MJFAS MALAYSIAN JOURNAL OF FUNDAMENTAL AND APPLIED SCIENCES
PRINT ISSN: 2289-5981 | ONLINE ISSN: 2289-599X

RESEARCH ARTICLE

Fabrication of polycarbonate-based polymer optical fiber cladding: Effect of different solvents

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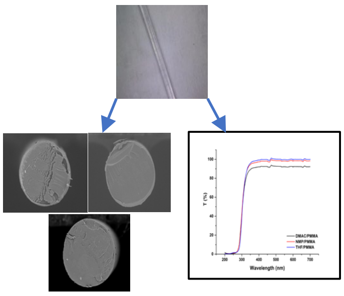
Article history
Received 5 January 2019
Revised 14 February 2019
Accepted 11 March 2019
Published Online 3 December 2019

Graphical abstract



Abstract

Normally, the cladding layer is fabricated by using heat through continuous extrusion, melt spinning, batch extrusion, and heat drawing technique. These techniques require high temperature precision in order to obtain the desired morphology of the cladding without compromising the quality of the polymer. Hence, in this study dip-coating method was utilized to coat the polycarbonate (PC) core with poly (methyl methacrylate) (PMMA) as a cladding part. The PC core was dipped into different cladding solutions using three types of organic solvent (i.e. Tetrahydrofuran (THF), N-Methyl-2-pyrrolidone (NMP), and Dimethylacetamide (DMAc) and subjected to post-treatment process. The thickness of fabricated cladding layer was ~10–15 µm for all coating solutions. The cladding prepared by THF exhibits transparent layer wrapping the core. However, the cladding layers for PMMA dissolved in NMP and DMAc showed translucent appearance. The THF/PMMA solvent displayed ~98 % transmittance at visible region which was higher than NMP/PMMA and DMAc/PMMA. The failure strain (3.6 %) and tensile strain (88.98 MPa) of THF/PMMA sample were higher compared to other solutions. Young's modulus which measures the stiffness and represents the breakability of a solid material was lower for THF/PMMA. Therefore, the THF is the most appropriate solution for fabrication of PC-based POF cladding layer.

Keywords: Polymer optical fiber, dip-coating, polycarbonate core, PMMA cladding

INTRODUCTION

Utilization of polymer optical fiber (POF) in sensor application has expended in the last few years due to its promising advantages such as immune to electromagnetic interference, low weight, and small dimension. Poly (methyl methacrylate) (PMMA) is one of the material that can be used for the fabrication of polymer optical fiber (POF). A research on the fabrication of POF sensor systems by using recycle PMMA material component in the electrical appliances by Prado *et al.* (2017) has showed that the fabricated fiber exhibits greater sensitivity and higher linearity than the available commercial POFs.

However, PMMA cannot be used in high operating temperature condition because it has low glass transition temperature of 105 ℃ (Zhong *et al.*, 2015). Leal-Junior *et al.* (2018) reported that the annealing and etching of PMMA-based optical fiber leads to the reduction of the mechanical strength of POF. In addition, the chemical and heat treatments also reduce the thermal expansion of the POF and caused a lower sensitivity of the PMMA fiber. High operating temperature in some application such as automotives, aircrafts, and electrical appliances demand selection of materials with high glass transition temperature (T_g). Therefore, various approaches have been developed to enhance the stability of the POF in a higher temperature condition. Doping the polymer with hydrophobic materials (Ishigure *et al.*, 2002) is one of the promising techniques to increase the stability of the POF. Based on a previous study, the result indicated that incorporating hydrophobic materials with PMMA increases the thermal stability of the fiber and decreases the absorption of water molecules while maintaining a low attenuation, even at high temperature and high humidity condition. In addition, copolymerization of 2,2,2-trichloroethyl methacrylate (TCEMA) and N-cyclohexyl maleimide (*c*HMI) as the core material has shown significant increment in the stability of POF (Nakao *et al.*, 2012).

Polycarbonate (PC) also has received a great attention from researchers to be used as the core material for the POF. PC has higher amorphous characteristic with high temperature resistance with similar characteristics as inorganic glass. Moreover, the arrangement of the polymer chains for PC is randomly oriented, therefore makes it highly amorphous structure and suitable for POF fabrication. Though the luminous transmittance for PC is almost similar to PMMA, PC exhibits a much higher glass transition temperature. Therefore, PC has been used in industrial applications where temperature is an important parameter. PC-based optical fiber was first introduced by Tanaka *et al.* (1988). They reported that the PC fiber has high thermal stability up to 130 ℃, high transparency and low attenuation loss which is 0.8 dB/m at 765 nm. Recently, Fasano *et al.* (2016) have fabricated PC-based optical fiber which was able to operate at temperature of 125 ℃ without resulting in malfunction.

