

# High-Bandwidth Graded-Index Polymer Optical Fiber With High-Temperature Stability

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**Abstract**—A newly developed graded-index polymer optical fiber (GI-POF) with high-temperature and high-humidity stability was proposed. As it was found that the high numerical aperture and high glass transition temperature ( $T_g$ ) at the core center of the GI-POF were key issues to achieve both high-temperature and humidity stability, a partially fluorinated polymer material was adopted to obtain both characteristics in the GI-POF. The newly developed GI-POF had low-loss (140 dB/km at 650-nm wavelength), high-bandwidth (higher than 1 GHz for 100 m transmission), high-temperature and humidity stability at 70°C, 80% relative humidity (R.H.) and low bending loss.

**Index Terms**—Graded-index polymer optical fiber (GI-POF), high-temperature stability, glass transition temperature, numerical aperture, partially fluorinated polymer.

## I. INTRODUCTION

THE development of personal computer and other audio-visual (AV) equipment technologies has increased the data volume to be handled. For instance, data for even several tens of minutes of motion pictures require several giga bits even if moving picture expert group II (MPEG II) compression technology is applied. To handle the increased demand to transmit such large volumes of data, the maximum data rate of the bus interface based on IEEE 1394 is currently upgraded to 400 Mb/s, and its port is installed in some computers and AV equipment. However, the maximum link length of the IEEE 1394 bus is limited to less than 4.5 m if metallic cable is used. On the other hand, graded-index polymer optical fiber (GI-POF) is one of the promising candidates for high-speed data transmission medium not only in IEEE 1394 home networks, but also gigabit Ethernet-based local area networks (LANs) [1]. In order to utilize the GI-POF in such areas, much higher temperature stability and lower bending loss become necessary [2]. We have already reported that [3] the types of dopant that are used for forming the refractive index distribution of the GI-POF strongly influence the temperature stability of the GI-POF. We have also reported an improvement in the temperature stability of the GI-POF by maintaining a high glass transition temperature ( $T_g$ ) of the core polymer [2], [3], which was enabled by designing an ideal dopant molecular structure and by decreasing the dopant concentration. However, the numerical aperture (NA) of the GI-POF is lowered by decreasing the feed concentration of the dopant, and thus a bending loss increment is a concern [4]. Therefore, a partially fluorinated polymer that has a lower refractive index than

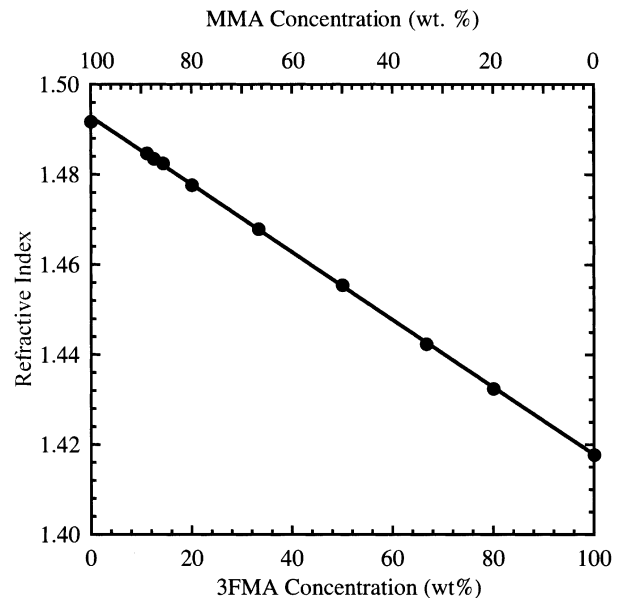


Fig. 1. Relation between the refractive index and the 3FMA concentration in the P(MMA-3FMA) copolymer.

poly(methyl methacrylate)(PMMA) was newly adopted as the cladding material of the GI-POF instead of using PMMA. As a result, a high NA GI-POF was successfully obtained even when the feed concentration of the dopant was drastically decreased.

