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Fluorescence characterization and gain studies on a dye-doped graded index polymer optical-fiber preform

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Abstract

Preparation of an appropriate optical-fiber preform is vital for the fabrication of graded-index polymer optical fibers (GIPOF), which are considered to be a good choice for providing inexpensive high bandwidth data links, for local area networks and telecommunication applications. Recent development of the interfacial gel polymerization technique has caused a dramatic reduction in the total attenuation in GIPOF, and this is one of the potential methods to prepare fiber preforms for the fabrication of dye-doped polymer-fiber amplifiers. In this paper, the preparation of a dye-doped graded-index poly(methyl methacrylate) (PMMA) rod by the interfacial gel polymerization method using a PMMA tube is reported. An organic compound of high-refractive index, viz., diphenyl phthalate (DPP), was used to obtain a graded-index distribution, and Rhodamine B (Rh B), was used to dope the PMMA rod. The refractive index profile of the rod was measured using an interferometric technique and the index exponent was estimated. The single pass gain of the rod was measured at a pump wavelength of 532 nm. The extent of doping of the Rh B in the preform was studied by axially exciting a thin slice of the rod with white light and measuring the spatial variation of the fluorescence intensity across the sample.

Keywords: Graded index (GI); Preform; Interfacial gel polymerization

1. Introduction

Graded-index (GI) polymers have recently attracted considerable attention as a result of their highly promising potential in optical fiber communications [1]. Although the loss in polymer optical-fiber (POF) is still much higher than that in silica optical fiber, recent progress has led to the development of polymer-based POF with a loss less than 100 dB/km, low enough for many short-range applications [2]. The vast majority of optical amplifiers are based on an optical fiber doped with a fraction of a percent of the rare-earth element erbium. Although rare-earth doping has been generally used in silica, many laboratories have been working to develop stable rare-earth-doped polymer lasers and amplifiers. The main issue with rare–earth-doped polymer lasers and amplifiers has been the pumping inefficiencies due to the de-excitation of the excited states caused by the IR absorption in the polymer. In bulk form, polymer hosts impregnated with certain dyes have now achieved 80% conversion efficiency, from pump power to signal power, with tuning range close to those in solution. Dye-doped POFs can be made into useful fiber amplifiers and lasers that operate in the visible region [3–6]. Optical amplifiers and lasers made of dye-doped fiber require much less pump power than in bulk material, because of the effective confinement and long interaction length available in the fiber. Since photo-bleaching increases with the increase of the exposure intensity, low pump intensity would increase the lifetime of the gain medium. Also, the thin and long geometry of the fiber is ideal for good thermal relaxation to minimize the thermally induced photo-bleaching as well [7]. In this paper, we are presenting the details of fabrication of a dye-doped graded-index

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polymer optical fiber (GIPOF) preform by interfacial gel polymerization and its characterization.

2. Theory and experiment

The dye-doped GIPOF preform was prepared by the well-known interfacial-gel-polymerization technique [8]. The monomer was purified as follows: inhibitors in the monomer were removed by rinsing with a 0.5-N-NaOH aqueous solution, followed by repeated washing with distilled water to remove the remaining NaOH. The monomer was dried over $CaCl_2$, distilled under reduced pressure and filtered through a 0.2 μm membrane filter. Initially a hollow polymer tube was prepared. There are many methods that may be used for the polymer tube fabrication. The first method is to rapidly rotate a glass tube containing the monomer using centrifugation (3000 rpm) in a furnace at 70 °C, which results in a polymethyl methacrylate (PMMA) tube after polymerization [9]. The second method is the "hole in rod" technique in which a hole is drilled into the cladding polymer rod [10]. The first method requires specialized equipment and the second one results in a preform with large scattering losses, since the core cladding interface may not be smooth because of the drilling. We have used the Teflon technique for the PMMA tube fabrication [11]. In this technique, we use a Teflon rod of diameter 6 mm that is properly fixed at the center of a glass tube of inner diameter 13 mm. One end of the tube is sealed and then it is filled with a mixture of monomer (MMA), 0.4 wt% of the polymerization initiator benzoyl peroxide (BPO) and 0.1 wt% of the chain transfer agent (*n*-butyl mercaptan). The thermal polymerization of the filled tube is carried out in an oil bath where its temperature is properly controlled. After the monomer was fully polymerized and heat treated, the Teflon was removed and we obtained a polymer tube. The non-sticking property of Teflon, as well its chemical and thermal stability, results in a smooth finish to the inner surface of the tube. The uniformity of the inner diameter of the tube was tested by making slices from the different parts of the rod. No non-uniformity was observed in the diameters, verifying the reliability of this technique.

