

# Hollow waveguides for gas sensing and near-IR applications

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## ABSTRACT

Coiled hollow glass waveguides (HGWs) were studied for use as gas sensors in the near and mid-IR regions of the spectrum. An analytic expression was obtained for the loss as a function of the number of turns,  $N$ , in the coil. The expected linear dependence of loss on  $N$  holds for radii greater than 13 cm, but deviates for smaller radii. The loss of HGWs is also shown to depend on silvering time and surface roughness.

**Keywords:** Hollow waveguides, gas sensing, bending loss, near-IR

## 1. INTRODUCTION

HGWs have been shown to be an attractive alternative to multipass absorption cells, such as the White cell.<sup>1</sup> Advantages over the White cell include flexibility, lower cost, and faster response times. An ideal hollow waveguide absorption cell consists of a large-bore, long-pathlength guide coiled into a small device. Large-bore silica tubing offers limited flexibility, so polycarbonate substrate waveguides offer an advantage over silica guides of similar dimensions. The metallic layer<sup>2</sup> can be deposited inside the polymer waveguide while the guide remains straight, and then the guide can be coiled at a later time. A typical polycarbonate waveguide can be coiled to a 4 cm diameter.

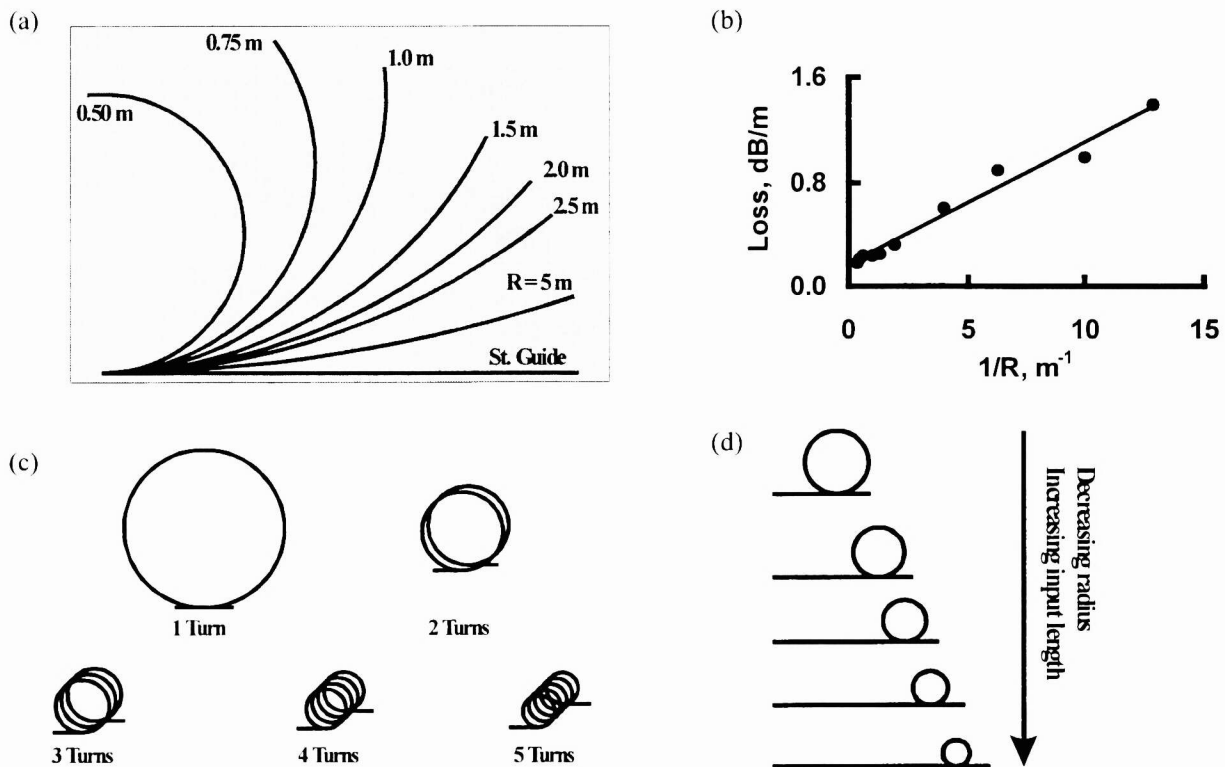
The behavior of polycarbonate waveguides at longer wavelengths has been previously reported.<sup>3,4</sup> It is the purpose of this paper to help characterize these waveguides at shorter wavelengths, particularly at the 2.94  $\mu\text{m}$ : the wavelength of the Er:YAG laser. In addition, the loss on bending of the waveguides is analyzed at both Er:YAG and CO<sub>2</sub> (10.6  $\mu\text{m}$ ) wavelengths in order to better understand what mechanisms might potentially limit a coiled absorption cell device.

