Fabrication of 100-μm-bore hollow fiber for infrared transmission

Katsumasa Iwai[1)], Mitsunobu Miyagi[1)], Yi-Wei Shi[2),*], Xiao-Song Zhu[2)], Yuji Matsuura[3)]

1) Sendai National College of Technology,
4-16-1 Ayashi-chuo, Aoba-ku, Sendai, 989-3128, Japan.
2) School of Information Science and Engineering, Fudan University,
220 Handan road, Shanghai 200433, China
* Email: ywshi@fudan.edu.cn, TEL/ FAX: +86-21-5566-4945
3) Graduate School of Engineering, Tohoku University,
Aramaki Aoba, Aoba-ku, Sendai 980-8579, Japan.

Abstract

Extremely flexible hollow fibers with 100 μm-bore size were developed for infrared laser delivery. The hollow fiber was inner coated with silver and a dielectric layer to enhance the reflection rate at an objective wavelength band. The silver layer was plated by using the conventional silver mirror-plating technique. And a thin dielectric layer was coated for low-loss transmission of Nd:YAG and Er:YAG laser light.

KEYWORD: hollow fiber, small-bore, infrared laser.

1. Introduction

Er:YAG laser, radiating at the wavelength of 2.94 μm, is an effective laser in biomedical field for incision and ablation. Low-loss hollow optical fiber for delivering Er:YAG laser light has been successfully developed [1-3], and the application of the Er:YAG laser is rapidly expanding in almost all the subfields in medicine. In many medical applications, there is a critical requirement for ultra-thin infrared fiber. Hollow fiber with an outer diameter less than 200 μm is a typical one. In dentistry, an edged tool with 160 μm outer diameter, being called *the Fiber*, is often used to treat the root of a bad tooth so as to keep the bad tooth from being removed. The infection parts at the root can be removed by using *the Fiber*. However, *The Fiber* is easily becoming blunt and the treatment time has to be prolonged. It is possible to shorten the treatment time to irradiate the infected part by using Er:YAG laser light. Therefore, the development of a patient-friendly thin hollow fiber for delivering

Optical Fibers and Sensors for Medical Diagnostics and Treatment Applications VIII,
edited by Israel Gannot, Proc. of SPIE Vol. 6852, 68520S, (2008) · 1605-7422/08/$18
doi: 10.1117/12.767934

ErYAG laser light has been one of the important research themes in laser dentistry.

We have been working on the research and development of infrared hollow fibers with bore-size ranging from 250 μm to 1000 μm for medical applications. Correspondingly, the outer diameters are ranging from 320 μm to 1500 μm. The hollow fiber was inner coated with a dielectric and a silver layer to reduce the transmission loss and obtain high-performance properties such as multi-wavelength delivery [4-6]. In the minimally invasive treatment of the tooth root by using an endoscope, we are trying to introduce a thinner hollow fiber for delivery the Er:YAG laser light. In this paper, we report on the fabrication of extremely flexible, low-loss, infrared hollow fiber with 100 μm-bore size.

2. Fabrication of the silver-coated ultra-thin hollow fiber

In the fabrication of a small-bore hollow fiber, a silica capillary tube is used as the base tube. The inner/outer diameters of the silica capillary are 100/170 μm, including a polyimide protective coating with 12 μm thickness on the outside surface to keep its flexibility and durability. The capillary can be safely bent with a bending radius as small as 2.5 mm, which is much more flexible than the fiber with 250 μm inner diameter that we developed before [4]. We coat silver layer and a dielectric layer as the high-reflection films on the inner surface of the capillary. In order to obtain a low-loss property at an objective wavelength, we have optimized the film thickness of the dielectric layer [7]. For the dielectric material, we used organic material such as cyclic olefin polymer (COP) and inorganic material such as SiO_2 (OC300 for the trade name of the coating material) [8].

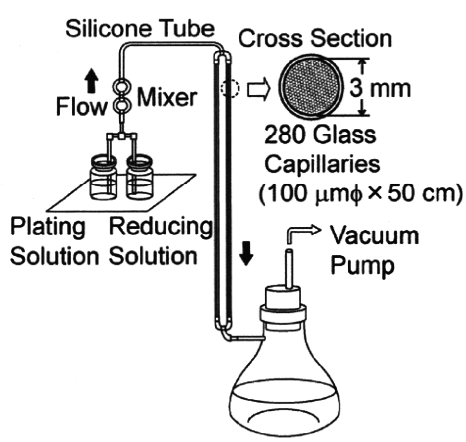

Figure 1. Experimental setup for depositing a silver layer inside the silica capillary.

Figure 1 shows the experimental setup for silver plating in the capillary tube. The silica capillary was with a length of 50 cm and an inner diameter of 100 μm. In the plating process, a silver film with the thickness of around 0.1 μm was formed. As shown in fig. 1, the plating solution and the reducing solution were forced to flow through the silica capillary. The two solutions were thoroughly mixed in the mixer. Silver reduced in the chemical reaction adhered on the inner surface and thus formed a silver layer. However, in fabrication of small-bore hollow fibers the flowing rate of the solutions became so lower that the two solutions could not be fully mixed before entering the capillary. This caused rough silver surface and even get stuffed by silver in the thin capillary.

In this research we have improved the fabrication techniques to solve these problems.

a. An $SnCl_2$ water solution was used as an activation solution to initialize the inner surface of the capillary before the silver-mirror plating. With the pretreatment, the reducing process was much more rapid and the silver layer could be formed in a much shorter time.
b. In order to increase the flowing rate, we made two bundles with 280 pieces of silica capillary (inner/outer diameters of 100/170 μm and length of 50 cm) to increase the cross-sectional area. The bundles were connected to silicone tubes with inner/outer diameters of 3/5 mm.

Concerning the silver plating parameters used up to now in the fabrication of hollow fibers with 700 μm bore-size, we targeted the flowing rate of the solutions at 15 ml/min. To achieve the target, we paralleled two bundles each with 280 pieces of capillary. The flowing rate was measured 14.5 ml/min. The silver mirror reaction process lasted for 3 minutes. An afterwards-treatment to evaporate water in the hollow core was also conducted, the fiber were kept in a 150°C furnace for 30 minutes, while nitrogen gas flowing through the hollow core at the rate of 300 ml/min.

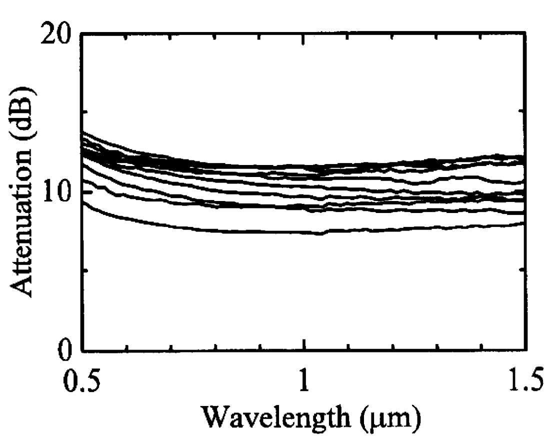

Figure 2. Loss spectra in the VI-NIR regions for the silver-coated hollow fiber with 100 μm-bore size and 35 cm length. The fiber was excited with a Gaussian beam with FWHM 10.6 °

Figure 2 shows the loss spectra in the visible and the near infrared (VI-NIR) regions for the hollow fibers with an inner silver layer. In the measurement, the fiber was excited by a Gaussian beam with a full width at half maximum (FWHM) of 10.6°. It can be seen that the attenuation of the fibers distributed in a relatively large range. The loss difference between the minimum and the maximum was 4.3 dB. The large difference might be caused by the imperfection in the fiber bundle preparation. When we made the bundle with 280 pieces of hollow fibers, it was difficult to keep the end face tidiness. Therefore, the flowing rate of the solutions in each fiber could be largely different. However, the lowest attenuation for the fiber with 35 cm length was 7.5 dB at the wavelength of 1 μm for Nd:YAG laser light, which was a quite low attenuation for the thin hollow fiber. Furthermore, the fabrication method can produce 560 pieces of silver-coated hollow fibers in one silver mirror reaction process, which is important to a batch production.

Up to now, we have fabricated hollow fibers with various inner diameters ranging from 250 to 1000 μm based on the commercially available silica capillary (inner diameters of 250, 320 540, 700 and 1000 μm). We used the cutback method to measure the attenuation properties of the hollow fiber. A reference fiber with 10 cm length and a measured fiber with 30 cm length were used. The outputs of the 10 cm long fiber and the 30 cm long fiber were measured and recorded. Then the loss spectrum of a 20 cm long fiber was calculated. The loss spectra in VI-NIR regions for the hollow fiber with various inner diameters were measured and calculated. Figure 3 shows their losses at the wavelength of 1 μm (excited by a Gaussian beam with FWHM=10.6°).

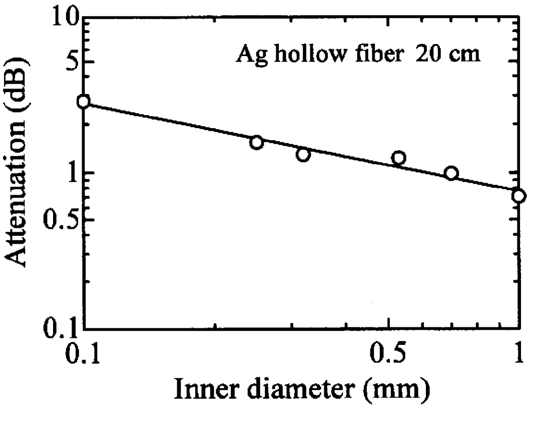

Figure 3. Transmission attenuation of the hollow fiber with various inner diameters. The fiber is with the length of 20 cm. The attenuation was measured at the wavelength of 1 μm and the fiber was excited by a Gaussian beam at FWHM 10.6°.

