Fabrication Process and Optical Properties of Perdeuterated Graded-Index Polymer Optical Fiber

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***Abstract*—We proposed a graded-index polymer optical fiber (GI-POF) enabling greater-than-gigabit data transmission in a short reach network by applying a two-step interfacial-gel polymerization process to a perdeuterated polymer material. Using this process, it is possible to fabricate a nearly optimum refractive-index profile with good reproducibility. In this paper, the preparation of a perdeuterated polymethyl methacrylate (PMMA-d8)-based GI-POF utilizing a PMMA cladding is described. This means that the low-loss PMMA-d8 material is used only for the core. Because the cladding transmits only a small fraction of the optical power, a low-loss material is not necessarily required for the cladding. It was verified that the low attenuation of the PMMA-d8 was maintained even if the cladding was composed of hydrogenated PMMA. As a result, a gigabit data transmission over 300 m was achieved by the PMMA-d8-core and PMMA-cladding GI-POF, which was impossible by the conventional PMMA-based GI-POF.**

***Index Terms*—Low attenuation, perdeuterated methyl metacrylate, plastic optical fiber, two-step interfacial-gel polymerization process.**

I. Introduction

THE demand for high-capacity data transmission in optical communication links such as office and home networks has increased. Currently, an unshielded twist pair (UTP) cable is used for most local area networks (LANs). However, it is hard for UTP to cover a bit rate higher than 1 Gb/s over 100 m. It can be achieved by single-mode and multimode glass fibers, which are made of high-quality silica glass. However, glass optical fiber cannot have a large-core diameter because of its fragility, which makes it difficult to connect fiber to fiber. Therefore, use of single-mode glass fibers in LANs or home networks is not practical.

Polymer optical fibers (POFs) have been recognized as a viable solution for short-distance applications. POFs can have a large-core diameter, because polymer materials have excellent flexibility and durability. Therefore, a system using POFs can use inexpensive connectors and can be connected easily to light sources. Consequently, the overall cost of this system is reduced compared with a system using glass fibers. Furthermore, graded-index (GI) POFs enable a higher data transmission compared with step-index (SI) POFs. The high bandwidth is achieved by a continuously varying refractive index in the core of the GI-POF, which reduces the modal dispersion. Several methods have been proposed as the fabrication process. One fabrication process is an interfacial-gel polymerization process [1], [2]. Recently, a modified interfacial-gel polymerization process was proposed to obtain a high-bandwidth polymethyl methacrylate (PMMA)-based GI-POF [3]. The GI-POF prepared by this process can transmit 3.0 Gb/s for 100 m. However, it was difficult to transmit over 200 m using the PMMA-based GI-POFs due to the inherent material absorption and scattering loss [4].

In this paper, a perdeuterated PMMA (PMMA-d8) was employed to fabricate the GI-POF to realize greater-than-200-m transmission, because the perdeuterated material has a low absorption loss. As the basic chemical properties such as polymerization reactivity are nearly the same as those of the hydrogenated one, the same interfacial-gel polymerization process can be applied to the MMA-d8 monomer. In addition to the polymerization reactivity, the refractive index of PMMA-d8 is close to that of the PMMA. Therefore, it was proposed to prepare the core of the GI-POF by using PMMA-d8. This means that the low-loss PMMA-d8 material was used only for the core. The reason why the cladding can be composed of the conventional PMMA with a large absorption loss is that the cladding transmits only a small fraction of the optical power. This structure is effective for material cost reduction, compared with an all-PMMA-d8-based GI-POF (both core and cladding) and even with a perfluorinated (PF)-polymer-based GI-POF. In the case of PF-polymer-based GI-POF, a PF polymer has to be used for both core and cladding, since its refractive index is much lower than other polymers. Because of this reason, the characteristics of the PMMA-d8-based GI-POF were investigated in detail.

II. Experimental

A. Formation of a Refractive-Index Profile by the Interfacial-Gel Polymerization Process

GI-POFs were obtained by the heat drawing of GI preform rods. A preform rod in which the refractive index gradually decreases from the center axis to the periphery was prepared by the interfacial-gel polymerization process whose procedure is described as follows [1], [2]. A PMMA tube was prepared by bulk polymerization from the purified MMA monomer. Its outer diameter was 22 mm, and its inner diameter was 60% of the outer diameter. The PMMA tube was filled with a mixture of MMA monomer, dopant, polymerization initiator, and chain transfer agent. The PMMA tube filled with this monomer mixture was heated from the surrounding to induce polymerization. The inner wall of the PMMA tube is slightly swollen by the monomer mixture. As a result, the polymer gel phase is formed

