Integration of blue–green electroluminescence structure in mid-infrared hollow optical fiber for targeting invisible CO₂ laser beam

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Abstract
Mid-infrared (IR) CO₂ lasers have important applications in laser medicine and material processing. However, the CO₂ laser beam is invisible to human eyes, it is not easy to accurately indicate the beam position in practical applications. In this work, we designed a PEDOT (Poly (3, 4-ethylenedioxythiophene))/ZnS: Cu/BaTiO₃/Ag electroluminescence (EL) structure and integrated it at the output end of the mid-IR hollow optical fiber. The blue–green light can be output along with the CO₂ laser beam in this EL structure integrated fiber, thus achieving the optical path indication and illumination of invisible laser beam. This work provides a possible guide for aiming the CO₂ laser beam in the mid-IR optical fiber.

Keywords Mid-infrared hollow optical fiber · CO₂ laser · Visible beam · Electroluminescence · ZnS: Cu

1 Introduction
Mid-infrared (IR) CO₂ laser is widely used in the fields of laser surgery, rehabilitation treatment, and material processing due to its high gain coefficient, energy conversion efficiency and output power (Hongo et al. 1991; Merberg 1993; Wang et al. 2017; Zhang et al. 2017). The CO₂ laser beam can be mainly transmitted through the various optical fibers. Among them, the hollow optical fiber has attracted much attention because of its excellent performance (Jaworski et al. 2020). Compared with the solid-core fiber, the hollow optical fiber has no end reflection, high laser threshold, low transmission loss, low divergence and good flexibility (Harrington 2004). Its unique air-core fiber can transmit high-power laser beam, with a low-cost and simple preparation method (Shi et al. 1999).
According to the transmission principle (Harrington 2000), the hollow optical fiber can be divided into attenuated total reflection (ATR) fiber and leaky-type fiber. For the former, the laser propagates through total reflection \((n_r < 1)\) between the reflective layer and the air interface in the ATR fiber. The leaky-type fiber is mainly composed of air, the dielectric layer with suitable thickness, and the metal high reflective layer (Yu et al. 2020). The laser beam is transmitted through continuous reflection at the metal-dielectric layer and the air-dielectric layer \((n_r > 1)\).

The mid-IR laser beam is invisible to human eyes, in several applications such as material processing and laser medicine, (Tichindelean 2016). So it is not easy to target the laser-beam position in the operation process. Moreover, the typical optical fiber used to transmit the mid-IR light has a low loss for CO\(_2\) laser but a high loss for the visible light band. The visible beam can hardly be transmitted to the target position when the CO\(_2\) laser is delivered. Therefore, it is urgent to explore an approach to aim the laser beam position. In the early years, many researchers focused on this problem by adding a visible light path. (Van Den et al. 1992) However, the red aiming beam used in this traditional auxiliary indicating light path method has low integration, and is difficult to be distinguished by human eyes in laser medical applications (Graham and Bartlett 1939; Sheinis et al. 1994). More importantly, when the direction of the CO\(_2\) laser-beam changes, it is not applicable to realize the collimated transmission light path of visible light through the optical lens group.

In 1996, James A. Harrington’s group (Rabii and Harrington 1996) proposed a dual-core fiber structure. The optical fiber consists of air core, Ag/AgI hollow glass-fiber layer, quartz-glass-tube layer, and the outermost fluorine-containing low refractive-index layer. The quartz glass tube layer and the outermost fluorine-containing low refractive-index layer can establish an ATR waveguide structure for visible-light transmission. The air core and the Ag/AgI layer on the inner surface build a leaky waveguide structure for transmitting the CO\(_2\) laser. This dual-core optical fiber has high structural integration. However, it requires to deposit a fluorine-containing low refractive-index layer on the inner surface of the quartz glass tube by the chemical vapor deposition method, and fabricate the capillary hollow glass fibers using melt-drawing process. Thus, the production cost is high, which limits the commercial applications.

In 2016, our research group and Harrington’s team (Jing et al. 2016) improved the integration method for manufacturing the dual-core fiber. This optical fiber was manufactured entirely based on two parts, including the standard quartz glass tubing materials available in the market and the standard manufacturing procedure of Ag/AgI hollow glass fibers, rather than the fluorine-doped quartz glass capillary. It significantly reduced the manufacturing cost of the dual-core fiber. Nevertheless, it is still challenging to couple both CO\(_2\) laser beam and visible laser beam simultaneously into the input end of dual-core fiber in practical applications.

