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Ageing and Degradation in Microstructured Polymer Optical Fiber

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ABSTRACT

Ageing and degradation has been accomplished on Microstructured Polymer Optical Fiber (MPOF) by changing simulated ambient environmental condition to ascertain their suitability in possible automotive application. Both single Moded Microstructured Polymer Optical Fiber (SM-MPOF) and Graded Index Microstructured Polymer Optical Fiber (GI-MPOF) has been studied for physical ageing and chemical degradation at elevated temperature in presence of humidification. An acceleration factor of approximately eight has been used to carry out accelerated ageing on specimen under test which is compatible to Australian winter environmental condition. Physical ageing exhibits hole expansion, defect in polymer side chain along hole and asymmetric hole expansion. Chemical ageing exhibits possibility of degradation product formation that is confirmed by observing peaks of different conformation functional groups (-OH, -CH, >CO/-COOH) involved in polymer chain in the both type fiber. Experimental data on chemical ageing satisfy good agreement with Arrhenius rate equation and that degradation products are likely to be accumulated in polymer matrix as sub molecular debris. Experimental data returns empirical correlation of durability versus ageing that manifests necessity of using cable sheath. Given that this would be resistance enough to absorb thermal shock retaining optical performance of the fiber concerned during the entire life span of the equipment.

Keywords: *Accelerated test, ageing, automotive application, degradation, durability, Microstructured Polymer Optical Fiber*

1. INTRODUCTION

Ageing and degradation on optic and thermoplastic polymer has been carried out by many researchers over last two decades but effect of ageing and degradation on microstructure of MPOF and on its light guiding capability is still quite uncertain. Several studies have been reported in published literature about effect of thermal ageing on polymer at elevated temperature [5-7]. These companion authors also observed possible mechanism of thermal ageing. According to these companion authors, thermal treatment of aliphatic polymers results in cyclic monomer and oligomers as dominant degradation product. There is ample evidence that thermal degradation of polymer is accompanied by formation of double bonds. Nevertheless, hydrolysis is known as a primary cause of degradation of thermoplastic polymer at elevated temperature. Effort has also been made to determine durability of thermoplastic polymer by loss of mechanical integrity of material [7]. This companion author observed degradation speed by measuring concentration of carboxylic end group concentration in the polymer.

MPOF can be regarded as 'next generation' fiber because of its enhanced light guiding capability and excellent compatibility in integrated optics. It is usually been made by either sleeving microstructured cane in hollow tube or by drilling rings of holes of required dimension in hexagonal symmetry around polymethyl methacrylate (PMMA) that is later been drawn down into fiber in a commercial draw tower [Figure 1,2]

Present study focuses on exploring suitability of this new class of fiber in possible automotive multimedia application. In automobile, optical performance of this fiber will depend on number of factors. These will include: physical and chemical properties of the fiber material and environmental factor with which this fiber has to come across during life span of the automobile. But this research is limited to study effect of environmental factors on optical performance of the fiber.

Ageing is basically physical phenomenon and can occur by absorption of moisture resulting in decline in optical transmission in MPOF. The absorption of moisture in MPOF can be seen in three distinct situations: in one case moisture/water accumulates in polymer matrix and leading to swelling which can be seen by transparent impression in polymer matrix under high resolution electron micrograph image (i.e., AFM, SEM), in second case it get absorbed into micro-pores and in third case absorbed water diffuses through mocropores and cling onto micro-holes that run along the length of the fiber. Kaino [17] comes to conclusion that a considerable increase in attenuation, varying wavelength (-OH absorption in 750-850nm), can occur when water is absorbed.

On the other hand degradation is a chemical transformation and can occur by thermal ageing in presence of moisture. Degradation results in decomposition which occurs due to thermal ageing. This consequences transfer of energy in

the form of heat which leads to degradation or splitting off of polymer chain. Result of this chemical transformation most probably by oxidation and hydrothermal oxidation give rise accumulation of degradation product as sub-molecular debris in polymer matrix. This phenomenon gives rise brittleness and loss of mechanical and optical integrity in MPOF fiber.

In possible automotive application, the MPOF optical performance should be above some threshold level within the lifespan of the automobile encountering associated environment involved. Producers of optical fiber and its component agree on some standards to have unified interface available. Most of these standards have been developed for Polymer Optical Fiber (POF). These standards have been reconciled with effective correction factor to adopt these for MPOF that is what we are interested in. Comparison of different standards has been summarized in Table 1.

2. EXPERIMENTAL

Experiment has been carried out according to standard method outlines by Palais et al., 1992 [1]. Two types of MPOF with different light guiding mechanism have been tested. In both scenario, simulated environmental condition in possible automotive application has been maintained so that usability and durability of these new classes of fibers be preserved.