Theoretically, the loss of the hollow fiber is in inverse proportion to the cube of the inner diameter. The theory has been well proved by the loss properties of the hollow fiber produced before. We can see from fig. 3 that the loss of the 100 μm-bore hollow fiber is on the loss-property line of hollow fiber with larger bore-size. We can conclude that the small-bore hollow fiber attained the same low-loss level as these fibers we had produced before.

3. The dielectric film coating for the thin hollow fiber

We coated dielectric film on the silver layer to reduce transmission loss. For the dielectric material, we used cyclic olefin polymer (COP) and OC300 (SiO_2) [8]. For COP film coating, a COP cyclohexane solution with a concentration of 8.5 wt% was used. The silver-coated hollow fiber was kept straight and vertically fixed on a stable stage. The COP solution was flown through the silver-coated hollow fiber with a constant speed. The flow speed of the COP solution was set at around 10 cm/min. As the afterwards treatment to evaporate the solvent cyclohexane in the liquid-phase film, we flew nitrogen gas through the hollow core at the flowing rate of 100 ml/min. Then a COP film was formed on the silver layer. However, the COP solution tended to be stuffed easily due to the ultra thin hollow core.

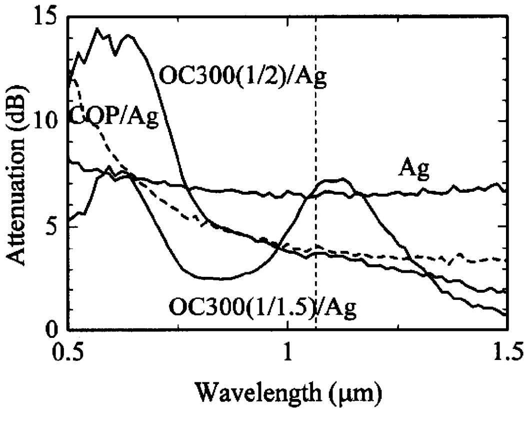

Figure 4. Loss spectra of COP/Ag and OC300/Ag hollow fibers (100 μmϕ×10 cm) in VI-NIR regions (FWHM = 10.6°). OC300 (1/2) and OC300 (1/1.5) mean that 10:1:20 and 10:1:15 mixing rate solutions were used in the fabrication.

We also tried to use an inorganic material OC300 [8] as the dielectric film. OC300 is the trade name of the coating material. It is a semi-inorganic polymer based on the structural unit R_2SiO, where R is an organic group. The material is characterized by wide-range thermal stability, water repellence, and physiological inertness. The OC300 coating material was originally developed for protecting the surface of a building or floor. It is a commercially available product and sold in a two-solution set. One is the main paint solution, and the other is a hardener. The two solutions are mixed at a certain rate. When the mixed solution is painted on a smooth surface, a solid SiO_2 film can be formed at the room temperature with the catalysis of water in the air.

There is another solvent that can be used to dilute the mixed solution. It is possible to modify the viscosity of the mixed solution by a certain amount of the solvent. In this research, the mixing rate among these three solutions (the main paint solution, the hardener and the solvent) was 10:1:20 or 10:1:15. The coating method was the same as what we used for COP coating. The flowing speed of the solution was 10 cm/min. After the liquid-phase coating, the fiber was treat at the room temperature with nitrogen gas flowing through the hollow core for 1 hour. The OC300-coated silver (OC300/Ag) hollow fiber was fabricated. Comparing with the COP solution, the OC300 solution has a lower viscosity. The OC300 solution moves smoothly inside the thin hollow core without stuffing at halfway. Therefore, it is comparatively easier for the OC300 coating in the small bore hollow fiber. Figure 4 shows the loss spectra of the hollow fibers in the VI-NIR regions. The fibers are 10 cm long, with 100 μm bore-size and excited with a Gaussian beam with FWHM at 10.6°.

Comparing with the silver-coated hollow fiber, the COP/Ag and OC300 (1/2)/Ag hollow fibers attained low loss at the wavelength of 1 μm (OC300 (1/2)/Ag means a fiber used the 10:1:20 rate solution in its fabrication). The COP/Ag hollow fiber (dashed curve) had a COP film with a thickness of 0.1 μm. It is possible to deliver Nd:YAG laser light with low-loss. OC300 (1/2)/Ag hollow fiber had OC300 film with a thickness of 0.15 μm. It is also obtained low-loss at the wavelength of Nd:YAG laser light. To deliver the Er:YAG laser light radiating at the wavelength of 2.94 μm, a thicker dielectric film is required. Normally a solution with higher concentration is need for a thicker film coating. Because COP solution has a high viscosity, it is almost impossible to drive a thicker solution through the 100 μm bore hollow core. While OC300 solution has a low viscosity, we increase the concentration by the mixing rate of 10:1:15. As seen in fig. 4, the OC300 film is evaluated as 0.25 μm. and the fiber was expected to have a low-loss property at the wavelength of 2.94 μm for the Er:YAG laser light. Furthermore, the clear interference peaks in the VI-NIR regions indicated that a uniform and smooth OC300 film had been achieved.

In order to evaluate the transmission loss for the Er:YAG laser light of the 100 μm bore hollow fiber, we used a tapered coupler to couple the laser light into the small core. In the measuring experiments, the fiber was 10 cm long with a 100 μm bore-size. The tapered coupler was 1 cm long and the inner diameters of the input and output ends were 700 and 100 μm, respectively. The Er:YAG laser was set at an output mode with 10 Hz repetition rate, 300 μs pulse width and 33 mJ energy per pulse. The tapered coupler was obtained by the glass-drawing method from a silica capillary with 700 μm bore-size [9]. The coupler was 6 cm long, in which 5 cm length was a uniform 700 μm bore hollow fiber and the rest 1cm tapered off to 100 μm bore-size gradually. The Er:YAG laser light was coupled into the 100 μm bore hollow fiber with the tapered coupler. The measured losses for the thin hollow fibers are summarized in table 1. The tapered coupler itself has a loss of 1.4 dB. And the minimum loss for the 100 μm bore hollow fiber was OC300 (1/1.5)/Ag hollow fiber, which was still as high as 8.8 dB. This might be cause by the tapered coupler. Because the taper coupler excited more high-ordered modes in the measured thin hollow fiber, which are of much higher attenuation coefficients than those of the low-ordered modes. It is necessary to optimize the shape of the tapered coupler to reduce the loss of the thin hollow fiber.

Table 1 Transmission characteristics for Er:YAG laser

	Energy (mJ)		Loss (dB)
	Input	Output	
Ag	24	0.7	15.3
COP/Ag	24	1.4	12.3
OC300 (1/2)/Ag	24	1.8	11.2
OC300 (1/1.5)/Ag	24	3.2	8.8

4. Conclusion

Small-bore hollow fiber with 100 μm inner diameter was fabricated. Comparing with the loss properties of the hollow fibers with larger bore-size produced before, the thin hollow fiber obtained low-loss property. The COP and OC300 were used as the dielectric film. For a Nd:YAG hollow fiber, dielectric film thickness of around 0.1 μm was coated and the loss was much lower than a silver-coated hollow fiber. A thicker OC300 film with 0.25 μm was also successfully coated, the fiber attained low-loss for Er:YAG laser light radiating at the wavelength of 2.94 μm. The loss for the 100 μm bore, 10 cm length OC300/Ag hollow fiber was 8.8 dB. The OC300 coated thin hollow fiber are of low-loss property, extreme flexibility and high durability, which is promising in many applications.

Acknowledgements

This research is supported by the Ministry of Education, Science, Sports and Culture of Japan through a Grant-in-Aid for Scientific Research (A) (17206033) and (B) (19760240) 2007, as well as by the National Nature Science Foundation of China (60608013) and the Shanghai Pujiang program (07pj14012).

Reference

1. J. Raif J, M. Vardi, O. Nahlieli, and I. Gannot, "An Er:YAG laser endoscopic fiber delivery system for lithotripsy of salivary stones," Laser in Surgery and Medicine **38**, 580-587 (2006).
2. S. Sato, Y. W. Shi, Y. Matsuura, M. Miyagi, and H. Ashida, "Hollow waveguide based nanosecond, near-infrared pulsed laser ablation of tissue," Lasers in Surgery and Medicine **37**, 149-154 (2005).
3. V. S. Johnson, B. Bowden, and J. A. Harrington, "Polymer/metal sulfide coated hollow waveguides for delivery of Er:YAG laser radiation," Proc. of SPIE **6433**, 64330E-1-64330E-8 (2007).
4. Y. Abe, Y. W. Shi, Y. Matsuura, and M. Miyagi, "Flexible small-bore hollow fibers with an inner polymer coating," Opt. Lett. **25**, 150-152 (2000).
5. M. Nakazawa, Y. W. Shi, Y. Matsuura, K. Iwai, and M. Miyagi, "Hollow polycarbonate fiber for Er:YAG laser light delivery," Opt. Lett. **31**, 1-3 (2006).
6. K. Iwai, Y. W. Shi, M. Miyagi, and Y. Matsuura, "Improved coating method for uniform polymer layer in infrared hollow fiber," Opt. & Laser Technol. **39**, 1528-1531 (2007).
7. M. Miyagi and S. Kawakami, "Design theory of dielectric-coated circular metallic waveguides for infrared transmission," IEEE J. Lightwave Technol. **LT-2**, 116-126 (1984).
8. K. Iwai, Y. W. Shi, M. Miyagi, X. S. Zhu, and Y. Matsuura, "Hollow infrared fiber with an inorganic inner coating layer with high durability," Proc. of SPIE **6433**, 64330L-1-64330L-7 (2007).
9. S. Narita, Y. Matsuura, and M. Miyagi, "Tapered hollow waveguide for focusing infrared laser beams," Opt. Lett. **32**, 930-932 (2007).