Electroluminescence (EL) technology (Picciolo et al. 2001) has a wide spectral range of the visible light region, high luminous intensity, self-luminescence, and fast response (tens of nanoseconds) (Allen 1994). This work aims to fabricate an EL structure-integrated mid-IR hollow optical fiber, which is capable of emitting high-reliability visible light and transmits CO\(_2\) laser by hollow optical fiber. We also investigate the fabrication process and the visible light targeting performance for the invisible CO\(_2\) laser beam. The results indicate that the EL integrated Ag/AgI hollow optical fiber is able to visualize the invisible CO\(_2\) laser output beam which may be potential in laser surgery and materials processing applications.
2 Experiments

Figure 1 shows the structure of the EL integrated Ag/AgI hollow optical fiber. The Ag/AgI air-core waveguide structure can serve for transmitting CO$_2$ laser. PEDOT (Poly (3, 4-ethylenedioxythiophene)) /ZnS: Cu /BaTiO$_3$ /Ag EL structure is integrated at the output end of the hollow optical fiber. Visible light is generated and enters the air-core and outputs along with the CO$_2$ laser beam. EL blue–green light is selected since it is sensitive to human eyes and easy to distinguish from the background color of blood red human tissue.

The quartz glass tubes were purchased from Ruifeng optical fiber Company (inner diameter: 530 µm, length: 1 m). The ZnS: Cu luminescent powders and epoxy resin were bought from Shanghai Kerun optoelectronics Co., Ltd.

Ag/AgI hollow optical fibers were prepared by using a dynamic liquid phase deposition (DLPD) method (Harrington 2004; Jing et al. 2016; Navarro-Cía et al. 2015) (See Fig. 2a). First, stannous chloride solution was pumped into a quartz glass tube by a peristaltic pump to pretreat the inner surface of the substrate tube. Then, the silver ammonia solution and reduced dextran solution were pumped into the glass tube to deposit a silver film on the inner tube wall. The thickness of the silver film is controlled by adjusting the reaction time. Finally, the iodine cyclohexane solution was pumped into the silver-coated glass tube and the top layer of the silver film was converted into a silver iodide layer. The
iodization process lasted for 6 min to obtain an AgI layer with an optimized thickness for 10.6 µm CO₂ laser.

1-cm long polyimide coating and the Ag/AgI film on the inner wall at the output end of the as-deposited Ag/AgI hollow optical fiber were removed by burning method. Then, the purchased PEDOT conductive liquid was coated on the 1.2 cm part of the outer wall at the optical-fiber end by dip-coating (See Fig. 2b). The extended 0.2 cm PEDOT conductive layer was exposed without contacting the subsequent EL structure layer, convenient to extract the copper wire for the front electrode. Finally, we put it into a 40 °C oven to dry (about 20 min).

0.4 g epoxy resin adhesive, 0.15 g dimethylformamide, and 0.4 g ZnS: Cu luminescent powder were mixed by stirring. The prepared luminescent powder mixture was coated on the transparent part of the hollow optical-fiber end by dip-coating method. The luminescent layer was coated three times by the same method with the 40 µm thickness of the three-layer fluorescent layer. It is noted that the prepared luminescent layer had no leakage point so that the front and back electrodes were not easy to break down and cause a short circuit. The main component of dielectric powder is barium titanate with a high dielectric constant, whose dilution ratio and coating method are the same as those of the fluorescent layer. The coating process was repeated four times, and the thickness of the dielectric layer was 60 µm. After each dip-coating, it was dried in an oven at 60 °C (about 30 min). The conductive silver paste was dipped with a brush and coated on the dielectric layer’s outer wall as a back electrode, then dried in an oven. Finally, the PEDOT-based front electrode and the conductive silver paste-based back electrode were extracted with 20-μm-diameter copper wire, respectively. The whole optical fiber was wrapped with a transparent insulating heat-shrinkable tube and heated at 60 °C to form a protective polymer insulating-layer to ensure the device’s insulation and chemical stability.

The surface morphology and crystal structure of ZnS: Cu were determined by X-ray diffraction (XRD: Rigaku, Ultima IV) and scanning electron microscope (SEM: Zeiss, Gemini 450). The visible EL spectrum was measured by the fluorescence spectrophotometer (LengGuang, F97PRO). The CO₂ laser beam was generated by the coherent C30a

Fig. 2 a Schematic DLPD method to prepare Ag/AgI hollow optical fiber device. b Dip-coating method
laser device and coupled into the waveguide sample after being focused by a ZnSe lens with a focal length of 10 cm. The output power was measured by the laser power detector (LP-3C). The transmission loss of the waveguide samples was calculated by the truncation method. The output laser beam profile and full divergence angle (FDA) of the waveguide samples were measured by the beam quality analyzer (LaserDec CL200). The loss spectra of waveguide samples was measured by a Fourier Transform IR spectrometer (FT-IR: BRUKER, VERTEX 80/80v).