## 2. EXPERIMENTAL SETUP

This paper is divided into three areas. The first involves the basic characterization of the polycarbonate waveguide with straight loss measurements and power handling measurements at 10.6  $\mu\text{m}$ . The second focuses on bending characteristics of glass and polymer waveguides at 10.6  $\mu\text{m}$ . The last section will discuss the bending losses of glass waveguides at 2.94  $\mu\text{m}$ .

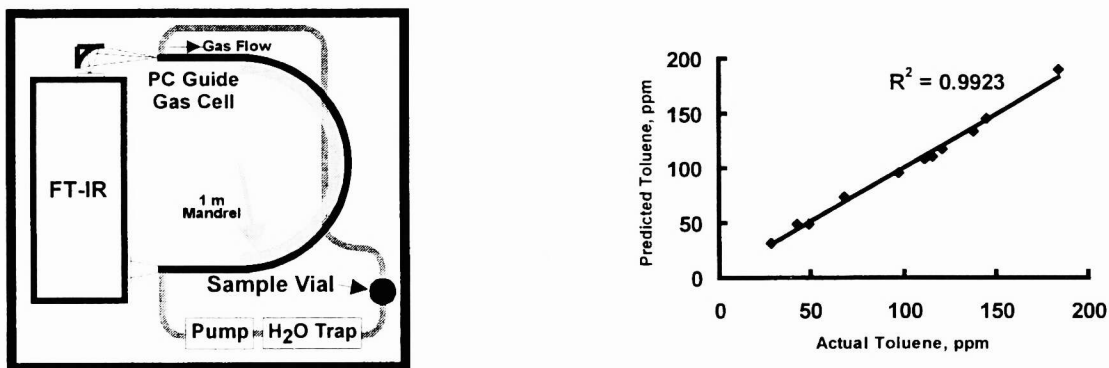
The bending characteristics of glass and polymer guides were measured using a bending board, as shown in Fig. 1a. Losses of the waveguide were then plotted against curvature. A typical loss-on-bending graph is shown in Fig. 1b. The slope of this curve,  $m$ , is an important parameter used in our analysis. One method of further testing involved keeping a constant length of the guide under bend. This method is shown in Fig. 1c. In a second method, a constant number of loops are maintained throughout the testing procedure, while simultaneously decreasing the radius of the loops. As each loop became smaller, it was moved to the distal end, minimizing the effect of higher-order modes.

In the final section, the silvering times of the liquid chemistry technique are studied. In this method<sup>5</sup> a metallic layer is deposited on the substrate and, in a second fabrication step, a dielectric layer is formed through a subtractive process. The final optical thin film coating consists of an Ag-AgI reflective-enhancing coating. The HGWs are designed for use at a specific wavelength or wavelength region. Longer wavelengths require thicker dielectric layers and, therefore, longer coating times. The thickness of the silver layer in the first step of the coating process should be thick enough to provide sufficient silver for reduction to AgI. Long silvering times, however, produce a rougher surface and increased scattering losses.<sup>6</sup> We have formed an optimum silvering time for producing guides with the lowest loss at the Er:YAG wavelength.