Generally, fabrication of graded index POF can be divided into two processes namely continuous and discontinuous processes (Beckers *et al.*, 2015). In continuous process, the POF can be produced via several methods such as co-extrusion, dry spinning, and melt spinning with water quench. Whereas, in discontinuous technique, the POF is fabricated via at least two separate processes such as interfacial-gel polymerization, chemical vapour deposition centrifugation, diffusion, and photochemical polymerization. In this study, the fabrication of POF core and cladding were conducted through the discontinuous technique in which the core was fabricated via melt spinning while the cladding was fabricated using dip-coating technique. To the knowledge of the authors, no previous study was conducted on this technique to fabricate the cladding layer of a polymer optical fiber. This technique was used in this study due to its simplicity, cost-effectiveness, and ability to form a uniform cladding thickness. In the dip-coating technique, the formation of the cladding layer is influenced by few parameters such as type of solvent, dipping rate, curing time, and viscosity of the solution. In this study, the effect of different solvents on formation of cladding layer was investigated while other parameters were kept constant. Three types of solvent (THF, DMAc, and NMP) were chosen due to their ability to dissolve PMMA polymer and produce a clear coating layer after the solvent has evaporated.

EXPERIMENTAL

Materials

Polycarbonate was chosen to be used as the core material for the fabrication of optical fiber. We purchased a commercial polycarbonate (PC) pellet from Chi Mei Corporation (Taiwan) with purity of ≥ 99 % and the relative density (H_2O=1) is 1.20 g/cm^3. PMMA (*Sigma-Aldrich* with a density of 1.188 g/cm^3) was used as the cladding material. Tetrahydrofuran (THF) (*Sigma*-Aldrich, ≥99.9 % purity, molecular weight of 71.11), N-Methyl-2-pyrollidone (NMP) (*Sigma-Aldrich,* ≥99.5 % purity, molecular weight of 99.13), and Dimethylacetamide (DMAc) (*Sigma*-Aldrich, ≥99.5 % purity, molecular weight of 87.12) were used as solvents to prepare the coating solution for the cladding layer.

Extrusion of polycarbonate core

The POF core was extruded using our in-house single screw extruder with screw diameter of 25 mm. The core was produced according to the technique by Prado *et al.* (2016). The temperature was set at above 250 ℃ in order to obtain the right morphology for the POF. Prior the extrusion process, the PC granule was dried in the polymer dryer at 120 ℃ for 4 hours to eliminate the moisture content in the polymer which may cause unfavorable morphology of the POF core. Both screw speed and take up speed were set at 10 RPM. The melt extrusion machines used to fabricate the PC core is shown in Fig. 1.

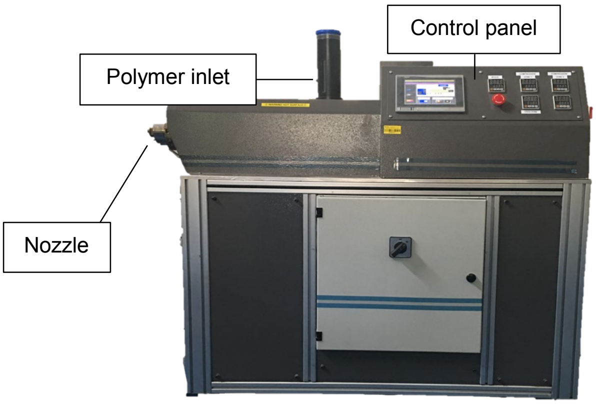


Fig. 1 Single screw melt extrusion machine.

Fabrication of cladding layer

The cladding layer was fabricated via the dip-coating technique. This technique was chosen due to its ability to produce coating layer with a uniform thickness through relatively simple approaches and does not require extensive equipment (Rout *et al.*, 2016). For the fabrication of the cladding layer, the polymer was first dissolved in different solvents namely THF, NMP, and DMAc. The mixture was stirred for 24 hours to obtain a homogenous solution. Prior to the coating process, the solution was ultrasonicated to remove the gas and bubbles within the solution. A POF core with a length of 15 cm was cut and dip-coated in the coating solution and then thermally cured in the oven for 24 hours at 40 ℃. The flow diagram for fabrication of the cladding layer is shown in Fig. 2 and the composition for each coating solution is shown in Table 1.

Table 1 Coating solution composition.

Solvent	Percentage (w/w)	Polymer	Percentage (w/w)
THF	80	PMMA	20
NMP	80	PMMA	20
DMAC	80	PMMA	20

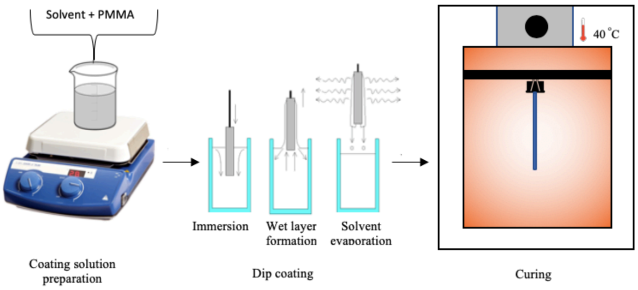


Fig. 2 Schematic diagram for coating process.