2.1 Fiber specification

Primarily two samples have been studied with change in simulated environmental conditions namely: temperature and humidity. Studied two samples have the followings specification:

- Sleeved ROHM GIMPOF having outer diameter 400µm (A type fiber).
- SM MPOF giving outer diameter 480µm (B type fiber).

Before environmental ageing, fiber dimension, i.e., diameter of core/cladding, d/ Λ (hole/spacing between hole) ratio has been measured under light microscopic examination.

2.2 Simulated Environment

MPOF in automobile will expose to elevated temperature, moisture, vibration and win flow moving past the automobile. During its operation, MPOF's performance will degrade with time. Typical adverse environmental conditions of MPOFs in automotive application are as follows:

- Temperature: -40°C~80°C, ± 0.5% variation[1]
- Humidity: 30~95RH%, ± 3% variation[1]
- Vibration frequency: 101~1500Hz[1,2]
- Air velocity: 0~4m/s[1]

2.3 Methodology

Accelerated ageing test has been devised to project future performance maintaining simulated environmental condition. This has been done in order to manipulate durability of MPOF at required performance level in automobile. International standard IEC 68-2-66 [18] has been used to carryout accelerated ageing developed by Tabai Espec, Japan. In this method, durability against degradation from temperature and humidity is evaluated using acceleration method. This is a function of chemical reaction and can be described by Arrhenius rate equation. External effect such as corrosion against moisture is avoided by using non-condensable option button from program set up menu in the environmental chamber equipment used for testing. Severity for this experiment has been devised to incorporate extreme operation conditions and resulting impact on light guiding capability of the MPOF. Severity schedule for the test fiber is given in the table 2. The exposure time in Table 2 has value that double for each temperature. The phenomena that are subject to this test are essential results of chemical reactions. From Arrhenius law of chemical reaction, it is clear that at constant attenuation (decrease in optical transmission), ageing time is inversely proportional to the absolute temperature (i.e., $t_A \propto \frac{1}{T}$, when decrease in optical transmission is constant).

Test cycle from beginning to end has been taken according to IEC 68-2-66 specification [18].

2.4 Experimental Set up

Ageing and degradation test on both A and B type sample has been carried out in Environmental chamber, "Radiometer" manufactured by vötsch Industrietechnik, Copenhagen, Danmark, model VC 4018 residing in sensor laboratory, Optical Fiber Technology Centre (OFTC), ATP. This environmental chamber has built in temperature, humidity and condensation protection system. Schematic diagram of experimental set up for ageing and degradation has been given in Figure 3.

Both A and B type fiber sample of approximately three meter length has been wound on a spool in three different slots and has been aged to temperature schedule given in column 2 of table 2. The exposure time is the maximum severity duration (III) but after exposure of the same fiber during severity

schedule (I) and (II), spool has been drawn out from the chamber for a very short period of time and has been cut off of approximately one meter length and the spool has been set back to the same chamber to be exposed during the rest of the time according to Table 2 schedule. This technique has been adapted to minimize fiber consumption for testing purpose. Fiber attenuation was measured by cut back method. In this method, an MPOF fiber of length L is connected to monochromatic NeHe laser light source that can emit wavelength of 633 nm. Collimating light beam from the fiber passes through objective lance, detector attached to an optical power meter and light power P_L is measured. The optical fiber is then detached from the receiver and cut back 0.1m beyond the receiver. Remaining fiber is connected to the receiver and light power emerging out of the fiber is measured again. Optical loss is then measured by the radio of the two power value divided by the length cut off of the fiber. A schematic of the power loss measurement is given in Figure 4.

2.5 Predictive Theory

On the basis of hypothesis that chemical reaction R is the underlying cause of the degradation of MPOF fiber, decay theory proposed by Arrhenius describes reaction rate taking into account reaction temperature according to the following rate equation:

$$\frac{dR}{dt} = A.e^{(\frac{-W}{k.T})}$$

Where, W is the thermal energy, k is the boltzman constant, T is the ageing temperature in K, A is a constant specific to the material and e is the base of natural logarithm.

For practical assessment of the thermal ageing process, this equation is set out and applied in the following from: [DIN ISO 2578: 1994]

$$t_A = A.e^{(\frac{B}{T})}$$

Where, t_A is the ageing time (h) up to a point of a given decline in transmission and A and B are constants.

Thus, in practice there is a power correlation between ageing temperature and ageing time required to effect a given decline in optical transmission. For a given decline in optical transmission, a plot of inverse temperature versus ageing time will give a moderate liner trend in semi logarithmic scale that can be used to predict durability at a given operating temperature for predefined criteria of ageing.

Logarithmic power ratio was used to measure optical power loss in MPOF fiber by cutback method according to the following formula:

$$Loss = 10.\log_{10}\frac{P_2}{P_L}$$

Where, P_L is the input power and P_2 is the output power of the fiber of specific length

A plot of cumulative length cut off versus cumulative relative power loss will give average loss in dB/m by measuring slope of the linear graph.