## II. EXPERIMENTAL

### A. Structure of GI-POF With High Temperature Stability

A methyl methacrylate (MMA)-2,2,2-trifluoroethyl methacrylate (3FMA) copolymer was adopted in the cladding of the GI-POF. In order to design the GI-POF using this copolymer cladding, the refractive index and the  $T_g$  of the copolymer were experimentally investigated by preparing eight MMA-3FMA copolymers (P(MMA-3FMA)) having different 3FMA content. Fig. 1 shows the experimentally measured refractive indices of these P(MMA-3FMA) copolymers. The refractive indices of the P3FMA and PMMA homopolymers are 1.418 and 1.492, respectively, and there is a linear relation between the refractive index and feed concentration. The refractive index of the cladding can be widely varied by controlling the feed concentration of the 3FMA to MMA monomers. Fig. 2 shows the relation between the feed ratio of 3FMA to MMA and the  $T_g$  of P(MMA-3FMA). Although it was preferable to use the P3FMA homopolymer with low refractive index as the cladding material to obtain a sufficiently high NA GI-POF, the low  $T_g$  (70°C) of the 3FMA homopolymer becomes a problem

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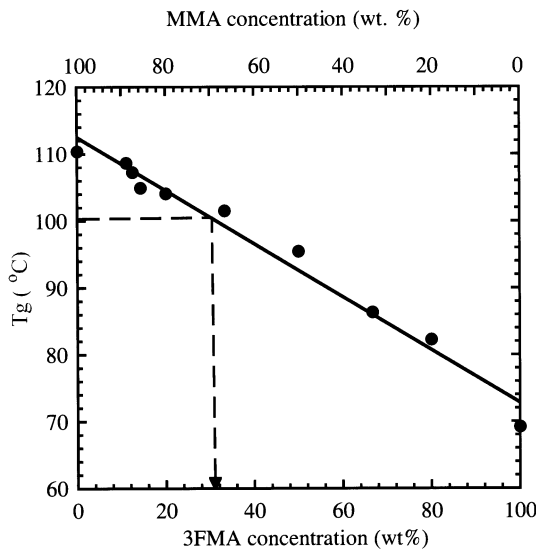


Fig. 2. Relation between the glass transition temperature ( $T_g$ ) and the 3FMA concentration in the P(MMA-3FMA) copolymer.

as shown in Fig. 2: the  $T_g$  of the copolymer is lowered by increasing the 3FMA feed ratio. In order to achieve a higher  $T_g$  than 100°C for the cladding of the GI-POF, we determined the maximum 3FMA feed concentration to be 33wt.% from Fig. 2. In this case, the refractive index of the copolymer is sufficiently low (1.46) to form a high numerical aperture GI-POF. In this paper, P(MMA-3FMA) in which the feed ratio of 3FMA was varied from 12.5 wt.% to 20 wt.%, was adopted as a cladding of the GI-POF. The preparation method of the P(MMA-3FMA)-clad GI-POF is briefly described.

### B. Preparation of MMA-3FMA Copolymer Tube

A GI preform rod was prepared by the interfacial-gel polymerization technique which we have previously described [1], [5]. For forming the core region of the GI-POF by the interfacial-gel polymerization process, the mixture of monomer and aromatic dopant (such as benzyl benzoate and diphenyl sulfide) is injected into a polymer tube having 18–30 mm outer diameter, and the tube is heated from the surrounding to polymerize the core region. During this process, the inner wall of the tube is slightly dissolved and swollen by the monomer mixture, and forms polymer-gel phase. This polymer-gel phase plays an important role in forming the concentration distribution of the dopant which is related to the refractive index distribution.

Since the cladding material of the GI-POF proposed in this paper is composed of P(MMA-3FMA), an excess scattering loss is a concern after the core polymerization because of the poor miscibility between the cladding materials (copolymer of MMA and 3FMA) and aromatic dopant. If the MMA-dopant mixture is directly injected into the copolymer tube and polymerized (i.e., the same way as in the interfacial-gel polymerization technique) the aromatic dopant must diffuse into the copolymer gel phase. Because of the poor miscibility between the aromatic compound and the MMA-3FMA copolymer, large heterogeneities are caused, which induces an excess scattering loss. Thus, a low-loss GI-POF is no longer obtained.

