

Direct femtosecond laser inscription of Bragg gratings in Ho³⁺/Pr³⁺ co-doped AlF₃-based glass fiber for 2.86 μm laser

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In this letter, we report the fabrication of fiber Bragg gratings (FBGs) in home-made Ho³⁺/Pr³⁺ co-doped single-cladding fluoroaluminate (AlF₃) glass fibers and its application for Watt-level lasing at the mid-infrared (MIR) wavelength of 2.86 μm. The FBGs were inscribed using an 800 nm femtosecond (fs) laser direct-writing technique. The FBG properties were investigated for different pulse energies, inscription speeds, grating orders and transversal length. A 2nd-order FBG with a high reflectivity of 99% was obtained at one end of a 16.5 cm long gain fiber. Under 1150 nm laser pumping, this fiber yielded a power greater than 1 W at 2863.9 nm with an overall laser efficiency of 17.7%. The fiber laser showed a FWHM bandwidth of 0.46 nm and a long-term spectral stability.

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Fiber lasers have been widely investigated for their numerous applications in medical surgery, communication, sensing, material processing, molecule spectroscopy, defense and pump sources for other lasers [1-6]. Fluoride glasses based on ZrF₄, InF₃ and AlF₃ exhibit low phonon energy and wide spectral transmission range, and thus are excellent host materials for long wavelengths (λ) fiber lasers. In order to achieve high efficiency, dichroic mirrors (DM) with high reflectivity at the laser wavelength are widely used at the pump end of the fiber laser, increasing the complexity and instability of the laser system. Fiber Bragg gratings are passive devices which can be embedded into the fiber and provide an all-fiberized structure, which can effectively solve these problems. FBGs with small and uniform pitch (Λ ~0.5-3 μm), high reflectivity, good wavelength selectivity and narrow reflection linewidth, have attracted more and more attention in the last decade. So far, the writing of FBGs in fluoride fibers have mainly focused on fluorozirconate fibers. In 2007, Bernier *et al.* obtained a FBG with refractive index modulation of Δn~10⁻³ in Tm³⁺ -doped ZBLAN fibers [7], and also showed that the formation of grating was related

to the negative refractive index change induced by femtosecond lasers. In 2018, Aydin *et al.* inscribed a 99.5% high reflectivity FBG and an 8% low reflectivity FBG in Er³⁺ -doped ZBLAN fiber, obtaining a laser output power of 41.6 W at 2.8 μm [8]: this is the highest laser output at ~3 μm, where FBGs played an important role in increasing the output power. The FBGs were written using a phase mask. While phase masks can only write gratings with a specific period or a small variation around it, the fs laser direct-writing technique allows to arbitrarily select the grating period (Λ), the Bragg wavelength (λ_B), the grating order (m), and its physical length (L), thus reflectivity (R) [9]. In 2017, Bharathan *et al.* wrote FBGs with R~0.5 in a Ho³⁺/Pr³⁺ co-doped double-cladding ZBLAN fiber using line-by-line direct-writing method, and achieved a laser efficiency of 17% at λ ~2.88 μm [10]. In 2018, Goya *et al.* obtained an FBG with R~0.97 and Δn~1.1×10⁻³ in Er³⁺ -doped ZBLAN fiber by plane-by-plane direct-writing technique, and obtained a laser efficiency of 29.1% at λ ~2.8 μm [11]. In 2019, Bharathan *et al.* wrote an FBG with a maximum coupling coefficient of κ ~464 m⁻¹ using the stacking direct-writing method [12]. FBGs were also inscribed in fluoroindate (InF₃) fibers: in 2020, Bharathan *et al.* wrote a grating with a high R at λ ~4 μm and κ ~275 m⁻¹ after annealing at a temperature of T ~150 °C for 90 minutes [13]. Up to date, however, there has been no investigation of FBGs written in AlF₃ fiber for lasing. From the perspective of the glass matrix, AlF₃ glass has a higher glass transition temperature (~370 °C) and better corrosion resistance than ZBLAN and InF₃ glasses [14, 15]. Therefore, AlF₃ fiber FBGs are potentially useful for high power fiber lasers. In 2019, Wang *et al.* obtained a 10.4% laser output efficiency at λ ~2.9 μm by pumping a Ho³⁺/Pr³⁺ co-doped AlF₃ fiber [16]. In 2021, Liu *et al.* obtained a room-temperature 1.13 W output power at λ ~2.9 μm in a Ho³⁺/Pr³⁺ co-doped AlF₃ fiber [17]. By replacing DM with FBGs, the laser power and efficiency could be further improved.