Fabrication and characterization of infrared hollow fiber with multi- SiO_2 and AgI inner-coating layers

Xiao Lin,[1] Yi-Wei Shi,[1,*] Ke-Rong Sui,[1] Xiao-Song Zhu,[1]
Katsumasa Iwai,[2] and Mitsunobu Miyagi[2]

[1]Department of Communication Science and Engineering, Fudan University, Shanghai 200433, China

[2]Sendai National College of Technology, 4-16-1 Ayashi-chuo, Aoba-ku, Sendai 989-3128, Japan

*Corresponding author: ywshi@fudan.edu.cn

Received 8 July 2009; revised 3 November 2009; accepted 5 November 2009;
posted 10 November 2009 (Doc. ID 113973); published 3 December 2009

We report the transmission characteristics of infrared hollow fiber with multi- AgI and SiO_2 inner-coating layers in the mid-infrared region. A three-dielectric-layer hollow glass fiber with a SiO_2–AgI–SiO_2–Ag structure was fabricated and low-loss property was obtained in the mid-infrared region. The SiO_2 films were coated by use of the liquid-phase coating method and a semi-inorganic polymer was used as the coating material. For deposition of the AgI film between the two SiO_2 films, a silver film was first plated by use of the silver mirror reaction method. Then the iodination process was conducted to turn the silver layer into silver iodide. A calculation method was also developed to estimate the film thickness of dielectric layers in each fabrication step according to the position of loss peaks in the measured loss spectra. Good agreement between calculated and measured loss spectra was demonstrated by taking into consideration material dispersion and surface roughness of inner-coating films. © 2009 Optical Society of America

OCIS codes: 060.2390, 230.4170, 040.6040, 160.5470, 260.2030.

1. Introduction

Hollow fiber [1–3] with inner coatings of a silver layer and a dielectric layer obtains low-loss property in infrared regions. It is capable of delivering key infrared lasers that are widely used in medicine and industry, including Nd:YAG, Ho:YAG, Er:YAG, CO, and CO_2 lasers. The low-loss property can be attained at a target wavelength by selecting an appropriate dielectric film thickness according to the design theory [4]. Furthermore, to increase the number of dielectric layers is also an effective means to lower the attenuation of the hollow fiber.

The structure composed of periodically deposited dielectric layers of low and high refractive indices [5,6] is one of the most commonly used structures for multiple dielectric layer hollow fibers. However, one encounters many difficulties in fabricating multilayered hollow fiber. One is the fabrication technique for a uniform and durable hollow fiber. For the application of a conventional glass-drawing technique, it is not easy to find two appropriate optical materials that can maintain the original structure after the co-draw process. Other fabrication methods that involve sputtering, electroplate coating, chemical vapor deposition (CVD), and liquid-phase chemistry are proposed. Recently, several different materials were used for multilayer hollow fiber fabrication. Both PbS–CdS [7,8] and Ta_2O_5–SiO_2[9] pairs were proposed, and fabricated three-layer hollow fiber showed low-loss property in the near-infrared regions. Limited by the coating techniques, thick films tend to have a rough surface and high nonuniformity. To avoid additional loss caused by the imperfections, these multilayer fibers need thin films targeted at near-infrared regions for Nd:YAG lasers.

We report the fabrication of multilayer hollow fiber with a SiO_2–AgI–SiO_2–Ag structure. AgI and SiO_2 were chosen because they have been studied

0003-6935/09/356765-05$15.00/0

extensively with regard to hollow fibers and the coating techniques have been well established. We carried out research using these two materials separately [10,11]. Coating techniques have been improved to obtain smooth and uniform optical films in the hollow air core surface. Furthermore, it is possible for the coating process to avoid high temperature curing that could destroy the flexibility of a hollow glass fiber. The calculated loss spectrum of multilayer hollow fiber was used to estimate the film thickness of each layer. Loss spectrum in each fabrication step for the SiO_2–AgI–SiO_2–Ag hollow fiber was measured and good agreement with the calculated results was demonstrated when we took into consideration material dispersion and surface roughness of inner-coating films.

2. Theoretical Loss Spectrum

The theoretical calculation for loss spectrum is based on the ray-optics theory [12]. For a hollow fiber of length z, the transmitted power $P(z)$ is

$$P(z) = \int_0^{\theta_{\max}} P_0(\theta) \exp\left[-\frac{1-R(\theta)}{2T\cot\theta} z\right] \sin\theta \mathrm{d}\theta, \quad (1)$$

where $P_0(\theta)$ is the angular distribution of the incident beam, $R(\theta)$ is the power reflection coefficient, T is the inner radius of the hollow core, and $\theta_{\max}$ denotes the maximum launching angle. For multilayer hollow fiber, $R(\theta)$ can be calculated from characteristic matrix M:

$$M = \begin{bmatrix} m_{11} & m_{12} \\ m_{21} & m_{22} \end{bmatrix} = \begin{bmatrix} \cos(k_0 nz \cos\theta) & -\frac{i}{p}\sin(k_0 nz \cos\theta) \\ -ip \sin(k_0 nz \cos\theta) & \cos(k_0 nz \cos\theta) \end{bmatrix}, \quad (2)$$

where $p = \sqrt{\frac{\varepsilon}{\mu}}\cos\theta$ for the TE mode and $p = \sqrt{\frac{\mu}{\varepsilon}}\cos\theta$ for the TM mode [13]. Then the reflective coefficient can be expressed as

$$r = \frac{(m_{11} + m_{12})p_l - (m_{21} + m_{22}p_l)}{(m_{11} + m_{12})p_l + (m_{21} + m_{22}p_l)}. \quad (3)$$

With regard to the multiple-layer calculation, matrix M_{total} can be calculated as

$$M_{\text{total}} = M_1(z_1)M_2(z_2 - z_1)M_n(z_N - z_{N-1}), \quad (4)$$

where M_1 is the characteristic matrix of the dielectric layer closest to the air core of hollow fibers, M_2 is that of the layer beneath the former layer, and M_n is that of the layer on the metal layer. Then the equivalent $R(\theta)_{\text{total}}$ for multiple dielectric layers can be derived from the equivalent characteristic matrix M_{total}. For the calculation, $R(\theta)$ depends on the refractive index of the dielectric film, and material dispersion has a significant influence on the shape and positions of low-loss valleys of the loss spectra. Therefore, material dispersion [14] for AgI and SiO_2 is also taken into consideration for the loss spectrum calculation.

A. Loss Spectrum Calculation

Figure 1 shows the calculated results for the loss spectra of hollow fiber with a single SiO_2 layer and fifteen dielectric layers (eight SiO_2 layers and seven AgI layers alternately). Both fibers attain low-loss property at the target wavelength of 2.94 μm. The bandgap effect of the multilayer coating can be observed and thus has a much lower loss than that of the single-layer coating hollow fiber. The calculated results also show that loss at the target wavelength decreases dramatically with an increase in the number of dielectric layers. When the number is greater than 100, loss does not decrease quickly, because the bandgap effect has been well established by the high reflection rate of the outer metallic layer [9].

B. Film Thickness Estimation

The optimized film thickness is important to realize low loss at a target wavelength. For single-layer hollow fiber, the film thickness can be easily calculated according to the wavelength of interference peaks in the measured loss spectrum [4]. For multilayer hollow fiber, it is impossible to simply calculate the film thickness of each layer using a certain formula. We used the above-mentioned loss spectrum calculation method to estimate the thickness of each layer. When calculated and measured loss spectra are in good agreement, we conclude that the actual film thickness is equal to the thickness value in the calculation. Fabrication parameters that control the film thickness should be carefully adjusted according to the estimated results so that optimized film thicknesses can be obtained. As we have shown in previous publications [11,14], the shape of the loss spectra relies greatly on the dispersion property of film material. For the calculation, we used the following Cauchy formula for SiO_2 and AgI material derived in [14]:

$$\text{AgI}: n(\lambda) = 2.0216 + 0.0878/\lambda^2 - 0.0024/\lambda^4, \quad (5)$$

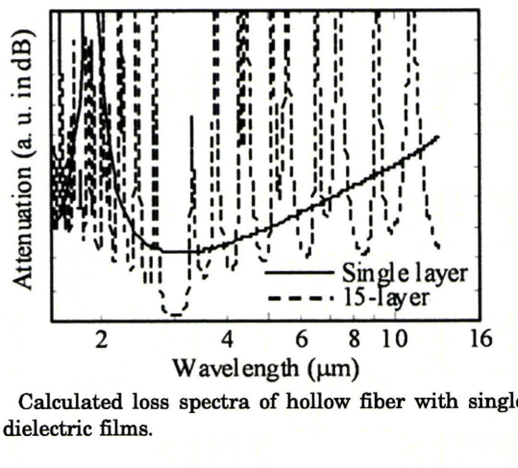

Fig. 1. Calculated loss spectra of hollow fiber with single and fifteen dielectric films.

$$SiO_2 : n(\lambda) = 1.42614 + 0.02729/\lambda^2 + 0.0001/\lambda^4. \quad (6)$$

Figure 2 shows the calculated loss spectra of hollow fiber with a SiO_2–AgI–SiO_2–Ag structure for Er:YAG laser delivery at 2.94 μm wavelength. The loss spectrum is shown when the three dielectric layers are all with the optimized thicknesses. When there is an error for the inner-coating layer thickness, the loss spectrum changes. Figure 2 also shows the calculated results when one of the three inner-coating layers becomes 20% thicker than the optimum. When the first layer (SiO_2) on the silver layer is 20% thicker, the loss spectrum is shown as the first layer in the figure. When the 2nd layer (AgI) or the third layer (SiO_2) is thicker, the loss spectra are shown as the 2nd layer and the 3rd layer in the figure, respectively. Considering the loss property at the target wavelength of 2.94 μm, we note that the second layer (the middle AgI layer) has the strongest impact on the transmission property when it has a thickness error. The first SiO_2 layer on the Ag film has the largest tolerance on the thickness error.