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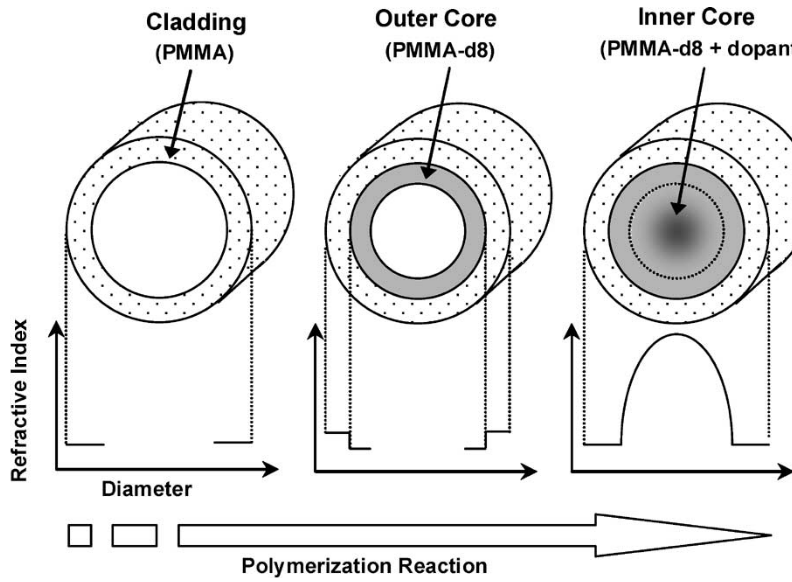

Fig. 1. Schematic representation of the two-step interfacial-gel polymerization process.

TABLE I
RATIO OF PMMA-D8 MATERIAL IN THE PMMA-D8-BASED GI-POFS

	Core Diameter (micrometer)	PMMA-d8 Volume Fraction	PMMA-d8 (gram per 100 meters)
PMMA-d8 core and cladding	525	1.00	52.6
PMMA-d8 core only	525	0.49	25.8
	375	0.25	13.2

on the inner wall of the tube. The polymerization reaction rate is generally faster in the gel phase due to the "gel effect." Therefore, the polymer phase grows from the inner wall of the tube to the center. During this process, the MMA monomer can easily diffuse into the gel phase compared to the dopant molecules because the molecular volume of the dopant, which has benzene rings in it, is larger than that of the monomer. Thus, the dopant molecules are concentrated in the center region of the core to form a nearly quadratic refractive index. The polymerization reaction rate plays an important role to control the refractive-index profile because it affects the diffusion process of the MMA monomer and dopant molecules in the polymer gel phase formed from the inner wall of the tube. The refractive-index profile of the GI-POF is controlled by changing the kind and concentration of the dopant, polymerization initiator, and chain transfer agent. The heat drawing of the GI preform was carried out at 220–250 $^\circ$C. The fiber diameter was controlled to be from 500 to 750 μm.

B. Two-Step Interfacial-Gel Polymerization Process

The PMMA-d8-based GI-POF has many advantages such as low attenuation and wide low-loss optical window in the near-infrared region [2]. However, the material cost of the perdeuterated material has been a great concern. A small fiber diameter was one of the viable solutions to reduce the material cost. The fiber diameter of the PF-polymer-based GI-POF is actually smaller than that of the conventional PMMA-based GI-POF. In order to maintain the ease of handling, which is the advantage of the large diameter POF, the PF-polymer-based GI-POF is surrounded by a reinforcing layer composed of the conventional inexpensive polymer material to increase the fiber diameter to 500 μm. On the other hand, by using the PMMA for its cladding, the PMMA-d8-based GI-POF can maintain a large fiber diameter with the material cost lower than the all-PMMA-d8-based GI-POF.

To fabricate the PMMA-d8-core and PMMA-cladding GI-POF, the two-step interfacial-gel polymerization process was adopted [3]. In this process, the polymerization of the core region was divided into two steps: the polymerization of the outer and inner core regions. The thickness of the outer core was controlled in this process. Therefore, the influence of the PMMA, which diffuses to the core region from the cladding, could be small. A schematic representation of this process is summarized in Fig. 1. After polymerizing the PMMA tube, a specified amount of MMA-d8 monomer mixture was injected into the PMMA tube, and the tube was rotated on its axis. After the polymerization, the polymer tube was obtained as shown in Fig. 1(b). Subsequently, the tube was filled with the MMA-d8 monomer and dopant mixture to form the refractive-index profile.