Next, the polymerized tube was heat treated at 100 °C in an oven for 24 h. The bottom of this tube is sealed and the hole is then filled with MMA, 0.4 wt% of BPO, 0.1 wt% of *n*-butyl mercaptan, 5 wt% of a high-refractive-index organic compound, diphenyl phthalate (DPP), the laser dye Rhodamine B (Rh B) (10^{-5} ml) and 1 wt% of dimethyl sulfoxide (DMSO). Here DMSO enhances the solubility of Rh B in MMA [12]. The filled tube was placed in an oven at 80 °C for 24 h and at 90 °C for another 10 h. Here the inner wall of the polymer tube is slightly swollen by the monomer and a gel phase is formed in this region. Since the rate of polymerization reaction inside the gel is faster than that in the monomer liquid owing to the gel effect, polymerization occurs on the inner wall of the tube. Due to the selective diffusion process, the dopant molecules are gradually concentrated along the axis of the tube [9]. Finally, the contents of the polymer tube are solidified up to the central axis. After this a heat treatment was carried out at 110 °C for 24 h to yield the desired preform.

To obtain any particular refractive index profile along the radial direction of a fiber core, a preform with the desired profile can be employed. The preform is a cylinder of polymer whose refractive index distribution can be made to coincide with that desired for the core of the POF. Interferometry is an accurate method for determining the refractive index profile of both preforms and fibers [13,14]. We have measured the refractive index profile of the solid rod using the slab method, where a thin slice of the preform was cut, polished and kept in one of the arms of a Mach–Zender interferometer. The light passing through the slab undergoes a phase shift, which depends on the optical path length. The fringe displacements for the points within the central portion of the disk are then measured with respect to the parallel fringes outside the core region. The difference in refractive index between various points in the core and cladding region can be calculated from the fringe shift $S(r)$ and the parallel fringe spacing D (in the cladding) according to the relationship [15].

$$n(r) - n_2 = \frac{\lambda S(r)}{Dd}, \tag{1}$$

where λ is the wavelength of the measuring light and d is the slab thickness. The parameter $S(r)$ is the central field deviation at a distance r measured from the baseline connecting the same cladding fringe at both sides of the core as shown in Fig. 1.

The profile parameter α of the rod was also calculated from the expression [16,17] for the variation of refractive index $n(r)$ with the distance from the axis for GI fibers:

$$n(r) = \begin{cases} n_0\left[1 - 2\Delta\left(\frac{r}{a}\right)^{\alpha}\right]^{1/2} & \text{for} \quad r \leqslant a, \\ n_0[1 - 2\Delta]^{1/2} & \text{for} \quad b \geqslant r \geqslant a, \end{cases} \tag{2}$$

where $n(r)$ is the refractive index at radius r, a is the core radius, b is the radius of the cladding, n_0 is the maximum