### 3 Results and discussion

The ZnS: Cu powder was used for the electroluminescent structure. Figure 3a shows that the particle size is about 17–55 µm. According to energy dispersive spectrometer (EDS), the elemental composition of the electroluminescent structure phosphor is Zn, S, and Cu.

Figure 3b shows the EL cross-sectional SEM morphology at the end of the EL structure-integrated mid-IR hollow optical fiber. The heat-shrinkable tube layer for protection has been removed to capture the cross-section of the optical fiber for the SEM investigation. It can be found that the device has a sandwich structure, and the PEDOT conductive film, the luminescent layer, the dielectric layer, and the silver back electrode layer are on the outer surface of the hollow fiber glass tube in turn. Since the adhesive used in the luminescent layer and the dielectric layer is the same epoxy resin adhesive, the hierarchical structure is closely bonded with blurred boundary.

Figure 4 shows XRD pattern of the ZnS: Cu powder. The XRD patterns of the samples were compared with the standard pattern of ZnS. The diffraction peaks of ZnS: Cu powder were partially consistent with the standard patterns of ZnS-Sphalerite and ZnS-Wurtzite-2H (Yang et al. 2010). The luminescent layer power used in the work includes cubic phase and hexagonal phase.

We measured the visible light emission intensity of waveguide sample by changing the driving voltage with the transformer and illuminometer (See Fig. 5a). It indicates that the light intensity is positively correlated to the voltage. When the voltage exceeds 110 V, the luminescence of the prepared EL structure will be unstable, and it can be as a prone to high-voltage breakdown. When the voltage is 110 V, the luminescence of the

![Fig. 3](image-url)  
**Fig. 3**  
a EDS element mapping of Zn, S, Cu for ZnS: Cu powder;  
b Cross-sectional SEM morphology of the EL structure at the end of the EL structure-integrated mid-IR hollow optical fiber
EL structure can maintain 168.6 cd/m² without breakdown. Although the required frequency and voltage are high, the load current is small (about 0.016–0.3 mA). Therefore, the power consumption is low. In addition, during the preparation of the EL structure, the heat shrinkable tube was used in this work to encapsulate it without the risk of leakage. Considering the luminescence and stability of the EL structure, 110 V AC voltage is selected to supply power for EL structure.

In the AC state, the T2 or E level of shallow donor electrons and copper ions in ZnS molecules will transition (Liang et al. 2017), thus emitting visible beam. At 110 V AC voltage, the EL spectrum analysis results are shown in Fig. 5b. It can be seen that the luminescent wavelength range of the EL structure is 400–600 nm, and the emission peak is located at 457 nm, belonging to the blue–green light. It is noted that the EL structure and Ag/AgI hollow optical fiber are separate. The spectrum generated by EL structure will not generate additional interference to CO₂ laser irradiation, burning or ablation.

Fig. 4 XRD pattern of ZnS: Cu phosphor

Fig. 5 a Visible light EL luminance intensity of EL structure upon a different driving voltage. b EL spectrum of the EL structure-integrated mid-IR hollow optical fiber
Figure 6 shows the loss spectra of the mid-IR fiber with and without the EL structure (the length: 30 cm). As expected, both of them possess low-loss windows at 10.6 µm, which are suitable for the transmitting CO₂ laser (Rabii et al. 1999). The transmission loss of the mid-IR fiber with EL structure is slightly higher than that of the mid-IR fiber without EL structure, and the shape and position of the loss spectrum of the two optical fibers are the same. It implies that the EL structure only increases the transmission loss of the hollow optical fiber, without affecting the position of the low-loss window of the optical fiber.