The PMMA-d8 volume required for preparing an all-PMMA-d8-based GI-POF is summarized in Table I compared with that of PMMA-d8-core and PMMA-cladding GI-POFs. In this calculation, the fiber diameter was assumed to be 750 μm. From Table I, even if the PMMA-d8-core and PMMA-cladding GI-POF has the same core diameter (525 μm) as that of the all-PMMA-d8-based GI-POF, the PMMA-d8 volume fraction is almost half compared with the all-PMMA-d8-based GI-POF. Furthermore, if the core diameter of the GI-POF is small, similar to that of the PF-polymer-based GI-POF, the PMMA-d8 volume fraction further decreases. Therefore, it was decided that the core diameter of the GI-POF should be almost the same as that of the PMMA-based GI-POF prepared by the two-step interfacial-gel polymerization process (PMMA-d8 volume fraction is 0.25.), because the PMMA-based GI-POF prepared by the two-step interfacial-gel polymerization process has an optimum refractive-index profile [3]. As a result, PMMA-d8 material fabrication in the GI-POF is one-fourth compared with that of the all-PMMA-d8-based GI-POF. Therefore, if the cost of PMMA-d8 monomer becomes 50 cents per gram, the price of GI-POF becomes five cents per meter, which is a quite reasonable material cost.

C. Refractive-Index Profile and Dispersion Analysis

The refractive-index profile formed in the core region of a multimode optical fiber plays a large role in determining its bandwidth, because modal dispersion is generally dominant in a multimode fiber. It has been reported that a GI-POF prepared by the interfacial-gel polymerization process enabled a gigabit data transmission rate [1]. Furthermore, the bandwidth potential of the GI-POF has been analyzed by taking modal, material, and profile dispersions into account. Material and profile dispersions are induced by the wavelength dependence of the refractive index of the fiber material. For instance, in the case of a PMMA-based GI-POF, it was theoretically shown that the large material dispersion limited the maximum bandwidth to be approximately 3 GHz for a 100-m distance at a 650-nm wavelength when the light source with a 2-nm spectral width was used [5]. The measured material dispersion of PMMA-d8 was almost the same as that of the PMMA material [6].

For a bandwidth analysis of the GI-POF, it was necessary to quantitatively approximate the refractive-index profile. For designing the optimum index profile of the GI-POF, the approximation of the index profile by the well-known power-law form described by (1) was adopted

$$\begin{aligned} n(r) &= n_1 \left[1 - 2\Delta \left(\frac{r}{a}\right)^g\right]^{\frac{1}{2}}, && 0 \le r \le a \\ &= n_2, && r \ge a \end{aligned} \quad (1)$$

where n_1 and n_2 are the refractive indexes of the core center and cladding, respectively, a is the core radius, and Δ is the relative index difference defined as

$$\Delta = \frac{n_1^2 - n_2^2}{2n_1^2}. \quad (2)$$

The parameter g, called the index exponent, determines the refractive-index profile.

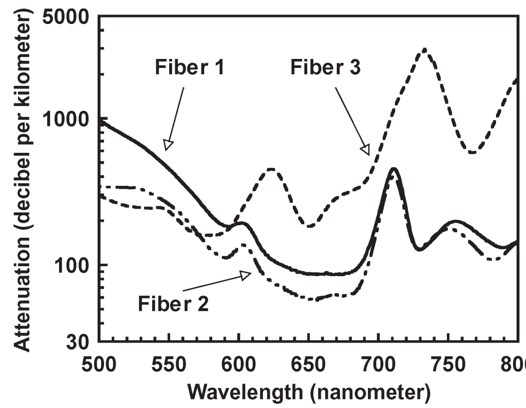

Fig. 2. Total attenuation spectra of the GI-POFs. (——) PMMA-d8-core and PMMA-cladding GI-POF prepared by the two-step interfacial-gel polymerization process (fiber 1). (— · · —) All-PMMA-d8-based GI-POF prepared by the conventional interfacial-gel polymerization process (fiber 2). (· · · · · · · ·) All-PMMA-based GI-POF prepared by the conventional interfacial-gel polymerization process (fiber 3).