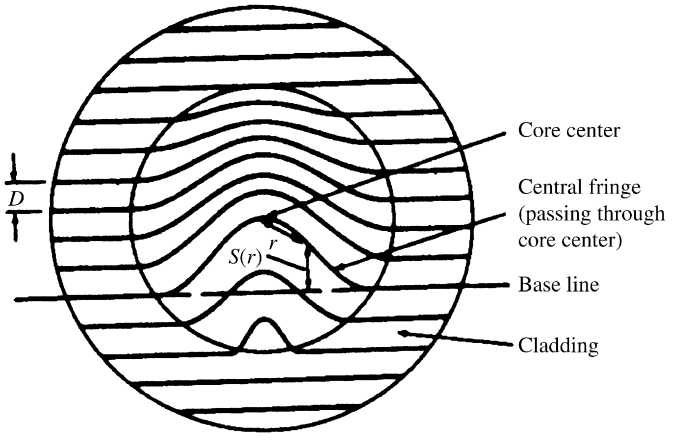

Fig. 1. Displacements of locus of the equal phase shift for different points in the slice.

value of refractive index along the axis of the core and Δ is the relative refractive index difference given by the expression [17].

$$\Delta = \frac{(n_0^2 - n_c^2)}{2n_0^2} \approx \frac{(n_0 - n_c)}{n_0}, \tag{3}$$

where n_c is the refractive index of the cladding. The value of α for the rod was found as the slope of the graph between $\ln(r/a)$ and $\ln g(r)$. where

$$g(r) = \frac{1}{2\Delta}\left(1 - \left(\frac{n(r)}{n_0}\right)^2\right). \tag{4}$$

The length of the preform fabricated was 3.5 cm and its diameter was 1.3 cm. Fluorescence emission was monitored using a (frequency-doubled) diode pumped solid-state (DPSS) laser emitting at 532 nm as the excitation source. The laser was mounted on a translational stage and the preform was kept stationary. The technique of side illumination fluorescence was used for recording the fluorescence spectrum. The laser beam was focussed using a convex lens along the axis of the preform and various points, and the fluorescence signal was collected from one end face of the preform [18]. At each point of illumination, the fluorescence spectrum was recorded using an Acton Spectrapro-500i spectrograph coupled with a CCD camera having a resolution of 0.03 nm.

The single pass gain measurement using the ASE method proposed by Shank et al. [19] is applied to calculate the gain in the polymer rod. The second harmonic output from an Nd:YAG laser was focused by a cylindrical quartz lens of focal length 5 cm on the rod. A 2-mm-thick aluminum sheet mounted on a micrometer arrangement served as the beam block. The rod was kept in a slanting position to avoid feedback and the output was recorded using an Acton Spectrapro-500i spectrograph coupled with a CCD camera. The dye-laser intensities I_1 and I_2 for the two lengths l_1 and l_2 of the active medium were measured for different pump intensities. The gain per unit length $G(\lambda)$ of the dye-doped polymer rod was determined using the Newton–Raphson numerical method:

$$\frac{I_1(\lambda)}{I_2(\lambda)} = \frac{[(\exp(G(\lambda)l_1) - 1]}{[(\exp(G(\lambda)l_2) - 1]}. \tag{5}$$

3. Results and discussion

Using the interfacial-gel-polymerization technique, a dye-doped graded index polymer fiber preform was fabricated. Fig. 2 shows the photograph of the preform.

A thin slice of the polished preform was prepared and placed in one of the arms of a Mach–Zender interferometer. The shift in the fringes for various positions of the slice was found by taking a photograph of the fringe pattern. Fig. 3 shows the photograph of the central portion of such a pattern obtained for a sample of thickness 1.19 mm.