Figure 7 shows the energy distribution (2D and 3D) of the CO₂ laser beam of samples. The two energy distribution profiles are almost circular, indicating that the output CO₂ laser beams’ energy distribution is close to Gaussian distribution. For the 530-µm-inner-diameter EL structure-integrated mid-IR hollow optical fiber, the laser beam is still transmitted in the fundamental mode (HE11 mode). The transmission loss value tends to be high, as the inner diameter of the hollow fiber decreases. Some higher-order modes with high loss gradually decay during the beam’s transmission, even disappear before reaching the output end of the fiber, and the lower loss of the fundamental mode has a large probability of output through the whole fiber core. This reflects the confinement effect of small-core hollow fiber on high-order modes, which is consistent with the results in Fig. 7. It also implies that the integrated EL structure at the end of mid-IR hollow fiber has no significant impact on the transmission mode of hollow fiber.

Then, we compared the straight transmission loss of CO₂ laser transmission in waveguide samples with and without the EL structure, and the measured transmission losses were 1.27 and 1.20 dB/m, respectively. The integrated EL structure has few influences on the transmission loss of the hollow fiber. The EL structure is only integrated within the 1 cm length range of the fiber from the output end. It is a very short length compared with the as-fabricated 100-cm-long Ag/AgI hollow fiber. The capillary tube with a 530-µm inner diameter used in this experiment was irregular in diameter. Besides, the vibration caused by truncation in the test of straight transmission loss made the optical fiber produce small displacement. These may have adverse effects on the transmission characteristics of the optical fiber. However, since the fiber’s diameter is only 530 µm, the fiber’s output can reach 90 mW, with the high energy density of the output CO₂ spot. The energy density can
be enough to meet several general laser medical treatments, such as skin surface treatment and laser rehabilitation physiotherapy. Moreover, the optical fiber with a laser transmission loss of less than 2 dB/m has practical value (Sun et al. 2018). Therefore, the as-fabricated EL structure-integrated mid-IR hollow optical fiber has the practical potential to transmit the CO₂ laser beam.

It should be noted that we can reduce the loss of this structure by increasing the inner diameter of the optical fiber sample. However, its flexibility and bending transmission performance may become worse upon the core diameter increases. It may reach a good balance as the fiber core size goes up to around 700 µm. Further study is undergoing.

In practical applications, the FDA is a crucial parameter for focusing and collimating the output beam, so it has an essential impact on the laser beam propagation ability and sufficient working distance (Hou et al. 2011; Hidaka 1982). By measuring the laser beam’s spot sizes along the optical propagation direction, for the mid-IR fiber with/without the EL structure, the FDA’s values are 26 and 16 mrad, respectively. When the EL structure is integrated on the mid-IR hollow fiber, the Ag/AgI waveguide reflection layer within 1 cm at the end of fiber is removed so that the visible light emitted by the EL structure can be...
penetrate into the quartz glass capillary of the fiber core and output from the end of the fiber. Therefore, when the CO$_2$ laser beam is transmitted through the core to the tail end, it will lose the constraint of the Ag/AgI waveguide reflector, and tend to diverge to a certain extent, resulting in an increase of the FDA value of output beam. However, considering that the divergence angle is 26 mrad, the beam can still be regarded as approximately linear propagation within a limited distance, which may have a negligible effect on the energy density of the spot in practical applications such as laser surgery.

In the material processing or laser surgery, it is likely to involve CO$_2$ laser-beam transmission under bending conditions. We measured the bending loss of CO$_2$ laser conducted by EL structure-integrated mid-IR hollow optical fiber with an inner diameter of 530 µm, by keeping the curvature radius R = 10 cm and input power of 108 mW, and changing the bending angle $\theta$.

Figure 8 depicts the experimental setup for measuring the bending loss of CO$_2$ laser beam. The test result can be seen in Fig. 9. When the bending angle increases from 0 to 240°, the optical fiber’s corresponding output power decreases from 90 to 50 mW, and the transmission loss increases from 1.27 to 5.33 dB/m. The transmission mode of optical fiber is very sensitive to the bending degree of optical fiber. With an increase of bending degree, the trend of exciting high-order modes increases. High-order modes generally have high transmission loss, so the bending loss of optical fiber will increase with the increase of bending angle. (Matsuura et al. 1994) Nevertheless, in the above bending test process, the power of output laser beam in optical fiber with an internal diameter of 0.53 mm can reach 50–90 mW. Considering that its internal diameter is only 0.53 mm with high energy density in the output CO$_2$ laser spot, it still has practical potential for the general laser medical surgery or laser irradiation rehabilitation therapy. When the bending radius is controlled at 10 cm and the bending angle is controlled at 0°–50°, the transmission loss is less than 2 dB/m. Therefore, it suggests the application potential of transmitting CO$_2$ laser under bending conditions.