D. Bandwidth Measurement of the GI-POF

The bandwidth of the GI-POF was measured by a time-domain measurement method, in which the bandwidth was estimated by measuring the output pulse waveform when a narrow pulse was inserted into the fiber. As the light source, an InGaAsP laser diode (LD) at a 650-nm wavelength and a 1-nm spectral width was used. The input pulse generated by the pulse generator was inserted into the GI-POF, and the output pulse was measured by a sampling head (Hamamatsu OOS-01) and recorded and analyzed by a sampling oscilloscope. The launch condition of the GI-POF is a very important issue in the measurement of the bandwidth. If the high bandwidth is achieved by the GI-POF even under an overfilled launch (OFL) condition, high-speed data transmission is enabled by the GI-POF with a wide range of the optical transceivers, because its high-bandwidth characteristic is independent of the launch condition. In this paper, a short SI-POF (1-m length) was used as the mode exciter to establish a uniform launching condition of all the modes. A pulsed signal was directly launched into a 1-m SI-POF followed by the tested GI-POF sample that is directly coupled on a V-groove. Since the power distribution at the output end of the 1-m SI-POF is uniform in its core region, and the numerical aperture of the SI-POF (0.5) is sufficiently higher than that of the GI-POF (0.2–0.3), the 1-m SI-POF is considered as an ideal mode exciter for a uniform launch.

III. Results and Discussion

A. Attenuation

The total attenuation spectra of the GI-POFs are shown in Fig. 2. Fiber 1 is the PMMA-d8-core and PMMA-cladding GI-POF prepared by the two-step interfacial-gel polymerization process, fiber 2 is the all-PMMA-d8-based GI-POF (both core and cladding are composed of PMMA-d8), and fiber 3 is the all-PMMA-based GI-POF. The attenuation of fiber 1 is 79.8 dB/km at a 650-nm wavelength. The attenuation curve of this GI-POF shows a slightly higher result than that of fiber 2

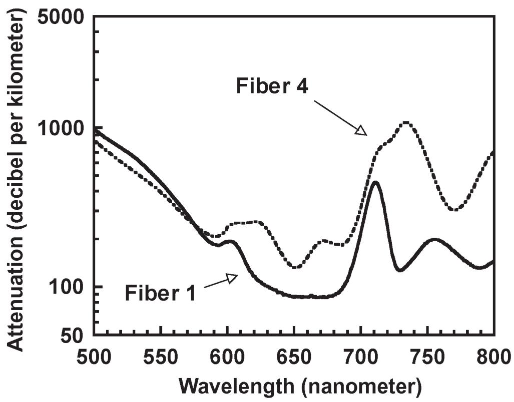

Fig. 3. Total attenuation spectra of fiber 1 and fiber 4. (———) PMMA-d8-core and PMMA-cladding GI-POF prepared by the two-step interfacial-gel polymerization process (fiber 1). (········) PMMA-d8-core and PMMA-cladding GI-POF prepared by the conventional interfacial-gel polymerization process (fiber 4).

particularly from a 500-nm to 700-nm wavelength. However, this GI-POF has a wide low-loss optical window similar to that of fiber 2, which is a great advantage. In the case of the PMMA-based GI-POF (fiber 3), the attenuation at a 650-nm wavelength is the lowest in the low-loss optical window, while if the wavelength is slightly shorter or longer than 650 nm, the attenuation abruptly increases. Therefore, there is a severe tolerance in the operating wavelength range of the light source that would be adopted in the PMMA-based GI-POF link. On the contrary, the low-loss optical windows of fiber 1 and fiber 2 at a 650-nm wavelength are much wider. Considering the emission wavelength shift of a light source such as LD or vertical-cavity surface-emitting laser, due to mode hopping, the wide low-loss optical window from a 650- to 680-nm wavelength in this GI-POF is very advantageous.

There is a concern that the slightly higher attenuation of fiber 1 compared with fiber 2 is caused by the PMMA diffused into the core region from the cladding. In order to clarify the effect of PMMA diffusion from the cladding, the PMMA-d8-core and PMMA-cladding GI-POF (fiber 4) was prepared by the conventional interfacial-gel polymerization process where the core region was directly polymerized in a PMMA tube (no outer core).

The total attenuation spectra of fiber 1 and fiber 4 are shown in Fig. 3. The attenuation of fiber 4 is 130 dB/km at a 650-nm wavelength, and this attenuation is higher than that of fiber 1 (90 dB/km at a 650-nm wavelength). In addition to the high attenuation at 650 nm, the wavelength range of the low-loss optical window of fiber 4 near 650 nm is close to that of the optical window of fiber 3. Furthermore, absorption peaks are observed at 630-, 680-, and 730-nm wavelengths in the spectrum of fiber 4. However, such peaks are not observed in the spectrum of fiber 1. Those peaks are attributed to the fifth and sixth overtone absorption peaks of carbon–hydrogen stretching vibration in the PMMA material [7] by which it was confirmed that the PMMA in the cladding diffused into the core. Therefore, the low-loss optical window in fiber 4 becomes narrower due to the fifth and sixth overtone absorption peaks of the carbon–hydrogen vibration.