Therefore, we modified the interfacial-gel polymerization with several steps. The schematic representation of several-step

interfacial-gel polymerization is shown in Fig. 3. In this process, a thin PMMA homopolymer layer is coated on the inner wall of the MMA-3FMA copolymer tube as shown in Fig. 3(b). After the polymerization of the PMMA homopolymer layer, the monomer/dopant mixture was injected into the tube, followed by the polymerization of the core region. By forming this PMMA homopolymer layer on the inner wall of the tube, the excess scattering is effectively eliminated. The GI-POF was obtained by the heat-drawing of the GI preform. Heat-drawing process of the P(MMA-3FMA)-clad GI-POF is almost the same as that of the PMMA-clad GI-POF we have proposed [1]. The heat-drawing of the P(MMA-3FMA)-clad GI preform was carried out at 220–230°C. The fiber diameter was controlled to be 750  $\mu\text{m}$ . In this fiber, core diameter becomes approximately 500  $\mu\text{m}$ .

### C. Characterization of GI-POF

In addition to the basic fiber characteristics such as refractive index profile, bandwidth, and attenuation, the  $T_g$  of the polymer, copolymer, and doped polymer was evaluated.

The refractive index profile of the GI-POF was measured by the transverse interferometric technique [1], [2]. Although several measurement methods to measure the refractive index profile were already proposed particularly for the silica based GI multimode fiber, it was verified that this interferometric method using an interference microscope is one of the best ways to measure the GI-POF with such a large core diameter (>500  $\mu\text{m}$ ).

The bandwidth of the P(MMA-3FMA)-clad GI-POF was evaluated by the time domain measurement method in which the bandwidth was estimated by measuring the output pulse waveform. As the light source, an InGaAsP laser diode at 655-nm wavelength was adopted. An 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 with a sampling oscilloscope.

The attenuation measurement of the P(MMA-3FMA)-clad GI-POF was carried out by using the cut-back method. The measurement method of temperature stability in the attenuation was clarified in [3] in detail. As the light source, a tungsten lamp was used and the optical output power change was measured through the aging with using the optical spectrum analyzer. The temperature stability of the attenuation was investigated by continuously measuring the output power change from the GI-POF through the aging, where the connections between the light source and the fiber, and between the fiber and the detector were fixed. Since the output power from the GI-POF is very sensitive to the coupling conditions between the light source and fiber and the fiber and detector, both coupling conditions were fixed through the aging. In this paper, the attenuation change at 650-nm wavelength was focused.

The  $T_g$  of the polymer, copolymer, and doped polymer was measured with using differential scanning calorimetry (DSC, Shimadzu Co.) The heating rate of the DSC measurement was 20°C/min. Since the dopant concentration was formed in the GI-POF in the radial direction, the  $T_g$  distribution exists in the GI-POF. This  $T_g$  distribution was indirectly measured by measuring the relations between the dopant concentration and  $T_g$ , and the dopant concentration and the refractive index. From these two relations the refractive index and  $T_g$  was obtained. This process was described in detail in Ref. [3].