**Figure 1:** (a) Standard bending board testing technique. (b) Typical loss-on-bending curve, showing linear increase in loss with increasing radius of curvature. (c) Constant length is kept under bend while increasing the number of turns in the guide. (d) Increasing input length of guide inherently decreases the radius and length under bend when the number of turns is kept constant.

The final product, as used in conjunction with Polestar Technologies, Inc., is shown in Fig. 2a.<sup>7</sup> It consists of a polymer waveguide coiled around a mandrel to a 1 m radius. An FTIR spectrometer launches light into the proximal end of the waveguide while a gas line from the sample vial brings gas into the guide. To calibrate the system, a known concentration of toluene is used. The actual and predicted amounts of toluene are shown in Fig. 2b, and we note the 99.2% accuracy of the curve fit.



**Figure 2:** (a) 1/2-turn hollow guide system for VOC sensing (Volatile Organic Compounds). (b) Guide accuracy in detection of trace-level toluene.

### 3. BENDING ANALYSIS OF POLYCARBONATE AND GLASS WAVEGUIDES

Polycarbonate waveguides have losses nearly as low as glass waveguides with similar bore sizes. Fig. 3 shows the dependence of bore size on glass, polycarbonate, and Teflon<sup>8</sup> waveguides. Glass has an extremely smooth surface, while Teflon is rougher than polycarbonate. Polycarbonate guides have inner diameters ranging from 840  $\mu\text{m}$  to 1500  $\mu\text{m}$ , with losses of 0.272 and 0.098 dB/m, respectively. The bore sizes of polymer waveguides can be considerably larger than that of glass guides, while still offering good flexibility. Polycarbonate guides also have a lower loss compared to Teflon due to the inherently smoother surface. For devices with long pathlengths, it would seem that one would like a larger bore guide for the lower loss which decreases as  $1/a^3$ , where  $a$  is the bore size of the waveguide. However, it will be shown that the lower-loss advantage of larger bore guides tends to diminish in the whispering gallery mode region of small bend radii. Polycarbonate waveguides also have the ability to transmit greater than 25 Watts of CO<sub>2</sub> power, as shown in the data for a 1500  $\mu\text{m}$  guide in Fig. 4. The slight increase in loss is assumed to be due to the decrease in beam quality of the laser with higher powers.

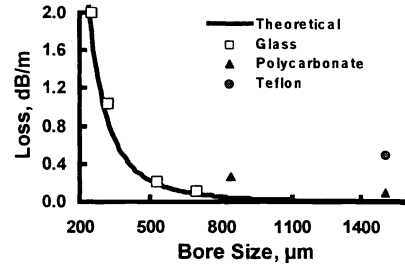


Figure 3: Experimental losses for glass and two kinds of polymer waveguides.

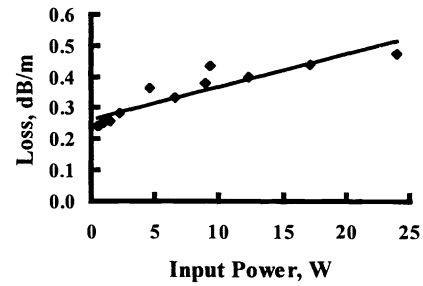


Figure 4: Power handling of polycarbonate guide.

In a previous paper, it was shown that the total loss of a coiled waveguide system, in dB, can be written in the form<sup>4</sup>

$$Loss_{tot} = \alpha_{st}(a)L_{tot} + 2\pi mN, \quad (1)$$

where  $\alpha_{st}$  is the loss, in dB/m, of a straight waveguide, which depends on the bore size as shown in Fig. 3.  $L_{tot}$  is the total length of the waveguide;  $m$  is the slope of the bending curve, as shown in Fig. 1b; and  $N$  is the number of turns in the coil. The bore-size dependence of the coil, calculated using Eq. 1 and shown in Fig. 5, indicates that a larger bore size does not have a significant advantage once it exceeds approximately 800  $\mu\text{m}$ . This is because the  $2\pi mN$  term dominates over the straight loss, which varies as  $1/a^3$ . The loss values in Fig. 5 are calculated based on an  $m$  value of 0.15 dB, a total length of 5 meters, and a straight loss of 0.5 dB/m for a 530  $\mu\text{m}$  bore size guide.