C. Surface Roughness

Limited by the film coating techniques, surface roughness exists for each layer. Normally, a thicker film has a rougher surface. The roughness causes additional loss whose property depends on the wavelength. Light reflected by a rough surface can be divided into two parts: diffuse reflection and specular reflection. By considering that the surface RMS roughness σ is much smaller than the operating wavelength, for simplicity, it is assumed that inclination of the surface is minimal and the energy of diffuse reflection could be disregarded. The effect of roughness on the reflected light can be regarded as the phase offset of the specular reflection of light [15]:

$$\Delta\phi = 2k_0 n_i \sigma \cos\varphi_i, \quad (7)$$

where φ_i represents the angle of incidence. Then the reflectance coefficient r on the rough surface can be expressed by the coefficient of the perfectly smooth surface r^0 [15]:

$$r = r^0 \exp[-(\Delta\phi)^2/2]. \quad (8)$$

Use of Eq. (8) for calculation of the theoretical loss spectrum yields the effect of roughness. Figure 3 shows the theoretical loss spectrum (black curve) of SiO_2–AgI–SiO_2–Ag hollow fiber when the three dielectric layers have perfectly smooth inner surfaces. Theoretical loss spectra were also added for comparison when one of the three layers had a rough surface. The green, red, and blue curves correspond to the calculation results when the first, second, and third layers have a RMS surface roughness of 30 nm. Note that, when the surface is rough, not only the loss increases but also the position of low-loss valley shifts to longer wavelengths. Similar to the influence of the film thickness error, the second layer of AgI has the strongest impact on the transmission property. The first SiO_2 layer on the silver film has the weakest influence. Therefore, much attention should be paid to the AgI coating during the fabrication process because the sensitivity of the transmission characteristics is more dependent on the property of the middle AgI film.

3. Experiments and Discussion

A. Fabrication Process

A glass capillary was used as the supporting tube for the hollow fiber because of its smooth inner surface and thus the potential for high quality optical film coatings. We fabricated a three-dielectric-layer hollow fiber with the structure of SiO_2–AgI–SiO_2–Ag. The thicknesses for each dielectric layer from air core to the metal layer are 0.75, 0.72, and 1.02 μm, respectively. Four steps are necessary for the fabrication process: the inner coating processes for Ag–SiO_2–AgI and SiO_2 films in sequence.

First, a Ag layer was deposited on the inner wall of the capillary tube by the liquid-phase chemistry method. The thickness of the silver layer was approximately 100 nm. A thinner layer is preferred

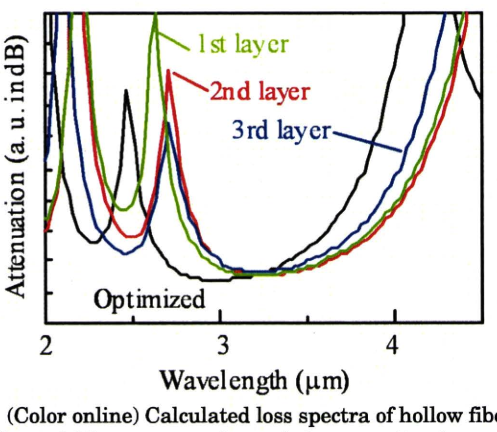

Fig. 2. (Color online) Calculated loss spectra of hollow fiber with and without thickness errors for each inner-coating layer.

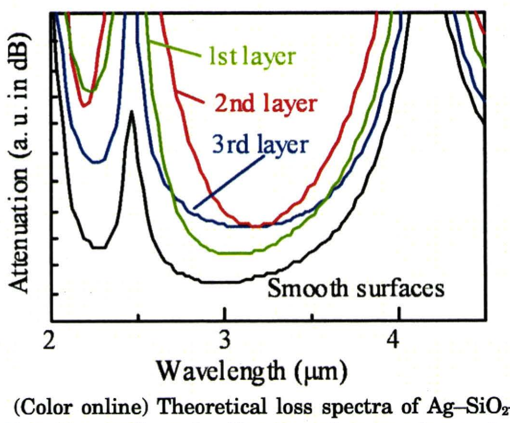

Fig. 3. (Color online) Theoretical loss spectra of Ag–SiO_2–AgI–SiO_2 hollow fiber with and without surface roughness for each inner-coating layer.

because it has less surface roughness. Second, we used OC300 [10] as the coating solution to form a SiO_2 layer on the silver layer. The OC300 solution is a semi-inorganic polymer based on the structural unit R_2SiO. The advantage of this material is that it can be formed at room temperature. Third, deposition of the AgI layer on the SiO_2 film, which can be divided into two separate parts: one part can be used to deposit a silver layer on the SiO_2 layer, the other is used to turn the silver layer into a silver iodide layer by the process of iodination. Finally, a process similar to the second one was used to form another SiO_2 layer on the AgI layer. By comparing the measured and the calculated loss spectra, we estimated the film thickness after each inner-coating process. Fabrication parameters that control the film thickness were carefully adjusted to obtain the expected film thicknesses.

B. Loss Property

The loss spectrum for each fabrication step is shown in Fig. 4. Calculated results based on the model developed in Section 2 are also added. By use of the fabrication process mentioned in Subsection 3.A, the silver and three dielectric layers were coated in order on the inner surface of the glass capillary tube.

Figure 4(a) shows the loss spectrum of the hollow fiber after the Ag and the first SiO_2 layer are deposited. The interference peak at the wavelength of 3 μm shows that the SiO_2 layer is 0.75 μm thick. The loss peak in the 8–10 μm wavelength band is the Si—O bond absorption in the thin film. The sharp peak at the 3.5 μm wavelength is the C—H bond absorption that is caused by the impurity in the silicone polymer. Figure 4(b) shows the hollow fiber with a Ag–SiO_2–Ag structure. The loss spectrum is typical for a Ag tube. Because the Ag layer on the SiO_2 film is thicker than the skin depth of the light and the SiO_2 layer provides minimal contribution to the optical property of the hollow fiber, no Si—O bond absorption can be observed, which means that no light penetrates the silver layer and transmits to the SiO_2 layer and is then reflected back to the air core. There are several sharp pulses in the spectrum that are caused by the noise of the measuring system and have no relation to the loss property. These sharp noise pulses are also shown in Fig 4(d).

The Ag layer near the air core in Fig. 4(b) was turned into a AgI layer during the next fabrication process. Figure 4(c) shows the hollow fiber with the AgI–SiO_2–Ag structure, which is the loss spectrum with two dielectric layers. The typical absorption of the Si—O bond can be observed in the loss spectrum, which means that light penetrates the AgI and SiO_2 layers and is then reflected back to the air core by the bottom Ag layer. The theoretical loss spectrum shows that the AgI layer is 0.72 μm thick. According to our calculations, there is an interference peak at 9.5 μm wavelength, which overlaps with the Si—O bond absorption.

Figure 4(d) shows the loss spectrum of the hollow fiber after the final coating process for the SiO_2 layer near the air core with the structure of SiO_2–AgI–SiO_2–Ag. We observed more high-loss peaks due to thin-film interference on both sides of the low-loss band around 5 μm. In comparison with the calculated results, we note that the Si—O bond absorption

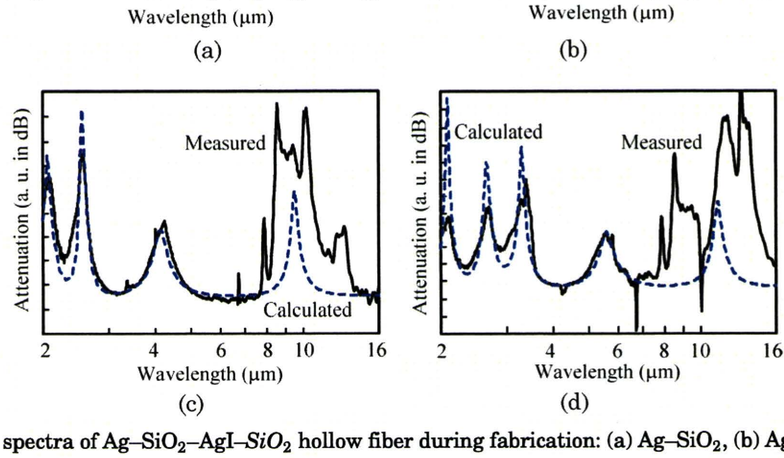

Fig. 4. (Color online) Loss spectra of Ag–SiO_2–AgI–SiO_2 hollow fiber during fabrication: (a) Ag–SiO_2, (b) Ag–SiO_2–Ag, (c) Ag–SiO_2–AgI, and (d) Ag–SiO_2–AgI–SiO_2.

appeared at the loss valley; the shape of typical Si—O bond absorption can be clearly seen.