Fs laser waveguide writing in various transparent materials requires little energy because of the multi-photon interactions [18-23], is not limited by the optical fiber photosensitivity, and provides extreme flexibility in terms of structure design. In this paper, FBGs with different parameters at λ_B ~2864 nm are successfully

inscribed into Ho³⁺/Pr³⁺ co-doped AlF₃-based glass fibers using a fs laser with a line-by-line direct-writing method. A 17.7% efficiency continuous wave laser is obtained owing to a highly reflective Bragg grating (HR-FBG). The laser shows a stable laser wavelength and a long-term work stability.

Fig. 1 (a) shows a schematic setup of the fs laser direct-writing system used in this work. A Ti: Sapphire femtosecond laser system (*Solstice Ace*) emitted pulses at $\lambda \sim 800$ nm with a repetition rate of 1 kHz and a duration of 100 fs (full width half-maximum, FWHM). A beam splitter with ratio of 9:1 was placed after the fs laser to reduce the laser energy. The laser beam was then focused into the homemade AlF₃ fiber core using a 40 \times objective with a numerical aperture (NA) of ~ 0.65 (*Olympus*). To avoid aberrations at the curved air-glass interface, the AlF₃ fiber was immersed in refractive index matching oil and then covered with a 100 μ m thick glass coverslip. The fiber was held straight by a customized, precisely-adjustable, three-dimensional translation stage (*X: XMS100-S; Y: XMS100-S; Z: VP-5ZA, XPS-D*). Each grating plane was inscribed by moving the fiber in relation to the focus of the laser beam for a distance of l (transversal length). As shown in Fig. 1 (b), each grating plane is separated by a distance Λ . The pulse train was blocked by a mechanical shutter during the movements represented by the dashed path. A mid-IR supercontinuum source was assembled and an optical spectrum analyzer (OSA, Yokogawa AQ6377) with a 200-pm resolution was used for the spectral characterization.

The AlF₃ fiber was fabricated by using the rod-in-tube method. The composition of the core was 30AlF₃-10BaF₂-19CaF₂-7.3YF₃-12.5SrF₂-2.5PbF₂-3.5MgF₂-3LiF-10ZrF₄-2HoF₃-0.2PrF₃ (mol%). The fiber had a core diameter of 10.5 μ m, a NA ~ 0.27 and a cladding diameter of 225 μ m. The cut-off wavelength at $\lambda \sim 3.69$ μ m effectively provided two modes at $\lambda \sim 2864$ nm. During the grating characterization experiments, the launched light beam offset was minimized to achieve the maximum coupling into the fundamental mode. The background loss of the fiber at $\lambda \sim 793$ nm was estimated to be ~ 1.6 dB/m by using the cut-back method.

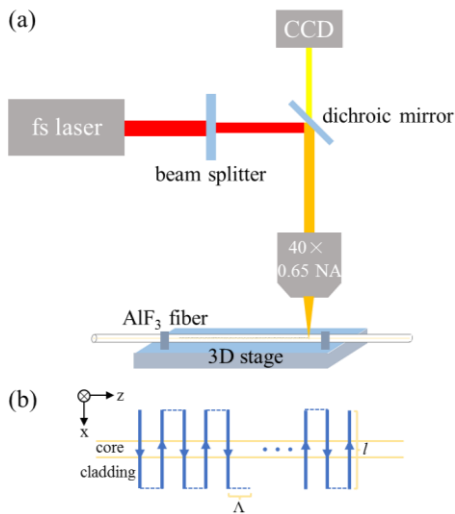


Fig. 1 (a). Schematic of fs laser fabricating FBGs system. (b). The pattern of FBGs processing path (top-view).