3. RESULTS AND DISCUSSION

Data has been generated on the basis of applied simulated environmental stress condition to predict durability of two types of fibers (A & B). Environmental condition included ambient to severe harsh environment to conduct enhanced ageing.

3.1 Qualitative Analysis

In this research project we generated lots of data in spectroscopic and gravimetric instrumentation. Some data have been reconciled to get good agreement with theory unless some adjustments have been made.

We used averaging technique to measure effective hole diameter in GIMPOF fiber according to the following formula:

$$d_{effective} = \frac{\sum_{i=1}^{k} n_i.d_i}{\sum_{i=1}^{k} n_i}$$

where n is the number of holes in a ring, d is the hole diameter in that ring and i is the number of rings around the core. Recalculated holes are assumed to be equally spaced around the central core region in hexagonal symmetry and that all holes in a ring are assumed to be same size [Figure 5].

Weight measurement has been done in a scale that can measure weight up to two decimal points down to gram scale. Power attenuation measurement has been carried out in a Optical Spectrum Analyzer (OSA) using two objective lances (10×0.25) and high intensity white light source that can emit visible light within the wavelength window of 400-1700nm.

3.2 Quantitative Analysis

Quantitative analysis in this project included dimensional analysis of the fiber (new and aged fiber of A and

B type) and graphical interpretation of data (gravimetric and spectroscopic). Details of dimensional analysis can be found in Table 3.

3.2.1 Effect of Ageing on Microstructure

Effect of ageing on microstructure was pronounced on the fiber (A & B type) under thermal ageing stress in presence of moisture at elevated temperature. Dominant effect was due to embedded micro-holes that run along the length of the fibers at different geometric orientation.

3.2.1.1 Hole expansion

Thermal expansion of embedded holes with propagation of time was observed. MPOF fiber, having thermal expansion coefficient $5.5\times10^{-4}K^{-1}$ and glass transition temperature 120°C got expanded since it been heated up with respect to reference temperature. That is taken as Australian typical summer temperature (25°C). As a result, original micro-holes (d) in both type of fiber got expanded. Since holes got expanded, density in micro-holes dropped off resulting contraction in spacing between holes. This results increase in density in solid polymeric region. This phenomenon can be described with conservation of volume-density relationship: $V.\rho = cons\tan t$. As a result d/Λ (d-hole diameter and Λ - spacing between hole) in aged fiber has increased with propagation of time at 100°C and 48.1RH%. From Figure 6 it is clear that d/Λ ration increases for both A and B type of fiber from 1.3 to 3.8 and 1.3 to 2.2 respectively. This difference in the ratio increase can be interpreted from local structural discrepancy between the fibers. A type of fiber has different size of holes embedded around the central core region where as hole in B type of fiber located in hexagonal symmetric orientation [Figure 1, 2]. As a result, local structural inhomoginity has caused more increase in d/Λ ration in A type fiber than that in B type fiber. This is because of different volume expansion between the two at constant temperature and relative humidity.

3.2.1.2 Defect in Polymer Side Chain along Hole

Side chain loosening occurred along the holes of MPOF fiber because of development of micro-cracks at the time of drilling and also during draw down process. This defect left week spots along polymer chain that could cause decomposition of the chain. Side chain is the lateral chain, for example, the substitutes that are connected to the main chains with low level of binding energy. For MPOF fiber there is particular risk of depolymerization. This term stands for splitting off of end groups and loosening of monomer component from the end chain. As the parts break off have unattached valencies – being known as free radicals – they try to form new combinations, for example with oxygen. This leads to oxidation and hydrothermal oxidation resulting in accumulation of degradation product as sub molecular debris (formic acid for instances via synthesis reaction). This consequences brittleness and disintegration with direct effect in mechanical and optical properties of the polymer optical fiber. This might be another cause of hole expansion and flaky structure in MPOF due to heating.

3.2.1.3 Asymmetric Hole Expansion

Hole expansion in A type fiber was greater than that in B type fiber because voidage $\left(\frac{V_{total}-V_{solid}}{V_{total}}\right)$ in A type of fiber was greater than that in B type of fiber in porous microstructural region. Nevertheless voidage in A type fiber varied along the length where as that in B type of fiber was constant along radial direction. As a result, rate of hole swelling and shrinkage at higher and lower temperature were not the same for A type fiber than that in B type fiber.

