### Fabrication and Amplification of Rhodamine B-Doped Step-Index Polymer Optical Fiber

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ABSTRACT: A step-index polymer optical fiber (SI POF) containing Rhodamine B in poly(methyl methacrylate) (PMMA) has been fabricated by a preform technique. Fluorescence of different fiber lengths were observed and discussed. A high gain (23 dB) for a SI POF with 60-cm length, 400- $\mu$ m diameter was obtained. The Rhodamine B content of the doped SI POF is 5 ppm-wt. The signal wavelength providing the highest gain for a 60 cm SI POF is around 630 nm, and the optimum fiber length is about 60 cm at 10 kW launched pump power. © 2004 Wiley Periodicals, Inc. J Appl Polym Sci 93: 681–685, 2004

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### **INTRODUCTION**

Polymer optical fibers (POFs) have attracted much attention in past decades because POFs have some unique characteristics, such as flexibility, they are easy to handle, and have a relative low cost in coupling due to their large-core diameter.<sup>1,2</sup> These characteristics make them especially suitable to be a transmission medium in local area networks (LANs) and short-haul optical communications. With the development of POF, increasing research activities have been also carried out in the field of active polymer optical fibers, most of which aim at making polymer optical fiber amplifiers or lasers.<sup>2-7</sup>

Organic dye-doped polymers have been widely investigated as gain media in solid-state dye lasers.<sup>6-10</sup> Dye molecules that have large absorption and induced emission cross sections due to allowed  $\pi$ - $\pi$  transitions are ideal active dopants for the generation and amplification of intense light pulse.<sup>2</sup> The first optical amplification of dye-doped POF was reported in 1993.<sup>7</sup> The maximum gain of 27 dB was achieved at 591 nm

wavelength by using a dye-doped gradient-index (GI) POF. However, the commercial POFs at present are step-index (SI) POFs, and the low loss window of poly(methyl methacrylate) (PMMA) POF is around 650 nm, so we present a technique of fabricating SI POF and report the experimental results of optical amplification using a Rhodamine B doped SI POF.

In this article, a SI POF containing Rhodamine B in poly(methyl methacrylate) (PMMA) has been fabricated by a preform technique. A high gain (23 dB) of a SI POF with 60-cm length, 400-µm diameter was observed. The Rhodamine B content of the doped SI POF is 5 ppm-wt. The optimum fiber length is about 60 cm at 10 kW launched pump power. The signal wavelength providing the highest gain for a 60 cm SI POF is around 630 nm, which is closer to the low loss window of PMMA POF.

### **EXPERIMENTAL**

### Materials

Rhodamine B was purchased from Acros Co. Ltd. Methyl methacrylate (MMA) was purified according standard procedures. 2,2-Azoisobutyronitrile to (AIBN) was recrystallized from methanol before use. Other chemicals were analytical grade.

### Preparation of Rhodamine B-doped PMMA and SI POÊ

Rhodamine B-doped PMMA was made by bulk polymerization. First, 15 mL purified MMA, 0.02 g 2,2-

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Azoisobutyronitrile (AIBN) as an initiator, 40  $\mu$ L 1-butanethiol as a chain transfer agent, and a specified amount of Rhodamine B dimethyl sulfoxide (DMSO) solution were mixed in a vessel. The above solution was filtrated through a microporous filter (d = 0.20  $\mu$ m) before being injected into a model, and then the thermal polymerization of the filled model was carried out in a water bath at 50°C for 48 h under 6 atm nitrogen and additionally heated at 75°C until solidification was fulfilled.

To fabricate Rhodamine B-doped SI POF, the preform can be also made by the procedure of bulk polymerization described above. The preform with a diameter of 10 mm prepared by this process was then heat-drawn into an optical fiber at 170°C by a taking up spool. The diameter of the Rhodamine B doped SI POF can be controlled by adjusting the ratio of the preform moving velocity and driving roll velocity. The cladding (GUV 1051 resin with low refractive index of 1.4105) was coated during the drawing process.

### Thermal gravimetric analysis of Rhodamine Bdoped PMMA

Thermal stability of the materials was studied by thermal gravimetric analysis (TGA) using a TGA-50H Shimadzu thermogravimetric analyzer, under nitrogen atmosphere, at heating rate of  $10^{\circ}$ C min<sup>-1</sup>.

# Measurement of spectra of Rhodamine B-doped PMMA and SI POF

The absorption spectrum of Rhodamine B-doped PMMA was recorded on a Shimadzu UV-2401 UV-Vis spectrophotometer. The fluorescence emission spectra of Rhodamine B-doped PMMA was recorded on a Shimadzu RF-5301PC spectrofluorophotometer.

