser light through the fiber.

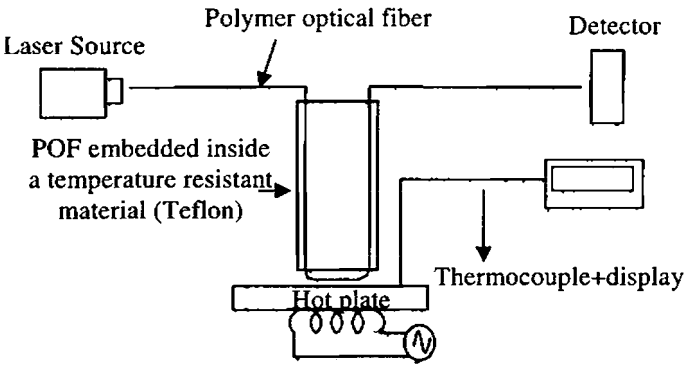


Figure 3.4 Case1: Experimental setup to study the temperature sensitivity of POF using a hotplate

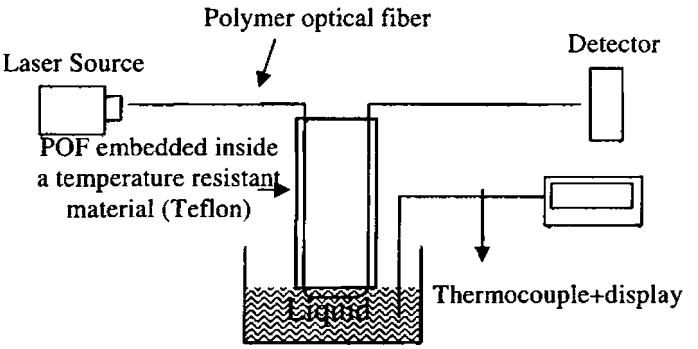


Figure 3.4 Case 2: Experimental setup to study the temperature sensitivity of POF using a water bath

The experimental setup consists of a laser source, a few centimeters of a bare core polymer optical fiber as the sensing element and a detector. The experiment was conducted in two different ways (figure 3.4). In the first case

gradually and the corresponding change in the transmitted output power of the fiber is noted. In the second case the POF sensor head is immersed in a small beaker of water and the temperature of the water is increased and the corresponding change in the output intensity is noted. In both these cases temperature measurement is carried out using a thermocouple to standardize the set up. The polymer fiber core material is polymethylmethacrylate (PMMA).The fiber used for our investigation is drawn from our polymer fiber drawing station and it has a diameter of 300micro meters. We used a teflon mould for holding the POF in such a way that only 5mm of the sensor head is exposed to the sample. The refractive index of the PMMA fiber core is 1.49.

The results obtained on the basis of the experimental data are as follows. Fig 3.5a shows the plot of the variation in output power as a function of the temperature of the object. The response is linear within the range of 35^0C-130^0C. Slope of the calibration graph (fig3.5a) gives the sensitivity of the sensor and is found to be $0.07\,\mu$ w/^{0}C. Fig 3.5b shows the plot of the variation in output power as a function of the temperature of water

We can see that the polymer fiber has a linear response from 35^oC to 130^oC for the measurements made on the hot plate while measurements made in water shows a temperature sensitivity only upto 45^oC.

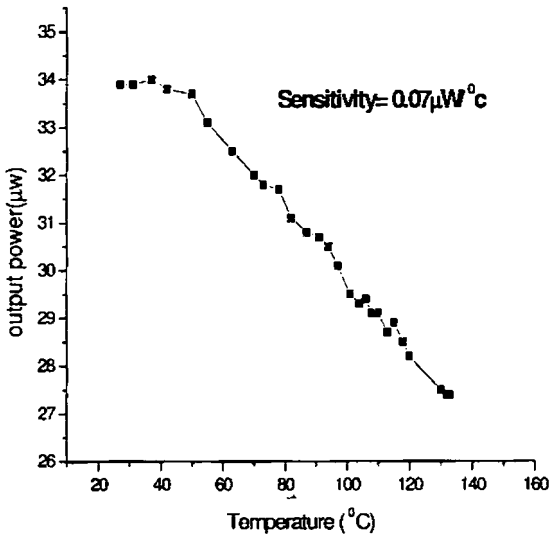


Figure 3.5a: Variation in output power with temperature of the object(hotplate)

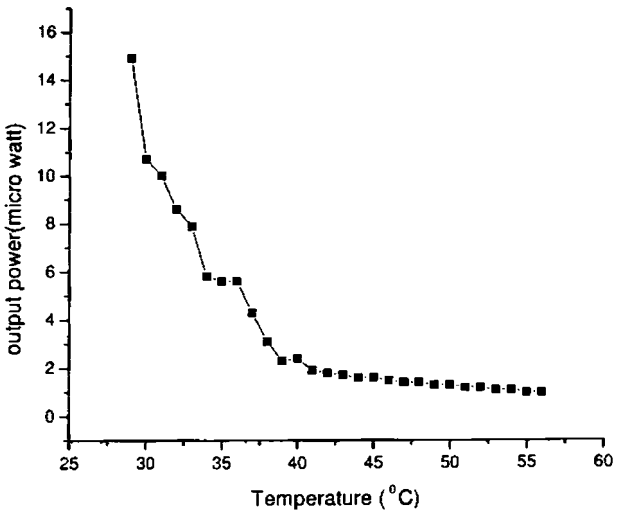


Figure 3.5b: Variation in output power with temperature of water.

3.3 Characterization of Rhodamine 6G doped polymer optical fiber

The length dependent tuning of the fluorescence spectra of a dye doped polymer optical fiber is described in this section. The fiber is pumped sideways and the fluorescence is measured from one of the ends. The excitation of a finite length of dye doped fiber is done by a diode pumped solid state laser at a wavelength of $532nm$. The fluorescence emission is measured from various positions of the fiber starting from a position closer to the pumping region and then progressing towards the other end of the fiber. We observe that the optical loss coefficients for shorter and longer distances of propagation through the dye doped fiber are different. At longer distances of propagation a decrease in optical loss coefficient is observed. The fluorescence peaks exhibit a red shift of 12nm from 589nm to 610nm as the point of illumination progresses away from the detector end. This is attributed to the self absorption and re-emission of the laser dye in the fiber [13-14].

3.4 Side illumination fluorescence studies on dye doped fiber

The total length of the fiber used for the loss measurements is 8cm with its ends cut and polished by usual techniques. To select the pump beam for exciting the dye doped fiber, we recorded the absorption spectrum (figure3.6) of the bulk sample using a spectrophotometer (JASCO UV/VIS/NIR V-570). The dye has a strong absorption at 530nm.

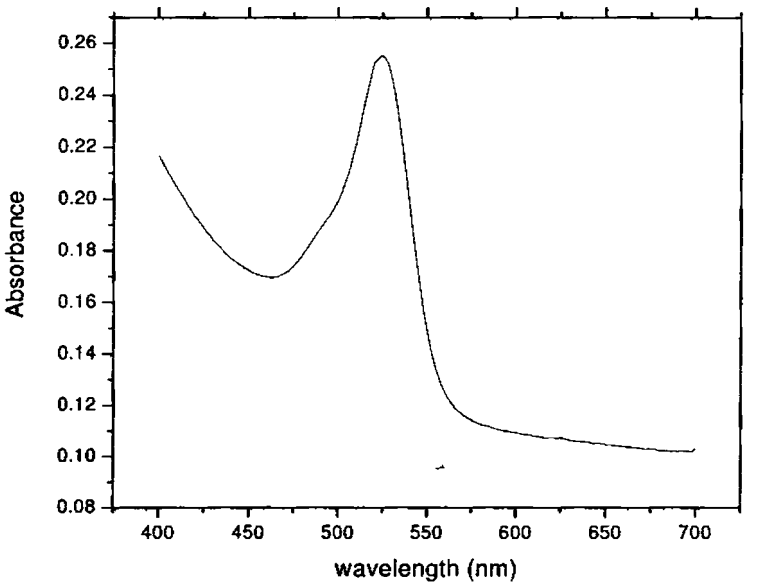


Figure 3.6: Absorption spectrum of Rh6G doped PMMA bulk sample

Radiation at 532 nm (beam spot size 1.5 mm) from a diode pumped solid state (DPSS) laser *(*Nd:YVO_4*)* is used as the pump source. A schematic diagram of the experimental set-up is shown in figure3.7.

The fiber is mounted normally on a translation stage with respect to the incident radiation. The side illumination of the dye doped fiber generates fluorescence emission. Light emission from one end of the fiber is collected by an optical fibre which is coupled to a monochromator–photomultiplier tube assembly coupled with a lock-in amplifier (Stanford Research Systems SR830) for signal analysis.

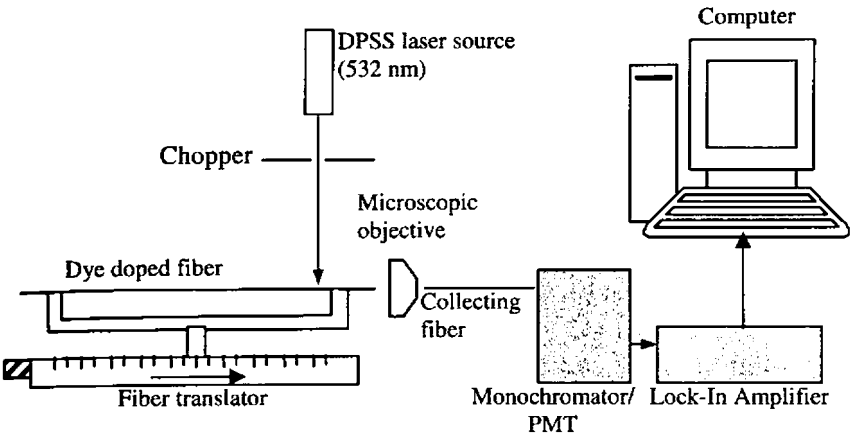


Figure 3.7: Experimental setup to record the fluorescence emission from the fiber end. Pumping is done transversely.

To measure the transmitted fluorescence as a function of propagation distance through the fiber, the illumination point on the fiber is varied by translating the fiber horizontally across the laser source. The direction of translation is indicated by the arrow mark in figure 3.7. At each point of illumination, the fluorescence spectrum is recorded. The experiment is repeated for three different pump powers. It is ensured that the sample is not bleached even at the highest pump power.

Figure 3.8 shows the spectra of transmitted fluorescence light measured as a function of the propagation distance through the fiber. As the propagation distance increases, the magnitude of the output intensity decreases due to loss mechanisms such as absorption and scattering of fluorescence emission. In addition, there is a red-shift for the peak fluorescence emission as the illumination distance from the detector edge of the fiber is increased. A similar red-shift in the fluorescence emission from side illuminated dye doped fibre has also been observed by other workers [8, 9].

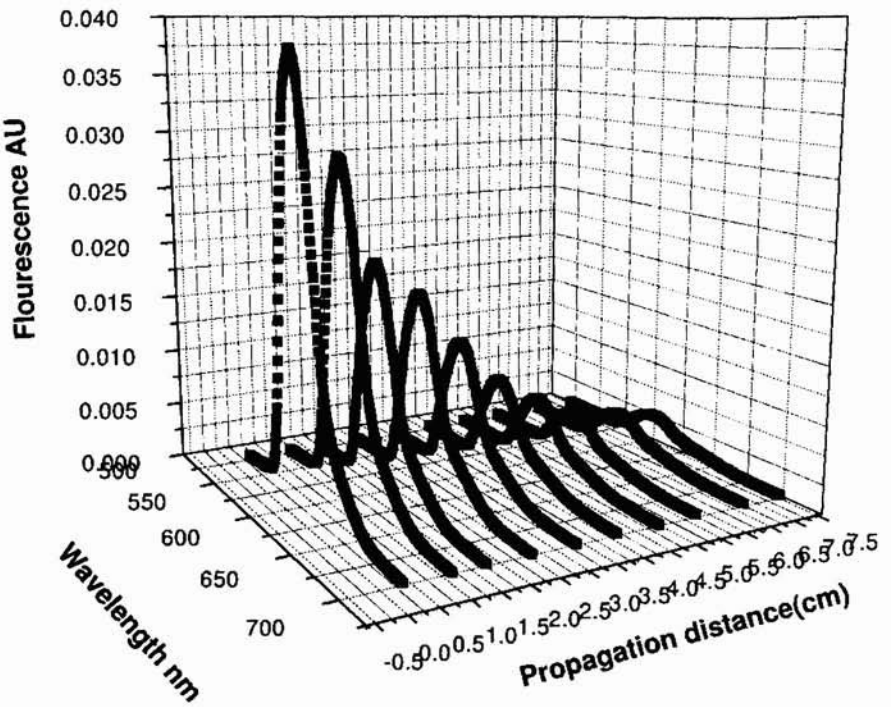


Figure 3.8: Transmitted fluorescence spectra measured as a function of the propagation distance

The red-shift of the fluorescence signal is produced by the self-absorption of the dye Rhodamine 6G caused by the overlap of the absorption spectra with the fluorescence spectra over a certain wavelength region [11].

As the fluorescence light is guided through the dye doped fiber, the effective pathlength is increased resulting in self-absorption and re-emission causing a red-shift in the observed spectrum. The farther the point of illumination is from the observation end, the larger is the effective path length.

This results in increased interaction between the dye molecules and beam propagating through the fiber causing enhanced fluorescence emission in comparison to that expected from Beer-Lambert's law. This results in an increased self-absorption of the fluorescence thereby shifting the emitted fluorescence peak towards the longer wavelength region. Figure 3.9 shows the variation of the fluorescence peak wavelength as a function of propagation distance through the fiber.