3.3.2 Effect of Ageing on Polymer Material

MPOF constituent material is made of synthetic organic PMMA. As a result vender wall force of attraction and bonding energy between constituent molecules and polymer chains are not as strong as inorganic silica material. This can be described by Gibb'e free energy of formation (ΔG_f^0) of both PMMA and silica. Gibb's free energy of formation of PMMA and free energy of formation of silica/quartz are approximately -65.6 and -190.4 Kcal/mole respectively at 25°C. This results in weak chemical stability in PMMA with respect to silica material. This Consequences splitting off of polymeric side chain and formation of degradation product in MPOF fiber under environmental stress condition.

3.3.2.1 Formation of Degradation Product

Thermal ageing can give rise formation of degradation product at elevated temperature in MPOF fiber. From figure 7, it is clear that formation of degradation product (R-OH, R-COOH) leading to increase in weight of MPOF fiber. This occurred due to thermal ageing. For more porous structure and weak tension surfaces, water vapor can get diffused and can

accumulate in polymeric matrix, leading to swelling of polymer chain. This can happen at relative lower temperature and at high relative humidity. In present of oxidant, oxidation reaction can dope hydroxyl group (-OH) in polymer chain and at the same time formic acid degradation product can accumulate in polymer matrix as sub molecular debris. This consequence is brittleness and loss of mechanical and optical properties of MPOF fiber. There is much evidence of oxidation reaction from observing –OH peak in –OH absorption and vibrational wave length window 700-850nm (Figure 8). At higher temperature, hydrothermal oxidation can occur leading to doping of carboxyl (-COOH)/Carbonyl (>CO) absorption peak in wavelength window of 1000-1100nm (Figure 8).

3.3.2.2 Underlying Chemical Transformation

Apart from possibility that water accumulate in polymer matrix, polymer material itself can take part in chemical transformation, This result in material degradation and accumulation of degradation product as sub molecular debris. Since polymer is quite inert in its present form and diffusion coefficient of water vapour in polymer is also small although at elevated temperature diffusion coefficient can increase in relation to material expansion, later possibility of getting chemical degradation due to thermal ageing in presence if humidification is more acceptable. One possibility is getting oxidation/substitution of side chain reaction at moderate temperature. This happens in presence of oxidant through coordination with unpaired valency electron of hydrogen attached to C-C side chain. This is due to splitting of methylene (-CH_2-) group from C-C side chain in PMMA's axial skeleton. This -CH_2- radical will then find oxygen molecule to pare up with formic acid which will accumulate in polymer matrix as sub molecular debris.

Another possibility is getting hydrothermal oxidation reaction at high temperature. This is sometimes called wet oxidation reaction. Here both oxygen and water vapour will react with polymer material. Since oxygen has higher potential in valency table than hydrogen, it will split hydrogen atom from the side chain of PMMA's axial skeleton resulting in doping of carbonyl group in C-C side chain and at the same time water vapour will from and will diffuse out of the material at elevated temperature.

(a)

$$\left[-C_5H_9O_2-\right]_n+\frac{3}{2}O_2\rightarrow\left[-C_4H_7O_3-\right]_n+HCOOH$$

MW(n=1)101 48 103 46

(b)

$$\left[-C_5H_9O_2-\right]_n+\frac{3}{2}O_2+2H_2O\rightarrow\left[-C_5H_7O_4-\right]_n+3H_2O$$

MW(n=1) 101 48 36 131 54

3.3.2.3 Characteristic of Weight Increase

From Figure 7, it is clear that weight increase from 0-21% in A type fiber at the same time this weight increase is 0-13% for B type fiber in between 0-96 hours of ageing at 100^0C and 48.1RH%. This weight increase can be described in terms of ageing at 100^0C and 48.1RH%. It can be interpreted in terms of chemical reactions stated in section 3.3.2.2. Since boiling point of formic acid is relatively high (101^0C), it will likely to adhere in polymer matrix and will not vaporize out of polymer material. So, if all molecular chains in MPOF take part in oxidation/substitution reaction, than maximum $\frac{149-101}{101}\times100\%$ or 50% in case of reaction (a) and $\frac{131-101}{101}\times100\%$ or 30% in case of reaction (b) weight increase is possible. This is due to oxidation/coordination substitution reaction between polymer, oxygen and water vapour in different synthesis pathways. We can predict from observing increased absorption peaks (in terms of increased attenuation in dB scale) in aged fiber in spectral attenuation that both type of reaction has occurred. This results weight increase in aged fiber.

From Figure 7 it is clear that, weight has increased linearly in both A and B type fiber in 25 hours of ageing at 100^0C and 48.1RH% after wards these curves get flattened. Stabilization of weight increase signifies saturation of degradation product formation in both type of fiber in 25-96 hours. After 96 hours of ageing we could not find any significance light guiding capability of either fiber. So, we can predict that beyond this period (96 hours) and above 100^0C and 48.1RH%, PMMA will no longer be able to withstand. This is because PMMA is chemically transformed due to ageing. At this particular stage, PMMA material will oxidize resulting degradation of optical properties. Since 50% decline in optical transmission is acceptable in optical fiber in most electronic device application, this extreme condition can be regarded as a limiting environmental condition for these particular A and B type of optical fiber during their application in electronic devices.