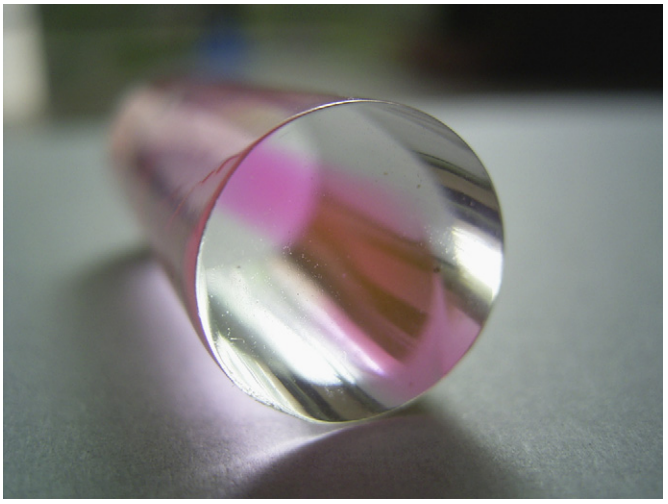

Fig. 2. Dye-doped graded index polymer rod.

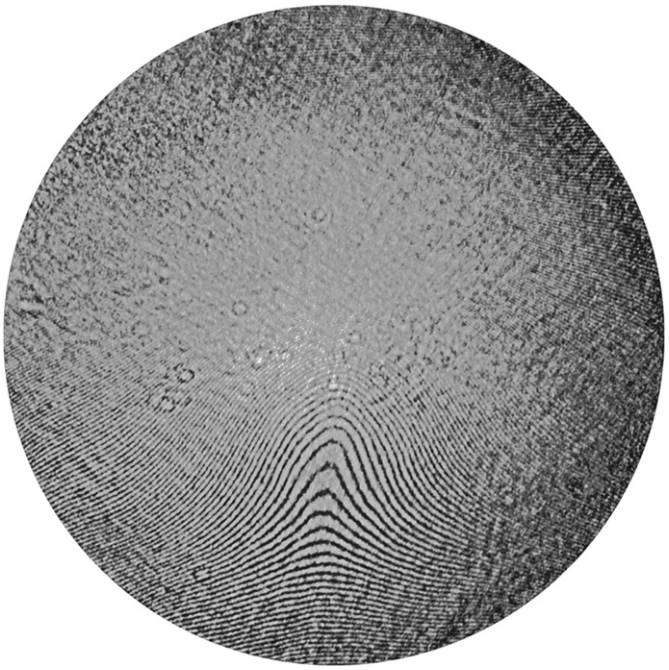

Fig. 3. The photograph of the fringe pattern.

Fig. 4 shows the transmitted fluorescence light as a function of the propagation distance through the preform. It is seen that the output intensity decreases with the propagation distance. It was also observed that the peaks exhibit a red shift of about 10 nm while varying the point of excitation through a distance of 3.2 cm.

The red shift of the fluorescence signal is produced by the self-absorption of the dye due to the overlapping of the absorption and fluorescence spectra of the Rh B. After a distance of 3.2 cm, the red shift shows saturation.

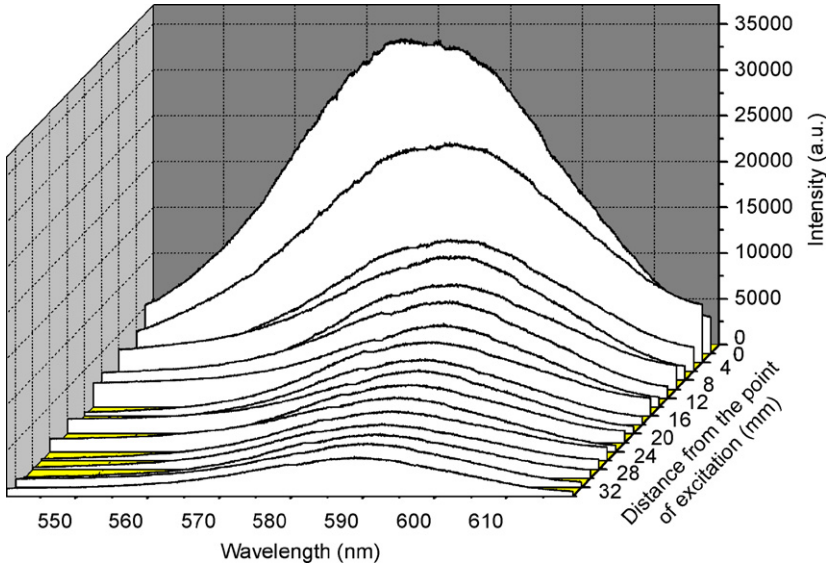

Fig. 4. Transmitted fluorescence light using side illumination.