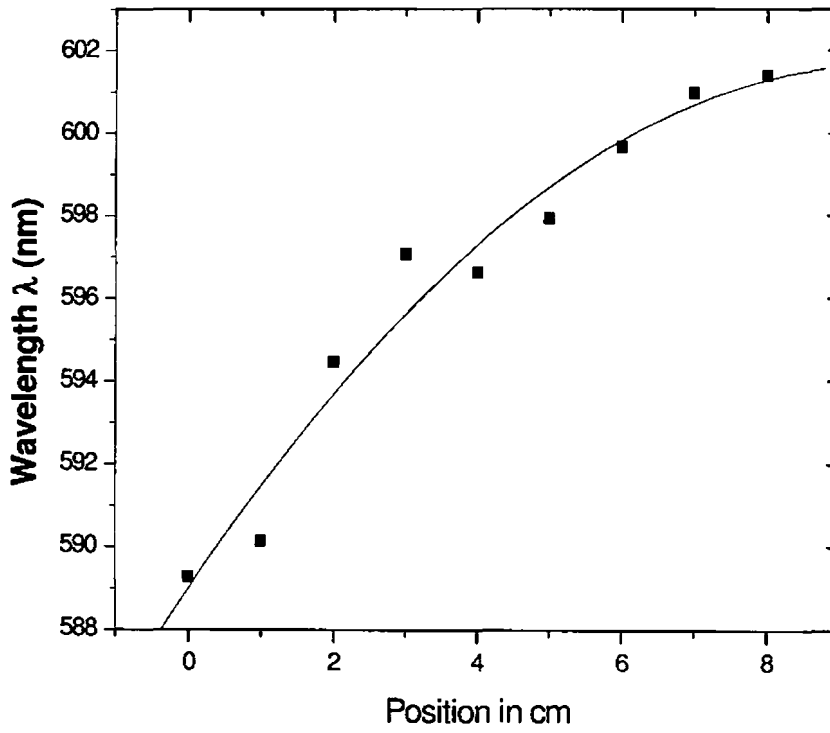


Figure 3.9: The variation of the fluorescence peak with propagation distance from collecting end of the fiber

For shorter propagation distances in the fiber, the red-shift shows a linear behaviour, whereas at longer distances, the shift tends to exhibit saturation behaviour. This mechanism is similar to the concentration dependent red-shift which is observed in dye solution [12]. The fluorescence collected from the dye doped fiber has a spectral width of about 40 nm. The transmitted fluorescence is measured as a function of the propagation distance so as to characterize the attenuation in the fiber. From Beer–Lambert's law for linear optical attenuation in a medium, $I(\lambda, z) = I_0(\lambda) \exp(-\alpha(\lambda) z)$ where $I(\lambda, z)$ and $I_0(\lambda)$ represent the intensity of the transmitted light at wavelength λ at propagation distances z and z=0(incident intensity) respectively and $\alpha(\lambda)$ is the linear attenuation coefficient. Figure 3.10 shows plots of the natural logarithm of the transmitted fluorescence intensity versus the propagation distance corresponding to various emission wavelengths.

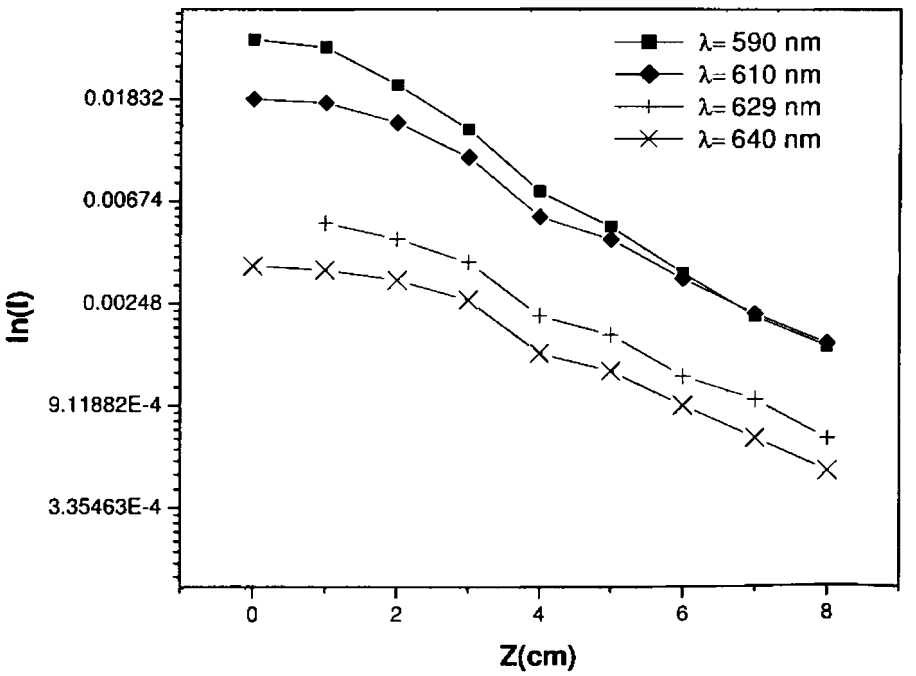


Figure 3.10: Plot of $\ln(I)$ versus z (z is the propagation distance through the fiber in cm)

An interesting observation is the nonlinear behaviour of these plots, which suggests that the loss coefficient is not a constant for the total length of propagation through the fiber. The nonlinear plot of ln I versus z can be fitted to a minimum number of straight lines (the method is known as peeling the curve) [15] which will provide the corresponding loss coefficients. Figure 3.11 show the application of this method to the semilog plot for emission at $\lambda = 610nm.$. For the lowest pump power, the plot can be fitted to a single straight line (figure 3.11 a), whereas for higher powers it has to be fitted to two straight lines (figure 3.11 b, figure 3.11 c).

Above results clearly suggest that as light propagates through the fiber, there arise some mechanisms which tend to alter the optical attenuation inside the fiber especially for larger propagation distances. It is observed that the spatial dependence of attenuation decreases for longer distances of propagation. One of the possible mechanisms for this behaviour is re-absorption of fluorescence light on the shorter wavelength side of the absorption spectrum and subsequent emission in the longer wavelength region. This re-emission in the longer wavelength side generates increased emission in the longer wavelength region resulting in enhanced intensity. Similar observations are made in polymer wave guide structures also [15]. The increase in the path length also causes different attenuation rates for various emission intensities. This explains the observation of difference in attenuation values with respect to distance for a given power. This also supports the observation that z dependent attenuation effect is more prominent in the longer wavelength region of the fluorescence spectrum. As the pump power is increased, the intensity of the fluorescence emission is also enhanced, which in turn causes increased probability of the re-absorption–emission proeess. This is exhibited as two linear parts in the ln I–z plots corresponding to higher pump powers.

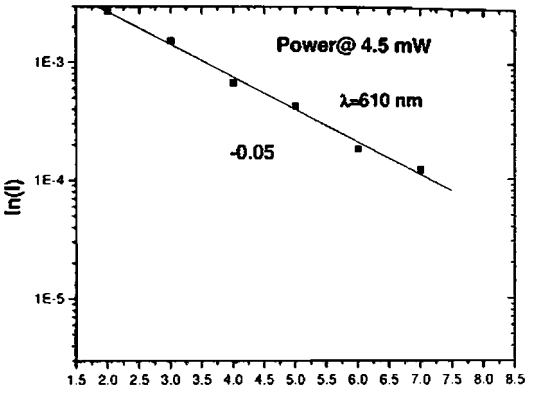


Figure 3.11 a

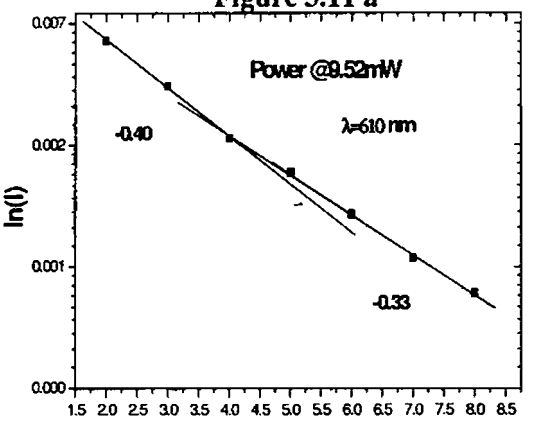


Figure 3.11 b

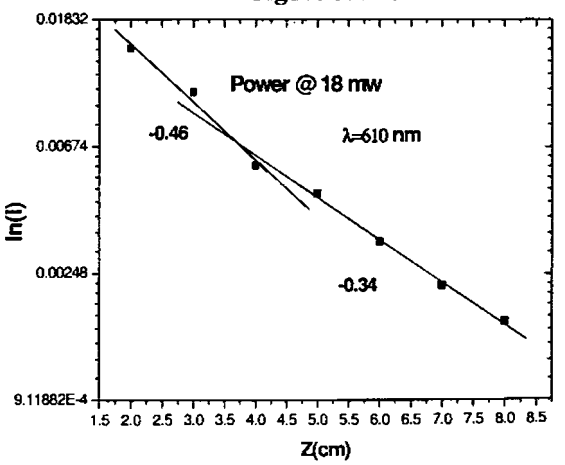


Figure 3.11 c

Figure 3.11 a, b, c): The 'peeling the curve' method applied to $\ln(I)$ versus z plot at λ = 610 nm at different pump powers. The solid lines represent the linear fits to the data

3.5 Homogeneity in fiber diameter and uniformity in dye concentration

The homogeneity of the fiber diameter as well as dye concentration are important parameters which has direct effects on the fiber fluorescence. An inhomogeneous dye doped fiber will have an inhomogeneous fluorescence distribution which directly affects the overall gain of the fiber. The homogeneity of the fiber diameter can be characterized by calculating the correlation length of the fiber as described below. If the fiber is not perfectly homogeneous in diameter or in dye concentration, the coefficient that describes the fluorescence source along the coordinate z is

$$C(\lambda_m, \lambda, z) = C(\lambda_m, \lambda)S(z) \quad \text{----------} \quad [3.1]$$

where: λ_m is the wavelength of the beam, $C(\lambda_m, \lambda)$ is the fluorescence yield

The deviation of S (z) from unity gives a measure of the inhomogeneity of the fiber [8, 9]. We can quantify the inhomogeneities by using the autocorrelation function, which measures the similarities between the intensity generated at one position in the fiber, z, and the intensity generated at a neighboring point, $z+\zeta$, over a length z of fiber and is defined for a fixed wavelength λ_0 as

$$\langle I(0)I(\zeta)\rangle = \lim_{z\to\infty}\left(\frac{1}{Z}\right)\int_0^z I(z)I(z+\zeta)dz, \quad \text{----------} \quad [3.2]$$

The form of the autocorrelation function is,

$$\langle \delta I(z)\delta I(z+\zeta)\rangle_{Norm} = \frac{\sum_{j=0}^{N} \delta I_j \delta I_{jk}}{\sum_{j=0}^{N} \delta I_j{}^2} \qquad \text{----------} \quad [3.3]$$

The autocorrelation function is often modeled as an exponential or Gaussian function of ζ .By fitting the autocorrelation function to one of these models, one determines the length scale of fluctuations and is called its correlation length. Mathematically, the correlation length (ζ_c) is the value of ζ when the correlation function is at 1/e of its magnitude. The correlation length is thus related to the homogeneity of the fiber and can be used to characterize the fiber quality.

Figure 3.12a shows the plot of fluorescence emission as a function of the pump position with respect to the fiber end from where emitted light is collected. As is clearly seen in the figure, fluorescence intensity fluctuates at shorter path lengths. Also at shorter lengths it is observed that there is slight deviation from the theoretical values in the florescence intensity. The dye doped fiber which is used for our studies does not have a cladding. Hence a considerable amount of scattered fluorescence intensity will be emanating from the sides of the fiber. This is collected by the lens which is used for coupling light to the monochromator which causes the intensity fluctuations at shorter lengths. Theoretical fit according to Beer–Lambert's law is also shown in the figure. To understand the inhomogeneity, autocorrelated function was evaluated for various space lags (figure 3.12 b). The 1/e point in the plot gives the value of space lag as 1.56mm which is the measure of the homogeneity of the fiber. Larger the correlation length better is the homogeneity of the fiber.

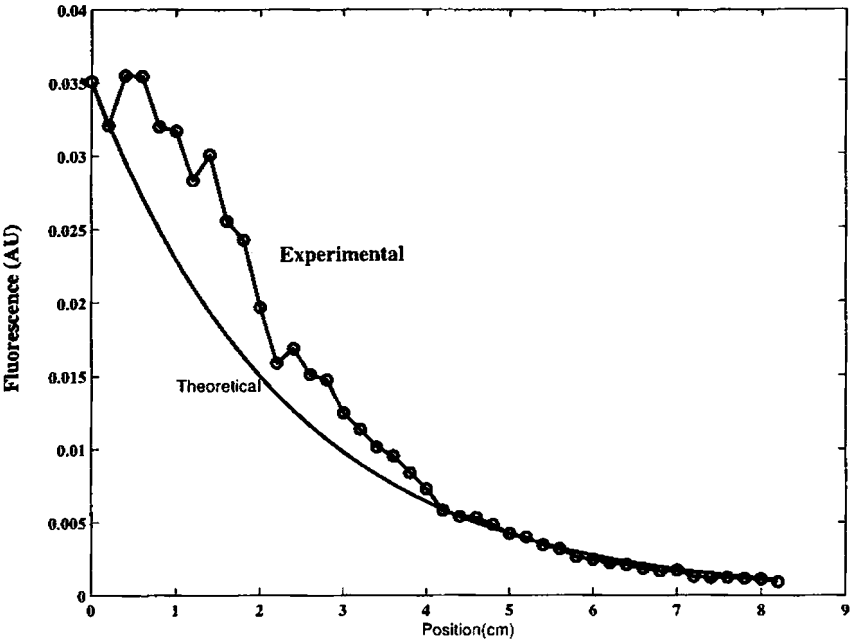


Figure 3.12 a: Fluorescence emission as a function of the pump position

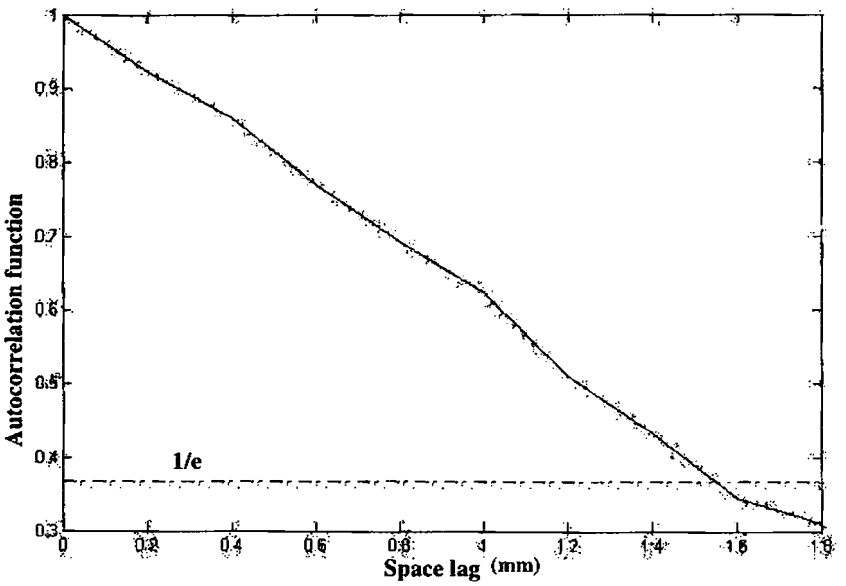
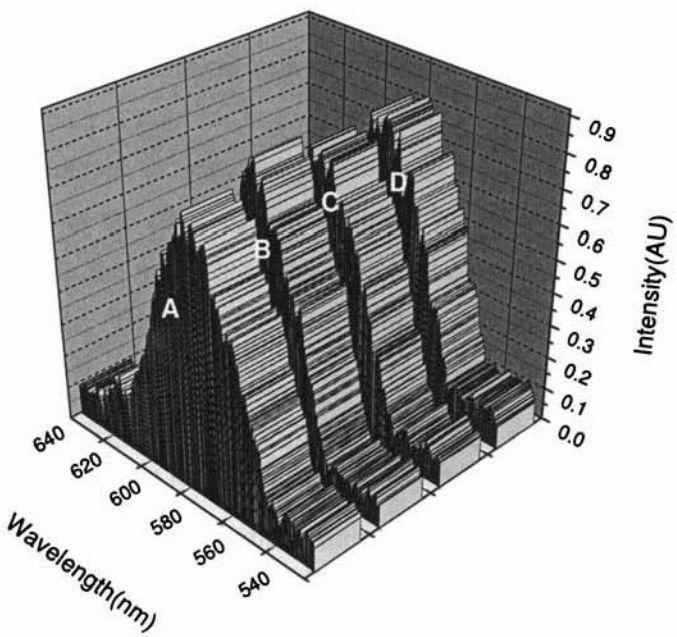


Figure 3.12 b: Autocorrelated function evaluated for various space lags

Diffraction technique can also be used to test the homogeneity in diameter of the fiber. Variation in diameter is found to be less than 1% so that the effect of inhomogeneity in loss mechanism can be neglected.