Theoretically, the loss of the fiber decreases with the increase in dielectric layers. The loss in Fig. 4(d) with three layers is approximately 1 dB smaller than that in Fig. 4(a) with a single layer. The loss differences are not exactly the same as the calculated results in Fig. 1 because each inner-coating layer has a certain surface roughness. Based on the previous fabrication results for hollow fiber with AgI–Ag and SiO_2–Ag film structures, AgI film has a rougher surface than that of a SiO_2 film. However, for the multilayer inner coating, the surface roughness of a film also depends on the surface status of the previously coated layer. The baseline of the loss spectrum moves up and down in each coating process due to the roughness of each coating layer.

4. Conclusions

Transmission characteristics of infrared hollow fiber with multi- AgI and SiO_2 films have been discussed experimentally and theoretically. Three-dielectric-layer hollow fiber with the SiO_2–AgI–SiO_2–Ag structure was fabricated and low-loss property in the mid-infrared regions was obtained. Fabrication parameters were carefully adjusted according to thickness estimation by use of the loss spectrum calculation model. Theoretical calculation results for each fabrication step demonstrated good agreement with the measured data when material dispersion and surface roughness of dielectric film are taken into consideration.

This research is supported by the National Natural Science Foundation of China (NSFC#) (60971014), State Education Ministry (the Scientific Research Foundation for the Returned Overseas Chinese Scholars), , and the 211 project for construction of key disciplines, as well as by the Ministry of Education, Sports, Science, and Technology of Japan (Basic and Fundamental Research and Development Projects B), and Health and Labor Science Research Grants, H20-nano-young-010, 2008.

References

1. N. Croitoru, A. Inberg, M. Ben David, and I. Gannot, "Broad band and low loss mid-IR flexible hollow waveguides," Opt. Express **12**, 1341–1352 (2004).
2. R. George and J. A. Harrington, "Infrared transmissive, hollow plastic waveguides with inner Ag—AgI coatings," Appl. Opt. **44**, 6449–6455 (2005).
3. Y. W. Shi, K. Ito, L. Ma, T. Yoshida, Y. Matsuura, and M. Miyagi, "Fabrication of polymer-coated silver hollow optical fiber with high performance," Appl. Opt. **45**, 6736–6740 (2006).
4. M. Miyagi and S. Kawakami, "Design theory of dielectric-coated circular metallic waveguides for infrared transmission," J. Lightwave Technol. **2**, 116–126 (1984).
5. B. Temelkuran, S. D. Hart, G. Benoit, J. D. Joannopoulos, and Y. Fink, "Wavelength-scalable hollow optical fibers with large photonic bandgaps for CO_2 laser transmission," Nature **420**, 650–653 (2002).
6. Y. Fink, D. J. Ripin, S. Fan, C. Chen, J. D. Joannopoulos, and E. L. Thomas, "Guiding optical light in air using an all-dielectric structure," J. Lightwave Technol. **17**, 2039–2041 (1999).
7. V. Gopal and J. A. Harrington, "Deposition and characterization of metal sulfide dielectric coatings for hollow glass waveguides," Opt. Express **11**, 3182–3187 (2003).
8. M. Ben David, M. Catalogna, J. A. Harrington, V. Krishnan, and I. Gannot, "Theoretical and experimental investigations of metal sulfide dielectric coatings for hollow waveguides," Opt. Eng. **47**, 045008 (2008).
9. T. Karagiri, Y. Matsuura, and M. Miyagi, "Metal-covered photonic bandgap multilayer for infrared hollow waveguide," Appl. Opt. **41**, 7603–7606 (2002).
10. K. Iwai, M. Miyagi, Y. W. Shi, X. S. Zhu, and Y. Matsuura, "Infrared hollow fiber with a vitreous film as the dielectric inner coating layer," Opt. Lett. **32**, 3420–3422 (2007).
11. K. R. Sui, Y. W. Shi, X. L. Tang, X. S. Zhu, K. Iwai, and M. Miyagi, "Optical properties of AgI/Ag infrared hollow fiber in the visible wavelength region," Opt. Lett. **33**, 318–320 (2008).
12. Y. Matsuura, M. Saito, and M. Miyagi, "Loss characteristics of circular hollow waveguides for incoherent infrared light," J. Opt. Soc. Am. A **6**, 423–427 (1989).
13. M. Born and E. Wolf, *Principles of Optics* (Cambridge U. Press, 1999).
14. K. R. Sui, X. S. Zhu, X. L. Tang, K. Iwai, M. Miyagi, and Y. W. Shi, "Method for evaluating material dispersion of dielectric film in the hollow fiber," Appl. Opt. **47**, 6340–6344 (2008).
15. Y. Matsuura and J. A. Harrington, "Hollow glass waveguides with three-layer dielectric coating fabricated by chemical vapor deposition," J. Opt. Soc. Am. A **14**, 1255–1259 (1997).

Fabrication and transmission characteristics of infrared hollow fiber based on silver-clad stainless steel pipes

Katsumasa Iwai,[1] Akihito Hongo,[2] Hiroyuki Takaku,[1] Mitsunobu Miyagi,[1,3] Jun-ichi Ishiyama,[3] Xiao-Xu Wu,[4] Yi-Wei Shi,[4,*] and Yuji Matsuura[5]

[1]Sendai National College of Technology, 4-16-1 Ayashi-chuo, Aoba, Sendai 989-3128, Japan

[2]Hitachi Cable, Limited, 880 Isagozawa, Hitachi 319-1418, Japan

[3]Miyagi National College of Technology, 48 Nodayama, Shiote, Medeshima, Natori 981-1239, Japan

[4]School of Information Science and Engineering, Fudan University, 220 Handan Road, Shanghai 200433, China

[5]Graduate School of Biomedical Engineering, Tohoku University, 05 Aoba, Sendai 980-8579, Japan

*Corresponding author: ywshi@fudan.edu.cn

Received 3 August 2009; revised 3 October 2009; accepted 7 October 2009; posted 9 October 2009 (Doc. ID 115103); published 3 November 2009

Silver-clad stainless steel pipe is used as the supporting tube for the fabrication of infrared hollow fiber. The hollow fiber has high mechanical strength and is highly durable for use in the medical sterilization process. Film of a cyclic olefin polymer layer or silver iodide (AgI) was coated internally to reduce the transmission loss. A liquid-filling method is proposed for coating the AgI layer. Multiple coating processes proved to be effective to increase the AgI film thickness. A treatment of sodium thiosulfate water solution is also proposed to reduce the film thickness. The film thickness can be accurately controlled by combining the coating and decoating techniques. A loss of less than 0.2 dB was obtained for CO_2 laser light for a hollow pipe with a length of 280 mm and an inside diameter of 0.75 mm. © 2009 Optical Society of America

OCIS codes: 060.0060, 120.7000, 230.7370, 310.6860, 060.2270.

1. Introduction

CO_2 laser radiation at a wavelength of 10.6 μm has found applications in industrial processing owing to its high output power. It is also one of the most commonly used lasers in the medical field for laser surgery because of its high water absorption coefficient at 10.6 μm wavelength. The laser power transmission system has been critically needed and actively developed [1–6] because of the wide application of the CO_2 laser. We developed hollow fiber with polymer and silver inside-coating films based on a glass capillary with high flexibility. Low-loss infrared hollow fibers with inner diameters from 320 to 1000 μm and a length of 2 m were successfully developed [6].

In medical applications, such as dentistry and otorhinolaryngology, a sterilization process must be done for the recycled output probe of the infrared laser delivery system. It has been shown that the dielectric-coated silver hollow tips were damaged after several sterilization cycles [7] by use of an autoclave. It was observed that the coated silver layer was detached from the inner surface of the glass capillary, which is caused mainly by the rather thin Ag film. To obtain a smooth silver surface for better optical film, the silver film had to be thin [8], which is normally from 50 to 200 nm thick.

Here we use metal supporting pipes for fabrication of the infrared hollow fiber [9]. Fabrication and

0003-6935/09/326207-06$15.00/0

transmission characteristics of infrared hollow fiber based on stainless steel pipes are experimentally discussed. Cyclic olefin polymer (COP) [10] and silver iodide (AgI) [11] were used as the dielectric coating material. COP is a nontoxic, transparent material in the infrared region and can be used to form high quality optical film. AgI is a traditional infrared material that is suitable for high power delivery because of its good adhesion with silver film and its heat resistance. These two kinds of dielectric film were used in the hollow output probe in the fabrication; the transmission properties were experimentally evaluated.

2. Supporting Tube

We used a silver-coated glass capillary as the metallic hollow fiber because of its smooth inner surface. To increase durability for the sterilization process, we propose to use a metal hollow fiber. There are several kinds of supporting tube that have the potential for high durability and are commercially available. They are stainless steel pipe (SUS) with a smooth polished inner surface, gold-coated stainless steel pipe (Au/SUS), and silver-clad stainless steel pipe (Ag/SUS). (SUS is the code name assigned to stainless steel by the Japanese Industrial Standard.) The Ag/SUS tube is made by inserting a silver tube into a stainless steel tube or by using the extrusion process to simultaneously form the silver and the stainless dual-clad pipe. The inner wall of the Ag-clad tube was carefully polished. The silver tube has a wall thickness of 100 μm and is expected to be of high durability and low loss when inner coated with dielectric optical films. The Au-coated tube is fabricated by electroplating. The longest length made up to now is 280 mm. The length for a Ag-clad tube is limited to approximately 1 m because of the polishing techniques. Both Ag-clad and Au-coated tubes are custom-made products.

