

Stability of High-Bandwidth Graded-Index Polymer Optical Fiber

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ABSTRACT: The properties of poly(methyl methacrylate) (PMMA)-based graded-index polymer optical fiber (GI POF), including the thermal stability, thermal humidity, and mechanical properties, were studied for polymer optical fiber research and applications. The glass-transition temperature of the fiber core was 103°C in the presence of the dopant, which was close to that of the PMMA matrix without the dopant. A special refractive-index profile derived from the distribution of the dopant was stable at 60°C.

Moreover, GI POF exhibited good mechanical properties. The excellent performance indicated that GI POF could be applied not only for indoor use but also for outdoor use. However, PMMA-based GI POF exhibited poor hot-water/humidity resistance. © 2003 Wiley Periodicals, Inc. *J Appl Polym Sci* 91: 2330–2334, 2004

Key words: optics; fibers; mechanical properties

INTRODUCTION

The recent explosion in the use of the Internet and the World Wide Web is demanding much higher bandwidth performance for short- and medium-distance applications.¹ There are three main methods for physically linking sources to transfer and communicate data: copper wire, glass fiber, and polymer optical fiber (POF). Currently, most short-distance data transmission in local area networks (LANs) and transmission to and in the home are based on copper wiring. However, copper wiring cannot support the ever-increasing bandwidth requirements of multimedia and Internet technologies. In addition, copper cabling is not a secure medium and is susceptible to electromagnetic interference and electromagnetic radiation, which can impair signal transfer and compromise critical data passing through a network.² Inorganic glass fibers are far superior to copper wire with respect to bandwidth, but their small core diameter and low numerical aperture make fiber installation difficult, and this increases the cost dramatically. Inorganic glass fibers have been proved to be unsuitable for LAN installations, being too fragile for horizontal subsystems. POF possesses a large core diameter and a relatively high numerical aperture; these facilitate installation and reduce the cost. Moreover, graded-index polymer optical fiber (GI POF), with a parabolic refractive-index gradient over the cross section, pos-

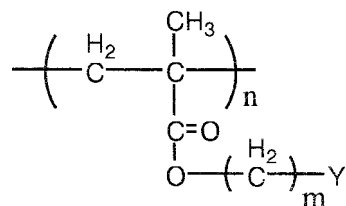
sesses a high bandwidth and is, therefore, an excellent candidate to replace copper wiring and glass fibers.³

As for POF, three optical plastics are usually used as the core material: poly(methyl methacrylate) (PMMA), polystyrene, and polycarbonate.^{4–6} PMMA-core optical fiber has some advantages over fibers with the other two polymers: high bandwidth and lower attenuation. As a result, PMMA optical fibers are useful in LANs for high-speed data communications. Obviously, the stability of POF properties is very important because it is crucial for the long-term reliability of LANs. In this article, some tests that we performed on PMMA fibers are described. The optical transmission properties under different conditions, such as heat and humidity, are evaluated. The mechanical properties of POF are also described.

EXPERIMENTAL

GI POF samples

The GI POF samples were prepared by our group⁷ and were made of a PMMA matrix doped with organic molecules 1 mm in diameter. Figure 1 shows a schematic structure of GI POF. The general structure of the dopant can be expressed as follows:⁸



where n is 1–20, m is 1–17, and Y is CX_3 (X is F, Cl, Br, or I). The molecular weight is 1000–20,000.

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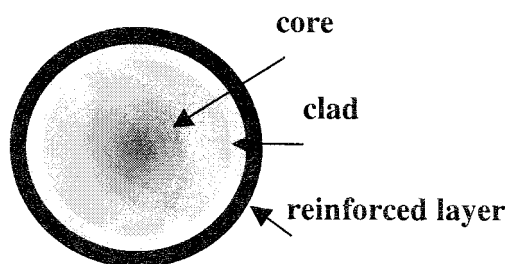


Figure 1 Schematic structure of GI POF.

Refractive-index distribution

GI POF was oven-aged at 60°C at room humidity. The refractive-index distribution of GI POF after aging was measured with the focusing method⁹ and was compared with the index profile of the original profile. The principle of the focusing method was given in our previous research.¹⁰ In a typical experiment, the samples were immersed in a matching oil, the refractive index of which was equal to that of the fiber cladding to eliminate light refraction at the outer boundary. Collimated light passed transversely through the fiber core and was detected with a microscope focused just above the plane of the core boundary. The intensity distribution in the image plane was recorded with the aid of a computer-controlled video analysis system. The refractive-index profile of the fiber core was then computed from this intensity distribution.