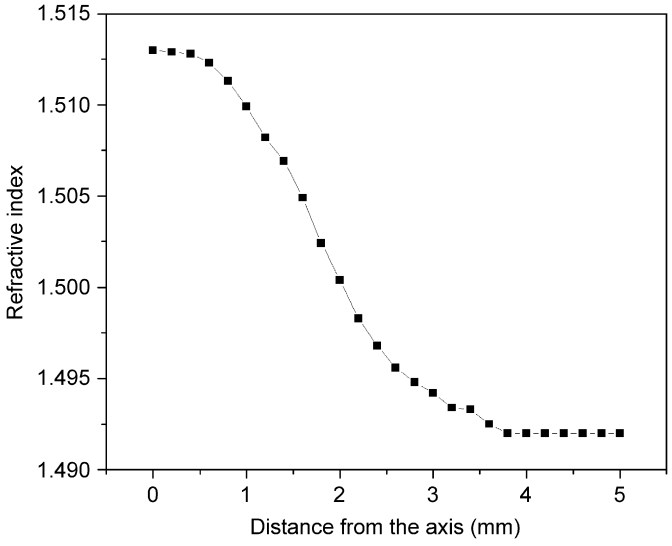

Fig. 5. Refractive index profile of the rod.

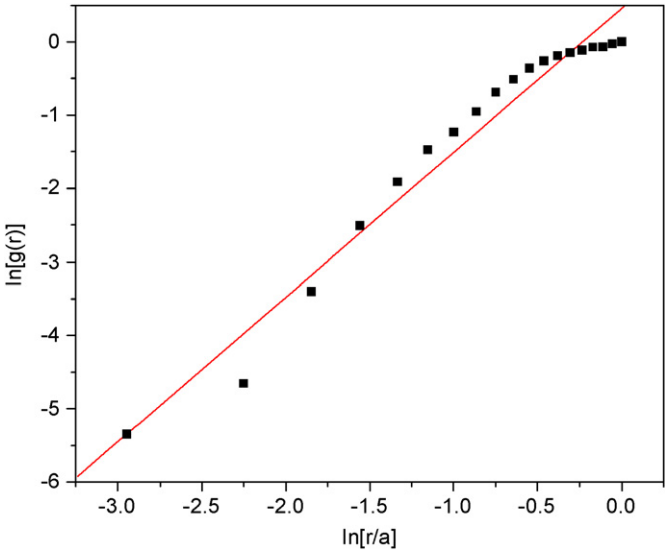

Fig. 6. Log–log plot of $g(r)$ vs r/a.

Fig. 5 shows the refractive index profile of a typical specimen of the fabricated preform. It was found that the maximum difference in the refractive indices between the core and the cladding is 0.021 ± 0.002, and it occurs along the axis of the preform and gradually decreases towards the cladding. Fig. 6 shows the curve obtained for the evaluation of the profile parameter. The curve is very close to a straight line up to an r/a value 0.47 and the profile parameter is 1.96. For higher values of r, there is a considerable reduction in the slope and the profile parameter varies much from 2. This drastic variation in the refractive index gradient can be attributed to the formation of aberrations at the inner wall of the polymer tube during the interfacial-gel-polymerization. This gives us an idea of the actual diameter of the graded index polymer rod that can be used as a good preform. The outer regions of the rod can be removed by machining before the fiber is drawn.

Fig. 7 shows the pump power dependence of the gain of the rod. From the figure it is clear that at lower pump powers the gain of the preform shows a linear increase, while above 0.04 W the gain tends to saturate.