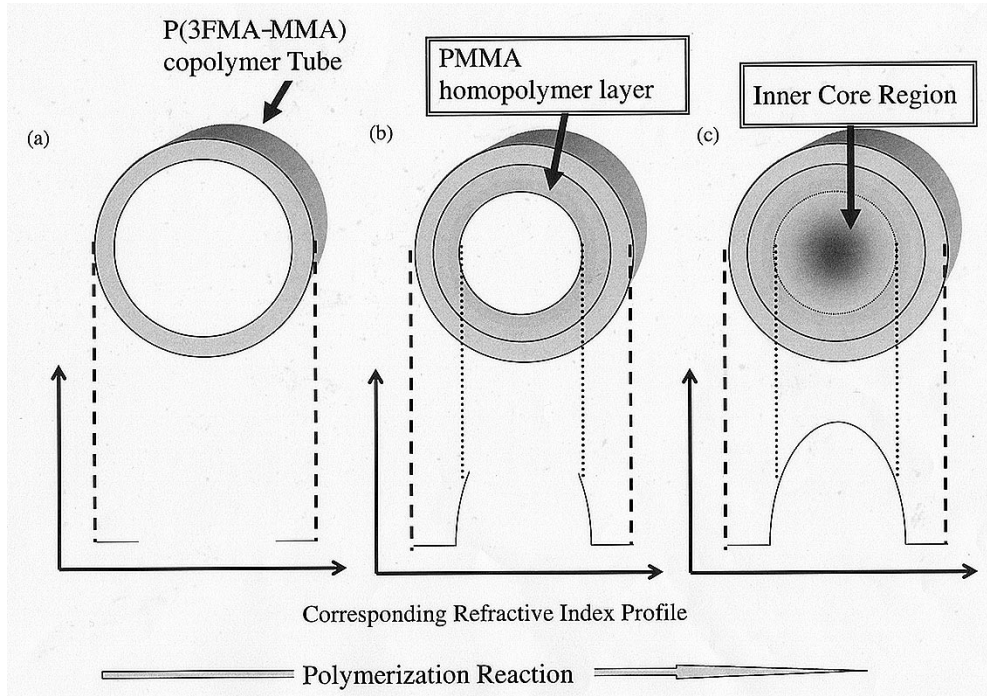


Fig. 3. Schematic representation of the several-step interfacial-gel polymerization process to obtain the P(MMA-3FMA)-clad GI polymer preform.

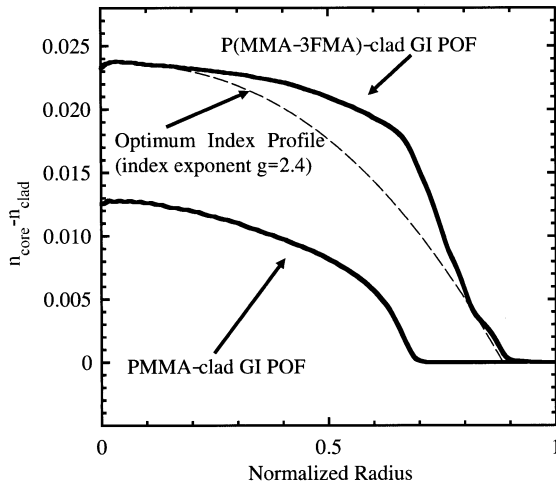


Fig. 4. Refractive index profile of the P(MMA-3FMA)-clad GI-POF compared with that of PMMA-clad GI-POF. Broken line shows the ideal refractive index profile providing highest bandwidth for the P(MMA-3FMA)-clad GI-POF.

### III. RESULTS AND DISCUSSION

#### A. Index Distribution

Fig. 4 shows an index distribution of the P(MMA-3FMA)-clad GI-POF compared with that of the PMMA-clad GI-POF. In the case of the PMMA-clad GI-POF, we already confirmed that the dopant concentration should be less than 11 wt.% in order to achieve high temperature stability (high  $T_g$ ). Consequently, the NA of the PMMA-clad GI-POF is also as low as 0.2 even if the dopant has a much higher refractive index than that ( $n_d = 1.492$ ) of PMMA such as diphenyl sulfide (DPS) ( $n_d = 1.633$ ). In contrast, a GI-POF having a high NA can be prepared despite the same or lower dopant concentration

by adopting the MMA-3FMA copolymer as the cladding as shown in Fig. 4. In fact, an NA as high as 0.27 was realized in the P(MMA-3FMA)-clad GI-POF with only 5 wt.% DPS doping. A greatly reduced dopant concentration allows the GI-POF to have the advantage of high-temperature stability.

On the other hand, since the core region is prepared by two steps (MMA homopolymer layer and PMMA-dopant layer), optimization of the index profile becomes more important. Actually, the index profile at the boundary of the core and cladding tends to be steep in the case of the P(MMA-3FMA)-clad GI-POF as shown in Fig. 4.