Bandwidth

The bandwidth of GI POF was measured with a pulse-broadening method in the time domain. A Spectra-Physics 3500 dye laser system (Solid-State Laser Co., Mountain View, CA) (synchronously pumped and cavity-dumped) was used as the pulse source at a repetition rate of 800 kHz, with a Spectra-Physics CW 3800 picosecond laser (frequency-doubled and mode-locked) used as the pumping source. The pulse was injected into the fiber with an optical coupler and measured by a diode with a response time of 900 ps; the output pulse was amplified with an HF8447F amplifier (Solid-State Laser Co.) and recorded with a Tektronix TDS620b 500-MHz digital real-time oscilloscope. The pulse broadening of the sample fiber was recorded as $P_2(t)$. The injection conditions were maintained, and the fiber was cut 2 m from the injection position as a reference; the reference pulse was recorded as $P_1(t)$. The bandwidth (B) was calculated with the corresponding full widths at half-maximum τ_2 and τ_1 (ns):

$$B = \frac{0.441}{\sqrt{\tau_2^2 - \tau_1^2}} \quad (1)$$

Attenuation

The attenuation of GI POF was measured by a cut-back method with a 650-nm light-emitting diode as the incoming light source and with a light-emitting diode as the output. The light sources emitted around 650 nm, which was in the gap in the red window of the PMMA fibers. This source was coupled to a 100-m initial length (L_1) of the fiber, which was then cut at $L_2 = 2$ m. The loss was thus obtained. The measurement was carried out in two stages: the measuring of power P_1 transmitted by a section of POF of length L_1 polished at both ends and, without changes in the launching conditions in POF, the cutting of the section, the polishing of the end, and the measuring of P_2 transmitted by the shorter length ($L_2 \sim 2$ m). The attenuation coefficient, at 650 nm, can be expressed (dB/m) by the following formula:

$$\text{Loss} = 10/(L_1 - L_2) \times \log_{10}(P_2/P_1) \quad (2)$$

Glass-transition temperature (T_g) of GI POF before and after heat aging

The thermal behavior of POF was analyzed by means of DSC. A physically aged sample of about 5.0 mg was sealed in an aluminum pan, and the thermal properties were measured with a Mettler-Toledo 822^e (Greifensee, Switzerland). Indium and tin were used for the temperature calibration, the heat capacity was evaluated with respect to sapphire as a standard, and a nitrogen gas purge with a flux of about 30 mL min^{-1} was used to prevent oxidative degradation of the sample during the heating run. The rate of heating for DSC was 20°C min^{-1} .

Mechanical properties

The tensile strength test, according to JIS-C-6861, was performed with a Minimat 2000 (Rheometric Scientific, Piscataway, NJ) with a gauge mark distance of 60 mm and a tensile rate of 10 mm/min.

RESULTS AND DISCUSSION

Thermal stability of the refractive-index profile

An investigation of the thermal stability of the refractive-index profile can offer important information on the long-term reliability of GI POF. The refractive-index distributions of organic-molecule-doped GI POF are shown in Figure 2. The dopant ratio was 2 wt % in the fiber. It is well known that profile stability depends strongly on the dopant distribution.¹¹ Figure 2 shows that little change in the profile can be observed after 360 days of aging at 60°C.

T_g of GI POF was 103°C, almost equal to that of pure PMMA (105°C). Thus, it could maintain the same refractive-index profile even after aging at 60°C for 360

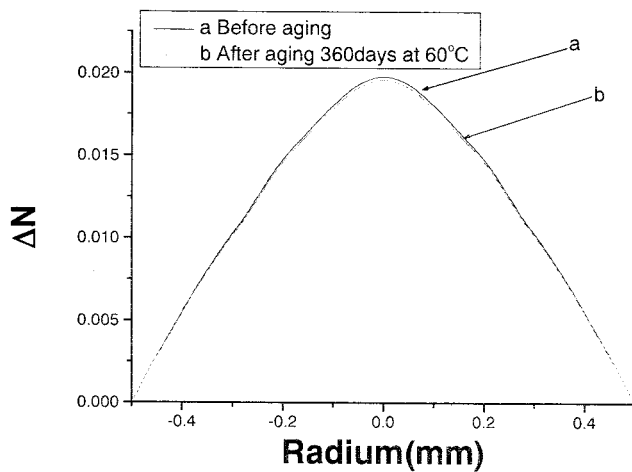


Figure 2 Refractive-index distributions of organic-molecule-doped GI POF before and after aging at 60°C for 360 days.