Characterization of the fiber

Scanning electron microscopy (SEM) was used to observe the morphology of the PC core and PMMA cladding. The sample was coated with platinum layer in vacuum condition and observed using Hitachi TM3000 microscope. Meanwhile, the mechanical behavior of the fiber was determined by the tensile test. The fiber sample was cut into small pieces with length of 15 cm. The loading rate used to test the mechanical behavior of the fiber was 1mm/min. The test was carried out using Instron 5566 at room temperature.

Another important aspect to observe in a fiber cladding is the transparency of the cladding layer. In the dip-coating technique, the cladding layer tends to form a cloudy appearance as the solvent evaporates during the curing period. Thus, the cladding layer was subjected to the light transmission test. The test was conducted by leaving the coating solution to dry on the surface of a glass and cured in the oven at 40 ℃ for at least 8 hours. The light transmission test was conducted using Shimadzu UV-3600 Plus at wavelength of 200 nm to 700 nm.

RESULTS AND DISCUSSION

SEM image for the PC core and cladding layer

Figure 3(a) and 3(b) show the cross section the SEM image and physical appearance of the core, respectively. The POF core exhibits high level of transparency with a very solid structure without any pore formation at any part of the fiber and the diameter is roughly around 464 µm. It is important to ensure that there are no bubbles within the POF core to reduce the extrinsic loss factors for the POF. The diameter of the POF is highly influenced by the take up speed during the extrusion process. In this study, the take up speed used was 10 RPM. A higher speed will produce a smaller fiber diameter.

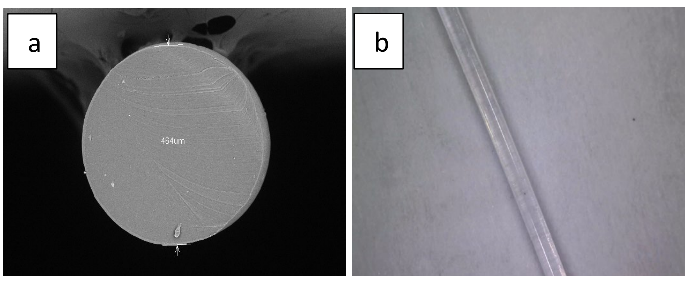


Fig. 3 (a) Sem image of the PC core; (b) Physical appearance of the PC core

After the dip-coating process, the cladding layer was observed by scanning electron microscope (SEM) as shown in Figure 4 to determine the thickness of the cladding. The cladding layers for DMAC/PMMA, THF/PMMA, and NMP/PMMA are shown in Fig. 4(a), Fig. 4(b), and Fig. 4(c), respectively. On average, the thickness of the cladding layer was around 5–10 μm as shown in Fig. 4(d). One of the factor that influenced the thickness of the cladding layer is the viscosity of the coating solution. In this study, the viscosity of the coating solution was roughly around 75–90 cp. Therefore, the right consistency of the coating solution must be obtained in order to have a uniform cladding thickness.

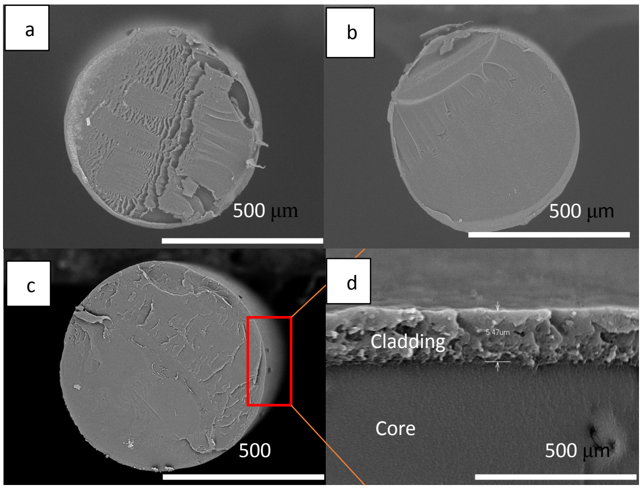


Fig. 4 Cross section images for the cladding layer (a) DMAc/PMMA; (b) THF/PMMA; (c) NMP/PMMA; (d) Cladding layer.

Mechanical properties of POF

Mechanical properties is an important factor to be considered for the fabrication of optical fiber. Application of optical fiber as a sensor is growing in the recent years. Therefore, it is important to determine this properties. For instance, fiber bragg grating (FBG) is depending on the mechanical deformation of the optical fiber to measure static or dynamic parameters like deformation, temperature or acceleration and thus it is vital to determine the mechanical properties of the optical fiber.