Figure 1 shows the structure of various metal pipes. Table 1 summarizes the size and transmission loss for CO_2 laser light of the metal pipes. Figure 2 shows the loss spectra for the metal pipes from the visible to the mid-infrared regions. The loss spectrum for a silver-coated glass capillary (Ag/SiO_2) was also added for comparison. Loss spectra for wavelengths less than or greater than 1.5 μm were measured with a spectrum analyzer and Fourier transform infrared spectroscopy (FTIR), respectively. SUS pipe has the highest loss because of its rough inner surface. Au/SUS pipe has a lower loss because of the gold film coating. The silver-clad Ag/SUS pipe in the SUS tube has the lowest loss. The property of the Ag/SUS pipe is the same as that of the silver-coated glass tube (Ag/SiO_2) in the mid-infrared region. The measured results are also shown in Table 1. Loss for CO_2 laser light is 1.7–1.8 dB for the Ag/SiO_2 and Ag/SUS pipes, respectively. During the measurement for CO_2 laser light delivery, a coupler was used to minimize the coupling loss between the laser light and the tube. The coupler was a Ag-coated hollow glass fiber with the same inner diameter as the measured tube. However, Ag/SiO_2 has low loss in the visible and near-infrared regions because the glass capillary has a smooth inner surface that guarantees a smooth Ag-layer plating and thus causes smaller additional loss in the shorter wavelength region. During fabrication for the metal pipes, a polishing process is needed to smooth the inner surface for optical film coating. According to observation with an atomic force microscope (AFM), the measured inner surface roughness has a 2 nm root mean square (rms) for a glass capillary and 50 nm for a well-polished stainless steel tube. Therefore, fabrication for long pipes is difficult. A short pipe has strong mechanical strength and is capable of withstanding sharp bending. A short pipe is adequate and safe for application as an output probe for the infrared delivery system.

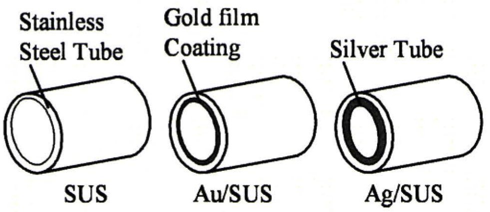

Fig. 1. Structure of various metal pipes.

Table 1. Size and Loss of a CO_2 Laser for Metal Pipes

	Size (mm)			
Material	Inner Diameter	Outer Diameter	Length	Loss (dB)
SUS	0.94	1.2	285	3.7
Au/SUS	0.94	1.2	285	2.1
Au/SUS	0.75	1.2	280	1.7
Ag/SiO_2	0.75	0.9	280	1.8

3. Polymer Inner Coating

Optical film coating on the metal surface can dramatically increase the reflectance rate of the surface. Therefore, it is commonly used to coat a dielectric film on the inner surface of a metal pipe to reduce transmission loss. COP and AgI are two successful dielectric coating materials for infrared hollow fiber.

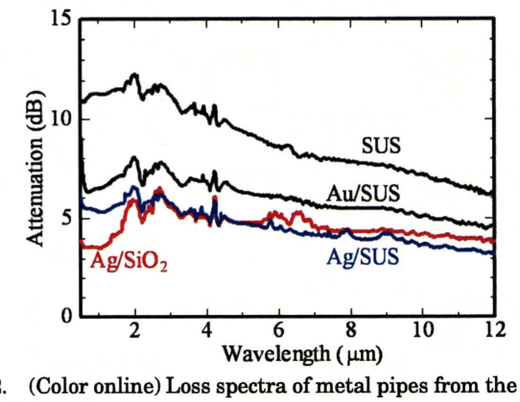

Fig. 2. (Color online) Loss spectra of metal pipes from the visible to the infrared regions.

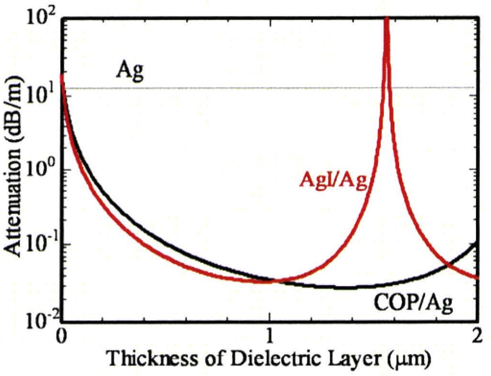

Fig. 3. (Color online) Theoretical losses of the HE_{11} mode in hollow fiber as a function of dielectric film thickness at a wavelength of 10.6 μm.

For a target wavelength, there is an optimum film thickness [12] for each dielectric material.

Figure 3 shows the theoretical loss of the HE_{11} mode in the dielectric-coated hollow fiber as a function of dielectric film thickness. The target wavelength is 10.6 μm for CO_2 laser light. For the calculation, the complex refractive index for Ag at the wavelength of 10.6 μm is $13.5 - j75.3$. The refractive indices for COP and AgI are 1.53 and 1.95 [13], respectively. We note in Fig. 3 that the optimum thicknesses for a minimum loss are 1.36 and 0.98 μm for COP and AgI layers, respectively. The loss of COP/Ag hollow fiber is slightly less than that of the AgI/Ag hollow fiber because the optimum refractive index for the dielectric layer is theoretically 1.41.

We used the COP, dissoved in cyclohexane, as the dielectric coating material. The solution was forced to flow through the metal pipe and a liquid-phase film was formed on the inner surface. After the curing process, the film was solidified to form a stable polymer layer. Detailed fabrication techniques and parameters to control film thickness have been published elsewhere [14]. Figure 4 shows the loss spectra for COP-coated metal pipes. The COP film thickness had been optimized for 10.6 μm wavelength. Table 2 summarizes the loss for CO_2 laser light of the COP-coated metal pipes.

In Fig. 4, loss peaks in the less than 5 μm wavelength region came from the interference effect of

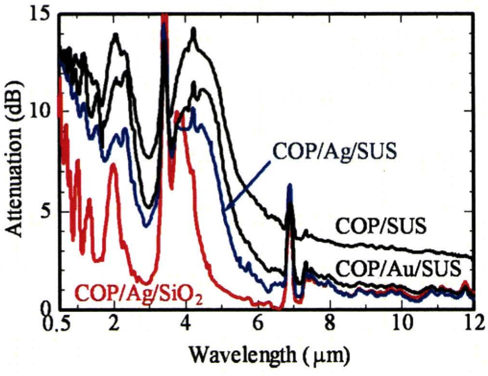

Fig. 4. (Color online) Loss spectra of COP-coated metal pipes.

Table 2. Loss of COP-Coated Metal Pipes for a CO_2 Laser

Material	Size (mm) Inner Diameter	Outer Diameter	Length	Loss (dB)
COP/SUS	0.94	1.2	285	0.6
COP/Au/SUS	0.94	1.2	285	0.22
COP/Au/SUS	0.75	1.2	280	0.18
$COP/Ag/SiO_2$	0.75	0.9	280	0.13

light in the thin COP optical film. According to the position of the interference peaks, the film thicknesses for the COP layer can be calculated. For the four kinds of COP-coated metal pipe, the film thickness ranges from 0.85 to 0.95 μm. In comparison with the theoretical optimum film, the coated film thickness is thinner. Because thicker film has a much rougher surface that causes additional loss, a thinner film is normally preferred for the fabrication process. Table 2 shows the measured results for CO_2 laser light. A low loss of around 0.2 dB was obtained for the hollow pipes, except for the COP/SUS pipe because the SUS pipe has a rough surface, which had been observed in the loss spectrum in Fig. 2.

4. Silver Iodide Coating

A. Liquid-Filling Method

The coating technique for the AgI layer is an iodination process that changes part of the Ag layer into a AgI layer. The iodine cyclohexane solution flows through the Ag-coated tube and the upper surface of the Ag layer changes into AgI. This AgI coating technique has worked well for hollow fiber with inner diameters ranging from 0.25 to 0.7 mm [15]. However, a rather high flow rate is necessary in the iodination process. To form the AgI layer, a 20 ml iodine solution is needed. We propose a new method that significantly reduces the waste of the iodine solution.

Figure 5 shows the new fabrication method. A syringe is used to fill the Ag/SUS tube with the iodine solution. The tube is completely filled, the solution is kept for a period of time for AgI layer formation, and then the iodine solution is flushed out. By using the newly developed method, the volume of the iodine solution is only 0.1 ml for the 280 mm long metal pipe with a 0.75 mm bore diameter. During the fabrication process, the iodine solution was made by dissolving iodine pellets in cyclohexane. Then the solution was stirred in a supersonic device for 10 min to mix the solution.