The uniformity in dye concentration has an important role to play in amplification. If the dye concentration is not uniform or if there are aggregate formation of the dye, it will quench the fluorescence propagating through the fiber. This will adversely affect the overall gain of the fiber amplifier. In order to study the uniformity in dye concentration through out the length of the fiber a fiber of total length of 20 cm was taken. The fiber was then cut into four equal lengths of 5 cm each and the fluorescence spectrum was charted for each of these individual lengths. The graph shown in figure shows the fluorescence intensity of the four individual fibers cut from the same fiber sample.



From the plots we can see that the four samples show the same fluorescence behaviour which implies that the dye is uniformly doped in the fiber for a total length of 20 cm.

3.6 Conclusion

Characterisation of undoped polymer optical fibers was carried out for its potential application in smart sensing. The sensitivity to temperature and microbends is found to be high in comparison with glass fibers.

Using the side illumination technique, position-dependent tuning of fluorescence light emitted from a Rhodamine 6G doped polymer optical fiber is observed. The fluorescence data collected from the fiber is used to characterize the loss mechanisms of the fiber. It has been observed that at longer wavelengths, there is a lowering of attenuation towards larger distances of propagation in the fiber. The mechanism for such position dependent loss parameter is attributed to re-absorption and re-emission process taking place along the length of the fiber. This suggests that appropriate design of the fiber will lead to a gain on the longer wavelength side. It is also found that the drawn fiber is having a good correlation length which confirms its homogeneity. The doping of dye in the fiber was found to be uniform from the fluorescence studies.

References

1. A Costela ,F Florido ,I G Moreno , R Duchowicz ,F AGuerri,J M Figuera and R Sastre "Solid-state dye laser based on copolymers of 2-hydroxyethyl methacrylate and methyl methacrylate doped with rhodamine 6G" Appl. Phys. B 60, 383–389(1995).
2. G D Peng,P L.Chu,Z Xiong,T W.Whitbread,and R P.Chaplin " Dye-Doped Step Index Polymer Optical Fiber for Broadband Optical amplification" Journal of Lightwave Technology 14 ,2215-2223 (1996).
3. K Kurki,T Kobayashi,N Imai,T Tamura,S Nishihara,Y Nishizawa,A Tagaya,and Y Koike "High –efficiency organic dye-doped polymer optical fiber lasers" Appl.Phys.Lett 77, 331-333(2000).
4. K Kurki,TKobayashi,N Imai,T Tamura,Y Koike and Y Okamoto " Organic Dye-Doped Polymer Optical Fiber Lasers" Polym.Adv.Technol 11, 612-616 (2000).
5. H Liang,Z Zengchang L J Xu,B Chen,H Zhao, Qijin Zhang and H Ming "Fabrication and Amplification of Rhodamine B- Doped Step-index Polymer Optical fiber" J.Appl.Polym.Sci 93, 681-685 (2004).
6. B. Chiron, "Highly efficient plastic optical fluorescent fibers and sensors" in *SPIE Proc. On Plastic Optical fibers*, Boston MA, vol, 1592, pp86-95,1991
7. M. Laguesse and M. Bourdinaud, "Characterisation of Fluorescent plastic optical Fibers for X-ray beam detection, "in *SPIE Proc. On Plastic Optical fibers*, Boston MA, vol, 1592, pp96-107,1992
8. S. Muto, H Sato and T Hosaka, "Fluorescent-POF humidity sensor; theoretical and experimental analysis", in *Proc. Third International Conference on Plastic Optical Fibers and Applications –POF'94,* Yokohama(Japan) pp.46-48,1994
9. S. Hadjiloucas,L S KAratzas, D A Keating and M J Usher, " Plastic optical fibers for measuring leaf water potential" , *Proc. Third International Conference on Plastic Optical Fibers and Applications –POF'94,* Yokohama(Japan) pp.64-68,1994
10. J.D Weiss, " The pressure approach to fiber liquid level sensors;" In *Proc. Fourth International Conference on Plastic Optical Fibers and Applications –POF'95,* Boston MA, vol, pp 167-170,1995
11. D W Garvey, Zimmerman K, Young P, Tostenrude J, Townsend J S, Zhou Z, Lobel M, Dayton M, Wittorf R and Kuzyk M G Single-mode nonlinear optical polymer fibers *J. Opt. Soc. Am. B* **13** ,2017–2023 (1996).
12. Kaino T 2000Waveguide fabrication using organic nonlinear optical materials *J. Opt. A: Pure Appl. Opt.* **2** R1–7
13. Kruhlak R J and Kuzyk M G " Side-illumination fluorescence spectroscopy I—principles" *J. Opt. Soc. Am. B* **16**, 1749–1755 (1999).
14. Kruhlak R J and Kuzyk M G " Side-illumination fluorescence spectroscopy II—applications to squaraine dye-doped polymer optical fiber" *J. Opt. Soc. Am. B* **16**, 1756–1767 (1999)
15. K Geetha, M Rajesh, V P N Nampoori, C P G Vallabhan and P Radhakrishnan "Loss characterization in rhodamine 6G doped polymer film

waveguide by side illumination fluorescence" *J. Opt. A: Pure Appl. Opt.* **6** 379–383 (2004)

16. John M Senior " Optical fiber Communications, Principles and practice" Prentice –Hall of India Private limited, New Delhi, 2004 second edition

Your vision will become clear only when you can look into your own heart. Who looks outside, dreams; who looks inside, awakes -- Carl Jung

Dye doped optical fiber as light amplifier

Two different schemes are employed to characterise the fiber as light amplifier. One is the stripe illumination technique to study the fiber as a gain medium and the other is its performance as an amplifier. We observed a spectral narrowing from 40nm to 5nm when the pump power is increased from 10kW to 50kW in the stripe illumination geometry. A gain of 18dB was obtained in the amplifier configuration. The effects of pump power and dye concentration on the performance of the fiber as an amplifier are also studied.

4.1 Introduction:

Polymer based waveguide structures and devices have been found to be suitable for short distance communications and optical integrated circuits [1-2]. Even though optical fiber networks are mainly focused on silica optical fibers (SOF), polymer optical fiber (POF) based systems are seriously being considered for short distance communication. This is due to the competitiveness of POF in fiber drawing and cost effectiveness with respect to silica fibers [3, 4.]. Although higher loss factor is a major handicap for POF, recently developed techniques for decreasing losses in polymethylmethacrylate based POF have raised much interest in this field [5-9]. Availability of laser sources in the visible region has increased the utilization of POF in data communication over LAN systems [10]. Implementation of optical communication in the visible region demands the development of suitable optical amplifiers working in this region [11-18]. POFs doped with dyes or rare earth elements are potential candidates for this purpose.

Laser dyes, which act as highly efficient media for lasing and amplification, have a wide range of tunability in the visible region [12-16]. For example the range of tunability of laser dyes like Rhodamine B and Rhodamine 6G lies between 570nm to 640nm. The advantage of incorporating laser dyes in solid matrices such as POF is that it is easier and safer to handle them than when they are in liquid form. From recent studies it is found that the dye doped polymer materials have better efficiency, beam quality and superior optical homogeneity when compared to other solid matrices [20].

In this chapter, we will focus on the line narrowing behavior and the amplification characterization of dye doped optical fibers.

4.2 Theory of dye doped polymer fiber amplifier

The polymer optical fiber is doped with an organic laser dye known as Rhodamine 6G for our studies on light amplification. A detailed overview of the organic laser band structure and its kinetics are discussed in chapter 1. As discussed in chapter 1, the laser dye called Rhodamine 6G [21] also has a typical energy level structure as shown in figure 1.2. Each electronic level is a band composed of a continuum of vibrational and rotational levels. The lowest energy absorption is due to the absorption from the electronic singlet ground state S_0 to the first excited singlet S_1. This property of strong absorption of dye usually in the visible region of the spectrum is used to color other substances such as cloth, food, plastics, etc.

There are several procedures to study absorption and emission of light in materials. In one of the methods, the system's density matrix is reduced to rate equations for incoherent light. In other method, rate equations can be extracted from the charge and stability that gives the equations relating the charge-density variations of the levels with pump and the signal power variations. To analyze the optical fiber amplifiers, we can numerically solve the time dependent rate equations to predict the results [22, 23].

4.2.1 Rate equations in dye doped polymer optical fiber amplifiers

It is assumed that this kind of amplifier is a three level system and that the third level decays fast. Due to this fast decay, the time dependent rate

equations in dye-doped optical fiber amplifiers can be expressed as a two level system [24].

$$\frac{\partial N_2(t,z,r)}{\partial t} = \frac{2\pi\sigma_p^a N_1(t,z) I_p(t,z)}{h\nu_p}\eta - \frac{N_2(t,z)}{\tau} - \frac{2\pi\left(\sigma_s^e N_2(t,z) - \sigma_s^a N_1(t,z)\right) I_s(t,z)}{h\nu_s}\eta, \quad \text{---------- (4.1)}$$

$$\frac{\partial I_s(t,z,r)}{\partial z} = 2\pi\left(\sigma_s^e N_2(t,z) - \sigma_s^a N_1(t,z)\right) I_s(t,z)\eta \quad \text{---------- (4.2)}$$

$$\frac{\partial I_p(t,z,r)}{\partial z} = -2\pi\sigma_p^a N_1(t,z) I_p(t,z)\eta, \quad \text{---------- (4.3)}$$

$$N_t = N_1(t,z) + N_2(t,z), \quad \text{---------- (4.4)}$$

Where N_1, N_2, and N_t are the ground, metastable, and total charge densities, respectively. I_p and I_s are the pump and signal intensities, respectively, σ_p^a, σ_s^a and σ_e^s are cross sections of the pump, signal absorbtion, and siginal emission, respectively, h is planks constant, τ is the steady-state life time of the dye in the metastable level, z is the direction of propagation of light in the fiber, η is the overlap integral:

$$\eta = \int_0^{a_0} \theta(r)\bar{\psi}(r) r dr \quad \text{---------- (4.5)}$$

Where $\theta(r)$ and $\bar{\psi}(r)$ are the dye and the pump distributions, respectively, and a_0 is the dopant radius.

In the steady state, the electron density of the second level can be assumed to be constant ($\delta N_2/\delta t = 0$). The time independent signal and pump evolutions can be expressed as ;

$$\frac{\partial I_s(z,r)}{\partial z} = \eta \frac{\left(\frac{\sigma_s^e \sigma_p^a}{h\nu_p}\right) I_p(z) + \frac{\sigma_s^a}{\tau}}{\left(\frac{\sigma_p^a}{h\nu_p}\right) I_p(z) + \left(\frac{\sigma_s^e + \sigma_s^a}{h\nu_s}\right) I_s(z) + \frac{1}{\tau\eta}} \times I_s(z) N_t - k_s I_s(z), \quad \text{---------- (4.6)}$$

$$\frac{\partial I_p(z,r)}{\partial z} = -\eta \frac{\left(\frac{\sigma_s^e \sigma_p^a}{h\nu_s}\right) I_s(z) + \frac{\sigma_p^a}{\tau}}{\left(\frac{\sigma_p^a}{h\nu_p}\right) I_p(z) + \left(\frac{\sigma_s^e + \sigma_s^a}{h\nu_s}\right) I_s(z) + \frac{1}{\tau\eta}} \times I_p(z) N_t - k_p I_p(z), \quad \text{---------- (4.7)}$$

Where ν_s and ν_p are the signal and pump frequencies, respectively and k_s and k_p are the fiber loss at signal and pump wavelengths, respectively.

The small signal gain factor of the amplifier is given by

$$g(z) = \eta \frac{\left(\frac{\sigma_s^e \sigma_p^a}{h\nu_p}\right) I_p(z) + \frac{\sigma_s^a}{\tau}}{\left(\frac{\sigma_p^a}{h\nu_p}\right) I_p(z) + \left(\frac{\sigma_s^e + \sigma_s^a}{h\nu_s}\right) I_s(z) + \frac{1}{\tau\eta}} \times N_t - k_s \quad \text{---------- (4.8)}$$

Since the pump power must be high in dye doped amplifier, the signal power can be ignored:

$$g(0)=\eta\frac{\left(\frac{\sigma_s^e\sigma_p^a}{h\nu_p}\right)I_p(z)+\frac{\sigma_s^a}{\tau}}{\left(\frac{\sigma_p^a}{h\nu_p}\right)I_p(z)+\frac{1}{\tau\eta}}\times N_t-k_s \qquad \text{----------} \quad (4.9)$$

Then, the signal variation is assumed to be $I_s(z)=I_0\exp(g_0 z)$ (4.10)

4.2.2 Gain saturation in a dye doped fiber amplifier

Gain achieved in a Dye Doped Fiber Amplifier (DDFA) is due to population inversion of the dopant ions. The inversion level of a DDFA is set, primarily, by the power of the pump wavelength and the power at the amplified wavelengths. As the signal power increases, or the pump power increases, the inversion level will reduce and thereby the gain of the amplifier will be reduced. This effect is known as gain saturation - as the signal level increases, the amplifier saturates and cannot produce any more output power, and therefore the gain reduces. Saturation is also commonly known as gain compression [25].

4.2.3 Optimum length of an amplifier

Pump absorption throughout the amplifier length results in a population inversion that varies with position along the fiber; this reaches a minimum at the fiber end opposite to the pump laser for unidirectional pumping, or a minimum at midlength for bidirectional pumping using equal pump powers [25, 26]. To achieve the highest overall gain, the length is chosen so that the fiber is transparent to the signal at the point of minimum pump power. For example, the optimum pump power is determined by requiring transparency

of the signal will occur beyond the transparency point with length shorter than the optimum, full use of the available pump energy is not made (figure 4.1.)