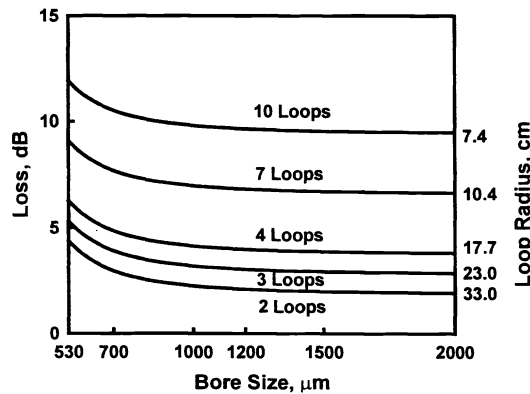


Figure 5: Loss calculations of a coiled hollow waveguide gas absorption cell system.

One approach used to study bending loss is to keep the length of waveguide under bend, as shown in Fig. 1c. This means that each additional loop decreases the bending radius. We consider only the additional loss of the waveguide for this case. That is, to consider a small  $\alpha_{st}$  in Eq. 1, such that

$$\text{Additional Loss} = 2\pi mN \quad (2)$$

Because the radius is determined by the number of turns in the guide for a constant  $m$ , we expect a simple linear relationship between the number of turns and the additional loss. This relationship is shown in Fig. 6. A 4 meter, 700  $\mu\text{m}$  glass waveguide was used with a  $\text{CO}_2$  laser source. The  $m$  value of 0.1 dB had been pre-determined through bending tests as shown in Figs. 1a and 1b. The expected trend shown in Fig. 6 was calculated using that  $m$  value. At zero turns the waveguide is straight and the additional loss is zero. From the data, one would expect an additional loss of about 0.63 dB per turn. The same guide was cut and then used again in the next experiment.

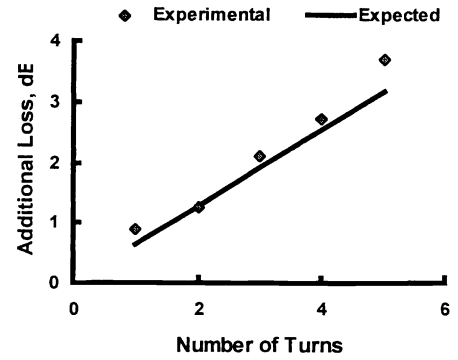


Figure 6: Linear relation of the additional loss and number of turns.

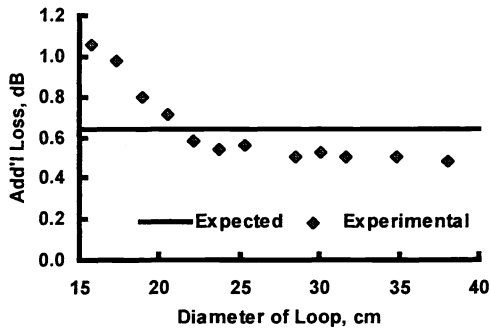


Figure 7: Effect of higher-order modes on tight bending radii.

The second approach involved keeping the number of turns constant while decreasing the length under bend. This study was shown schematically in Fig. 1d. Since there is no length term in the additional loss equation, there should then be a constant additional loss of  $2\pi m$  for each turn, irrespective of the diameter of the coil. Using a single loop, the diameter was decreased from 38 to 16 cm. The total length of the guide was 1.78 m. It can be observed in Fig. 7 however, that there is more loss for the smallest radii. This may be attributed to the generation of higher-order modes in the tightly bent regions of the guide. The losses of larger radii that are lower than the expected values are attributed to reasonable diameter sizes. This might be a region where the radius of the coil is large enough to prevent higher-order modes from propagating through the guide, yet small enough to be of an easily maintainable size. The propagation of higher-order modes can decrease the transmission of a waveguide.