days.¹² The high T_g value is one of the main reasons for the high stability of GI POF. Another factor should be considered. An organic molecule with a high molecular weight were selected as the dopant of GI POF. It had a larger molecular volume than ordinary small organic molecules. The large molecular volume could prohibit the diffusion of the dopant molecules. Furthermore, the dopant and PMMA had similar groups and possessed good miscibility. As a result, the dopant molecules could not easily diffuse to degrade the index profile of GI POF.

The bandwidth of an optical fiber determines the maximum information transmission capacity. It is expressed as the product of the frequency and distance (MHz km or GHz km). The bandwidth properties of optical fibers are best understood in terms of the minimum time that must be used between pulses. The width of a pulse propagating in an optical fiber increases with increasing distance of propagation caused by dispersion in the optical fiber. Therefore, if insufficient time is allowed between pulses, the pulses will overlap and not be distinguished by the detector.

The degradation of the refractive-index profile of GI POF mentioned previously directly influenced its bandwidth characteristics. The pulse-broadening phenomenon was investigated for 100-m GI POF before and after oven-aging at 60°C for 360 days. The fiber used in this experiment was 2 wt % organic-molecule-doped GI POF, and the index profile stability at 60°C is shown in Figure 2. The bandwidth measurements of GI POF are shown in Figure 3. A pulse width change was hardly observed for GI POF even after aging for 360 days. The good thermal stability of GI POF resulted mainly from the high T_g value of the core center of the fibers. The distribution of the refractive index decreased gradually from the core to the cladding and was similar to that reported in the literature for PMMA-based GI POF.^{3,13–15} However, the T_g values of the samples in this work almost equaled those of the

polymer matrix. The organic molecules of the dopants had higher molecular weights than the dopants of the references reported. The plasticization effect of the dopants had very little influence on the polymer. Thus, T_g of GI POF was close to that of the PMMA homopolymer.

Attenuation is the optical loss induced as a signal is propagating along a fiber; that is, while light is guided through a fiber, the intensity of the light decreases. Attenuation determines the distance over which a signal can be transmitted, and the transmission distance is inversely proportional to the attenuation. Figure 4 shows the heat-aging properties of GI POF measured at 60°C with a 650-nm wavelength. The transmission loss increased slightly with heat aging. This result could be due to the following reasons. When a fiber sample is exposed to a high temperature, a transmission loss occurs from the degradation, that is, oxidized degradation of the core polymer. In chemical terms, carbonyl groups may be formed at this time, and crosslinking and double bonding also occur because of molecular dissociation or an elimination reaction. The formation of carbonyl groups, crosslinks, and double bonds will, in turn, induce an increase in the electron