Table 2 shows the mechanical properties of the polycarbonate optical fiber with different cladding blending. It can be seen that THF/PMMA cladding has the highest failure strain which is 3.5 % and DMAc/PMMA has the lowest failure strain. The maximum tensile stress before it breaks also shows that THF/PMMA has the highest value. Thus, this indicates that PC coated with THF/PMMA has the strongest mechanical properties. This is probably because as the solvent evaporated, the polymer can change its state from amorphous to crystalline which influenced by the evaporation rate of the solvent (Pramod and Gangineni, 2015). Hence, in the event of the different solvents, THF has the lowest boiling point and highest evaporation rate. Thus, it remains in amorphous state whereas NMP and DMAc have relatively slower evaporation rate and the polymer molecules has longer time to rearrange its chain and form more crystalline microstructure. In comparison, an amorphous state polymer has higher flexibility and therefore THF/PMMA cladding has higher elongation before it breaks as compared to NMP/PMMA and DMAc/PMMA cladding. In this test also, in order to avoid slippage of the fiber from the grip, a masking tape was applied at the end of the optical fiber and was put in a U-shape. In addition to that, Young's modulus was also calculated at the slope of stress-strain curve. Young's modulus measures the stiffness of a solid material. In this study, PC coated with DMAc/PMMA has the highest Young's modulus value. This is also a much lower value than the silica based optical fiber which is around 80 GPa (Bundolo *et al*., 2014). The higher the value of Young's modulus the easier for the fiber to break when forced is applied (Jin and Granville, 2016).

Table 2 Coating solution composition.

Cladding	Failure Strain (%)	Tensile stress (MPa)	Young's Modulus (GPa)
THF/PMMA	3.6	88.98	2.47
NMP/PMMA	3.3	82.23	2.49
DMAC/PMMA	3.1	80.25	2.58

Light transmittance for the cladding layer

Determining the opacity of the cladding layer is necessary because opacity of the cladding layer can increase by different mechanisms such as rearrangement of the PMMA chains during solvent evaporations or thermodynamical stabilization. The thickness of the cladding layer also can affect the transparency of the cladding. Fig. 5 shows the dependence of light transmission of PMMA in different solvents. The optical transmission for all coating layer compositions is more than 90% at visible region in which shows a good transparency for a cladding layer. This high transparency is attributed to the phase dimensions of the solvent/PMMA blend which are well below wavelength of light and leads to high transparency. However, amongst the three blends, THF/PMMA shows better transparency for fabricating of the cladding layer. During the solvent evaporation process for fabrication of the cladding, the transparency of the polymer may be compromised. Therefore, any parameter that can accelerate the phase of inversion will lead to a higher transparency. Among the three types of solvent studied, THF has the highest evaporation rate. Thus, THF/PMMA has the highest transparency amongst the three blends. An optical fiber should has transparent appearance because it determines the extent of light loss. This is because, an opaque surface will tend to absorb the light when it travels in the optical fiber instead of reflected it back and will cause a large optical loss (Choi *et al.*, 2004).

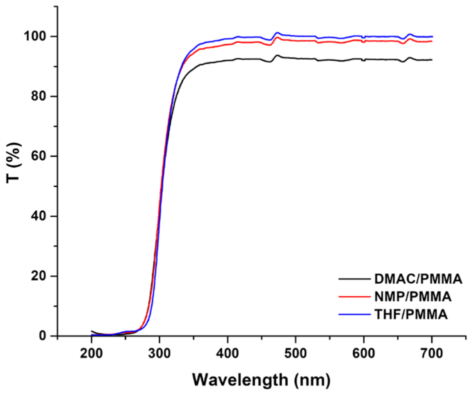


Fig. 5 Light transmittance for PMMA at different solvent.

CONCLUSION

PC based optical fiber core and cladding had successfully fabricated via our in-house extruder and dip coating technique respectively. The fiber was characterized and the results show that PC core coated with THF/PMMA is the most flexible due to its higher tensile stress and lower Young's modulus value compare to the other solutions. Besides that, though the transmission for all coating layer are more than 90 %, THF/PMMA has the highest light transmission percentage which is more than 98 % which indicate THF/PMMA blend is the most suitable candidate to be used as the cladding layer. Furthermore, the technique to produce the cladding layer is straightforward and less complex approach which can be utilized to produce polymer optical fiber.

ACKNOWLEDGEMENT

The authors gratefully acknowledge technical and management supports from the Research Management Centre (RMC), Universiti Teknologi Malaysia and financial support from Universiti Teknologi Petronas (Project Number: R.J130000.7609.4C112). The authors also would like to acknowledge the contributions from Alpha Matrix and financial support from the Petroleum Research Fund (PRF) (0153AB-A32).

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