

Article

# Characteristics and Laser Performance of Yb<sup>3+</sup>-Doped Silica Large Mode Area Fibers Prepared by Sol–Gel Method

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**Abstract:** Large-size 0.1 Yb<sub>2</sub>O<sub>3</sub>–1.0 Al<sub>2</sub>O<sub>3</sub>–98.9 SiO<sub>2</sub> (mol%) core glass was prepared by the sol–gel method. Its optical properties were evaluated. Both large mode area double cladding fiber (LMA DCF) with core diameter of 48  $\mu$ m and large mode area photonic crystal fiber (LMA PCF) with core diameter of 90  $\mu$ m were prepared from this core glass. Transmission loss at 1200 nm is 0.41 dB/m. Refractive index fluctuation is less than 2  $\times$  10<sup>-4</sup>. Pumped by 976 nm laser diode LD pigtailed with silica fiber (NA 0.22), the slope efficiency of 54% and "light-to-light" conversion efficiency of 51% were realized in large mode area double cladding fiber, and 81 W laser power with a slope efficiency of 70.8% was achieved in the corresponding large mode area photonic crystal fiber.

**Keywords:** fiber laser; sol–gel method; Yb<sup>3+</sup>-doped silica glass; LMA DCF; LMA PCF

### 1. Introduction

In recent years, the demands on high-power fiber lasers applied for Inertial Confinement Fusion (ICF) [1] and industrial laser processing application make Yb<sup>3+</sup>-doped silica fiber laser a hot research area [2–5]. Compared with the traditional double-clad fibers, large mode area double cladding fiber (LMA DCF) and large mode area photonic crystal fiber (LMA PCF) have unparalleled advantages, such as low power density at the pump end, high thermal damage threshold when pumped at high power, and single-mode transmission at certain designed structures [6,7].

The fabrication of LMA Yb<sup>3+</sup>-doped silica fiber needs large size core glass. It challenges the traditional modified chemical vapor deposition process (MCVD) method of active fiber preform. The MCVD, combined with solution doping with the merit of high purity and low optical loss, has been today's standard production method of fiber preform for industrial applications [8]. However, it is difficult to make large-diameter fiber core for LMA fiber under the condition of sustaining the initial homogeneity [9]. The sol–gel method can address this issue. It has great flexibility to prepare the geometry of silica fiber core and different species of chemicals can be mixed at the molecular scale through the sol–gel process. Therefore, large-size cores with good doping-homogeneity can be realized by using the sol–gel method.

In this work, Yb<sup>3+</sup>-doped silica core glass was prepared using the sol–gel method. Al<sup>3+</sup> was co-doped in silica glass to promote the solubility [10] of Yb<sup>3+</sup> ions. The basic properties of this core glass were evaluated. LMA DCF and LMA PCF were prepared from this core glass. The 51% optical–optical efficiency wad realized in Yb<sup>3+</sup>-doped silica LMA DCF. We acquired 81 W laser output from Yb<sup>3+</sup>-doped silica LMA PCF, with a slope efficiency of 70.8%. To the best of our knowledge, this is the highest laser output power report for Yb<sup>3+</sup>-doped silica LMA PCF prepared by the sol–gel method.

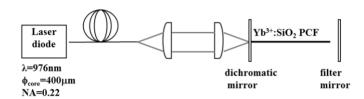
### 2. Experimental Section

Starting from the sol–gel method, we made a fiber preform core glass rod sized  $\Phi$  5  $\times$  80 mm with a composition of 0.1 Yb<sub>2</sub>O<sub>3</sub>–1.0 Al<sub>2</sub>O<sub>3</sub>–98.9 SiO<sub>2</sub> (moL%). The preparation processing has been published in our previous work [11,12]. Slice glass cut from this rod with 2 mm thickness was used to test absorption spectrum, emission spectrum, fluorescent lifetime and refractive index. FTIR spectrum in 400–4000 cm<sup>-1</sup> was detected to evaluate the residual hydroxyl in glass. Double cladding fiber preform was prepared by rod in tube method and Yb<sup>3+</sup>-doped silica LMA DCF was drawn in 2000  $^{\circ}$ C with plastic coating. It has a core diameter of 48  $\mu m$  with core NA of 0.08, and outer diameter of 340  $\mu m$  with NA of 0.46. Yb<sup>3+</sup>-doped silica LMA PCF was prepared by the stack-capillary-draw method at the drawing temperature of 2000  $^{\circ}$ C. It has a core diameter of 90  $\mu m$ , and the outer diameter is 720  $\mu m$  without plastic coating. The core NA is 0.08; and the inner cladding NA is 0.22.

To assess the homogeneity of the dopant distribution in the fiber core, the radial refractive index distribution of single cladding fiber prepared from the same core glass was measured (S14 Refractive Index Profiler, Photon Kinetics, Beaverton, USA). The transmission loss of the fiber core was also measured on the single cladding fiber by the standard cut back technique. The laser behaviors of both

DCF and PCF fiber were tested on setup shown in Figure 1. The details are discussed in the succeeding sections.

Figure 1. Fiber laser experimental setup.