In order to estimate the optimal pulse energy (E_p) for FBG writing, a series of second order gratings with $\Lambda \sim 1.92$ μ m, $l \sim 30$ μ m, $L \sim 8$ mm and $\lambda_B \sim 2864$ nm were written with $E_p \sim 2.4$ μ J, 2.8 μ J, 3.2 μ J, respectively. The inscription speed of all the FBGs was 60 μ m/s. The total inscription time of each FBG was approximately 1 h. Fig. 2 (a) and (b) show microscope images of the FBGs inscribed with $E_p \sim 2.4$ μ J and 3.2 μ J. The grating plane inscribed at $E_p \sim 2.4$ μ J exhibited a small visibility, while that at $E_p \sim 3.2$ μ J was clear. Fig. 2 (c), (d) show the top and side views at 2.8 μ J. The side view shows that the grating was written into the fiber core, leading to a certain depth. Fig. 2 (e) shows the FBGs transmission spectra at different pulse energies. At increasing E_p , the corresponding resonance dip increase from 11.9 dB to a maximum of 19.6 dB and then fell to 13.6 dB. The increase was attributed to the rising Δn caused by the gradual E_p increase, while the fall resulted from damage in the core, possibly due to the energy exceeding the damage threshold of the AlF₃ fiber. Correspondingly, the insertion losses introduced by the gratings increased for increasing E_p from 0.6 dB to 1 dB and 1.28 dB respectively. The side peak at $\lambda \sim 2852$ nm can be ascribed to cross-coupling between modes, i.e., fundamental mode to second-order modes or second-order modes to fundamental mode [24-26].

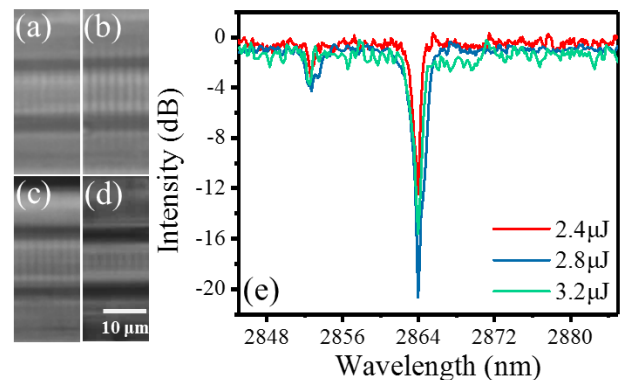


Fig. 2. Microscope images of FBGs inscribed with E_p of (a) 2.4 μ J (top view), (b) 3.2 μ J (top view) and (c, d) 2.8 μ J, (c: top view; d: side view). (e) Transmission spectra of the FBGs at different pulse energies.

The inscription speed is also an essential parameter for FBG writing: values of 30 μ m/s, 60 μ m/s and 120 μ m/s were used for inscribing 2nd-order gratings with $E_p \sim 2.8$ μ J, $\Lambda \sim 1.92$ μ m, $l \sim 30$ μ m, and $L \sim 8$ mm. As shown in Fig. 3 (a), a dip of 13.6 dB was achieved when FBGs was inscribed at 30 μ m/s, smaller than that of 19.6 dB at 60 μ m/s, but larger than that of 10.6 dB at 120 μ m/s. Moreover, as the speed increased, the insertion losses of FBGs were 0.53 dB, 1 dB and 1.2 dB, respectively. Fig. 3 (b), (c) and (d) show microscope images of the transverse cross section at speeds of 30 μ m/s, 60 μ m/s and 120 μ m/s (the red dashed line is the profile of the core): a smoother grating plane was written into core at lower speeds because of sufficient spatial overlap of pulses at 30 μ m/s compared with the irregular grating structure at 120 μ m/s. The speed of 60 μ m/s was chosen as an overall balance, considering also the FBG uniformity and resonance strength.

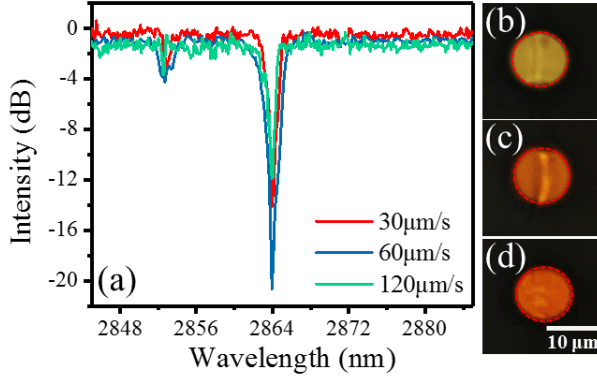


Fig. 3 (a). Transmission spectra of the FBGs at different inscription speeds. (b-d). Transverse cross section of the modification in AlF₃ fiber with different inscription speeds.