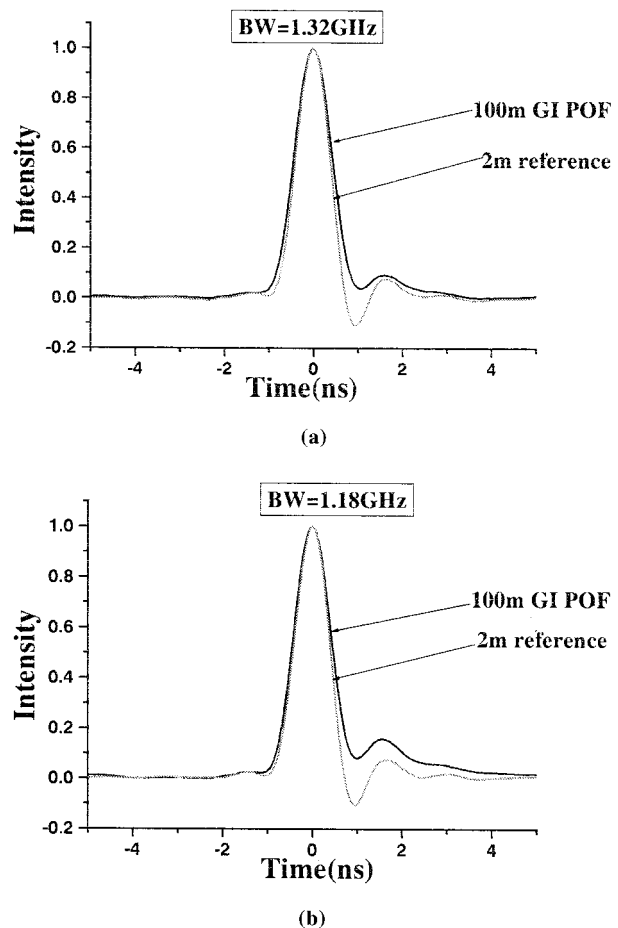


Figure 3 Bandwidth of organic-molecule-doped GI-POF (a) before and (b) after aging at 60°C for 360 days.

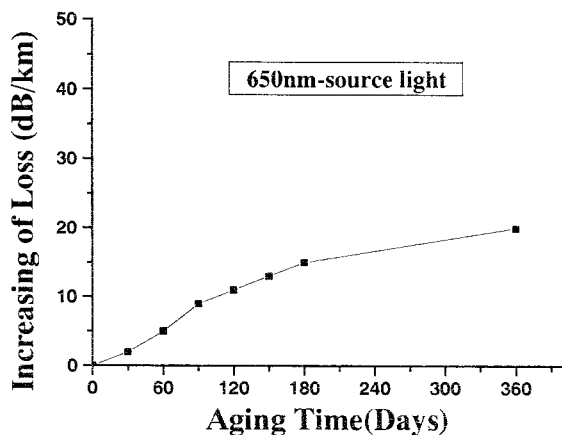


Figure 4 Effect of the aging time on the loss of GI POF at 60°C.

transition absorption and an adaptive shift toward a longer wavelength band.¹⁶ There are several reasons for the high stability of the samples in this work. First, the PMMA core had a T_g value of 103°C, which was much higher than the heat-aging temperature. Under this condition, oxidized degradation of the core polymer did not occur. Second, PMMA and the dopant used constitute a good miscible system, and no large heterogeneous structure could be formed in GI POF through aging. All these factors resulted in the Rayleigh scattering loss decreasing. As a result, the loss due to heat aging increased a little. As shown in Figure 4, an increase of approximately 20 dB/km in the attenuation could be observed in GI POF through aging at 60°C for 360 days.

Hot-water/humidity resistance

The water molecules absorbed in the polymer matrix affected the stability of the fibers.¹⁷ All of the afore-

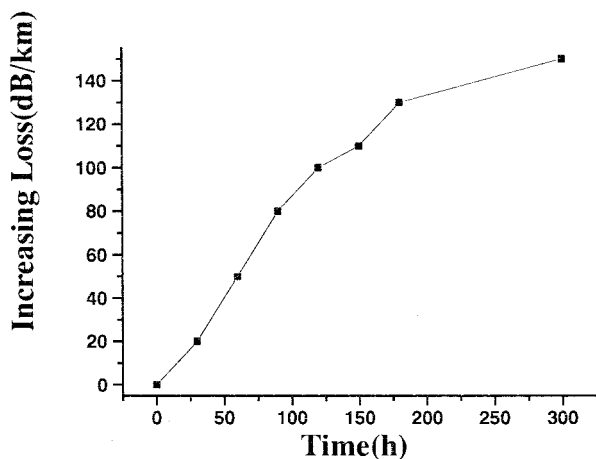


Figure 5 Dependence of the loss of GI-POF on the aging time during exposure to hot water (60°C).

TABLE I
Mechanical Properties of GI POF Before Aging

Sample	Yield strength (MPa)	Breaking strength (MPa)	Yield elongation (%)	Breaking elongation (%)
1	93.8	97.7	10.4	28.8
2	94.7	98.7	10.3	27.9
3	93.2	97.3	10.3	27.8
4	93.2	97.8	10.0	27.5
5	93.3	96.7	9.9	26.9

mentioned stability tests were performed at room humidity.