3.2.3 Spectral Attenuation in MPOF Fiber

Functional group absorbs light at a specific wavelength. In PMMA, carboxyl (-COOH)/carbonyl (>CO) groups are attached to axial skeleton of polymer chain. At different wavelength, these functional groups are excited to their vibrational state where surface electron are excited from lower orbital state to higher orbital state absorbing specific wavelength. When these electrons retard to ground state, they liberate excess absorbed energy in the form of radiation that is later on lost to the environment. Absorption of functional groups in MPOF fibers by white light (400-1700nm wavelength) was observed at different wavelength windows. In wavelength window 700-850, 850-1000 and 1000-1100nm –OH, ≡CH and –COOH/>CO group respectively get absorbed [23]. This was revealed by observing peaks in absorption spectrum (Figure 8). Structural inhomoginity in graded index A type (GIMPOF) fiber give rise more optical power loss than B type fiber (SMMPOF). Thermal ageing leading to decline in optical transmission was also observed in Figure 8, where negative sign signifies power loss.

3.3 Accelerated test

Plot of inverse ageing temperature and ageing time return moderate straight lines in exponential least square curve fitting technique. Nature of these curves is down hill slopes that satisfy good agreement between experimental data and theoretical trend of Arrhenius rate equation. Where chemical reaction is result of degradation product due to ageing at elevated temperature* in presence of humidification. At accelerated state, experimental data return empirical correlation: $t_A = 3.4382\times10^{-4} e^{(\frac{4179.5}{T(K)})}$ for test fiber. Following manner of this experiment, an acceleration factor of eight with respect to Australian average winter environmental temperature can be used $(12+24+48+96\cdots = a+ar+ar^2+ar^3\cdots)$. Taking this into account, our test fiber was durable (at projected ambient condition) nearly about three months in experimental case and that for theoretical case 17.5 months (for activation energy 39.5 KJ/mol [22]) for predefined criterion of ageing (i.e., time to 50% decline in optical transmission). A discrepancy between theoretical and practical data was however observed due to use of uncertain activation energy for PMMA. This varies a lot from literature to literature (in the range of 21.9 - 78 KJ/mol). Use of experimental data to evaluate pre-exponential factor of Arrhenius rate equation may also cause for this discrepancy. Hence by curve fitting, activation energy of 43.9 KJ/mol will give prefect coincidence between theory and practice.

3.4 Comparison of MPOF optical performance with required standards

An attempt was made to measure attenuation of test fiber by cutback method. Our test fiber exhibited loss nearly about 4dB/m where as requirement set by D2B, MOST, IEEE1394 standards are 0.433dB/m, 10dB and 0.072dB/m respectively [11]. This result encourages further development of this new class of MPOF fiber to minimize optical power loss for its application in integrated optics.

4. CONCLUSION

Ageing and degradation experimentation on MPOF fiber exhibits expansion behavior and formation of degradation product. This consequences loss of mechanical and optical integrity of the fiber. We find that total optical power loss is due to both mechanisms occurring simultaneously as mentioned above. However, dominant loss mechanism was due to chemical degradation which is observed by degradation product formation at elevated temperature in presence of humidification. Still considerable work need to be carried out to develop right type of cable sheath in order to incorporate this new class of fiber in its diverse potential areas of application and automotive multimedia network is one of them.

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TABLES

Table 1: Comparison of different standards for MPOF

Performance requirement	Standard		
	D2B	MOST	IEEE1394
Maximum LED power	-15.833dB	-11dBm	-8.5 to -2.1dB
Maximum MPOF attenuation	422dB/km	9.5dB	72dB/km
Range	7.56m	---	47m
System margin	5.28dB	9.5dB	---
Coupling loss	1.3722dB	2.6dB	---
Decoupling loss	0.32dB	2.6dB	---

Add-on coupler	1.3dB	---	---
Receiver sensitivity	-27.43dBm	-25.3dBm	-22.2dBm
User data range	5.29Mbit/s	21.25Mbit/s	118.1 Mbit/s
Temperature	-37 to +80^{0}C	-37 to +80^{0}C	-37 to +80^{0}C

Table 2: Severity

Conditions			Severity		
			Exposure time, h[3]		
	Temperature (^{0}C[1])	Relative Humidity, RH%[2]	I	II	III
A	100	~50	24	48	96