FBGs of different m orders were investigated. All the FBGs were written with $E_p \sim 2.8 \mu\text{J}$, speed of $60 \mu\text{m/s}$, $l \sim 30 \mu\text{m}$ and $L \sim 8 \text{mm}$, while the grating pitch was changed from $\Lambda \sim 1.92 \mu\text{m}$ to $2.88 \mu\text{m}$ and $3.84 \mu\text{m}$, for $m = 2, 3$, and 4 , respectively, corresponding to the central wavelength of $\lambda_B \sim 2.86 \mu\text{m}$. The first-order grating was difficult to inscribe probably due to the overlap of adjacent grating planes. Fig. 4 (a) suggests that the biggest resonance dip of 19.6 dB was obtained for $m = 2$. The strengths for $m = 3$ and 4 were 16.9 dB and 13.4 dB, respectively. As increasing m , the Bragg resonance of the FBG gradually decreases. The reason could be that for higher-order gratings, nonsinusoidal refractive index perturbation is formed, which is related to the expansion of Fourier series. And the order of the Fourier series and perturbation are negatively correlated [27,28]. Thus, the 2nd-order gratings caused the strongest perturbation, and biggest resonance strength. Besides, 2nd-order gratings of different transversal lengths (l) were discussed in AlF₃-based fiber. The grating with $l \sim 20 \mu\text{m}$ showed a dip of 17 dB, which was smaller than that of 19.6 dB observed at $l \sim 30 \mu\text{m}$ (see Fig. 4 (b)). The result involved a slightly smaller modulation depth along the y -axis and shallower modulation traces when the transversal length was shorter.

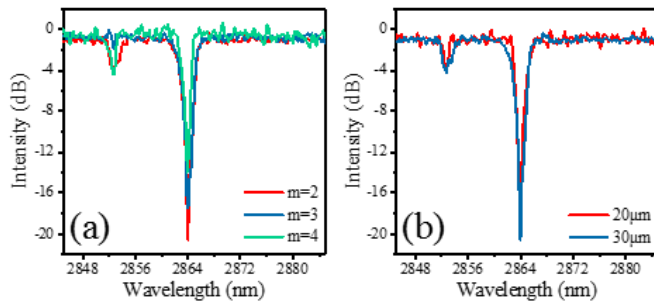


Fig. 4 (a). Transmission spectra of the FBGs with different orders m . (b). FBG transmission spectra for different transversal length l .

Fig. 5 shows a schematic of experimental setup used for $2.86 \mu\text{m}$ laser testing. A $\lambda \sim 1150 \text{nm}$ Raman fiber laser was used as pump source and launched through a commercial HI1060 standard fiber with a fiber collimator at the end. L3 had a focal length of 6 mm. A length of 16.5 cm Ho³⁺/Pr³⁺ co-doped AlF₃-based glass fiber was selected as the gain medium of the laser cavity. The laser cavity mirrors were formed by inscribing a high reflectivity grating (HR-FBG, $R \approx 99\%$) into the fiber at one extremity and exploiting Fresnel reflection ($\sim 4\%$ at $2.86 \mu\text{m}$) at the other extremity. Most of the fiber was placed on a copper plate with active cooling to reduce heat load. A 2 m long InF₃-based fiber cable (Thorlabs MF21L2) with a core diameter of $200 \mu\text{m}$ and $NA \sim 0.26$ was connected to the OSA (AQ6377) to detect the output laser spectra. A power meter (Thorlabs PM100D) was used to measure the output power value.

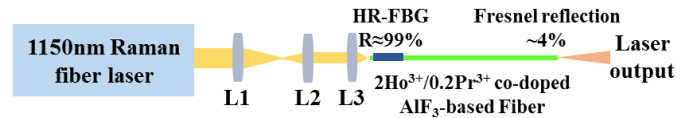


Fig. 5. Schematic diagram for the fiber laser experiments at $\lambda \sim 2.86 \mu\text{m}$.