After proper calibration, the spatial variation of the doping concentration of the dye Rh B was also studied. The axial excitation of a thin slice of the rod using white light and the measurement of the fluorescence signal at 590 nm at various points showed maximum fluorescence along its axis. Fig. 8 shows the fluorescence-intensity variation across the dye-doped region. The result indicates that the maximum extent of doping of the dye molecules occurs along the axis of the rod.

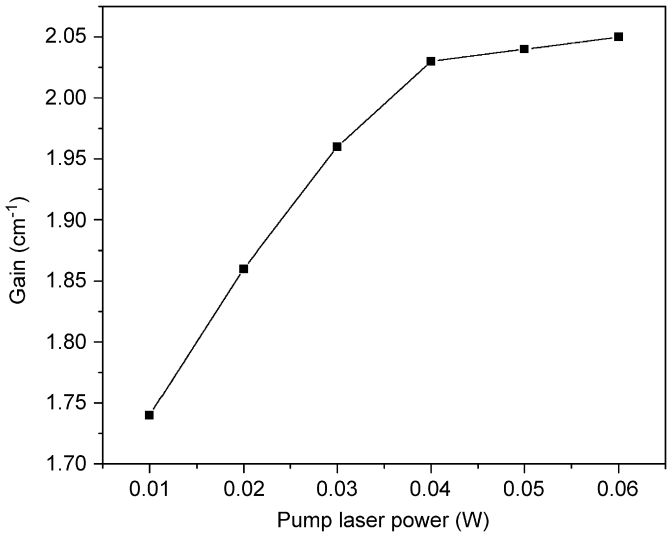

Fig. 7. Variation of gain with Nd:YAG laser power.

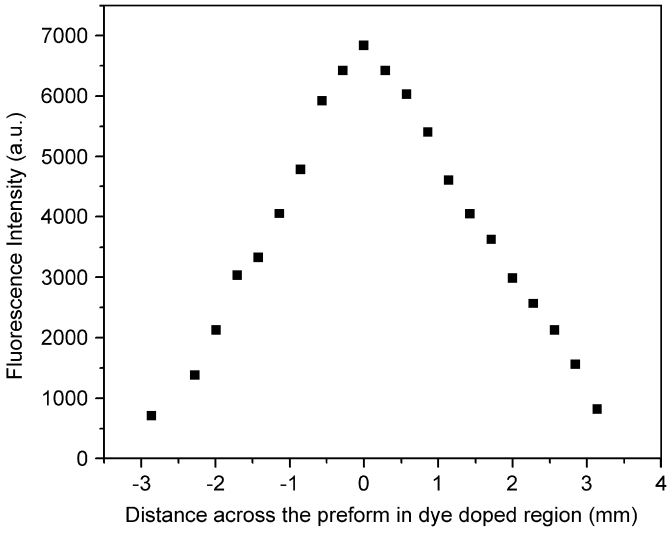

Fig. 8. Fluorescence intensity variation across the dye-doped region of the preform.

4. Conclusions

A graded index polymer optical-fiber preform was fabricated using the interfacial-gel-polymerization technique, and its refractive index profile was measured using the interferometric technique. It was seen that the refractive index varies from 1.492 in the cladding to 1.513 at the axis. The profile parameter of the preform was found to be 1.96 for the inner 47% of the GI region. This suggests that one can fabricate a dye-doped fiber GIPOF using the above kind of preforms. Such dye-doped optical fiber can be employed to develop fiber amplifiers in the visible region. The fluorescence characterization of the preform shows that it exhibits a red shift of about 10 nm, while varying the point of excitation through a distance of 3.2 cm. It was also seen that the dye concentration and the fluorescence intensity were maximum along the axis of the preform. The pump power dependence of the gain of the rod shows that the gain of the rod tends to saturate above 0.04 W.

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