The measurement of fluorescence spectra of the doped SI POF was carried out as follows: the pump beam from an Ar<sup>+</sup>-laser operating at 457.9 nm wavelength was launched into a doped SI POF by a microscope objective. The fluorescence spectrum at the end of the POF was collected by a convex lens and recorded by a spectrum analyzer.

## Experimental arrangement for optical amplification of Rhodamine B-doped SI POF

The experimental arrangement for optical amplification of Rhodamine B-doped SI POF is shown in Figure 1.

The second harmonic at 532 nm form a 1064 nm YAG laser, which is used as the pump source. The input signal light from 610 to 645 nm is obtained from the tunable output of a dye cell, which is also pumped by the second harmonic at 532 nm from the same YAG



**Figure 1** Experimental arrangement for optical amplification. BS, beamsplitters; DC, dye cell; ML, mirror; AT, attenuator; MO, microscope objective; MONO, monochromator; PD, photodiode; OSC, oscilloscope.

laser. The full-width at half maximum of both signal and pump pulses is about 10 ns.

The pump light and the signal light were combined by a beamsplitter and were coaxially launched into the doped SI POF through a microscope objective. An optical attenuator was used to adjust the intensity of the pump light. The delay of the optical path of the pump light was about 1.9 m, to keep the synchronization of the pump light and the signal output form the dye cell. The output of the signals from the SI POF was detected by a photodiode connected to a digital oscilloscope.

### **RESULTS AND DISCUSSION**

### Fabrication of Rhodamine B-doped PMMA perform

MMA has relatively low polarity, and many of the laser dyes show low solubility in it. In the case of Rhodamine B, it is insoluble in MMA,<sup>11</sup> but the addition of a high-polarity agent can enhance the solubility of the dyes in MMA. Here, DMSO was selected as the agent for its high polarity (3.9-D dipole moment) and high boiling point (189°C). The high boiling point is important, because the temperature in the fiber drawing process is about 170°C.

As to the fabrication of the preform, the polymerization was carried out under pressure. Bulk polymerization of methyl methacrylate is a highly exothermic one. Because viscosity increases early in the reaction, the problem of heat transfer leads to difficulty in control and a development of localized "hot spots" and "runaways."12 This may result in undesirable mechanical properties. To reduce heat evolution and avoid autoacceleration (also referred to as Trommsdor effect) that takes place during the polymerization, in general, a syrup of partially polymerized methyl methacrylate is used. However, we have found even in this way, the formation of bubbles cannot be prevented. It is a serious problem in fabricating polymer optical fiber preforms, especially in the drawing process of the fiber. So we adopted another method to solve the problem. The polymerization was carried



**Figure 2** TGA traces of PMMA and Rhodamine B-doped PMMA (the concentration of Rhodamine B is 5 ppm-wt).

out in a cylinder under pressure. At the same time, the amount of initiator and the reaction temperature should be properly controlled. It is very effective to prevent bubbles formation even without prepolymerization. A preform fabricated in this way shows good processability in fiber drawing.

TGA traces of PMMA and Rhodamine B doped PMMA (the concentration of Rhodamine B is 5 ppm-wt) are shown in Figure 2.

According to Figure 2, the degradation temperature for Rhodamine B-doped PMMA is about 180°C, while the pure PMMA has a degradation temperature of 290°C. It is obviously that the incorporation of Rhodamine B is accounted for by the decrease of thermal stability. To avoid the degradation of Rhodamine B, the temperature of the fiber drawing process should be under 180°C. It is well known that the processability of a polymer material is related to its molecular weight. In bulk polymerization of PMMA, the molecular weight is mainly controlled by the amount and the kind of the chain transfer agent. In this case, 1-butanethiol was used as chain transfer agent to adjust the fiber drawing temperature to about 170°C; thus, the degradation of Rhodamine B can be avoided.

# Spectra of Rhodamine B doped PMMA and the doped SI POF

The spectra of Rhodamine B doped PMMA and the doped SI POF with different fiber lengths are shown in Figure 3. The doping concentration of the PMMA and the SI POF are both 5 ppm-wt.