Figure 6 shows the AgI film thickness versus liquid-filling time. The concentrations of the iodine solution are also shown. The dashed line indicates the optimum film thickness for the Er:YAG laser that irradiates at 2.96 μm. The fabrication parameter for Er:YAG hollow pipe could have a 60 s filling time for an iodine solution with 0.5% concentration. The film thickness tends to be thicker when the filling time is prolonged. However, a saturation time exists for a certain concentration as can be observed in Fig. 6

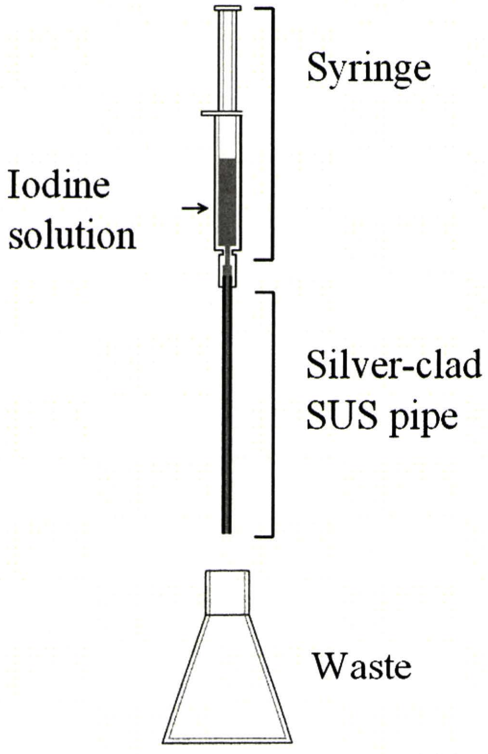

Fig. 5. Schematic experimental setup for the liquid-fill iodination process.

because concentration of the iodine solution decreases with the chemical reaction time and the AgI formed on the surface also controls the speed of the chemical reaction.

To form a thicker AgI film for CO_2 laser light, we used a solution with 1% concentration and multiple liquid-fill processes. Since one process could not achieve enough film thickness, we repeated the same filling process three to five times. Figure 7 shows the initial AgI film thickness versus the film thickness after repeated filling times. The filling times for each filling process are also shown in Fig. 7. The dashed line indicates the optimum film thickness for CO_2 laser light. It is obvious that we need to repeat the

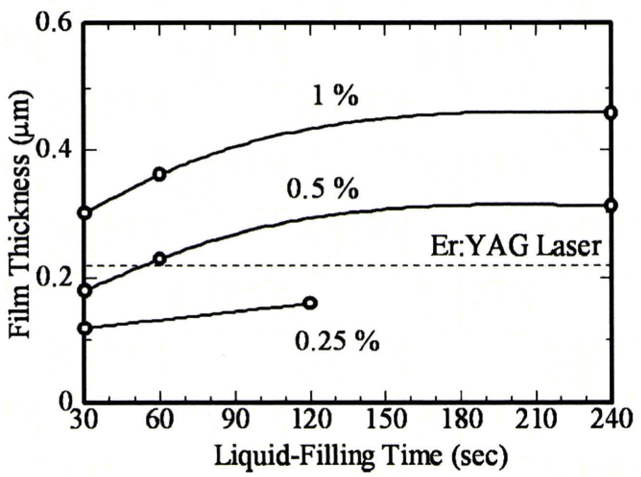

Fig. 6. AgI film thicknesses versus liquid-filling time for solutions with various concentrations.

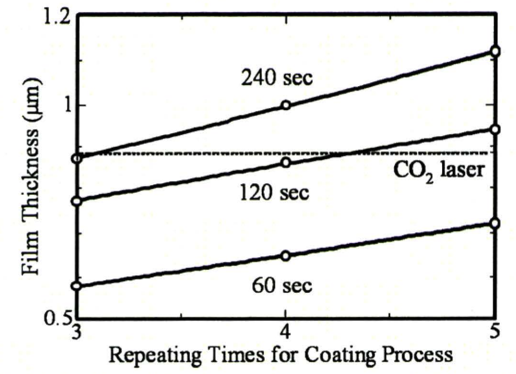

Fig. 7. AgI film thicknesses versus repeat times for the liquid-filling process.

process three times for a 240 s filling time or four times for a 120 s filling time. Since 240 s is a long filling time, the concentration of the iodine solution decreased significantly. The surface of the AgI film tends to be rough. We selected a 120 s filling time and repeated the process four times.

Figure 8 shows the loss spectra of the fabricated AgI/Ag/SUS pipes. For pipes with a two to four repeat filling time, the interference peaks shift to longer wavelengths, which means that the AgI film becomes thicker. When the process is repeated two, three, or four times the film thicknesses are 0.59, 0.72, and 0.81 μm, respectively. A one-time filling process increases the film thickness by approximately 0.1 μm. The film thickness of 0.72 μm in Fig. 8 for a three-time filling process is slightly thinner than the result of 0.78 μm in Fig. 7 because we used a higher fill-and-flush speed in Fig. 8. In Fig. 8 we used 7.1 cm/min and 4 s to completely fill the 280 mm long tube with iodine solution; in Fig. 7 we used 2 cm/min and 14 s.

B. AgI Film Decoating

The AgI film thickness can be controlled by adjustment of the fill-and-repeat times or the AgI film can be dissolved in a sodium thiosulfate solution. Figure 9 shows the variation of the AgI film thickness with the treatment of a sodium thiosulfate water solution. The original AgI/Ag hollow fiber has an interference peak at the wavelength of 6.5 μm,

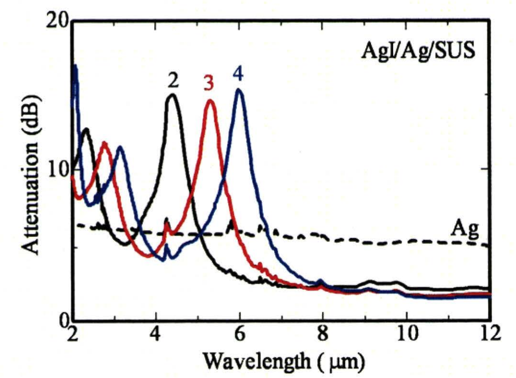

Fig. 8. (Color online) Loss spectra of AgI/Ag/SUS pipes using multiple iodination processes. The concentration of the solution was 0.1%. The process had a 120 s fill time; the process was repeated two, three, and four times.

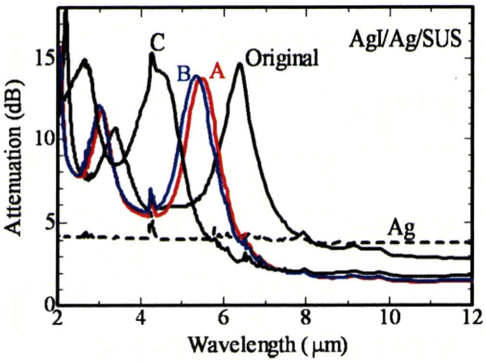

Fig. 9. (Color online) Loss spectra of AgI/Ag/SUS pipe treated with a sodium thiosulfate decoating process.

which means that the AgI film thickness is 0.86 μm. Spectra A, B, and C were measured results after a series of decoating processes on the original AgI/Ag/SUS pipe. Spectrum A was the measured result after the first sodium thiosulfate water solution treatment. The solution flowed through the fiber for 30 s at a speed of 3 cm/s; the concentration of the solution was 0.1 ml/L. Spectrum B was the measured result after the second treatment when the solution flowed through the fiber for 300 s; the concentration was 0.05 ml/L. Spectrum C was the measured result after the third treatment when the solution flowed through the fiber for 180 s; the concentration was 0.1 ml/L. The film thicknesses for the A, B, and C spectra were 0.73, 0.72, and 0.59 μm, respectively.

From spectrum A to spectrum B we conclude that film thickness decreases only slightly by use of a sodium thiosulfate water solution at a concentration of 0.05 ml/L. The film thickness variation from the original to spectrum A and from spectrum B to spectrum C was the same at 0.13 μm, with treatment times of 30 and 180 s, respectively. We therefore conclude that a longer than 30 s flow time is not effective when using a 0.1 ml/L sodium thiosulfate water solution.

Figure 10 shows the loss spectra for AgI/Ag/SUS pipe with various AgI film thicknesses. The film thicknesses for the three pipes are 0.71, 0.91, and

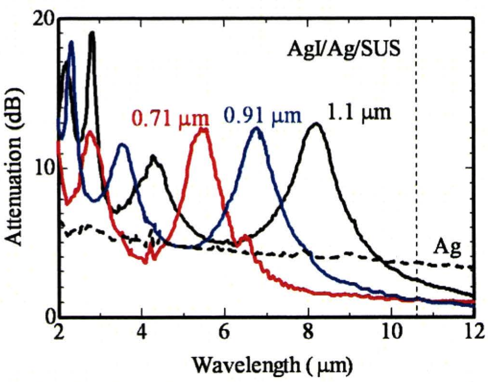

Fig. 10. (Color online) Loss spectra for AgI/Ag/SUS pipes with various AgI film thicknesses.

Table 3. Loss of AgI/Ag/SUS Pipes for a CO_2 Laser

Material	Size (mm) Inner Diameter	Outer Diameter	Length	Loss (dB)
AgI/Ag/SUS	0.75	1.2	280	0.18

1.1 μm, respectively. Sharp interference peaks showed that uniform and smooth AgI films were formed on the inner surface of the Ag/SUS pipe. According to the theoretical calculation, the pipe with 1.1 μm thick AgI film should have low loss at the target wavelength. However, the tube with a thinner AgI film, 0.71 μm or 0.91 μm, has less loss at the CO_2 laser wavelength because thicker film needs long iodination time or more repeated processes that cause a rough film surface or nonuniformity. It can be seen that the AgI/Ag/SUS pipe with thicker AgI film has a higher loss at a shorter wavelength region. We therefore prefer a slightly thinner film for the actual fabrication process.

Table 3 shows the measured loss of AgI/Ag/SUS pipe for a CO_2 laser. The loss of 0.18 dB in comparison with the loss of 1.7 dB for the Ag/SUS pipe shows that the AgI film is effective in reducing the transmission loss. This is a reasonable loss for an output probe of an infrared laser power delivery system. The transmittance was 85% (0.7 dB) when the tube was bent to an angle of 90 deg with a bending radius of 4 cm.