### 3. Results and Discussion

### 3.1. Optical Properties of Yb<sup>3+</sup>-Doped Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> Glass

Figure 2a shows the absorption and emission cross-sections of Yb<sup>3+</sup>-doped Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> slice glass calculated using the Beer-Lambert equation [13] and Fuchbauer-Ladenburger method (F-L) [14], according to the measured absorption and fluorescence spectra, respectively. The maximum absorption cross section ( $\sigma_{abs}$ ) at 976 nm is 2.4 pm<sup>2</sup>, and the emission cross section ( $\sigma_{emi}$ ) near 1025 nm is 0.8 pm<sup>2</sup>. The corresponding fluorescence lifetime at 1025 nm is 896 µs by single exponent fitting of the measured photoluminescence decay curve. As can be seen from the FTIR spectrum shown in Figure 2b, there is almost no absorption peak at 2700 nm due to lower hydroxyl (OH) content. The hydroxyl content in our glass is calculated to be 1.45 ppm according to the method in reference [12].

**Figure 2.** (a) Absorption and emission cross-sections and (b) FTIR spectrum of Yb<sup>3+</sup>-doped Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> glass.

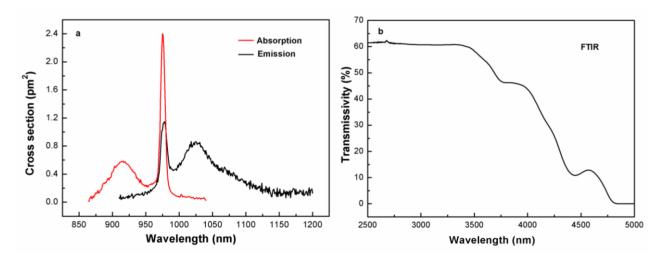


Table	1.	Spectral	parameters	of	Yb <sup>3+</sup> -doped	silica	glass	derived	from	different
prepara	atior	n methods.								

<b>Preparation Method</b>	Sol-Gel	MCVD	Sol-Gel	Heraeus
$Yb_2O_3$ (mol%)	0.1	0.15	0.21	0.25
$\sigma_{\rm abs}$ (976 nm) (pm <sup>2</sup> )	2.4	2.7	1.74	
$\sigma_{\rm emi}~(\sim 1~\mu {\rm m})~({\rm pm}^2)$	0.8	~0.6		
τ (μs)	896	830	840	
OH (ppm)	1.45	0.5		2
Reference	this work	[15,16]	[17]	[18]

Table 1 lists spectral parameters of Yb<sup>3+</sup>-doped silica glass derived from different preparation methods for comparison. It is found that Yb:Al:SiO<sub>2</sub> glass prepared in this work shows longer fluorescent lifetime and higher stimulated emission cross section compared with those prepared by MCVD and other sol–gel method [17]. The hydroxyl content (1.45 ppm) in our glass is compatible with that obtained in silica fiber prepared by Heraeus Conpany (2 ppm) and MCVD method (0.5 ppm).

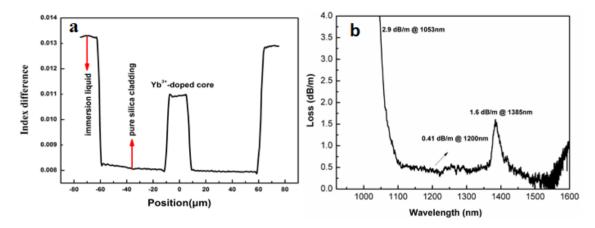
## 3.2. Optical Loss Profile and Homogeneity of Yb<sup>3+</sup>-Doped Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> Fiber

In order to assess the homogeneity of core glass, the single cladding fiber was drawn using the same  $A1^{3+}$ ,  $Yb^{3+}$  co-doped silica glass as the core. The radial refractive index distribution of the fiber core was measured and shown in Figure 3a. The maximum refractive index fluctuation  $\Delta n$  in the core is smaller than  $2 \times 10^{-4}$ . It indicates good optical homogeneity of the fiber core prepared by sol–gel method. Figure 3b shows the optical loss of the fiber core. The background attenuation at 1200 nm is 0.41 dB/m. Furthermore, there is an additional attenuation band at 1383 nm caused by OH. The OH-content of the fiber core is calculated to be 26 ppm from this absorption band [19]. It is higher than that in slice glass (1.45 ppm). This may be caused by moisture during the fiber drawing process.

### 3.3. Laser Behavior of Yb<sup>3+</sup>-Doped Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> LMA DCF and PCF Fibers

A 976 nm laser diode pigtailed with a spot diameter of 400  $\mu$ m and NA of 0.22 was used as pump source for both double cladding and PCF fibers. The pump light was collimated and focused by a couple of lenses (Figure 1) and then entered into the inner cladding of either LMA DCF or PCF fiber. The resonator is formed by a dichromatic mirror and the cleave fiber end. The dichromatic mirror is butt-coupled to the incident end of the fiber, which possesses high reflectivity (R > 99%) at 1040 nm and high transmittance (T > 99%) at 976 nm. The filter mirror (T > 99 at 1040 nm, R > 99% at 976 nm) is used when laser power is measured by the power meter.