The  $1/R$  relationship for a waveguide under bend is dependent on the existence of the whispering gallery mode, or edge-guided light propagation. This is when the light, within some critical radius of curvature, propagates only along the outer edge of the waveguide. Therefore, there is no bore-size dependence of the waveguide in the whispering gallery mode. Krammer<sup>9</sup> and Miyagi<sup>10</sup> have separately calculated the effects of higher-order modes in bending. Numerical results in Fig. 8a were given by Krammer, while Miyagi gave the mode perturbation approximations as

$$\alpha = \alpha_{\infty} \left[ 1 - \frac{2}{3} \left( 1 - \frac{15}{4u_{\infty}^2} \right) \left( \frac{n_0 k_0 a}{u_{\infty}} \right)^4 \left( \frac{a}{R} \right)^2 \right],$$

or

$$\alpha = \alpha_{\infty} \left[ 1 - \frac{2}{3} \left( 1 - \frac{15}{4u_{\infty}^2} \right) \left( \frac{n_0 k_0 a}{u_{\infty}} \right)^4 \left( \frac{a}{R} \right)^2 - \frac{5}{9} \left( 1 - \frac{105}{2u_{\infty}^2} + \frac{495}{4u_{\infty}^4} \right) \left( \frac{n_0 k_0 a}{u_{\infty}} \right)^8 \left( \frac{a}{R} \right)^4 \right],$$

depending on which order of the perturbation is chosen. Both equations are shown in Fig. 8a, along with the numerical results obtained by Krammer. For the solutions to the equations,  $n_0 k_0 a = 300$ , where  $a$  is the bore size of the waveguide ( $a = 506 \mu\text{m}$ ). The number of modes traveling in the waveguide is denoted by  $j$ , with

$$u_{\infty} = \frac{(j+1)\pi}{2},$$

and

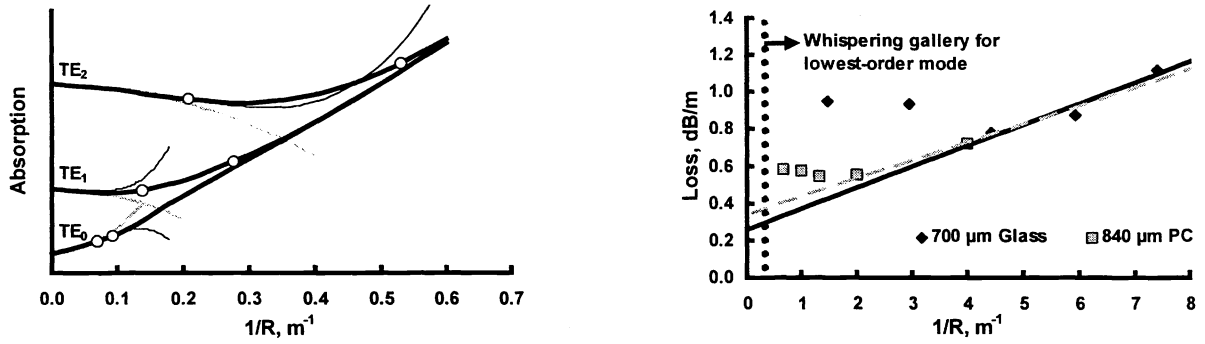
$$\alpha_{\infty} = n_0 k_0 \frac{u_{\infty}^2}{(n_0 k_0 a)^3} \operatorname{Re} \left( \frac{1}{\sqrt{(n - ik)^2 - 1}} \right),$$

where  $n - ik$  is the complex index of refraction. For aluminum, Krammer and Miyagi used  $n - ik$  equal to  $20 - i59$ .