5. Conclusion

To obtain a strong mechanical strength for the probe of the infrared power delivery system, we fabricated a 280 mm long infrared hollow pipe based on various metal tubes. The tubes are stainless steel pipe (SUS), gold-coated stainless steel pipe (Au/SUS), and silver-clad stainless steel pipe (Ag/SUS). We have shown that fiber with an inner optical film coating based on Ag/SUS has the lowest loss property. We developed a liquid-fill method to minimize waste and multiple iodination processes to form a thicker film. A decoating process was also proposed to accurately decrease the AgI film thickness. Loss for the COP or AgI-coated Ag/SUS hollow pipes is less than 0.2 dB. The tube has a length of 280 mm and an inner diameter of 0.75 mm. The Ag/SUS tube is mechanically strong and experiences low loss.The Ag/SUS tube can be used as the output probe for the long, flexible, infrared laser power delivery system.

This research is financially supported by the Ministry of Education, Sports, Science, and Technology of Japan (Basic and Fundamental Research and Development Projects B) as well as by the National Natural Science Foundation of China, 60971014, and the Fudan Undergraduate Research Opportunities Program (FDUROP), China.

References

1. M. Alaluf, J. Dror, R. Dahan, and N. Croitoru, "Plastic hollow fibers as a selective infrared radiation transmitting medium," J. Appl. Phys. **72**, 3878–3883 (1992).
2. T. Abel, J. Hirsch, and J. A. Harrington, "Hollow glass waveguides for broadband infrared transmission," Opt. Lett. **19**, 1034–1036 (1994).
3. I. Gannot, S. Schründer, J. Dror, A. Inberg, T. Ertl, J. Tschepe, G. J. Müller, N. Croitoru, "Flexible waveguides for Er-YAG laser radiation delivery," IEEE Trans. Biomed. Eng. **42**, 967–972 (1995).
4. Y. Wang, A. Hongo, Y. Kato, T. Shimomura, D. Miura, and M. Miyagi, "Thickness and uniformity of fluorocarbon polymer film dynamically coated inside silver hollow glass waveguides," Appl. Opt. **36**, 2886–2892 (1997).
5. Y. Matsuura, A. Tsuchiuchi, H. Noguchi, and M. Miyagi, "Hollow fiber optics with improved durability for high-peak-power pulses of Q-switched Nd:YAG lasers," Appl. Opt. **46**, 1279–1282 (2007).
6. Y. W. Shi, Y. Wang, Y. Abe, Y. Matsuura, M. Miyagi, S. Sato, M. Taniwaki, and H. Uyama, "Cyclic olefin polymer-coated silver hollow glass waveguides for the infrared," Appl. Opt. **37**, 7758–7762 (1998).
7. K. Iwai, M. Miyagi, Y. W. Shi, X. S. Zhu, and Y. Matsuura, "Infrared hollow fiber with a vitreous film as the dielectric inner coating layer," Opt. Lett. **32**, 3420–3422 (2007).
8. C. D. Rabii, D. J. Gibson, and J. A. Harrington, "Processing and characterization of silver films used to fabricate hollow glass waveguides," Appl. Opt. **38**, 4486–4493 (1999).
9. A. Hongo, H. Takamiya, and T. Koike, "Hollow fiber using silver-clad stainless steel pipe with inner dielectric layer for CO_2 laser light transmission," Chin. Opt. Lett. **5S**, 70–72 (2007).
10. Y. W. Shi, K. Ito, Y. Matsuura, and M. Miyagi, "Multiwavelength laser light transmission of hollow optical fiber from the visible to the mid-infrared," Opt. Lett. **30**, 2867–2869 (2005).
11. R. George and J. A. Harrington, "Infrared transmissive, hollow plastic waveguides with inner Ag-AgI coatings," Appl. Opt. **44**, 6449–6455 (2005).
12. M. Miyagi and S. Kawakami, "Design theory of dielectric-coated circular metallic waveguides for infrared transmission," J. Lightwave Technol. **2**, 116–126 (1984).
13. R. George and J. A. Harrington, "Cu/CuI-coated hollow glass waveguides for delivery of infrared radiation," Opt. Eng. **45**, 055004 (2006).
14. Y. W. Shi, K. Ito, L. Ma, T. Yoshida, Y. Matsuura, and M. Miyagi, "Fabrication of a polymer-coated silver hollow optical fiber with high performance," Appl. Opt. **45**, 6736–3670 (2006).
15. Y. Matsuura, T. Abel, and J. A. Harrington, "Optical properties of small-bore hollow glass waveguides," Appl. Opt. **34**, 6842–6847 (1995).

Transmission characteristics of terahertz hollow fiber with an absorptive dielectric inner-coating film

Xiao-Li Tang,[1] Yi-Wei Shi,[1,*] Yuji Matsuura,[2] Katsumasa Iwai,[3] and Mitsunobu Miyagi[3]

[1]*School of Information Science and Engineering, Fudan University, Shanghai 200433, China*

[2]*Graduate School of Engineering, Tohoku University, Sendai 980-8579, Japan*

[3]*Sendai National College of Technology, Sendai 989-3128, Japan*

**Corresponding author: ywshi@fudan.edu.cn*

Received May 6, 2009; revised June 12, 2009; accepted June 12, 2009; posted June 22, 2009 (Doc. ID 111031); published July 14, 2009

We report calculation results for the transmission characteristics of terahertz hollow fibers with inner coatings of absorptive dielectric and metal layers. The absorption property of the dielectric film has an obvious influence on the transmission property of terahertz hollow fiber, because the optimum thickness of the dielectric layer is several tens of micrometers. Calculations were conducted on the loss properties of the hollow fiber with and without the absorptive dielectric layer. Important results were obtained, such as the optimum refractive index for the absorptive dielectric layer and the absorption tolerances for hollow fibers with various inner diameters. © 2009 Optical Society of America

OCIS codes: 060.2310, 350.4010, 060.2280.

Dielectric-coated metallic hollow fiber has been extensively studied for the application to the mid-infrared (MIR) region [1–4]. In recent years, this kind of fiber is receiving much attention for its use in the terahertz frequencies [5–7]. Approaches to fabricating the hollow fiber are also well established. They concern the wet chemistry method for the metal film and the liquid-phase coating for the dielectric film. To reduce the transmission loss, a dielectric coating over the metallic film is often necessary, as has been well proved in the MIR region both theoretically and experimentally. Ito *et al.* developed a 1 mm bore Ag-coated terahertz hollow fiber [5]. The measured loss was 7.5–8 dB/m at the wavelength band from 190 to 250 μm, which had good agreement with their theoretical results. They concluded that the TE_{11} mode dominates in Ag-coated hollow fibers when a linearly polarized beam is launched. Bauden *et al.* [6] and Themistos *et al.* [7] reported the polystyrene (PS)-coated silver (Ag/PS) terahertz hollow fiber. A low loss of 0.95 dB/m at the wavelength of 119 μm (2.5 THz) was obtained for the 2 mm bore 90-cm-long Ag/PS hollow glass fiber. Their experimental results showed that the HE_{11} mode dominates in the Ag/PS hollow fiber. Also, a detailed theoretical discussion [1] has shown that the dominant transmission mode transits from the TE_{11} mode for the metal-only fiber to the HE_{11} mode for the dielectric-coated metal fiber. Normally, the HE_{11} mode in the dielectric-coated metal hollow fiber has much lower loss than that of the TE_{11} mode in the metal-only hollow fiber. However, the dielectric layer brings an additional loss owing to its material absorption. In this Letter, we report the influence of dielectric absorption on the transmission characteristics of the terahertz hollow fiber.

When dielectric absorption is taken into consideration, the loss of the HE_{11} mode in the dielectric-coated metallic hollow fiber can be expressed as

$$\alpha'_{\mathrm{HE11}} = \alpha_{\mathrm{HE11}} + \Delta\alpha, \quad (1)$$

where α_{HE11} is the attenuation constant of the HE_{11} mode without considering the dielectric absorption, which is given by [1]

$$\alpha_{\mathrm{HE11}} = \frac{1}{2}\left(\frac{\mu_0}{2\pi}\right)^2 \frac{\lambda^2}{T^3} \frac{n}{n^2+k^2}\left(1+\frac{n_d^2}{(n_d^2-1)^{1/2}}\right)^2, \quad (2)$$

where μ_0 is the first zero of the Bessel function $J_0(x)$, λ is the transmission wavelength, T is the inner radius, n and k are the real and the imaginary parts of the complex index for the metal layer, and n_d is the refractive index of the dielectric layer.

$\Delta\alpha$ is the additional loss introduced by the absorption of the dielectric layer. When the fiber is inner coated with dielectric and metallic layers, a corresponding transverse transmission line model [8,9] can be used. After some calculations, $\Delta\alpha$ can be expressed as

$$\Delta\alpha = \alpha_{\mathrm{HE11}} k_0 d \frac{1}{F_m} k_d F_d, \quad (3)$$

where

$$F_m = \frac{n}{n^2+k^2}, \quad (4)$$

$$F_d = \frac{\dfrac{n_d}{n_d^2-1} + \dfrac{n_d}{\sqrt{n_d^2-1}}}{1+\dfrac{n_d^2}{\sqrt{n_d^2-1}}}. \quad (5)$$

Here k_0 is the angular wavenumber, d is the thickness of the dielectric layer, and k_d is the imaginary part of the complex index of the dielectric layer.

Figure 1 shows the loss of the HE_{11} mode in a Au/PS-coated hollow fiber as a function of the PS layer thickness. In the calculation, the complex refractive indices of Au and PS at the wavelength of 200 μm is

0146-9592/09/142231-3/$15.00