Table 3: Physical Examination of MPOF Fiber before and after Ageing

No.	Cond	Core dia, (µm)	Hole Dia, D (µm)	Spacing, Λ (µm)	d/ Λ	Hole expansion/ shrinkage	Fiber colour
1.	A new[4]	50	2.33	1.74	1.34	new	Transparent
2.	A[5]	47	3.48	1.74	2.0	+30%/sq of microstructured cane	Transparent
3.	A[6]	45	4.1	1.2	3.41	+40%/sq of microstructured cane	Slightly tarnished
4.	A[7]	44	4.65	1.2	3.88	+50%/sq of microstructured cane	Transparent
5.	B new[8]	3.15	0.9	0.7	1.28	new	Transparent
6.	A[5]	2.76	1..74	1.16	1.5	+40%/outer ring of holes	Transparent
7.	B[6]	2.36	1.75	0.87	2	+50%/outer ring of holes	Slightly tarnished
8.	B[7]	2.35	1.76	0.81	2.15	+60%/outer ring of holes	Transparent

FIGURES

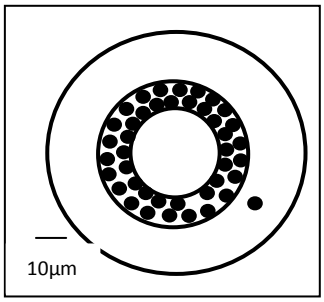

Figure 1: Schematic cross section of A type GIMPOF @ 400Ø

[1]±2^{0}C (in work place); [2]±5RH%; [3]0,+1.5h; [4]A type fiber: 100µm outer dia GIMPOF,
[5]Aged at 1000C & 48.1RH% in 25 hours; [6]Aged at 100^{o}C & 48.1RH% in 48 hours;
[7]Aged at 910C in 96 hours; [8]Btype fiber: 480µm outer dia SMMPOF

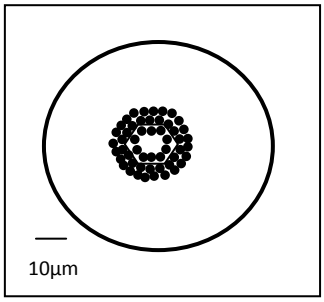

Figure 2: Schematic cross section B type SMMPOF @ 480Ø

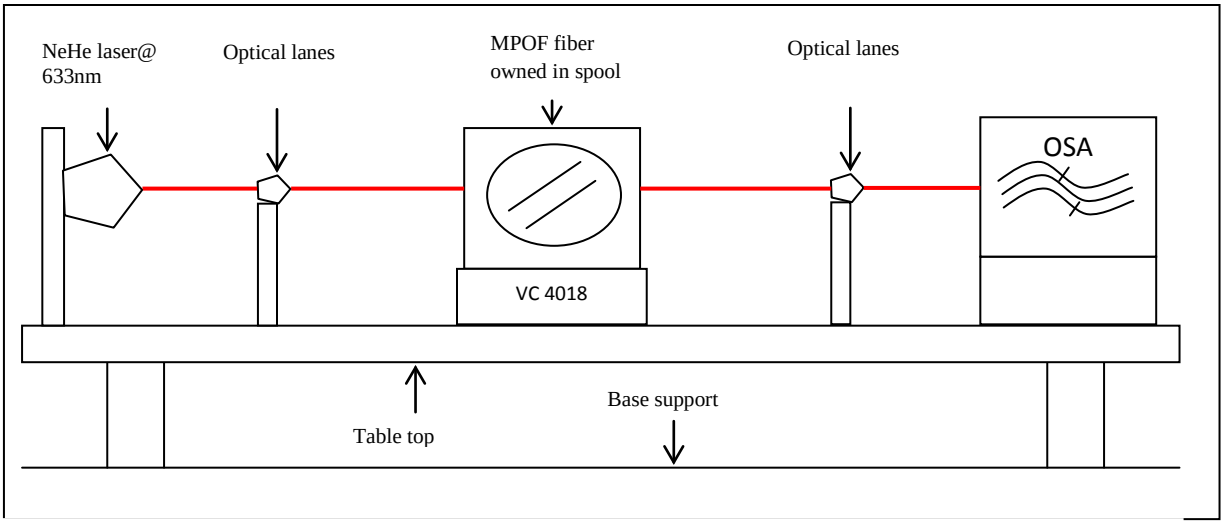

Figure 3: Schematic Diagram of Ageing and Degradation Experiment. Note That Due to Constraint Aging and Optical Loss Measurement Experiments were Carried Out Separately