It was already shown that [6], [7] the optimum refractive index profile for a GI-POF that exhibits the largest bandwidth should be described by the well known power-law form shown in Eq. (1).

$$n(r) = n_1 \left[ 1 - 2\Delta \left( \frac{r}{a} \right)^g \right]^{\frac{1}{2}} \quad 0 \leq r \leq a$$

$$= n_2 \quad r \geq a \quad (1)$$

where,  $n_1$  and  $n_2$  are the refractive indices of the core center and cladding, respectively,  $a$  is the core radius, and  $\Delta$  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. It was already found that the optimum index exponent ( $g_{opt}$ ) of the PMMA-DPS system GI-POF is 2.5 [7]. However, the index profile of the P(MMA-3FMA)-clad GI-POF deviates from the ideal one that is shown by the broken line in Fig. 4. Since the index profile of the GI-POF directly influences the bandwidth of the GI-POF, reduction of bandwidth is a greater concern with the P(MMA-3FMA)-clad GI-POF compared with the conventional PMMA-clad GI-POF, which

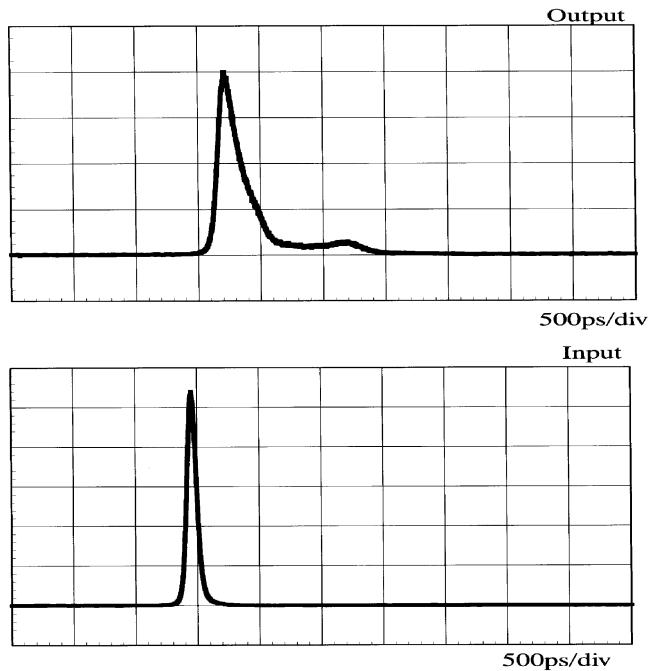


Fig. 5. Experimental result of the bandwidth measurement of the 100-m P(MMA-3FMA)-clad GI-POF by the time domain measurement method at a 650-nm wavelength.

has an almost optimum index profile. Compared to the ideal index profile shown by the broken line, the measured index profile of the P(MMA-3FMA)-clad GI-POF has a steep index profile at the core and cladding boundary. This is because the polymerization of the core region was divided into two steps. If MMA monomer diffuses into the P(MMA-3FMA) cladding, obtained polymer includes a heterogeneous structure to cause an excess light scattering loss. Therefore, the gradual refractive index profile is not formed at the boundary of core (PMMA homopolymer) and cladding (P(MMA-3FMA)). Detailed investigations of the bandwidth of the P(MMA-3FMA)-clad GI-POF are described in the following sections.

### B. Bandwidth

One of the recent trends in digital home network is in the high-speed data transmission based on the IEEE 1394b (S400) format using a large bandwidth GI-POF [8]. As a light source of such an optical home network, a laser diode and/or a resonant cavity light emitting diode [9] emitting at 650-nm wavelength are promising candidates.

The result of the output pulse measurement from a 100-m GI-POF is shown in Fig. 5 compared with the waveform of the input pulse. Although a slight tail is observed in the output pulse particularly in the falling part (right edge), the  $-3$  dB bandwidth calculated by Fourier transform was as high as 1.5 GHz for 100 m, which ensures coverage of the required bit rate in the IEEE 1394 S400, S800, and gigabit Ethernet. The reason of the slight output pulse broadening (tail and second peak) was analyzed by measuring the differential mode delay and differential mode attenuation. For these analyses, the same method written in our previous paper [10] was adopted. As the result, it was found that the long tail and second peak of the output pulse shown in Fig. 5 was caused by the late arrival of the high order mode. Such a group delay in the high order mode is observed because