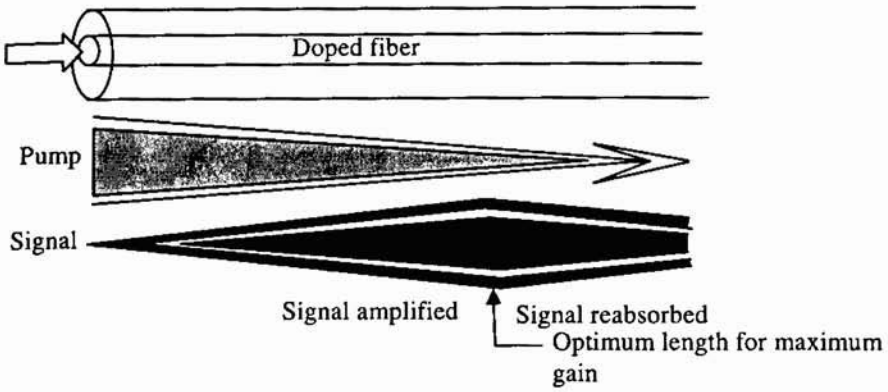


Figure 4.1 Schematic diagram showing variation of pump and signal powers with distance along the doped fiber

4.3 Fabrication of dye doped optical fiber

As explained in the previous chapters the base material used for the fabrication of polymer preform is methyl methacrylate (MMA) monomer [11-15]. Methyl methacrylate is a suitable candidate for the fabrication of polymer preforms since it has good optical quality and is compatible with most of the organic dopants. We concentrated on only core fibers (air cladding), neglecting the scattering losses associated with it due to the absence of cladding. Commercially available methyl methacrylate will contain inhibitors like hydroquinons. Inhibitors are used for transporting MMA without polymerizing. Inhibitors are removed by repeatedly washing the monomer with 5% NaOH solution followed by flushing with distilled water. The remaining water is removed by adding suitable drying agents like $CaCl_2$. The monomers are purified by distillation under reduced atmosphere.

The initiator used for the fabrication of dye doped polymer fiber is benzoyl peroxide. The chain transfer agent used is n-butyl mercaptan along with the

initiator. As described in chapter 2 the optimum molecular weight (typically between 60,000 and 1, 00,000) is fixed by a number of trial and error methods. We used Rhodamine 6G dye [17, 18] for our current studies since it has relatively good photostability and high fluorescence efficiency. Rhodamine 6G at various concentrations is chosen for the present studies. The resulting mixture is stirred well so as to avoid aggregate formation. The resultant mixture is heat treated to get the polymerized MMA preform.

The prepared preform is now ready for drawing the fiber in a custom-made fiber drawing tower (figure 2.1). By using a preform feeder, the preform is lowered into a furnace which is maintained at a stable temperature of 180°C, and fiber is drawn at this temperature. The fiber diameter can be varied by adjusting the feed rate of the preform and draw rate of the fiber. Using diffraction technique the homogeneity in diameter of the fiber is tested.

4.4 Characterization of dye doped fiber as light amplifier

The current use of fiber amplifier is based on erbium doped silica fibers working in the IR region (in the communication wavelength). Recent introduction of polymer fibers in short distance communication and network designs like LAN has brought amplifiers and repeaters using polymer fibers to the forefront of R&D activities. Possibility of large bandwidth available in the visible wavelength-based data communication has encouraged the studies exploring the possibility of using dye doped optical fibers as optical amplifier [11-15] Details of the work done in our laboratory in this direction are given in the following sections.

4.5 Experimental setup for the characterization of dye doped polymer fiber as a gain medium

Schematic diagram of the experimental setup to characterise the dye doped polymer fiber as an optical gain medium is given in figure 4.2. The main component of the experimental setup is the Rhodamine 6G doped polymer optical fiber of length 5cm. The optical emission from the dye doped fiber was excited using 532nm radiation from a frequency doubled pulsed Nd:YAG laser (Quanta Ray DCR11). The transverse pumping was done with the pump beam profile as a stripe obtained by employing a cylindrical lens of appropriate focal length [27, 28]. The spectral emission characteristics and the gain studies were carried out by collecting the fluorescence signal from the fiber medium using a detector fiber coupled to a monochromator-CCD system. Experiments were carried out using different fiber lengths and pump powers.

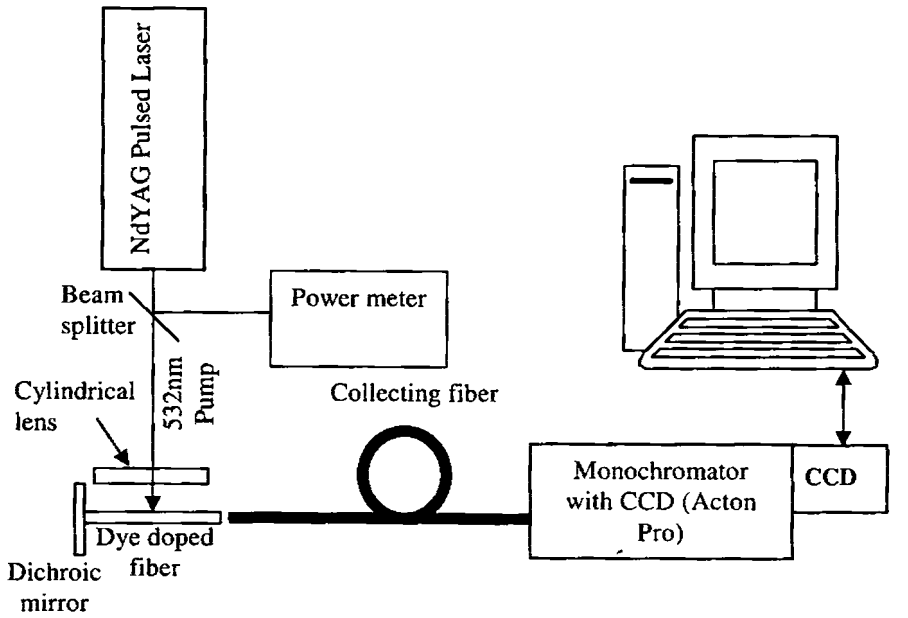


Fig4.2. Experimental setup for gain narrowing studies in DDPOF

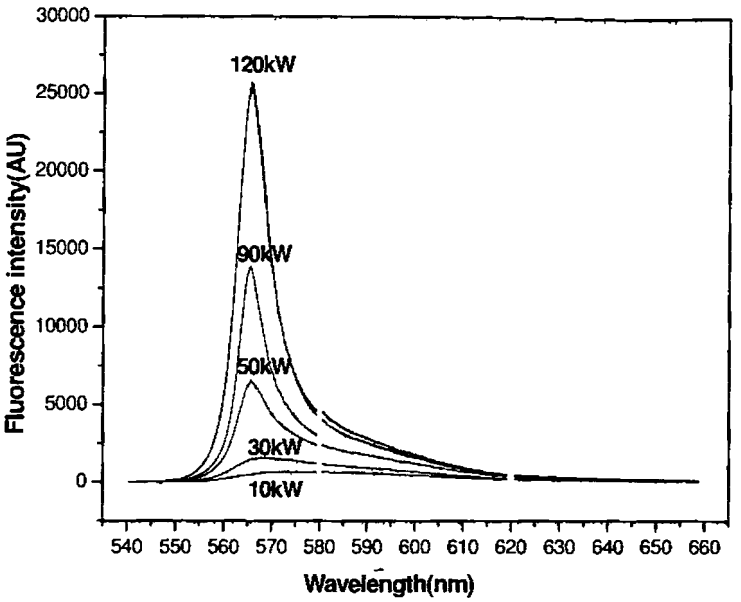


Figure 4.3: Fluorescence emission from DDPOF at various pump power

In order to investigate the effect of feedback, a reflecting mirror was butt coupled to the other end of the fiber. Experiments were carried out with and without the mirror to compare the results obtained from both the cases. Spectral line narrowing as a function of pump power is the signature of light amplification. Fluorescence emission spectra at various pump powers are shown in figure.4.3 which shows a clear line narrowing as pump power is increased, similar to the line narrowing observed in dye doped thin film waveguide structures [29]. Figure 4.4 is the plot of the FWHM of the spectral emission as a function of pump power. As is clear from the figure, spectral narrowing from 42 to 7nm is observed when the pump power is increased from 10kW to 60kW above which no further line sharpening is observed. The value of gain at the line center becomes very large as compared to the values away from the line center. This results in an enhanced emission near the line center owing to the line narrowing effect [30].

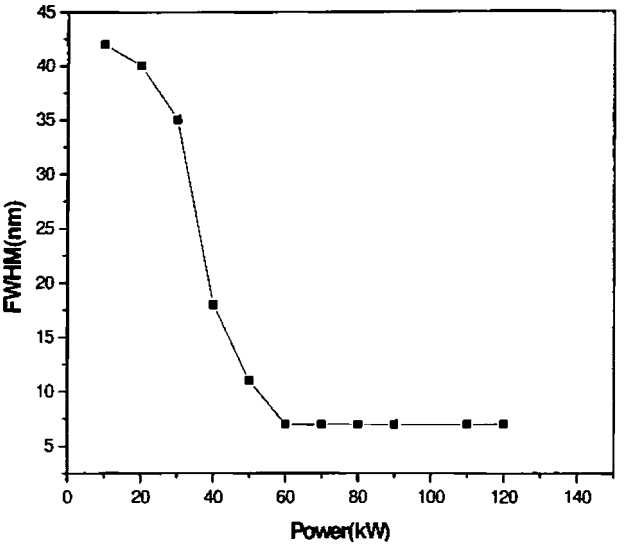


Figure 4.4: Line narrowing with respect to increase in pump power without using the reflecting mirror

Above a certain pump power, the enhanced emission near the line center further will not show any further reduction in the line width as shown in figure 4.4. This is due to the gain saturation effect discussed earlier in this chapter at higher pump powers.

Figure 4.5 shows the emission spectra at 120kW with and without the mirror which does not show any significant change in FWHM. However, one can see that the signal power is enhanced to more than two times. Figure 4.6 is the plot exhibiting the variation of peak intensity as a function of pump power when a mirror is introduced at one end of the fiber. Above 60kW one can see a dramatic enhancement in the emission intensity which is due to the feedback effect.

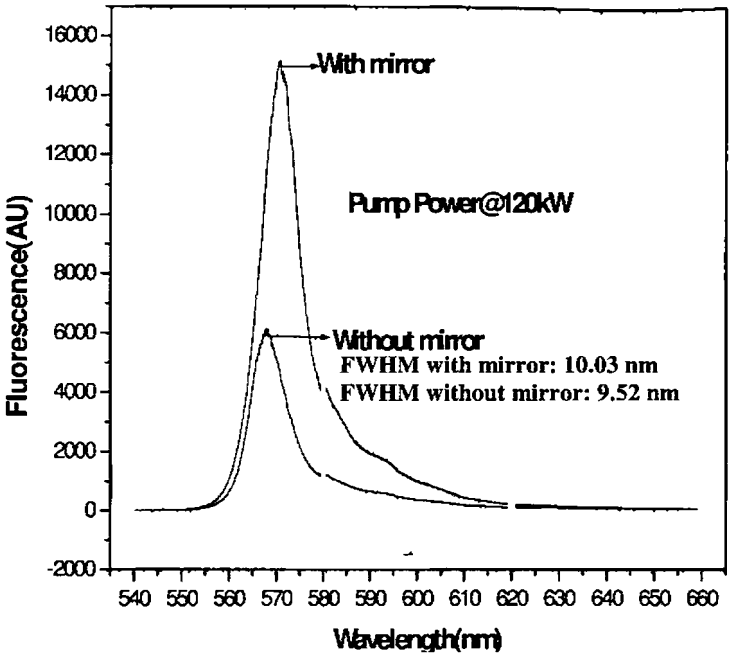


Figure.4.5: Amplification from the DDPOF obtained by introducing the feed back mirror.

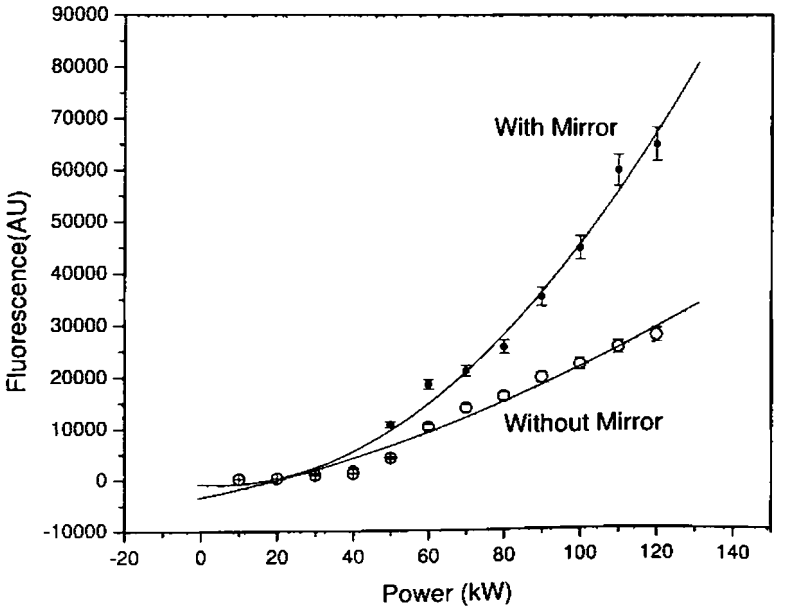


Figure 4.6: Plot showing the peak intensity at various pump powers from DDPOF with and without feed back mirror

4.6 Dye doped POF as optical amplifier

In communication system one has to amplify weak optical signals. If L is the length of the amplifying medium, then

$$I(L) = I_i e^{gl} \qquad \text{----------} \quad (4.11)$$

or

$$\ln \frac{I(L)}{I_i} = gL \qquad \text{----------} \quad (4.12)$$

or

$$g = \frac{1}{L} \ln \frac{I(L)}{I_i} \qquad \text{----------} \quad (4.13)$$

When a signal of intensity I_i is injected into an amplifying medium it gets amplified while propagating along the gain medium and the output intensity will then be

$$I_0 = I_i e^{g(\nu)z} \qquad \text{----------} \quad (4.14)$$

Where g (ν) is the spectral amplification constant.

To check the efficiency of the dye doped polymer fiber as an optical amplifier, experiment was conducted to measure amplification factor by injecting a weak signal and measuring the output intensity with and without the pump. Details are given in the following sections.