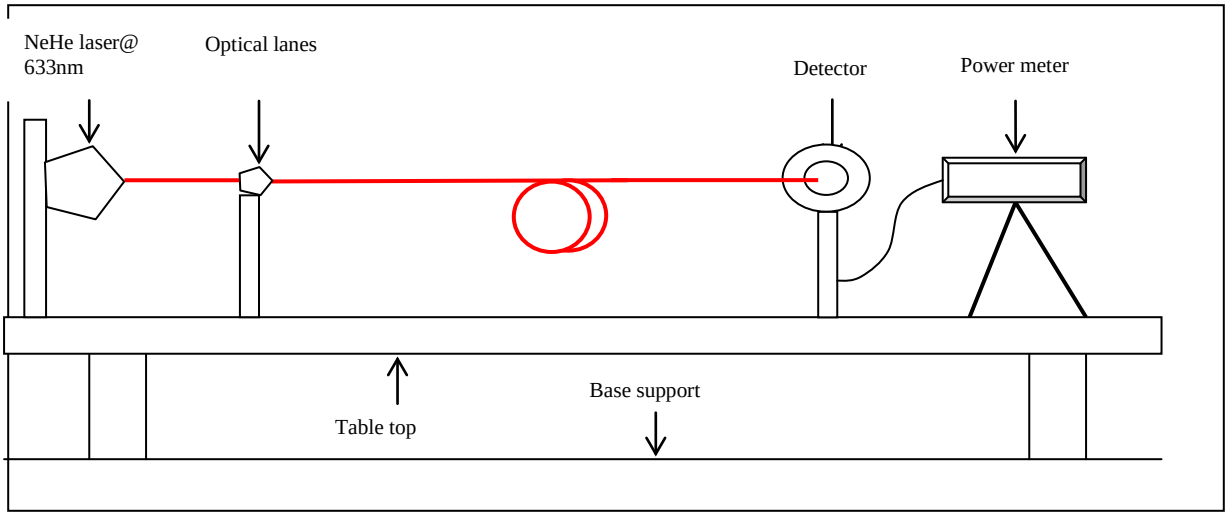

Figure 4: Schematic Side View of Experimental Setup for Optical Loss Measurement in MPOF Fiber