Miyagi has also defined two radius values that are upper and lower limits of the whispering gallery approximation. For radii greater than the lower-limit radius,  $R_l$ , the waveguide is not in the whispering gallery mode and behaves as if it were a straight waveguide. For radii tighter than the upper-limit radius,  $R_u$ , the waveguide follows the predicted  $1/R$  relationship. The upper and lower limits are given by the equations

$$R_l = \frac{(n_0 k_0 a)^2}{2u_{\infty}} a \quad \text{and} \quad R_u = \left[ \frac{3}{8} \left( j + \frac{3}{4} \right) \pi \right]^{-2} \left[ 1 + \left( 2j + \frac{3}{2} \right)^{-2/3} \right]^{-3} (n_0 k_0 a)^2 a.$$

Fig. 8b shows that waveguides not in the whispering gallery region show losses higher than would be expected if only low-order modes were to propagate through the waveguide. The data were obtained through a careful experiment in which the waveguide sagged between supports from its own weight. This generated higher-order modes in the waveguide; the result is shown in Fig. 8b. As a general rule, bending losses of a coiled waveguide can be minimized when the generation of higher-order modes is prevented.

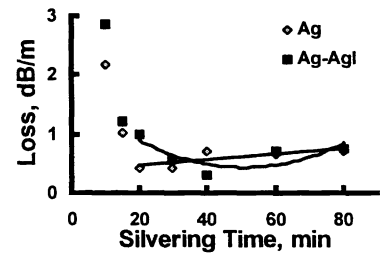


**Figure 8:** (a) Bending losses for several modes in a metallic hollow waveguide. Darker lines represent numerical results. Lighter lines represent results predicted by mode perturbation approximations. The small circles correspond to  $R_l$  and  $R_u$ . (b) Experimental observation of higher-order modes in polymer and glass waveguides. Dotted line approximately represents  $R_l$  and  $R_u$  for  $TE_0$  mode of both 700 and 840  $\mu\text{m}$  Ag-AgI guides.

#### 4. PROPERTIES OF GLASS WAVEGUIDES AT 3 $\mu\text{m}$

Previous AFM (atomic force microscopy) data showed that the surface roughness of silver increases on microscope slides with increasing processing time.<sup>6</sup> We have studied the effect of silver roughness and the additional dielectric layer on silvering times. Seven waveguides were fabricated with silvering times of 10, 15, 20, 30, 40, 60, and 80 minutes. After measuring the straight loss, each guide was then coated with AgI using a constant AgI processing time. They were tested again, this time straight and under bend.

Fig. 9 shows the straight losses of silver only and silver-with-dielectric waveguides fabricated with various silvering times. Even for the Ag guides, 10 and 15-minute silvering times were too short to provide a layer thick enough to efficiently reflect laser light. For silvering times between 20 and 80 minutes, an almost linear relation was observed between loss and silvering time. It is interesting to note that the guides with silvering times between 10 and 30 minutes increased in loss with the addition of AgI. The 40-minute Ag guide improved when the dielectric layer was added, and the 60 and 80 minutes guides remained approximately constant in loss. Fig. 10 shows what occurs for the short and long silvering times. Schematically, for very short silvering times, a smoother surface can be achieved, but the silver layer does not provide a sufficient thickness for an AgI layer to form. For longer silvering times, the Ag film thickness is



**Figure 9:** Silver only (Ag) and silver with dielectric (Ag-AgI) losses of various silvering times.

sufficient to produce an AgI layer, but the roughness of the surface increases the losses for these guides. For straight loss testing, the optimal silver processing time that accommodates the requirements of surface roughness and film thickness of the silver layer is 40 minutes.