Although both fiber 1 and fiber 4 are composed of a PMMA-d8 core and PMMA cladding, the total attenuation of fiber 4 is higher than that of fiber 1. From these results, it was verified that forming an outer core layer in the two-step interfacial-gel polymerization process by PMMA-d8 is effective for reducing the PMMA diffusion from the cladding. Therefore, a slight increment of the attenuation of fiber 1 from a 500-nm to 700-nm wavelength is caused by excess scattering.

B. Refractive-Index Profile

The refractive-index profiles of fiber 1, fiber 2, and fiber 3 are shown in Fig. 4(a), (b), and (c), respectively. The core polymerization conditions (polymerization temperature, polymerization times, and concentration of the dopant) of these GI-POFs were almost the same. However, the index exponent g value of fiber 1 is smaller than that of fiber 2, and the g value $(g = 2.3)$ of the fiber 1 is close to the optimized value $(g_{\rm opt} = 2.4)$.

A refractive-index dip is observed at the boundary of core cladding in the PMMA-d8-core and PMMA-cladding GI-POF [Fig. 4(a)]. This index dip is caused by a slight refractive-index difference between PMMA-d8 and PMMA. It was already clarified that such an index valley at the core–cladding boundary provided another advantage in modal dispersion compensation [8]. On the contrary, the refractive-index profile of the PMMA-d8 core and PMMA cladding prepared by the conventional interfacial-gel polymerization process does not have a dip at the boundary of core cladding [Fig. 4(c)]. The PMMA dissolved and diffused from the cladding to the core is confined to the PMMA-d8 and dopant mixture, particularly around the core–cladding boundary by which the refractive index increased. Consequently, the refractive index of the outer core is not observed to be higher than that of the cladding. As a result of the refractive-index profile in Fig. 4(a) and (c), it was verified that the PMMA in the cladding rarely diffused to the core in the PMMA-d8-core and PMMA-cladding GI-POF prepared by the two-step interfacial-gel polymerization process. The result is consistent with the results of the attenuation measurements.

C. Bandwidth

An experimentally measured output pulse from a 300-m PMMA-d8-core and PMMA-cladding GI-POF is shown in Fig. 5 compared with that of the input pulse. The low attenuation of the PMMA-d8 core shown in Fig. 2 allows a 300-m transmission. The output pulse measured under OFL agrees with the one under UFL. The -3 dB bandwidth of fiber 1 under OFL was 1.2 GHz for 300 m. It was experimentally demonstrated that the PMMA-d8-core and PMMA-cladding GI-POF enables a gigabit transmission over 300 m.

In order to verify the modal dispersion compensation effect by the refractive-index profile, the differential mode delay (DMD) of fiber 1 was also investigated [5]. As shown in Fig. 4(a), the refractive-index profile was accurately approximated by a power-law form by adopting $g = 2.3$, which is slightly smaller than the optimum value. Therefore, the DMD results show that the high-order modes exhibit faster arrival than the lower order modes (Fig. 6). This is typical for an overcompensated fiber in which the g value is smaller than