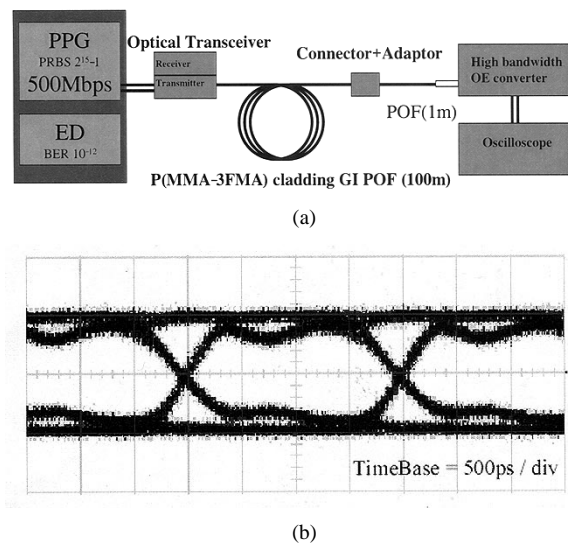


Fig. 6. (a) Eye pattern measurement setup for the GI-POF link. (b) Measured eye diagram after 100-m P(MMA-3FMA)-clad GI-POF.

the refractive index profile is deviated from the optimum one. However, the bandwidth of the GI-POF shown in Fig. 5 is much higher than the conventional SI type POF.

Next, a 100-m P(MMA-3FMA)-clad GI-POF link was constructed with a 650-nm laser diode and an Si-PIN PD, and a 500-Mb/s transmission was carried out. Then the eye-pattern of P(MMA-3FMA)-clad GI-POF link was measured. Since the actual waveform transmitted through the GI-POF is examined, the eye-pattern measurement is suitable for troubleshooting any problems with the link performance. Fig. 6(a) shows the schematic representation of eye measurement. The fiber input signal intensity was  $+1.91$  dBm. Good eye opening was observed in Fig. 6(b) after the 100-m length 500-Mb/s data transmission.

### C. Attenuation

Fig. 7 shows the attenuation spectrum of the P(3FMA-MMA)-clad GI-POF. A low-loss (130–160 dB/km at 650-nm wavelength) as a PMMA-clad GI-POF is realized.

Fig. 8 shows the bending loss of the P(MMA-3FMA)-clad GI-POF compared with the conventional GI-POF. The bending loss was measured by perpendicularly bending the fiber, and all modes of both fibers were uniformly launched for the bending loss measurement. It is generally known that low bending loss is achieved by increasing the NA of the fiber. As shown in Fig. 8, the bending loss of the P(MMA-3FMA)-clad GI-POF is improved in spite of the half or lower feed concentration of the dopant, because the P(MMA-3FMA)-clad GI-POF used in the bending loss measurement has an NA (0.27) that is higher than that (0.21) of the PMMA-clad GI-POF in Fig. 8.

### D. High-Temperature Stability

The temperature stability both in the index profile and the attenuation of the P(MMA-3FMA)-clad GI-POF was evaluated. Temperature stability in the index profile is shown in Fig. 9. It is noteworthy that the refractive index profile originally formed (shown by open circles) was maintained after 3500 h of aging even at  $85^\circ\text{C}$  shown by the solid line. In Fig. 9, the  $T_g$  distribution in the core region is also plotted. In order to obtain this

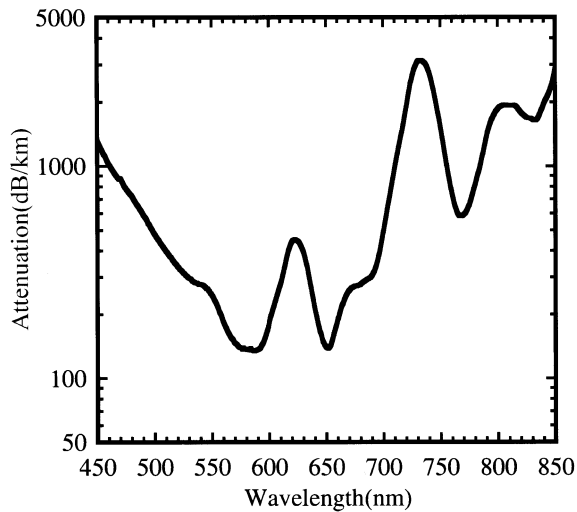


Fig. 7. Total attenuation spectrum of the P(MMA-3FMA)-clad GI-POF.