4.6.1 Experimental setup

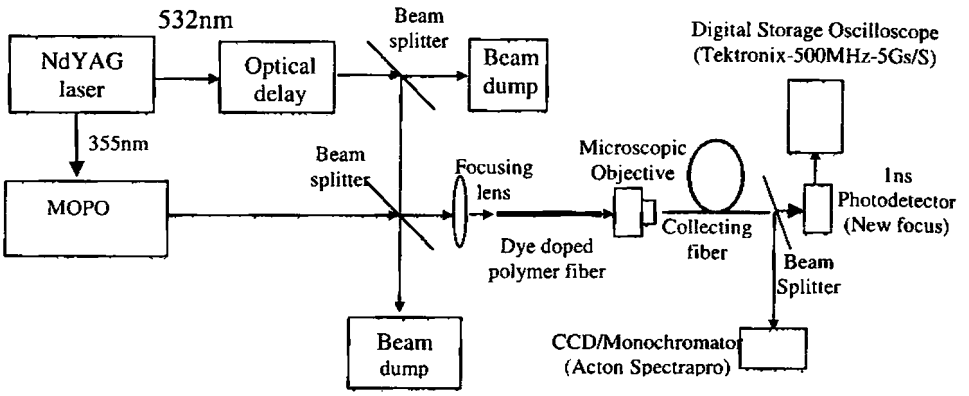


Figure 4.7: Experimental setup for amplification studies of dye doped fibers

Frequency tripled beam of Nd: YAG laser, at 355nm was used to pump the MOPO while the frequency doubled beam at 532 nm was used to pump the dye doped POF. The pumping was done axially and the amplified signal was coupled using a collecting fiber using microscope objective (Fig 4.7).

The pump beam (532nm) was combined with the signal beam (at 598nm) both temporally as well as spatially using appropriate optical delay system and beam splitter. The optical delay was introduced by multiple mirror reflection technique by which we can precisely adjust the time delay between signal and pump in the order of a few nanoseconds. A photo detector of 1ns response time (Newfocus) along with a Tektronix DSO (500MHz) was used to monitor the amplified output. A monochromator-CCD assembly (Acton Spectrapro) was used to study the spectral response of the output signal. Experiments were repeated for various pump powers and fiber lengths.

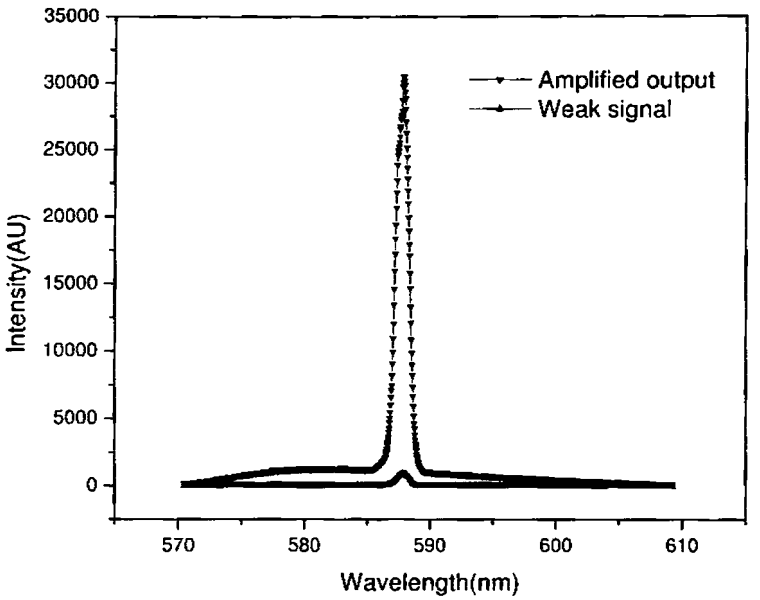


Figure 4.8: Amplification of a weak signal in a dye doped fiber

4.6.2 Results and discussion

As can be seen in figure 4.8, there is clear signal amplification in the presence of the pump source. This proves beyond doubt that the dye doped fiber can be used effectively as an amplifier in communication link in the visible spectral region. Figure 4.9 shows that the maximum gain of 18dB is attained for a length of 10 cm which reveals that there is an optimum length to attain maximum amplification (at 10^{-3} molar dye concentration). The fact that we can have a gain of 18dB for a 10 cm length of fiber is an advantage of dye doped fiber in comparison with an EDFA, which needs a length of several meters to attain optimum gain.

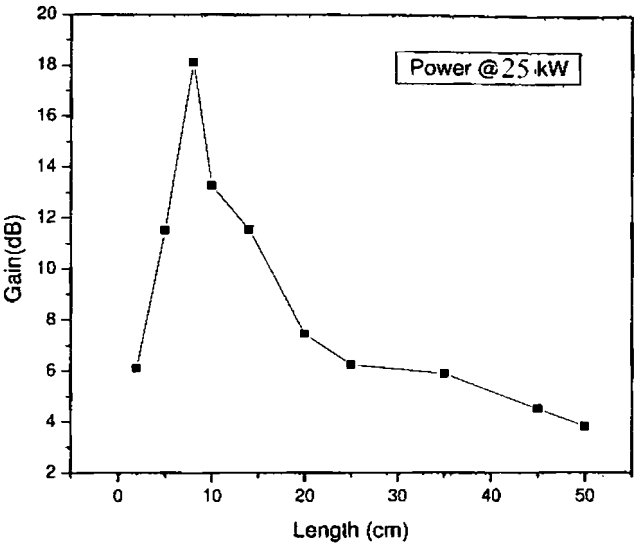


Figure 4.9: Gain vs length of a dye doped fiber amplifier

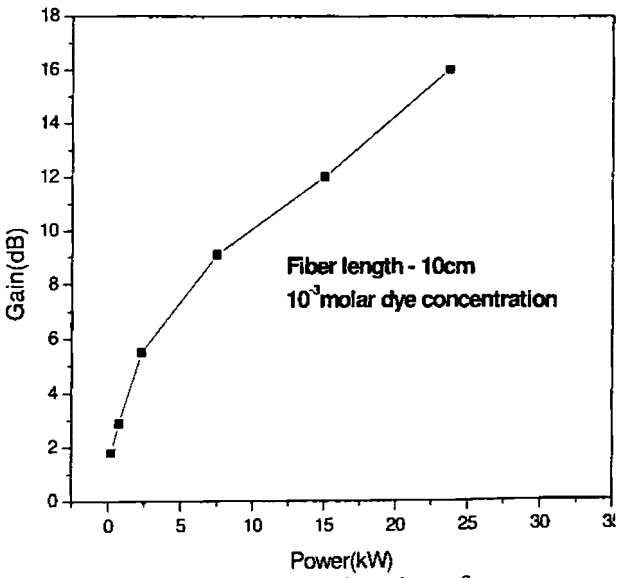


Figure 4.10: Gain as a function of pump power.

Figure 4.10 shows the gain attained by a dye doped fiber of length 10 cm as a function of pump power. The gain varies as quadratic function of pump power. The pump power has to be limited below a certain level because of the bleaching of the dye molecules contained in the fiber. Figure 4.11 shows the intensity distribution of amplified signals at different wavelengths at a constant input signal power (<0.2W peak power). The peak pump power was 1kW.

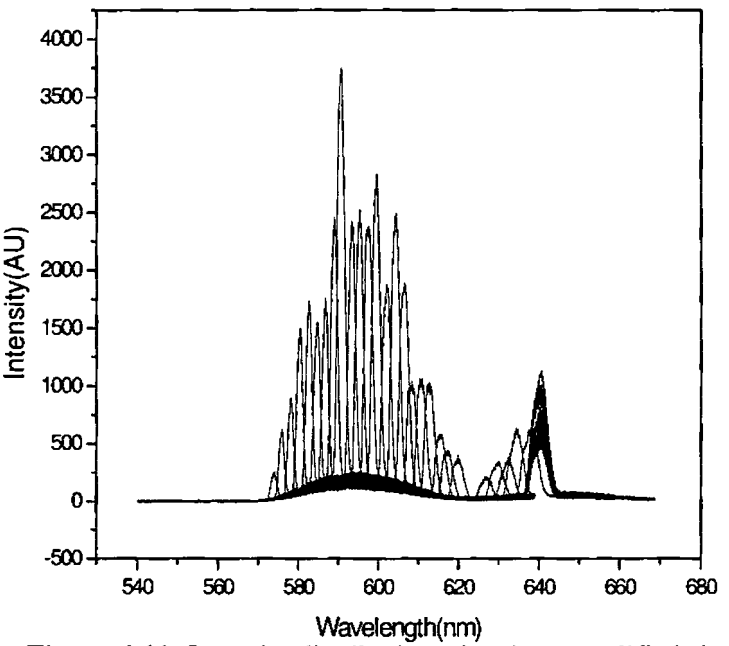


Figure 4.11: Intensity distribution of various amplified signal wavelengths

The spectrum shows maximum gain at 586nm and another maximum at 640nm. The peak around 586nm is due to the characteristic emission of Rhodamine 6G. The emission around 640nm is attributed to either aggregate formation or due to emission followed by intersystem crossing. As suggested by John and Pang [19] (1996) nearest probable origin of the 640nm emission

is the triplet-triplet transition. One can consider the 640nm emission as an added feature in the laser emission from the sample.

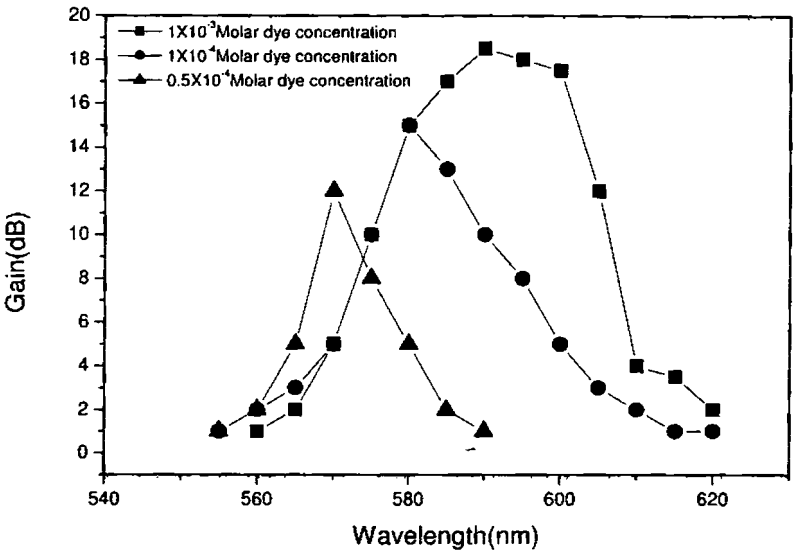


Figure 4.12: Plot showing the gain values for different wavelengths for three different dye concentrations. The fiber length was 10cm with a diameter of 250 μm.

The gain of a dye doped polymer fiber amplifier is dependant on the doping concentration as shown in figure 4.12. From the figure we can clearly see that the gain is increased from 12dB to around 18.5 dB as the dye concentration is increased from $0.5x10^{-4}$ molar to $1x10^{-3}$ molar. There is also a noticeable shift in the peak wavelength from 570 nm to 590 nm. This is due to the absorption and re-emission mechanism taking place in the dye molecules [21]. At higher dye concentrations the absorption and re-emission rates will be higher which tends the wavelengths to red shift.

Figure 4.13 depicts spectral dependence of fluorescence output as a function of pump power in the core of the dye doped fiber of 320micrometer in diameter. There is a narrowing coupled with the blue shift in the emission

spectra as pump power is increased. This blue shift is due to the selective excitation of those modes which have sufficient gain for amplification [21, 29].

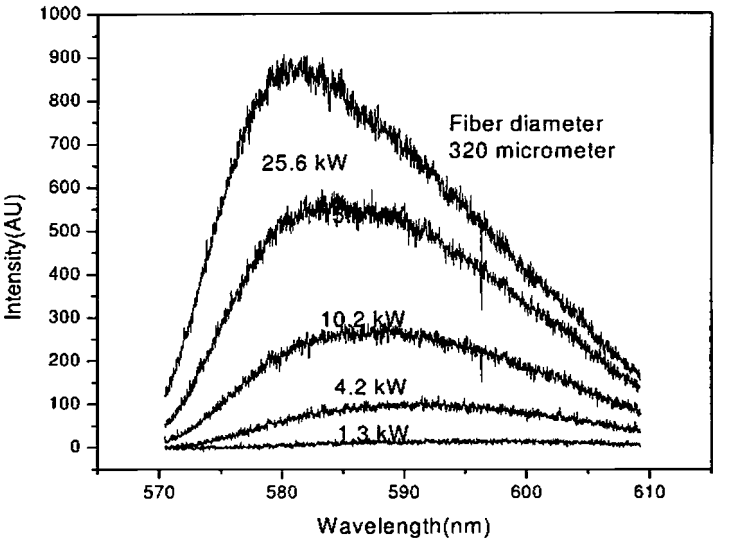


Figure 4.13: Fluorescence blue shift observed as the pump power is increased

Figure 4.14 shows the variation of gain as a function of pump power for three different dye concentrations and three diameters. One of the obvious results is the enhancement of gain with dye concentration. Saturation of gain is observed at higher concentrations and moreover, the gain characteristics are sensitive to fiber diameter at lower concentration.

At higher concentration there is not much dependence of gain with fiber diameter. This is due to the fact that increase in the diameter is equivalent to enhancement in number of molecules involved in the excitation process. This will result in an effect equivalent to the concentration enhancement resulting in gain saturation.