The transmission loss of the fiber exposed to a hot-water environment at 60°C was also studied, and the results are illustrated in Figure 5. An important factor causing large attenuation was the scattering loss due to some aggregation of water molecules in GI POF, which depended on the affinity of water to the polymer matrix. It was reported that PMMA, the polymer matrix of GI POF, absorbed 2 wt % water when the PMMA bulk was placed in hot water at 60°C¹⁸ because the carbonyl groups in PMMA could form hydrogen bonds with hydroxyl groups in water. In addition, the dopant molecule had groups similar to those of PMMA and, therefore, would not affect the absorption of water significantly. From Figure 5, it is clear that the loss increased to 150 dB/km after 300 h of aging in hot water (60°C). As a result, the attenuation increased greatly at a high temperature and a high humidity. In view of this large increase in the transmission loss (Fig. 5), the PMMA-based GI POF in this work was not expected to have sufficiently high heat and humidity resistance in hot water.

Mechanical properties

Mechanical properties are significant to optical fiber applications. We studied the mechanical properties of GI POF before and after 360 days of aging at 60°C, and the obtained results are given in Tables I and II, respectively. The five samples after 360 days of aging at 60°C had a tensile yield strength of 93.5 MPa, a tensile yield elongation of 9.9%, a breaking strength of 97.4 MPa, and a

TABLE II
Mechanical Properties of GI POF After Aging for 360 Days at 60°C

Sample	Yield strength (MPa)	Breaking strength (MPa)	Yield elongation (%)	Breaking elongation (%)
1	93.5	97.5	10.2	28.5
2	94.5	98.5	10.1	27.8
3	93.0	97.0	10.0	27.5
4	93.5	97.5	9.8	27.3
5	93.0	96.5	9.7	26.8

breaking elongation of 27.6%; this qualified the samples as safe optical fibers. It was also verified that GI POF permitted an easy termination treatment by a hot-plate method or a grinding method.

CONCLUSIONS

The stability of PMMA-based GI POF was determined. T_g of the fiber core was kept at a temperature as high as 103°C in the presence of the dopant. A special refractive-index profile was stable at 60°C. Moreover, GI POF had mechanical properties similar to those of the PMMA matrix. This outstanding performance mainly came from the dopant, which had a higher molecular weight. It was suggested that GI POF could be applied to both indoor and outdoor uses. However, the PMMA-based GI POF in this work exhibited poor hot-water/humidity resistance.

References

1. Koike, Y.; Ishigure, T.; Nihei, E. *J Lightwave Technol* 1995, 13, 1475.
2. Yabre, G. *J Lightwave Technol* 2000, 18, 869.
3. Ishigure, T.; Nihei, E.; Koike, Y. *Appl Opt* 1994, 33, 4261.
4. Toshikuni, K.; Michiya, F.; Shigo, N. *J Appl Phys* 1981, 52, 7061.
5. Yamashita, T.; Kamada, K. *Jpn J Appl Phys* 1993, 32, 2681.
6. Emslie, C. *J Mater Sci* 1988, 23, 2281.
7. Han, B. X.; Xu, J. *Chin. Pat. Appl.* 00134132.4 (2001).
8. Liu, Z. M.; Dai, X. H.; Xu, J. *Chin. Pat. Appl.* 02157893.1 (2002).
9. Marcuse, D. *Appl Opt* 1979, 18, 2073.
10. Dai, X. H.; Ai, X. C.; Liu, Z. M.; Yang, M. J.; Yang, G. Y.; Han, B. X.; Xu, J. *Chin Sci Bull* 2002, 47, 982.
11. Masataka, S.; Takaaki, I.; Yasuhiro, K. *J Lightwave Technol* 2000, 18, 952.
12. Ishigure, T.; Sato, M.; Nihei, E.; Koike, Y. *Jpn J Appl Phys* 1998, 37, 3986.
13. Koike, Y.; Nihei, E.; Tanio, N.; Ohtsuka, Y. *Appl Opt* 1990, 29, 2686.
14. Yang, S. Y.; Chang, Y. H.; Ho, B. C.; Chen, W. C.; Tseng, T. W. *Polym Bull* 1995, 34, 87.
15. Koike, Y.; Hatanaka, H.; Ohtsuka, Y. *Appl Opt* 1984, 23, 1779.
16. Takezawa, Y.; Tanno, S.; Taketani, N.; Ohara, S.; Asano, H. *J Appl Polym Sci* 1991, 42, 2811.
17. Sato, M.; Hirai, M.; Ishigure, T.; Koike, Y. *J Lightwave Technol* 2000, 18, 2139.
18. Sato, M.; Ishigure, T.; Nihei, E.; Koike, Y. *Proc Int Plast Fibers Conf* 1998, 7, 108.