In our work, we found that the power handling capability of the fiber sample can be further increased by increasing the core size of the Ag/AgI hollow fiber, but its bending performance may become worse upon the core diameter increases. It may reach a good...
balance as the fiber core size goes up to around 700 µm, which can achieve an output CO₂ laser beam with an energy density up to 519.7 W/cm². Further study is undergoing.

Figure 10a shows the encapsulated optical fiber. The EL structure-integrated mid-IR hollow optical fiber can emit visible guiding light continuously under 110 V AC voltage. It should be noted that the circuit is safe and stable under the encapsulation state, which retains the optical fiber’s original flexibility and has high integration. When the optical fiber is transmitted in any direction, the position of the output CO₂ laser spot can still be indicated.

Through the experiment of surgical burning with CO₂ lasers, we measured the visible light indication effect of IR hollow fiber with inner diameter of 0.53 mm for CO₂ laser transmission, as shown in Fig. 10b, c. Since the EL structure is only integrated within the length of 1 cm from the tail end of the mid-IR optical fiber, the bending state of the optical fiber has little effect on the output of visible light. Red paperboard is selected to simulate the red blood environment during CO₂ laser surgery. Two copper wires are externally
connected with AC power supply to excite the EL structure to produce indicator light. The 
CO₂ laser is coupled into the interior of the EL structure-integrated mid-IR hollow optical 
fiber for beam transmission. It is found that the EL structure can stably emit the blue–green 
visible beam while transmitting CO₂ lasers.

It should be noted that the visible light source has a certain divergence, but the light is 
constrained inside the hollow core fiber by the capillary glass-based tube wall. Then, the 
light will propagate divergently outside and form a round visible light profile (see Fig. 10b, 
c, below). It can be seen that the centers of the CO₂ laser spot and the output visible light 
spot are still coincident, and the visible light spot can indicate the Gaussian region of CO₂ 
laser. When the distance is 5 mm away from the output end, the spot diameter of the visible 
light is about 1.2 mm (see Fig. 10c). In practical application, the CO₂ laser contacts the 
location to be ablated within about 0.5 cm from the output end of the hollow fiber. The out-
put visible light spot can meet the requirements for indicating the optical path of invisible 
CO₂ laser radiations in practical application.

In general, for a higher power CW laser, heating indeed may become an issue. It is 
worth noting that the thermal effect mainly occurs at the input end of this kind of hollow 
optical fiber because of coupling loss. According to the studies reported by Miyagi et al. 
(Karasawa et al. 1987), the coupling loss in the process of laser input into the fiber will 
produce additional thermal effect, resulting in an increase in the temperature of the incident 
end of the hollow core fiber, which is much higher than that of other parts of the fiber. The 
heat energy generated by coupling loss generally has an impact only within a certain 
distance (about 10 cm) range of the optical fiber close to its input end. Thermal damage 
probably occurs at the input end of the Ag/AgI fiber when a high power CO₂ laser beam 
(i.e. hundreds or tens of Watts) is delivered through the fiber. However, heating will not 
become an issue in current work since only milliwatt to watt level of CO₂ laser power is 
needed for infrared radiation therapy or ablation of human tissues. In this work, no thermal 
damage was observed at the input end of the fiber under normal coupling condition and 
the waveguide temperature at the output end remained near room temperature. Because the 
luminescence of the EL structure integrated at the output end of the fiber only plays a light-
ing effect, it works under a very low power condition (1.76–33 mW) and the thermal effect 
is negligible.

4 Conclusion

By integrating the EL structure into Ag/AgI hollow optical fiber, we successfully fabri-
cated the EL structure-integrated mid-IR hollow optical fiber. The fiber could accurately 
indicate the output position of CO₂ laser with the visible light intensity of 168.6 cd/m². The 
results indicate that the optical fiber could continuously carry out visible light indication 
with CO₂ laser output. Besides, it can stably emit blue–green light sensitive to human eyes 
and possess a good visible light indication effect. It is expected to be applied in CO₂ laser 
medical treatment and material processing.

The results showed that the output of 530 µm inner diameter EL structure-integrated 
mid-IR hollow optical fiber presented the Gaussian distribution. The straight transmission 
loss of CO₂ transmission of the waveguide sample was as low as 1.27 dB/m, and the output 
beam FDA was 26 mrad. The output CO₂ laser had high quality, and the bending transmis-
sion ability could meet practical application requirements.
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Declarations

Conflict of interest  The authors declared no potential conflict of interest with respect to the research, authorship and/or publication of this article.

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