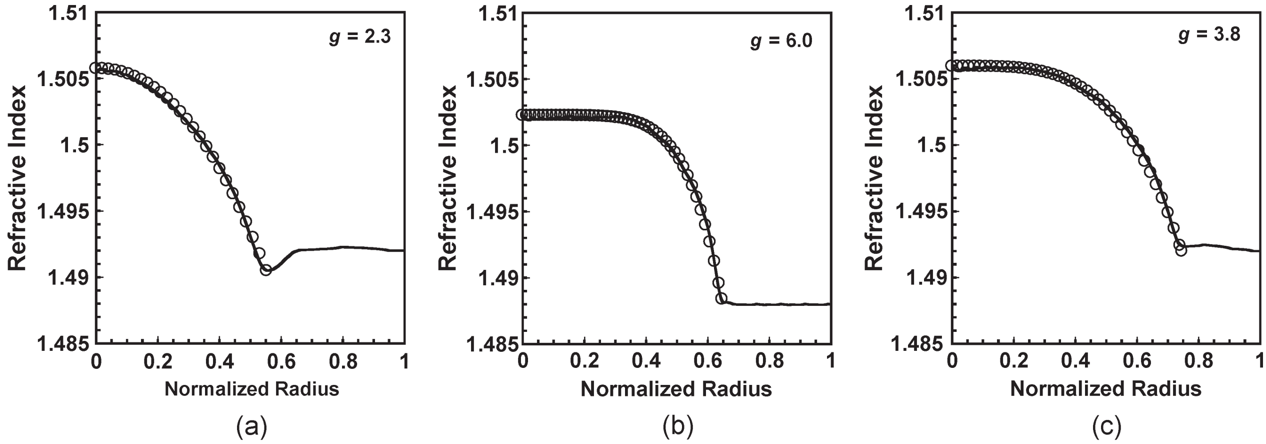

Fig. 4. Measured (solid line) and approximated (open circle) refractive-index profile of the GI-POFs. (a) PMMA-d8-core and PMMA-cladding GI-POF prepared by the two-step interfacial-gel polymerization process. (b) PMMA-d8-based GI-POF prepared by the conventional interfacial-gel polymerization process. (c) PMMA-d8-core and PMMA-cladding GI-POF prepared by the conventional interfacial-gel polymerization process.

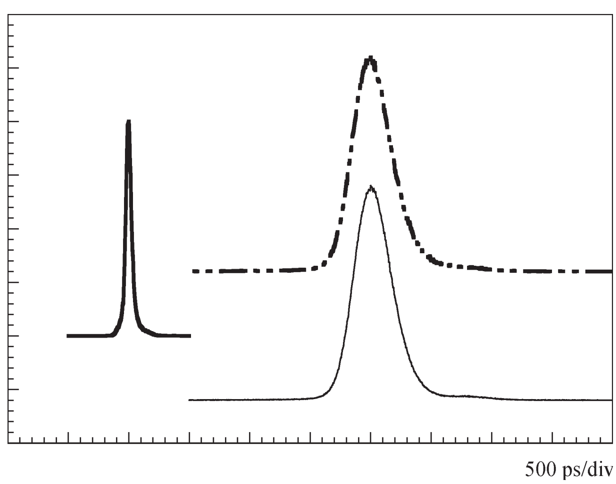

Fig. 5. Output pulse waveform from 100-m PMMA-based GI-POF compared with the input pulse. (———) Measured input pulse. (———) Measured output pulse (launched partially by LD). (— · · —) Measured output pulse (all modes were launched); output pulse waveforms through 100 m of fiber 3 under underfilled launch (UFL) and OFL.

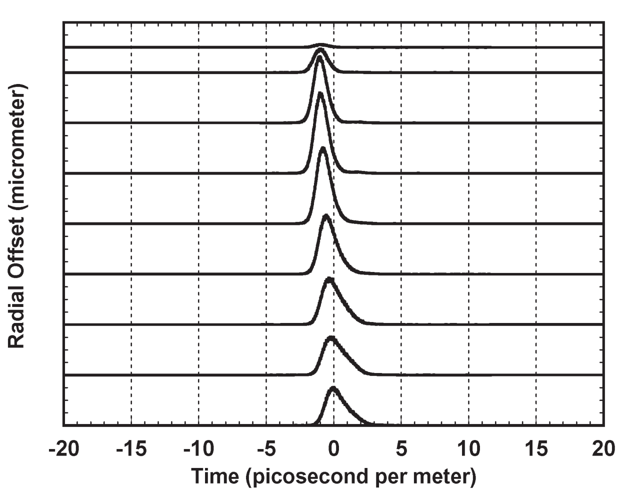

Fig. 6. Differential mode delay in a 300-m PMMA-d8-core PMMA-cladding GI-POF at 650-nm wavelength.

optimum. It was verified that the PMMA-d8-core and PMMA-cladding GI-POF prepared by the two-step interfacial-gel polymerization process in which the g value was not optimum can transmit greater than 1 Gb/s for 300 m.

IV. Conclusion

A low-loss PMMA-d8-core- and PMMA-cladding-based GI-POF with an almost optimum refractive-index profile was fabricated by using the two-step interfacial-gel polymerization process. The attenuation of the GI-POF was 79.8 dB/km at a 650-nm wavelength. In addition to a low attenuation, the GI-POF had a wide low-loss optical window from 620 to 680 nm, which is similar to that of the all-PMMA-d8-based GI-POF. It was indicated that the PMMA polymer diffusion from the cladding to the core had a small effect on the attenuation in the GI-POF. The GI-POF enabled a gigabit transmission over 300 m.

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