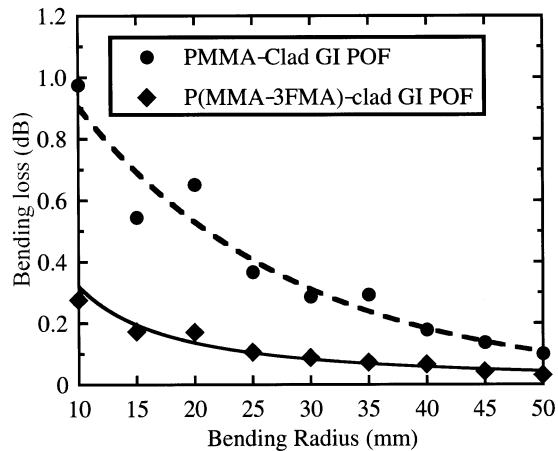


Fig. 8. Bending loss measurement of the P(MMA-3FMA)-clad GI-POF compared to the PMMA-clad GI-POF.

$T_g$  distribution, the relation between the  $T_g$  and dopant concentration, and the refractive index and dopant concentration were measured in bulk polymer samples; subsequently, the relation between the refractive index and the  $T_g$  of the doped polymer was obtained. From this relation and the refractive index profile experimentally measured, the  $T_g$  distribution shown in Fig. 9 was calculated. The detailed process for obtaining the  $T_g$  distribution was described in Ref. [3]. The  $T_g$  at the core center is as high as 100°C which is sufficiently higher than the aging temperature (85°C). It was possible to maintain a high  $T_g$  at the core center by decreasing the dopant concentration as we already described in Ref. [3]. However, the numerical aperture of the GI-POF was lowered by decreasing the dopant feed concentration, which induces the high bending loss. On the other hand, the P(MMA-3FMA)-clad GI-POF allows a low dopant concentration and a high numerical aperture. Consequently, high temperature stability and low bending loss are simultaneously achieved as shown in Figs. 9 and 7, respectively.

The result of the stability in the attenuation is shown in Fig. 10 compared with the PMMA-clad GI-POF. The GI-POF was oven aged at 70°C, 80% relative humidity (R.H.). When the 20-%

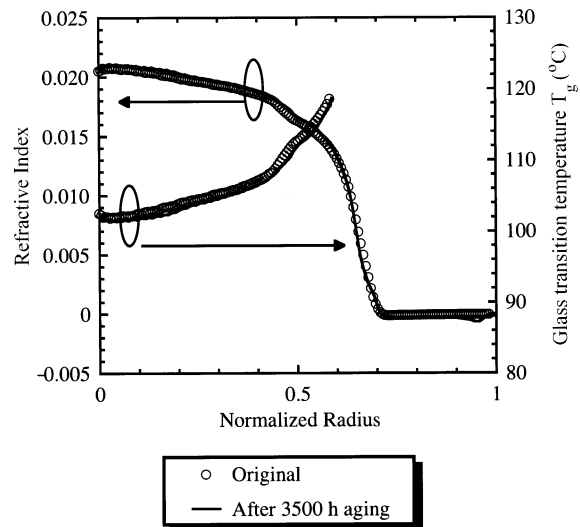


Fig. 9. Temperature stability of the refractive index profile of the P(MMA-3FMA)-clad GI-POF compared with the glass transition temperature ( $T_g$ ) profile in the core region.

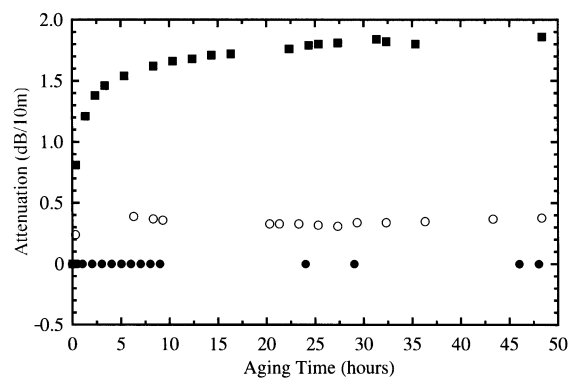


Fig. 10. Attenuation increment of the P(MMA-3FMA)-clad GI-POF at 70°C, 80% R.H. compared to that of the PMMA-clad GI-POF. ●: P(MMA-3FMA)-clad GI-POF (less than 10% DPS doped.), ○: 12.5-% DPS doped PMMA-clad GI-POF, ■: 20-% DPS doped PMMA-clad GI-POF.