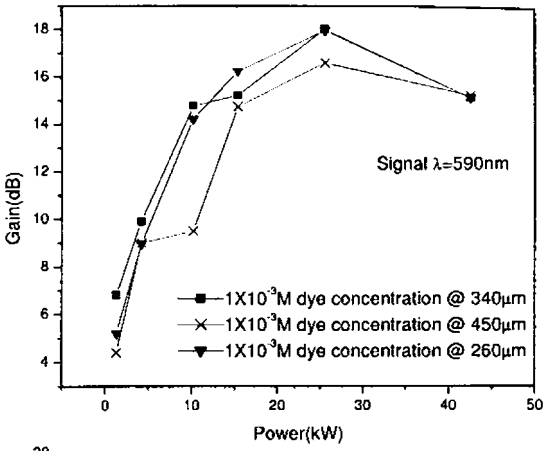


Fig 4.14a

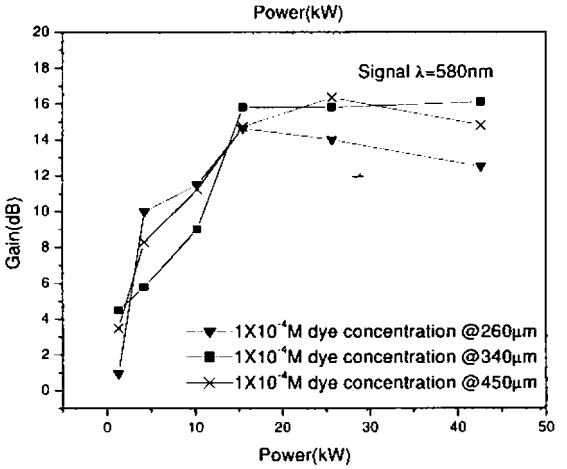


Fig 4.14b

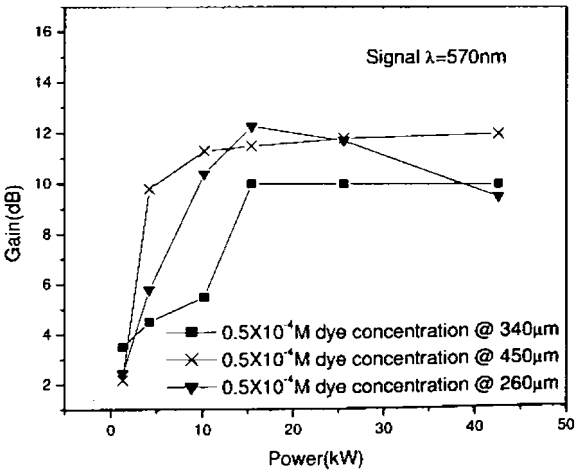


Fig 4.14c

Figure 4.14a, b, c: Optical gain plots of various doping concentrations and fiber diameters. The length of the fiber is taken as 10cm.

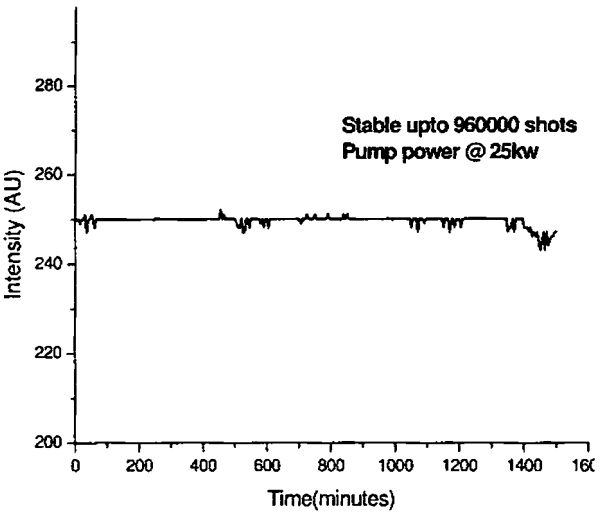


Figure 4.15: Graph showing fluorescence intensity vs. time of exposure of dye doped optical fiber to pump (532nm)

It is found that the fluorescence output from the fiber was stable up to 960000 shots of pump pulse without considerable fluorescence bleaching at a peak pump power of 25kW as can be seen from the figure 4.15.

4.7 Conclusion

Good quality dye doped polymer optical fibers were drawn. Studies described in this chapter show the performance and stability of Rhodamine 6G doped polymer optical fiber as an amplifying medium. An important observation is that there is an optimum length at which amplifier gain is a maximum. For example with 25kW pump power a maximum 18dB gain is attained for 0.1meter length of fiber. One of the important advantages to be noted is that the fiber length needed is very small unlike in the case of EDFA which requires several meters of fiber. The polymer optical fiber amplifier working in other spectral regions like the communication wavelength can also be developed by using appropriate dyes or rare earth dopants.

References:

1. M.B.J. Diemeer, P. De Dobbelaere, and R. Flipse , in " Integrated Optical Circuits and Component, Design and Application", Dekker, New York,1999
2. L. Eldada and L.W. Shacklette, IEEE, J. Select, Topics Quantum Electron, 16,54(2000)
3. Hari Singh Nalwa,"Polymer optical fibers, American Scientific Publishers,2004
4. G.P Agarwal,"Fiber optic Communication systems"2nd ed. Wiley-Interscience, New York.1997.
5. F.P Kapron ,D.B. Keck, and R.D Maurer, "Radiation losses in glass optical fibers", *Appl. Phys Lett*.17,423(1970)
6. T.Kaino,KJinguji and S.Nara, "Low loss poly(methamethacrylate-d8) core optical fibers"*Appl.Phy.Lett*,42.567(1983)
7. Y.Ohtsuka,T. Senga, and H Yasuda, "Light–focusing plastic rod with low aberration"*Appl.Phy.Lett*,25.659(1974)
8. Y.Ohtsuka,T. Sugano and Y Terao," Studies on the light-focusing plastic rod. 8: Copolymer rod of diethylene glycol bis(allyl carbonate) with methacrylic ester of fluorine containing alcohol "*ApplOpt,12.2319*(1981)
9. Y.Koike,H Hatanaka, Y.Ohtsuka "Studies on the light-focusing plastic rod. 17: Plastic GRIN rod lens prepared by photocopolymerization of a ternary monomer system",*ApplOpt,23,1779*(1984)
10. Werner Daum, Jurgen Krauser,Peter E Zamzow,Olaf Zeimann,"POF-Polymer optical fibers for data communication"Springer-verlag,2002
11. A Costela ,F Florido ,I G Moreno , R Duchowicz ,F AGuerri,J M Figuera and R Sastre "Solid-state dye laser based on copolymers of 2-hydroxyethyl methacrylate and methyl methacrylate doped with rhodamine 6G" *Appl. Phys.* B **60**, 383–389(1995).
12. G D Peng,P L.Chu,Z Xiong,T W.Whitbread,and R P.Chaplin " Dye-Doped Step Index Polymer Optical Fiber for Broadband Optical amplification" *Journal of Lightwave Technology* **14** ,2215-2223 (1996).
13. K Kurki,T Kobayashi,N Imai,T Tamura,S Nishihara,Y Nishizawa,A Tagaya,and Y Koike "High –efficiency organic dye-doped polymer optical fiber lasers" *Appl.Phys.Lett* **77,** 331-333(2000).
14. K Kurki,TKobayashi,N Imai,T Tamura,Y Koike and Y Okamoto " Organic Dye-Doped Polymer Optical Fiber Lasers" *Polym.Adv.Technol* 11, 612-616 (2000).
15. H Liang,Z Zengchang L J Xu,B Chen,H Zhao,Qijin Zhang and H Ming "Fabrication and Amplification of Rhodamine B- Doped Step-index Polymer Optical fiber" *J.Appl.Polym.Sci* **93,** 681-685 (2004).

16. K Geetha, M Rajesh, V P N Nampoori, C P G Vallabhan and P Radhakrishnan "Loss characterization in rhodamine 6G doped polymer film waveguide by side illumination fluorescence" *J. Opt. A: Pure Appl. Opt.* **6** 379–383 (2004)
17. Drexhage K H " Structure and properties of laser dyes *Dye Lasers" (Springer Topics in Applied Physics* vol 1) ed F P Sh¨afer (Berlin: Springer) chapter 5, p 155 (1990)
18. Sh¨afer F P (ed) "Principles of dye laser operation *Dye Lasers" (Springer Topics in Applied Physics* vol 1) (Berlin: Springer) chapter 1, p 1 1990
19. Sajeev John and Gendi Pang "Theory of lasing in a multiple-scattering medium" *Phy.Rev. A, 54, 4 (1995)*
20. Costela A, Florido F, Garcia-Moreno I, Duchowicz R, Amat-Guerri F, Figuera J M and Sastre R 1995 Solid-state dye laser based on copolymers of 2-hydroxyethyl methacrylate and methyl methacrylate doped with Rhodamine 6G *Appl. Phys. B* **60** 383–9
21. C.V. Shank, "Physics of dye lasers", *Rev. Mod. Phy. 47,649, 1975*
22. T. Kobayashi, K Kurki, N. Imai, T. Tamura, K Sasaaki, Y Koike. *Proc. SPIE,3625,206,1999*
23. A. Tagaya, S. Teramoto, T. Yamamoto,K. Fujii, E Nihei, Y. Koike, K Sasaki, *IEEE J Quantum Electron, QE-31,2215,1995*
24. M Karimi, N Granpayeh, M. K Morraveg Farshi, " Analysis and design of dye-doped polymer optical fiber amplifier" , *Appl.Phy B 78,387-396,2004*\
25. Optical Society Of America, Fiber optics Handbook, McGraw –Hill Professional , , 2001
26. A.Ghatak, K. Thyagarajan, Introduction to fiber optics, Cambridge University Press, ,2004
27. Kruhlak R J and Kuzyk M G 1999 Side-illumination fluorescence spectroscopy I—principles *J. Opt. Soc. Am. B 16 1749–55*
28. Kruhlak R J and Kuzyk M G 1999 Side-illumination fluorescence spectroscopy II—applications to squaraine dye-doped polymer optical fiber *J. Opt. Soc. Am. B 16 1756–67*
29. K. Geetha, M. Rajesh, V.P.N. Nampoori, C.P.G. Vallabhan and P.Radhakrishnan, "Laser emission from transversely pumped dye-doped free-standing polymer film", ***J.Opt. A: Pure Appl. Opt. 8 , 189-193 (2006),***
30. O.Svelto, "Principles of Lasers" Plenum Press. New York and London, 1998

Chapter 5

Do not draw the conclusion from your apprentice studies that you have nothing left to learn, but rather that you have infinitely more to learn.- Blaise Pascal

A fiber optic smart sensor to characterise cement paste

Fiber optic smart sensors are used to monitor the civil structures. One of the important parameters in civil engineering is the setting characteristics of concrete made of cement. This chapter describes how optical fiber can be used to characterise the setting dynamics of various grades of cement. The results explain the comparative performance of polymer fiber over silica fiber.

5.1 Introduction

Over the last century, concrete [1, 2] has changed the way we dwell on this planet. It has been established as the most popular building material with unmatched properties. It is such a familiar material that quite often we ignore the remarkable process by which cement and water are mixed with a wide range of aggregates to form a plastic mass which ultimately sets into a strong and durable material.

Moisture or water plays an important role in the setting and strength development of concrete. The cement hardening is due to the chemical process called hydration. This means that the silicate and aluminate [3] minerals in the cement react and combine with water to produce the 'glue' that holds together the aggregate which forms the concrete. The water-cement ratio is always much in excess of that required for hydration of the cement in the attainment of its final form in the concrete. The excess water used for workability will be lost as the cement gradually attains its final form. The cement paste has several other important properties such as strength and porosity, which are determined by the ratio of water to cement in the original mix. The water-cement ratio is an index of strength and design of cement mixes. The lower the water cement ratio, the higher will be the strength of the hardened paste and that of the concrete.

Many studies have been done in the past for the optimization of cement paste used for civil structures [4, 5, 6]. Conventional technique for the measurement of setting time of cement mix is by using the Vicat and Gillmore needles [1, 2]. These measurements can provide only the initial and

final setting time. Since the characteristics may change from batch to batch of the same grade of cement it is very important to study the setting behavior of the cement paste. There are many chemical and physical reactions taking place during the setting of concrete, which are not fully understood till now. This is due to the fact that wide ranges of chemical substances exist in the cement. The chemical reactions may continue slowly over a long period of time and others may be initiated by various environmental parameters into which concrete is subsequently exposed. For these studies, continuous measurement of these setting and curing characteristics of cement are very important. Conventional methods do not provide continuous measurement of setting characteristics. But with the present fiber optic sensor we can continuously monitor setting and hardening pattern of different grades of cement.

Optical fiber sensors are capable of playing an important role in the health monitoring of civil structures such as bridges [7], dams,[8] buildings and so on. The main features of the optical fiber sensor are its immunity from electromagnetic noise, very good sensitivity and compactness [9]. Since optical fibers are very sensitive to strain and bending losses, a variety of intensity modulated sensors can be used to carry out *insitu* studies of civil structures [10]. Studies have been carried out to determine the cracks in concrete structures using displacement techniques [11].Moreover, many interferometric sensors based on Bragg gratings have been developed for more sensitive measurements, such as vibrations and strain [12,13]. Distributed fiber optic sensors can also be used as smart sensors for the determination of cracks in concrete structures [14, 15].

The optical fiber sensor can be incorporated into such structures during their construction or can be adhered to them after the completion of construction. The sensitivity of the sensor largely depends on the bonding properties of the construction materials [2]

The main advantage of the present method is that no mechanical parts are involved in the measurements, and this reduces the instrumental error to a great extent. Moreover, the experimental setup is very simple and straightforward and this can be installed at the work site with no sophisticated instruments for measurements. Commonly available high luminescent LEDs and photodiodes can be employed for these studies, which make the sensor very cost effective. The use of plastic optical fiber will further reduce the cost considerably.

5.1.1 Setting and hardening of cement paste:

Setting is described as the stiffening of the cement paste, i.e. setting refers to a transition from fluid to a rigid state. In current practice the terms 'initial setting' and 'final setting' are used to describe the stages of setting, which take place within ten hours. The setting process is accompanied by temperature changes in cement paste. Initial set corresponds to a rapid rise in temperature and final set to a peak in temperature. The usual method for finding the setting time of cement is by Vicat apparatus. Usually a measure of the above said two setting times, viz the initial and the final setting times follow a definite procedure. The period elapsing between the time when water is added to the cement and the time at which the Vicat needle fails to pierce the test block by 5± 0.5mm is taken as the initial setting time [2].

The period elapsing between the time when water is added to the cement and the time at which the needle makes an impression on the surface of the test block while the attachment fails to do so is taken as final setting time[2]. Typically, the initial setting value for ordinary porcelain cement should be higher than 30 minutes while final setting time should be less than 600 minutes [2].

This means that in the usual description, the setting of the cement will be completed within 600 minutes. However, the dynamics of cement setting continues for duration of more than 600 minutes, the characteristics of which cannot be studied using the Vicat apparatus. This is one of the important drawbacks of the Vicat-based measurements. Since the Vicat measurements provide only the information about the setting times within 600 minutes, the curing and rapidity with which cement achieves hardness with different environmental conditions cannot be studied. Although, during the setting process, the paste acquires some strength, for practical purposes, it is the hardening, which indicates the gain of strength of set cement paste. The speed of setting and rapidity of hardening are entirely independent of one another. Using conventional techniques, the speed of setting and hardening cannot be traced. The fiber optic based measurements will be helpful to study the complete setting characteristics of the cement paste.

5.1.2 Principle

The basic principle underlying the sensor is that as the cement sets, it exerts a stress on the sensing fiber, which is laid within the cement paste. This stress

induces strain on the optical fiber, which can be thought of as a series of aperiodic microbends on the surface of the fiber. This in turn changes the characteristics of the light signal transmitted through the fiber and can be viewed as stress induced modulation of light in the fiber. By monitoring the intensity variation with time we can accurately determine the cement setting rate. This can be used as an effective tool for quality testing of commercially available cements of different grades.