Fig. 6 showed the dependence of the output power at $\lambda \sim 2864 \text{nm}$ with respect to the absorbed pump power at $\lambda \sim 1150 \text{nm}$. As the absorbed pump power increased to 30.67mW , lasing at 2863.9nm was observed. A maximum output power of 1006mW (with no 1150nm residual pump power) was achieved when the absorbed pump power was increased to 5.7W . The linear fit provided a slope efficiency of 17.7% . Fig. 7 shows the output laser spectra as a function of wavelength at output powers of 5mW , 367mW , 604mW and 977mW . The center wavelength remained constant under different output powers. While at low power the central wavelength was 2863.9nm , at 977mW , it shifted to 2863.88nm , within the spectrometer measurement error range. The inset shows the laser spectrum in the wavelength range of $2820 \text{nm} - 2910 \text{nm}$ at an output power of 977mW : the laser line FWHM was 0.46nm .

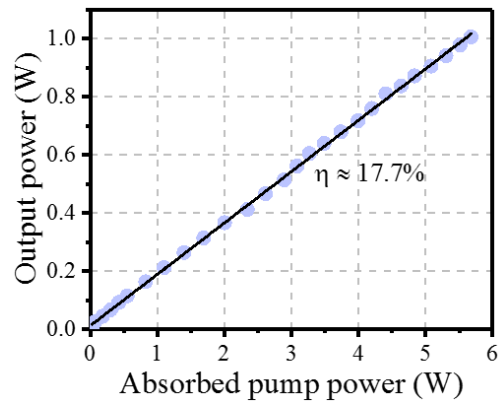


Fig. 6. Laser output power at $2.86 \mu\text{m}$ as a function of the absorbed pump power at 1150nm .

The stability of output power was also tested over a period of 30 minutes, as shown in Fig. 8. The RMS fluctuation for the output power is 0.52%. As the gain fiber at the pump side was not fully protected by the heat sink, the thermal expansion of fiber with a $\sim 10.5 \mu\text{m}$ core caused a change in the focus position of the pump beam, thus the value of the output power presented a decreasing trend.

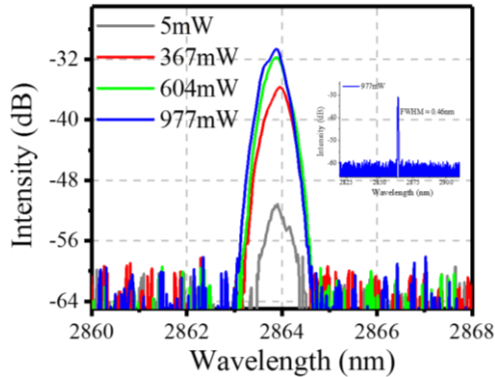


Fig. 7. Laser spectra at output powers of 5, 367, 604 and 977 mW (inset: laser spectrum at the output power of 977 mW).

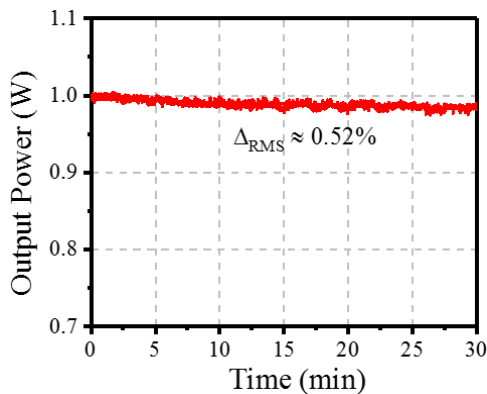


Fig. 8. Output power temporal stability.

In conclusion, FBGs with different parameters were first successfully written into AlF_3 -based fibers at the MIR wavelength of $\lambda \sim 2.86 \mu\text{m}$. The best FBG writing conditions were achieved for pulse energy of and speed of $2.8 \mu\text{J}$ and $60 \mu\text{m/s}$, respectively. A 2nd-order FBG with $30 \mu\text{m}$ transversal length showed a strong dip, and was exploited to inscribe a high reflectivity mirror for the laser cavity. A 16.5 cm long stretch of $\text{Ho}^{3+}/\text{Pr}^{3+}$ co-doped single-cladding AlF_3 -based fiber was used as lasing medium and produced a maximum output power of 1006 mW with an overall slope efficiency of 17.7%.

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Data availability. Data underlying the results presented in this paper are not publicly available at this time but may be obtained from the authors upon reasonable request.

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