DPS-doped PMMA-clad GI-POF was aged at 70°C, 80% R.H., an abrupt attenuation increment was observed even after several hours of aging as shown by the squares (■) in Fig. 10. It was shown that this attenuation increment was caused by an excess scattering loss that was induced by the aggregation of water molecules absorbed in the core of the GI-POF [11]. Because the DPS used as the dopant is more hydrophobic than the PMMA polymer matrix, as the dopant concentration is increased, the core region of the GI-POF becomes more hydrophobic. Therefore, the water absorption into the DPS doped PMMA is less than that into the PMMA homopolymer. However, this small amount of water cannot be uniformly dispersed but is aggregated in the core region. Consequently, the 12.5-% DPS doped PMMA-clad GI-POF shows less attenuation increment than the 20-% DPS doped PMMA-clad GI-POF as shown by the open circles (○) in Fig. 10. Details of the high-temperature and high-humidity stability of the PMMA-clad GI-POF is described in [11].

Furthermore, the stability at high temperature and high humidity of the P(MMA-3FMA)-clad GI-POF (shown by closed

circles (●) in Fig. 10) is greatly improved compared with that of the PMMA-clad GI-POF. Fig. 10 shows that no attenuation increment was observed during aging at 70°C 80% R.H. This is because the dopant concentration could be decreased to less than 10% while maintaining sufficiently high NA.

The temperature and humidity stability in bandwidth was also investigated by continuously measuring the output pulse broadening during aging. It was confirmed that high bandwidth as 1.5 GHz for 100 m was maintained even after 5000 hours aging at 70°C, 80% R.H. As it was reported by our previous paper that [3] the thermal stability in the refractive index profile of the PMMA based GI-POF was remarkably improved by decreasing the dopant concentration. In the case of P(MMA-3FMA)-clad GI-POF, the dopant concentration can be much lower than that of PMMA-clad counterpart. Therefore, the thermal stability of the refractive index profile is higher than that of the PMMA-clad GI-POF. In addition to the thermal stability, the high humidity stability of the refractive index profile was verified by measuring the bandwidth stability at high temperature and humidity.

As described in the above sections, although the control of the refractive index profile is difficult compared with the PMMA-clad GI-POF, the P(MMA-3FMA)-clad GI-POF can have sufficiently high bandwidth (the order of GHz for 100 m) and low-attenuation comparable to the PMMA-clad GI-POF and the conventional PMMA core step-index (SI) POF commercially available. Furthermore, high-temperature and high-humidity stability in both attenuation and bandwidth can be achieved by adopting the copolymer of MMA and 3FMA as the cladding material.

#### IV. CONCLUSION

A P(MMA-3FMA)-clad GI-POF having high-temperature stability in the attenuation and bandwidth was demonstrated for the first time. By adopting the P(MMA-3FMA) as the cladding, the refractive index of the cladding can be decreased compared with the PMMA homopolymer-clad GI-POF we already demonstrated. This low refractive index of the cladding allows a reduction in the dopant feed concentration of the core compared with the PMMA-clad GI-POF ensuring the same or higher numerical aperture. Although the dopant concentration was decreased, the high NA was maintained because of the low refractive index of the P(MMA-3FMA) cladding. Therefore, the P(MMA-3FMA)-clad GI-POF can solve both temperature stability and bending loss problems existing in the PMMA-based GI-POF we already developed. We believe that the improved temperature stability of the GI-POF opens the way for great improvements in the high-speed optical home network.

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Dr. Koike received the International Engineering and Technology Award of the Society of Plastics Engineers in 1994, and Fujiwara Award in 2001.