Figure 3(a) is the normalized absorption and fluorescence spectra of Rhodamine B-doped PMMA. It can clearly be seen that there is an overlap between absorption and fluorescence spectra. The absorption and the fluorescence peaks are 560 and 577 nm, respectively. Figure 3(b) is the normalized fluorescence spectra of Rhodamine B-doped SI POF with different fiber lengths. An Ar<sup>+</sup>-laser operating at a 457.9-nm wavelength was used as excitation light source. According to Figure 3, there is a difference in the fluorescence spectra between the fiber output and the bulk output that caused by the self-absorption of the Rhodamine B.<sup>13</sup> In Figure 3(b), the fluorescence peak wavelength shifts from 603 to 619 nm, with the fiber length increased from 20 to 80 cm. This spectra shift of fluorescence on length has also been observed by other research workers.<sup>14</sup> The fiber attenuation contributes to the red-shift of the fluorescence at the output end. This character of spectra shift with fiber length is also related with the optimum signal gain wavelength.

### Amplification of Rhodamine B-doped SI POF

The experimental setup for the optical amplification is shown in Figure 1. The diameter of the fiber sample



**Figure 3** (a) Absorption and fluorescence spectra of Rhodamine B in PMMA. (b) Fluorescence spectra of Rhodamine B-doped SI POF with different fiber lengths.

200 Output power(w) 150 100 50 0 6 8 10 Launched pump power(KW) (a) 25 20 Signal gain(dB) 15 10 5 0 10 4 6 8 Launched pump power(KW) (b)

**Figure 4** Signal output power and gain dependence on the launched pump power. (a) Signal output power dependence on the launched pump power. (b) Gain dependence on the launched pump power.

used in this work was 400  $\mu$ m. The Rhodamine B content of the doped SI POF is 5 ppm-wt. The refractive index of the core was 1.471. The index difference between the core and cladding region was 0.072. Therefore, the numerical aperture of POF is 0.45. The POF ends were mounted in a special clamp and polished mechanically.

Figure 4 shows signal output power and gain dependence on the launched pump power of a 60-cm length SI POF. The input power of signal light at 630 nm wavelength is 1 W.

According to Figure 4(a), there is nearly a linear relation between the signal output power and the launched pump power. The maximum power of output power 180 W (the signal gain of 23 dB) was obtained with the launched pump power of 10 kW. It can clearly be seen from Figure 4(b) that at lower launched pump power, the signal gain increased drastically with the increase of the launched pump power,

while at higher launched pump power, the increase of the signal gain is relatively slow and there appears a certain saturation of the gain. Under a certain pump power, this kind saturation of the gain is due to the effect of the medium inversion and fiber absorption.

The signal gain dependence on signal wavelength of the same SI POF is shown in Figure 5. The input power of the signal light is 1 W, and the launched pump power is 10 kW.

According to Figure 5, the maximum gain of 23 dB of the 60 cm SI POF is around 630 nm wavelength. The signal wavelength providing the highest gain is a slightly longer compared to the fluorescence peak showed in Figure 3(b), which is about 615 nm. The reason is the overlap between the absorption and fluorescence spectra, because at the wavelength of the fluorescence spectrum peak, although the fluorescence intensity is high, the absorption is also high. Due to that, amplification in the SI POF at the signal wavelength at which the fluorescence intensity is high and the absorption is relatively low provides a high gain. The signal gain is determined by both the fluorescence and the absorption from a particular fiber length.

The signal gain as a function of the fiber length is shown in Figure 6. The input power of signal light at 630 nm wavelength is 1 W, and the launched pump power is 10 kW.

According to Figure 6, there is the existence of an optimum length  $L_{opt'}$  for which the signal gain is maximized. Because the effect of pump decay along the SI POF results in nonuniform medium inversion. The medium is absorbing when not inverted, the fiber becomes lossy beyond a certain length. For fiber length  $L > L_{opt'}$  the signal is absorbed along the fiber. This optimum length actually depends on the input pump power, because a longer length of inverted







(b)

**Figure 6** Signal gain dependence on fiber length (the input power of signal light at 630 nm wavelength is 1 W, and the launched pump power is 10 kW).

medium can be achieved by a higher pump power.<sup>15</sup> In this case, under the 10-kW launched pump power, the optimum fiber length is about 60 cm.

### **CONCLUSION**

In conclusion, a preform technique has been presented and used to fabricate Rhodamine B-doped SI POF. A high gain (23 dB) for a SI POF with a 60-cm length, 400- $\mu$ m diameter was observed. The Rhodamine B content of the doped SI POF is 5 ppm-wt. The optimum fiber length is about 60 cm at 10 kW launched pump power. The signal wavelength providing the highest gain for a 60-cm SI POF is around 630 nm, which is closer to the communication window (650 nm) of PMMA-based POF. The results showed that the Rhodamine B-doped SI POF can be used as optical amplifier near the low loss window of PMMA POF.

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