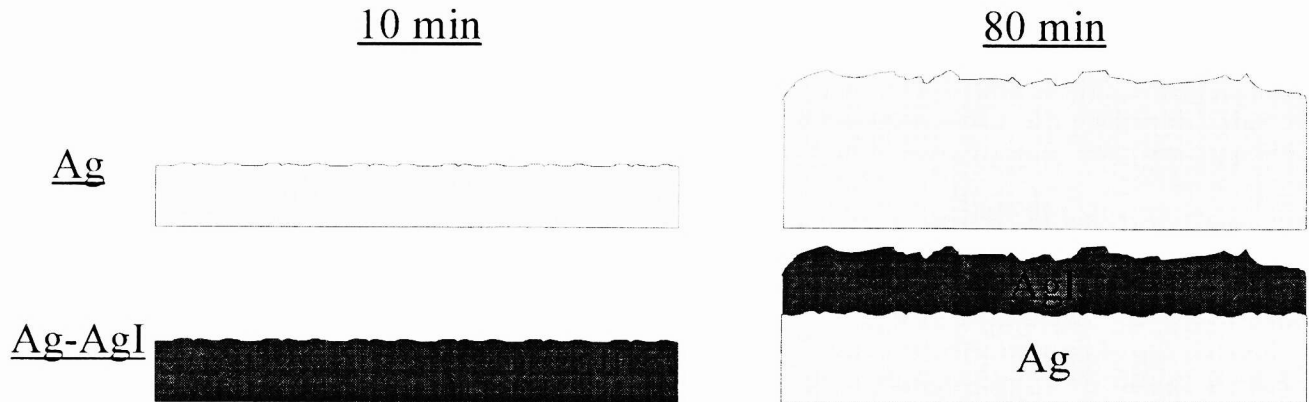


Figure 10: Short and long silvering times, showing the effects of both surface roughness and required thickness for AgI layer.

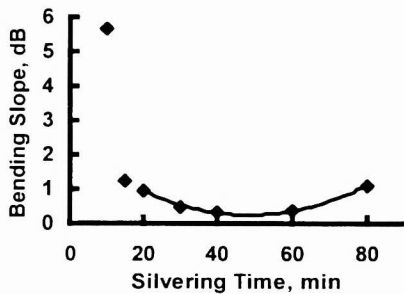


Figure 11: Effect of silvering time on bending slope of an Er:YAG waveguide.

Each of the seven guides was tested for bending loss. Each bending test was performed as shown in Fig. 1b. The slope of the bending curve was obtained from each graph, and the summary of the results is plotted in Fig. 11. The 40-minute silvering time again showed the lowest bending slope (0.336 dB) although the 60-minute guide nearly as low (0.366 dB). We conclude from the data for straight and bent losses that the optimum silvering time for Er:YAG waveguides is about 40 minutes. It should also be noted that the lowest bending slope, 0.336 dB is about 2-4 times greater than bending slopes observed with a CO<sub>2</sub> laser. This is because of increased surface roughness that gives rise to higher losses for 3 μm waveguides.

## 5. CONCLUSIONS

Hollow waveguides, and in particular, polycarbonate guides, are an alternative to White cells for gas sensing applications. They offer faster response times, increased flexibility, and a cost low enough for disposable applications. In order to further understand their properties, both glass and polymer guides were tested straight and bent at Er:YAG and CO<sub>2</sub> laser wavelengths. At CO<sub>2</sub> wavelengths, with a constant length under bend consisting of one to five loops, we formed a linear increase in loss of 0.63 dB/turn. In another test, the guide was coiled with one full turn to radii varying from 8 to 19 cm. There should have been a constant loss for each test, but the presence of higher-order modes dramatically increased the loss when the radius became smaller than about 13 cm. Finally, seven glass guides were fabricated with silvering times between 10 and 80 minutes. The guide that was silvered for 40 minutes showed the lowest straight loss as well as the lowest slope of the bending curve. However, even the lowest Er:YAG bending slope was higher than that of a typical CO<sub>2</sub> bending slope, due to the sensitivity of the guide to scattering at shorter wavelengths.

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