Microbends on the fiber arise as the variable pressure exerted on it distorts the fiber [16, 17, 18]. These bends are so small that the bend radii are of the order of the diameter of the fiber. If the mechanical perturbation is severe, a major part of the light is coupled to the cladding and is lost as radiation modes.

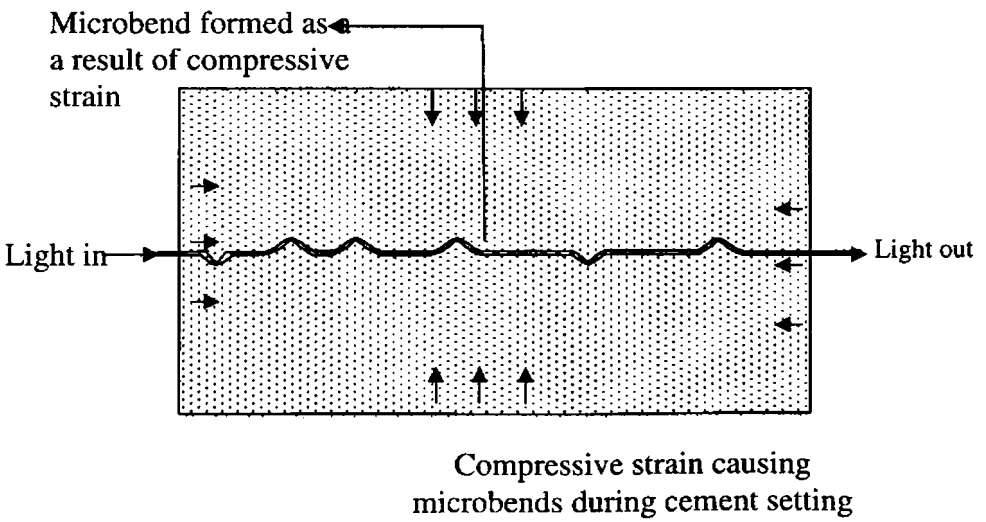


Figure 5.1: Stress induced microbends in an optical fiber

Essentially, a continuous succession of such small bends may cause a significant enhancement of attenuation in the fiber (figure 5.1). The small

variations in the core diameter due to stress induced deformation on the fiber during the setting of the cement mix, give rise to a scattering mechanism, which accounts for part of the loss. The microbends formed cause the coupling of energy between various guided modes and leaky modes (both cladding and radiation modes), the latter giving rise to a loss during transmission of light.

5.2 Experimental setup

The experimental set-up essentially consists of a super luminescent LED source, a certain length of multimode plastic clad silica fiber (200/230μm) or a polymer optical fiber (PMMA) and a photo detector. The epoxy lens of the commercially available LED (λ=670 nm.) is removed and the end face is thoroughly polished, so as to make a very efficient coupling to the fiber. The experimental setup is as shown in figure 5.2.

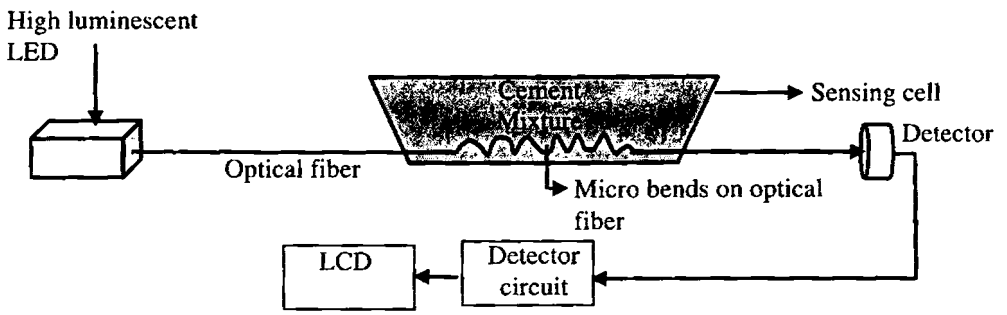


Figure 5.2: Experimental setup

Initially the sheath in the sensing region of the fiber is removed. This ensures the development of micro-bends on the fiber due to local strain. The ends of the fiber are polished to obtain efficient coupling from the LED and to the

power meter. The whole setup is aligned perfectly with one or two loops of the fiber residing in the cement container (size 12cm X 9 cm) as shown in figure 5.3.

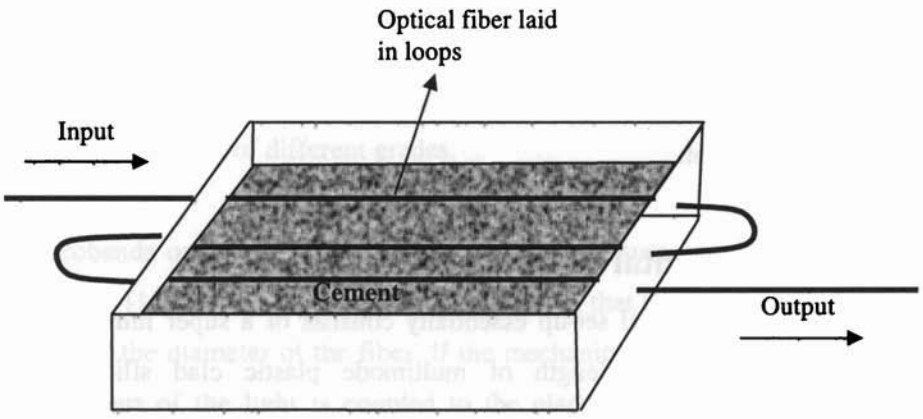


Figure 5.3: Schematic of the sensing head

The total length of the fiber within the cement mix is around 15 cm. This looping provides a greater surface area so as to enhance the sensitivity. The fiber is placed in such a manner that its unsheathed sensing region is well within the container. A cement mixture with a specific water cement ratio (cement: water is 4:1) is made and it is poured into the container (50mm x 50mm x 20mm) such that it fully covers the sensing region. Only a small quantity of the cement mix is required for this purpose. The output power measurements are taken at regular intervals of time using a Newport 1815C power meter. Data acquisition and processing are done using a Labview card and a PC.

5.3 Results and discussion

The output variations with time for cement mix prepared for three different grades of cements are studied. Typical plots of power *vs* setting time are shown in figures.5.4a, 5.4b &5.4c. As can be clearly seen from the figure, the dynamics of the setting of cement mix involves three phases, viz., an initial slow phase, the second rapid phase and the final slow setting phase [2].

During the initial phase, the cement loses its plasticity and becomes a thick paste. Here, we can infer that the cement mix starts the process of setting. During this phase, the cement passes through the initial and final setting processes as described in literature [1, 2]. Then it enters the second phase, which is the most important and the fastest process in the whole phenomenon of setting of cement. For example the semi-log plot of transmitted power vs the setting time in the second phase (figure.5.5) provides the following information.

In the case of blended and 53 grade cements, a power law behavior can be obtained as, viz. $I(t) = C_1 t^{-0.96}$ and $I(t) = C_2 t^{-0.93}$ respectively while in the case of 43 grade the corresponding relation is $I(t) = C_3 t^{-0.12}$, where C_1, C2, C_3 are constants. The setting times for the 53 and the blended grades are lower than that in the case of 43-grade sample. The commercial specifications also show that 53-grade and blended grade cements have similar setting properties.

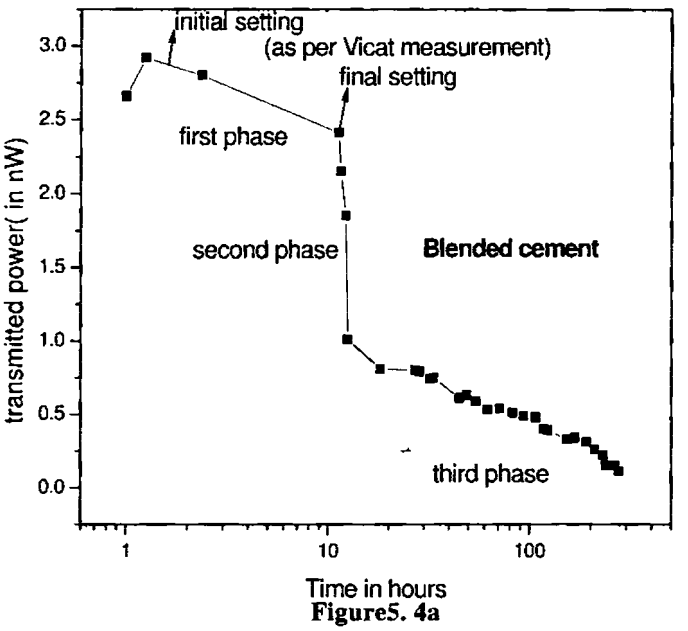


Figure5. 4a

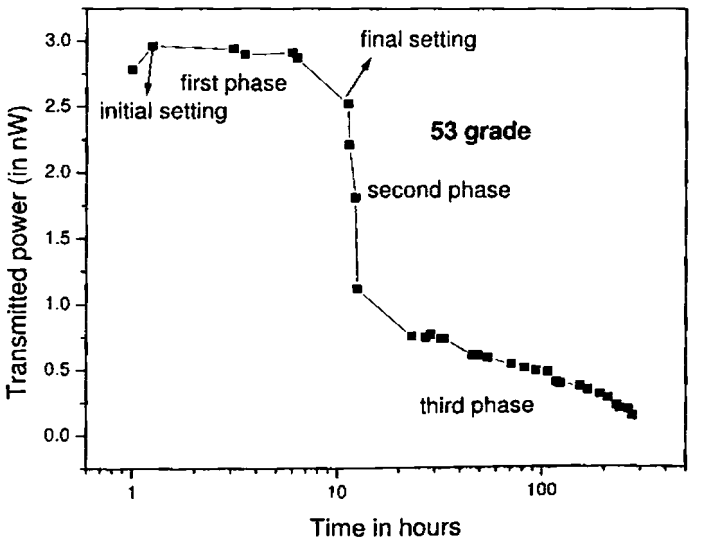


Figure5. 4b

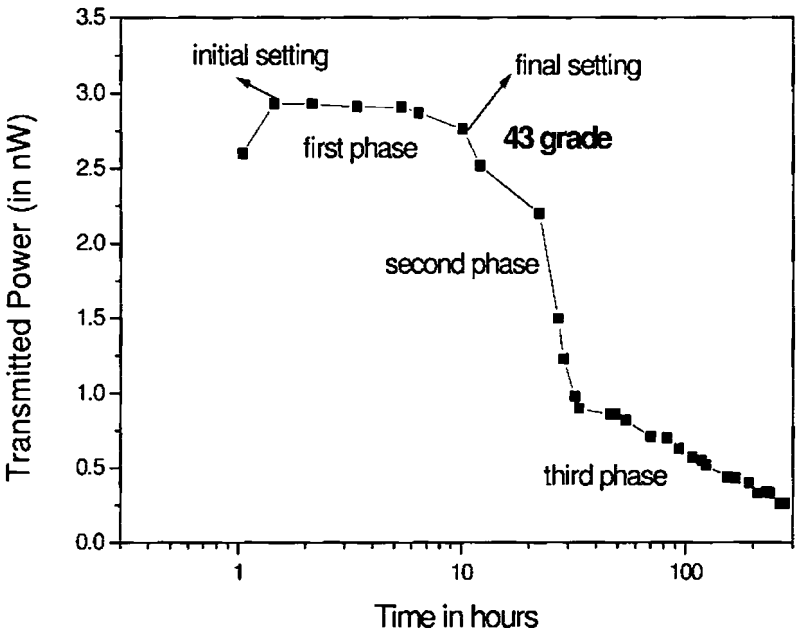


Figure 5.4c

Figure 5.4 a, b, c: A typical graph showing time vs. transmitted intensity through a silica fiber laid in the cement paste.

As per commercial specification, the 43-grade cement is slow setting, which is confirmed by our investigations [19].

In summary, by evaluating the setting properties of the second stage one can determine whether the sample in question is showing the properties of the required grade. Thus for verifying the data provided by the manufactures, we can determine the setting times and hence the quality of the cement samples. Observation during the third and final setting phase reveals that the cement mix attains its full strength in almost a month's time.

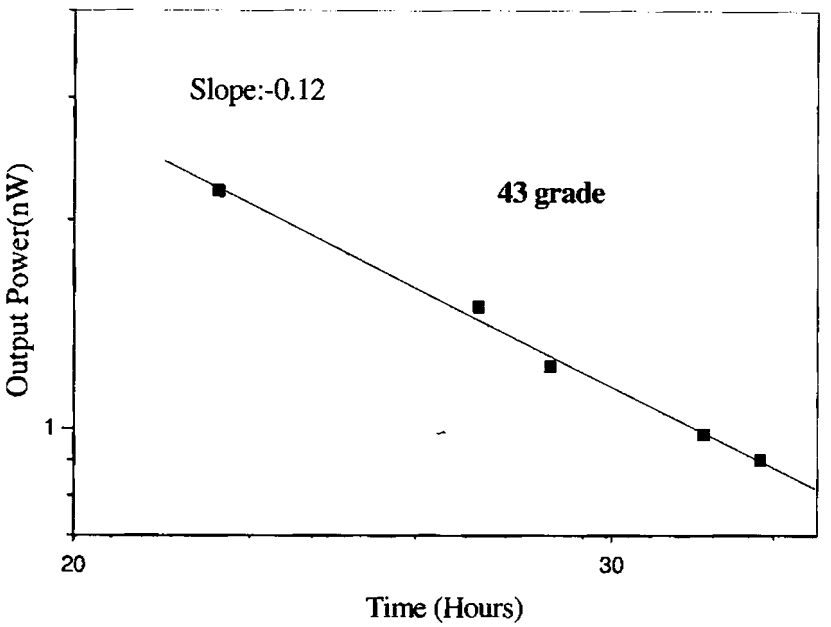


Figure 5.5: Time vs. log (I) plot showing the power law dependency

As mentioned before, the results from the Vicat experiment provides the details of the initial and final setting times. However, the results from the fiber optic smart sensor show not only the setting times given by Vicat apparatus but also the setting characteristics extending upto a few weeks. The so-called initial and final setting times are 160 and 420 minutes respectively for 43-grade cement, which agrees with the Vicat test. The differences in setting behavior of cement mix have far reaching implications in civil works related to building constructions.

A carefully incorporated fiber optic sensor during building construction will be helpful in identifying proper phases of setting.

5.4 Modification of Porosity during cement setting

To study the modification of porosity in a concrete mix during the setting period, water is added at different setting stages of the curing process. This causes discontinuities in the curing curve. The amount of shift in the discontinuity on the curing curve directly reflects the reduction in the number of micropores in the cement paste as the setting progresses. To study the setting process continuously the experimental setup was interfaced to a computer with the help of LabView software.