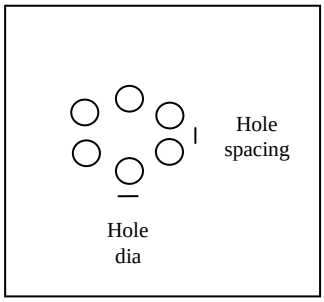

Figure 5: Dimension of Hole and Spacing

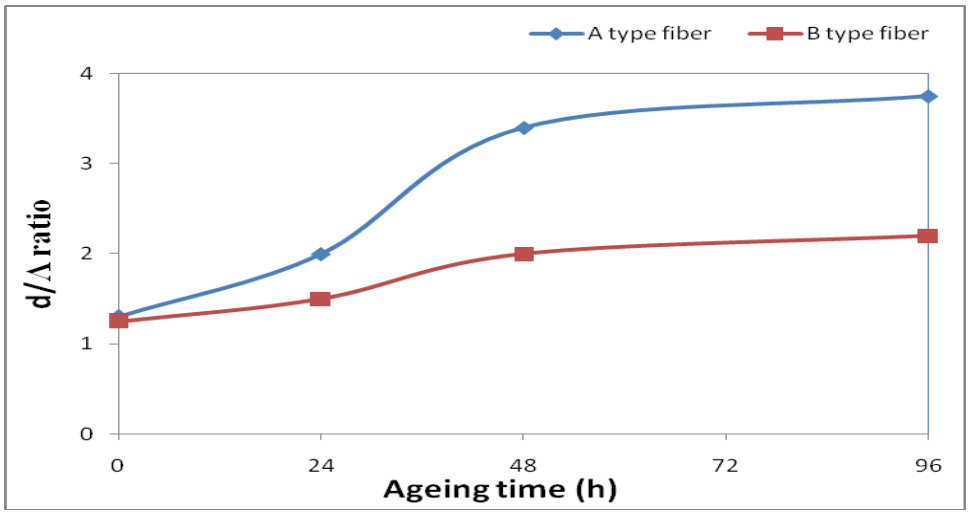

Figure 6: Effect of Ageing on Microstructure

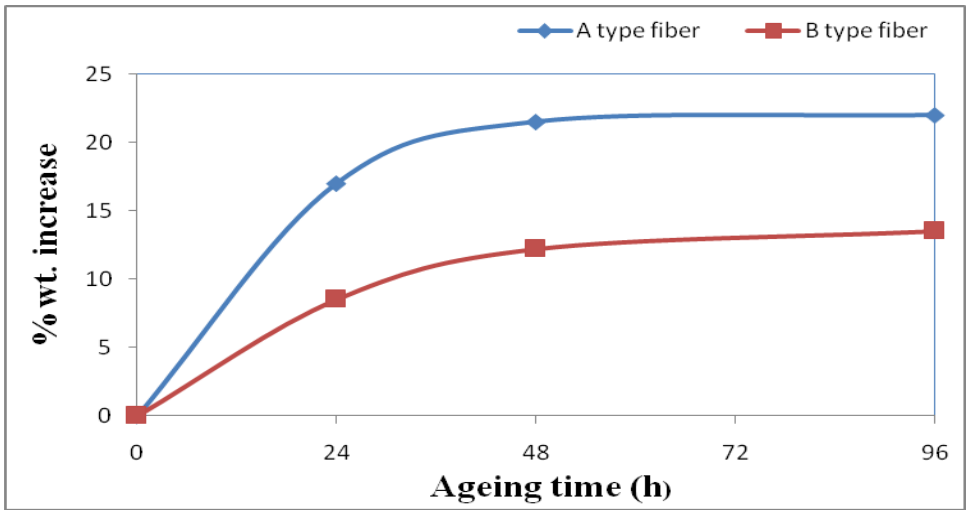

Figure 7: Effect of Ageing on Polymer Material

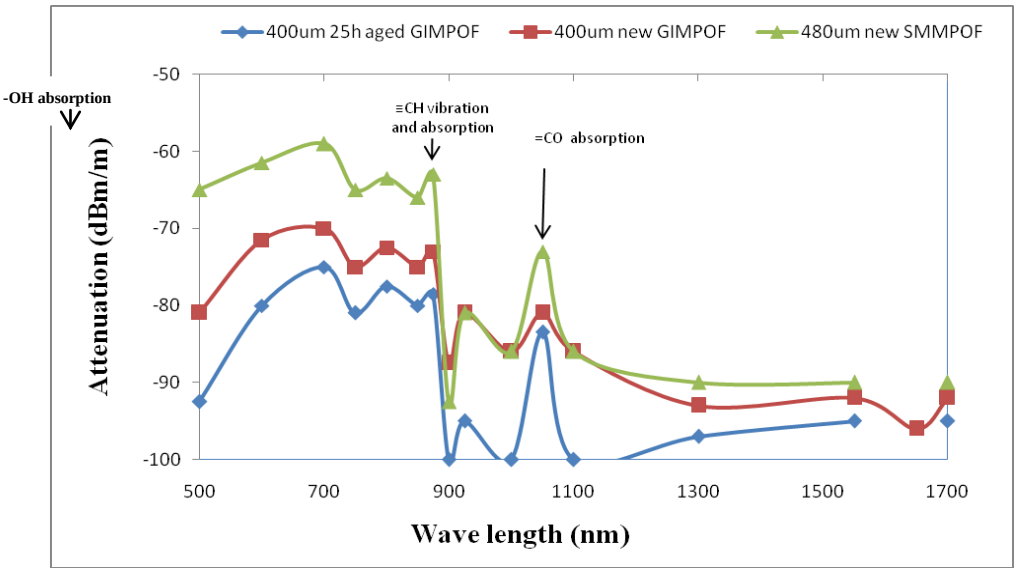

Figure 8: Effect of Ageing on Transmission Spectrum

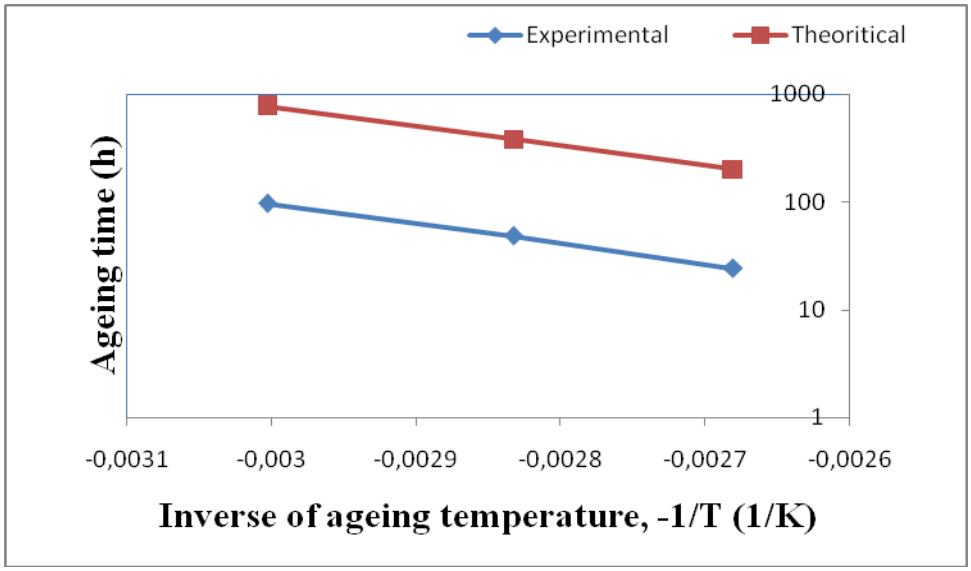

Figure 9: Ageing Time-Temperature for a given Decline (50%) in Optical Transmission for GIMPOF Fiber (400/50µm)