When water is added at specific times, the water percolates through the micropores onto the fiber laid inside the cement paste. Due to the presence of water instead of air, the cladding modes will be guided back into the fiber, thus increasing the output optical power. Setting of cement mix involves three phases, viz., an initial slow phase, the second rapid phase and the final slow setting phase [2]. During the initial phase, the cement loses its plasticity and becomes a thick paste. Here, we can infer that the cement mix starts the process of setting. During this phase, the cement passes through the initial and final setting processes as described in literature [1, 2]. Then it enters the second phase, which is the most important and the fastest process in the whole phenomenon of setting of cement. Conventional technique followed by civil engineers provide information regarding the dynamics of cement setting during first phase, where both initial and final set completes.

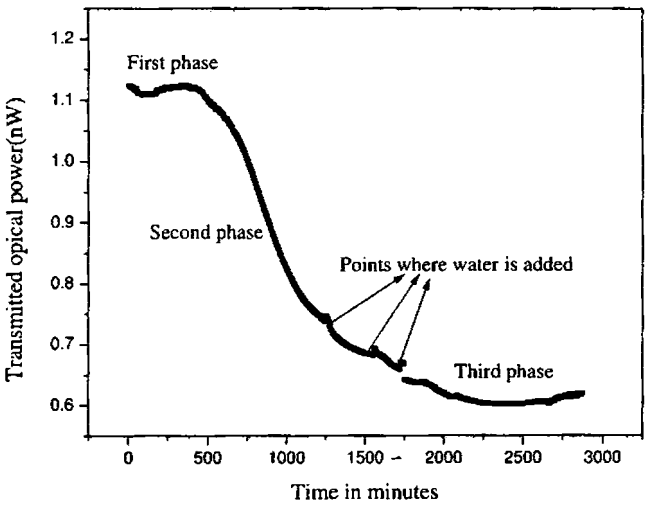


Figure 5.6a

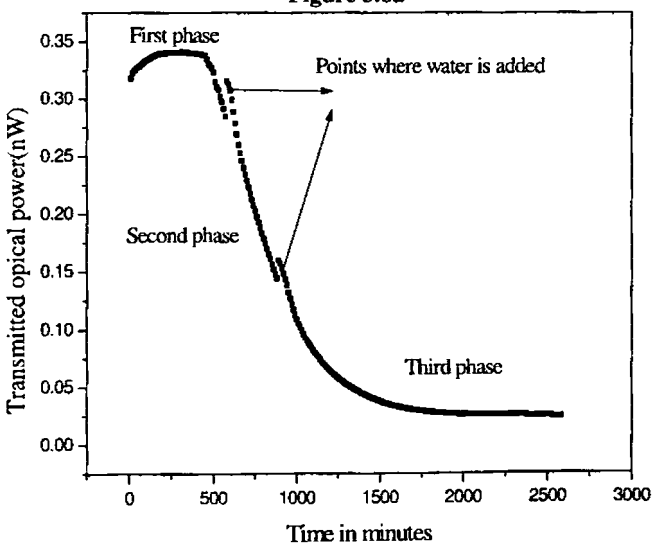


Figure 5.6b

Figure 5.6a, b: Plot showing the output optical intensity vs. time : 43 grade cement was used for the study.

However, lack of knowledge regarding the setting dynamics after the first phase will affect the stability and strength of the concrete structure .One of the methods to monitor this is to study changes in porosity in the cement mix during setting time. During the initial period, after the first phase the cement has a large distribution of porosity, which can be identified by introducing water into the mix.

Water will percolate into the mix through these pores, which will affect the transmitted optical intensity through the fiber. This change in transmission characteristics will appear as a peak in the output characteristics of the sensor (fig5.6a, b). When this experiment is repeated at a later stage, it has been found that variation in the output transmission is reduced due to the reduction in the porosity of the mix. Thus complete disappearance of porosity can be monitored unambiguously using this technique. Thus FOS can be used to monitor the complete setting of cement mix and minimum degree of porosity. This type of sensor will have considerable application in monitoring the health of civil structures.

5.5 Results using POF

The same experiment is repeated by incorporating polymer optical fiber which was fabricated by using the fabrication techniques described in the previous chapter. A 43 grade ordinary Portland cement was used for the study. The fiber was laid inside the cement mixture having a cement water ratio of 4:1. As in the previous experiment the power transmitted through the fiber was constantly monitored by using LabView interface and the plots were obtained as shown in the figures5.7a & 5.7b. Two sets of experiments

were carried out, one without hydrating and the other by hydrating at regular intervals. It can be clearly seen from the graphs that by using polymer optical fiber as the sensing element the sensitivity is increased to a greater extent. Moreover, the initial setting region which had a very low sensitivity in the first setting phase by using glass fibers has been enhanced to a greater extent when polymer optical fibers were used. One can clearly see that the initial setting phase also has a gradual setting pattern. But this was not evident from the experiments based on glass fiber.

In the second set of experiments the cement mix was allowed to set and was hydrated at regular intervals.

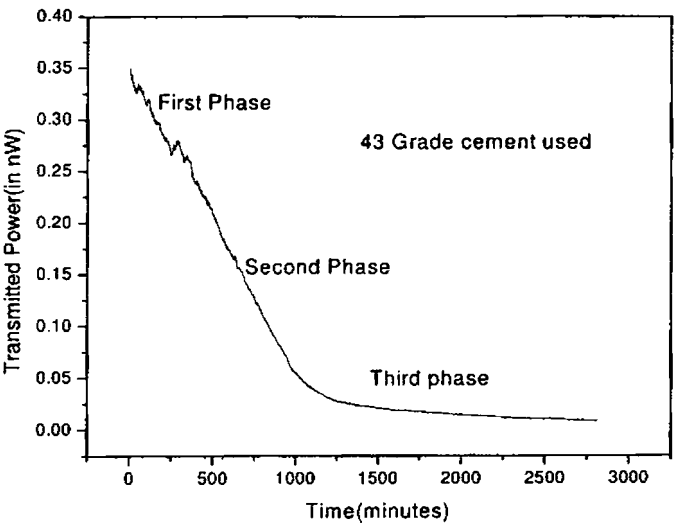


Figure 5.7a: Setting characteristics of 43grade cement monitored by using POF

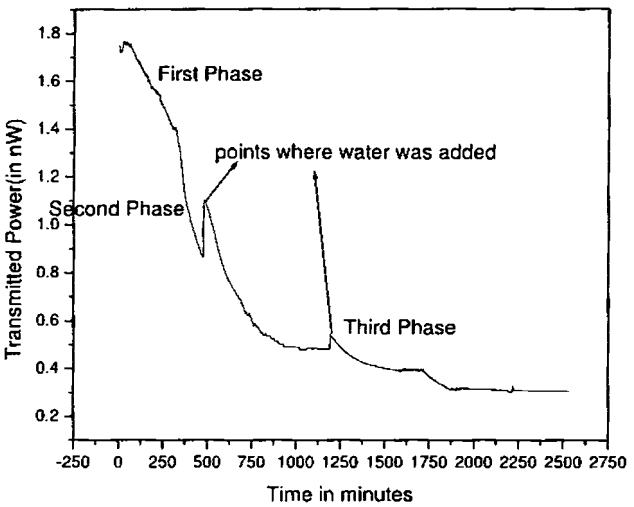


Figure 5.7b: Setting characteristics of cement monitored by adding water at specific intervals of time

It can be clearly seen from the graph that there is a definite increase in the output intensity as the cement is hydrated. Comparing the results with those obtained with glass fiber, plastic fiber shows more sensitivity to moisture. This shows that the polymer optical fiber based experimental results are reliable and are more sensitive than the results involving glass fibers.

5.6 Conclusion

In conclusion, we have described an optical fiber smart sensor that can be used to monitor the setting characteristics of cement mix during civil construction activities. The optical fiber sensor described here is inexpensive and simple to implement. The sensor can be incorporated during the construction process and it has the advantage of being nonobtrusive while

providing the possibility for continuous measurement during the entire lifetime of the civil structure. Detailed analysis of the results with special reference to civil construction is in progress. Continuous monitoring of the civil structures using the embedded fiber optic sensor as described in this paper will be helpful in the deterioration measurements of the concrete structures as well.

References

1. Concrete Technology by George R. White. 3rd edition. Albany, NY: Delmar, 1977.Hobar Publications.
2. B.C.Punima "Reinforced concrete structures." Vol 1. (Standard publishers distributors, Delhi) 1985
3. Effect of aluminium phosphate as admixture on oxychloride cement, M P S Chandrawat and R N Yadav, Bull. Mater. Sci., Vol. 23, No. 1, February 2000, pp. 69–72., Indian Academy of Sciences.
4. Bresler, B., Reinforced Concrete Engineering, Wiley-Interscience, New York (1974)
5. Free and Restrained Shrinkage of Normal and High-Strength Concretes, Ronit Bloom and Arnon Bentur ,ACI Materials Journal, Vol.92,No2,1195,pp.211-217
6. http://www.mnconcretecouncil.org/2003meetings/03-Nancy%20Paper_MCC.pdf
7. Samuel Vurpillot, Nicoletta Casanova, Daniele Inaudi, Pascal Kronenberg, "Bridge spatial displacement monitoring with 100 fiber optic sensors deformations: sensors network and preliminary results" SPIE conference on smart structures and materials , San Diego, USA, 1997
8. Pascal Kronenberg, Nicoletta Casanova, Daniele Inaudi, Samuel Vurpillot, "Dam monitoring with fiber optics deformation sensors." SPIE conference on smart structures and materials , San Diego,USA, 1997
9. Brian Culshaw, John Dakin, "Optical fiber sensors, volume three, components and subsystems." (Artech house publishers, London) 1996
10. Pierre Choquet, François Juneau, john Bessette, "New generation of Fabry-perot optic sensors for monitoring structures." Proceedings , SPIE 7th Annual international symposium on smart structures and materials, Newport Beach ,CA, March 2000, pp.5-9
11. Insang Lee, Yuan Libo, Farhad Ansari & Hong Ding. "Fiber-optic crack-tip opening displacement sensor for concrete". Cement and concrete components .Vol 19 ,1997, pp.59-68
12. Xianglin Gu and Farhad Ansrai "Distributed monitoring of structural cracks by interferometric fiber optic sensors." EM2000, Fourteenth Engineering Mechanics Conference ,The University of Texas at Austin May 21-24, 2000
13. Qinngbin Li and Farhad Ansari "Circumferential strain measurement for a concrete cylinder in uniaxial compression by fiber optic sensor". Experimental mechanics, Vol 42, no 1, March 2002, pp. 37-42.
14. M A Davis, D G Bellemore & A D *Kersey* "Distributed fiber Bragg grating sensing in reinforced concrete structural components". Cement and composite Vol 19, 1997, pp.45-57.

15. Brian Culshaw, John Dakin "Optical fiber sensors, components application, volume four, analysis and future trends". (Artech house publishers, London). *1997*
16. John Gower, "Optical communication systems." Second edition. Prentice-Hall of India Private ltd. 1995
17. S Thomas Lee, K. Geetha, V P N Nampoori, C P G Vallabhan and P Radhakrishnan "Microbent Optical Fibers as Evanescent Wave Sensors" Optical Engg. **41**, 3260-3264,2002
18. Thomas, Lee S.; George, Nibu Sureshkumar, P.; Radhakrishnan, P.; Vallabhan, C. P. G.; Nampoori, V. P. N. "Chemical sensing with microbent optical fiber" Opt. Lett. 26(20), 1541-1543, 2001
19. *http://www.indiacements.co.in/*

Chapter 6

General conclusion and future prospects

General conclusions and future prospects are discussed in this concluding chapter.

Conclusion

Polymer optical fiber based systems are strongly being considered as a potential replacement for silica fiber based systems. This is due to the various advantages of polymer optical fiber over silica optical fiber such as low weight, low processing temperature, economical viability, ease of fabrication and so on. The five chapters of this thesis presented the fabrication and characterisation of polymer optical fibers in their applications as optical amplifier and smart sensors.

Optical polymers such as PMMA are found to be a very good host material due to their ability to incorporate very high concentration of optical gain media like fluorescent dyes and rare earth compounds. High power and high gain optical amplification in organic dye-doped polymer optical fibers is possible due to extremely large emission cross sections of dyes. Dye doped (Rhodamine 6G) optical fibers were fabricated by using indigenously developed polymer optical fiber drawing tower. Loss characterization of drawn dye doped fibers was carried out using side illumination technique. The advantage of the above technique is that it is a nondestructive method and can also be used for studying the uniformity in fiber diameter and doping. Sensitivity of the undoped polymer fibers to temperature and microbending were also studied in its application in smart sensors.

Optical amplification studies using the dye doped polymer optical fibers were carried out and found that an amplification of 18dB could be achieved using a very short fiber of length 10cm. Studies were carried out in fibers with different dye concentrations and diameter and it was observed that gain stability was achieved at relatively high dye concentrations irrespective of the fiber diameter.

Due to their large diameter, large numerical aperture, flexibility and geometrical versatility of polymer optical fibers it has a wide range of applications in the field of optical sensing. Just as in the case of conventional silica based fiber optic sensors, sensing techniques like evanescent wave, grating and other intensity modulation schemes can also be efficiently utilized in the case of POF based sensors. Since polymer optical fibers have very low Young's modulus when compared to glass fibers, it can be utilized for sensing mechanical stress and strain efficiently in comparison with its counterpart. Fiber optic sensors have proved themselves as efficient and reliable devices to sense various parameters like aging, crack formation, weathering in civil structures. A similar type of study was carried out to find the setting characteristics of cement paste used for constructing civil structures. It was found that the measurements made by using fiber optic sensors are far more superior than that carried out by conventional methods. More over,POF based sensors were found to have more sensitivity as well.

Future prospects

Polymers like PMMA are good host material for various amplifying media such as organic laser dyes and rare earths. Since the doping concentration can be increased to a greater degree than in the case of inorganic glass, amplifiers using polymers as the host medium is short in length and thereby reducing the overall system size. Studies can be extended to different stable laser dyes and rare earth elements by doping these materials at very high concentration to the polymer matrix. This enables light amplification studies on all possible visible wavelengths and even communication wavelengths. Prospect of using dye mixtures in tunable fiber amplifiers is also very good. Polymer optical fiber lasers at both visible and IR communication regime can also be envisaged.

Fiber nonlinearity is very high in POF when compared to glass fibers and the nonlinearity can be controlled by adding appropriate materials to the polymer. Nanoparticle can be doped in polymer fibers to tailor the nonlinear properties of the fiber for studies using ultra short pulses.

Both polymer fiber Bragg and long period gratings can be fabricated to realise more sensitive fiber optic sensors. Fiber optic active devices like optical diodes and computational elements can also be investigated.

Another interesting area which has a bright prospect is the fabrication of speciality fibers like microstructured and photonic band gap fibers. Its operations in the ultrashort regime are also worth investigating.