Figure 3. (a) Radial profile of the refractive index and (b) optical loss spectrum of the single cladding fiber.



Laser performance of a 5.5 m long LMA DCF Yb<sup>3+</sup>-doped Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> fiber was tested and its output *versus* input curve is given in Figure 4. The output power was limited to 30W due to the limit of pumping source. The slope efficiency of 54% and "light-to-light" conversion efficiency of 51% were achieved in this fiber. The overall absorption coefficient (including background loss) of the DCF at the pump wavelength (976 nm) was tested to be 1.6 dB/m.

The laser experiment was carried out on a 210-cm-long Yb<sup>3+</sup>-doped Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> LMA PCF. The overall absorption coefficient (including background loss) of the fiber at the pump wavelength (976 nm) was tested to be 3 dB/m.

**Figure 4.** Laser input and output curve of Yb<sup>3+</sup>-doped Al<sub>2</sub>O<sub>3</sub>–SiO<sub>2</sub> large mode area double cladding fiber (LMA DCF).

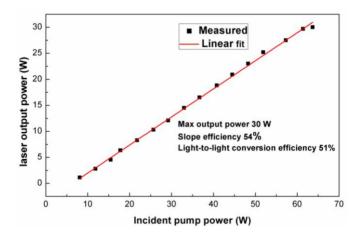
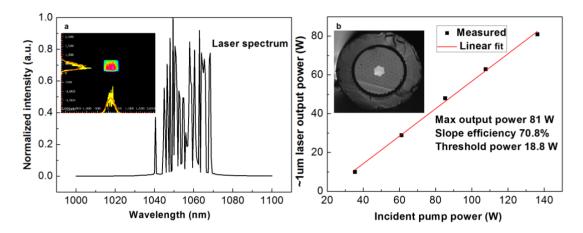


Figure 5a shows laser spectrum of  $A1^{3+}$ ,  $Yb^{3+}$  co-doped silica LMA PCF. The laser wavelength is at 1040–1070 nm. The inset shows the laser mode pattern in the far field. It is multimode. The micrograph of LMA PCF cross section is given in the inset of Figure 5b. Figure 5b indicates the laser input-output curve of LMA PCF. The slope efficiency is 70.8%. The laser threshold is approximately 18.8 W evaluated from the linear fitting. The maximum output power is limited to 81 W by the available pumping power. At this laser lever, we did not observe the photodarkening in our silica fiber. But codoping moderate  $P_2O_5$  with  $Al_2O_3$  in  $Yb^{3+}$ -doped silica fiber is necessary to greatly

suppress photodarkening effect when the fiber is operating continuously at high-power. Further work to reduce optical loss and improve mode quality of output laser is continued.

**Figure 5.** Laser behaviors of Al<sup>3+</sup>, Yb<sup>3+</sup> co-doped silica large mode area photonic crystal fiber (LMA PCF), (a) laser spectrum and mode pattern in the far field (inset); (b) laser output and micrograph of fiber cross section (inset).



### 4. Conclusions

Large-size 0.1 Yb<sub>2</sub>O<sub>3</sub>–1.0 Al<sub>2</sub>O<sub>3</sub>–98.9 SiO<sub>2</sub> (mol%) silica fiber preform core glass sized  $\Phi$  5 × 80 mm was prepared by the sol–gel method. The spectral properties of this glass were discussed. The maximum absorption cross section ( $\sigma_{abs}$ ) at 976 nm is 2.4 pm<sup>2</sup>, and the emission cross section ( $\sigma_{emi}$ ) near 1025 nm is 0.8 pm<sup>2</sup>. The fluorescence lifetime at 1025 nm is 896  $\mu$ s. Evaluated by single cladding fiber, the refractive index homogeneity of fiber core prepared from above core glass reaches 2 × 10<sup>-4</sup>; and the optical loss at 1200 nm is about 0.41 dB/m. The corresponding LMA DCF and LMA PCF were prepared by rod in tube and stack-capillary-draw, respectively. The NA values of core and cladding are 0.08 and 0.46 for LMA DCF. The NA values of the core and inner cladding are 0.08 and 0.22 for LMA PCF. The absorption coefficients of the LMA DCF and PCF at the pump wavelength (976 nm) are 1.6 dB/m and 3 dB/m, respectively. Slope efficiency of 54% and "light-to-light" conversion efficiency of 51% were achieved in Yb<sup>3+</sup>-doped silica LMA DCF. The maximum output power was limited to 81 W by the available pumping power with a slope efficiency of 70.8% in Yb<sup>3+</sup>-doped silica LMA PCF fiber .To the best of our knowledge, this is the highest laser power report for Yb<sup>3+</sup>-doped silica LMA PCF prepared by the sol–gel method.

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### **Conflicts of Interest**

The authors declare no conflict of interest.

### References

1. Glenzer, S.H.; MacGowan, B.J.; Michel, P.; Meezan, N.B.; Suter, L.J.; Dixit, S.N.; Kline, J.L.; Kyrala, G.A.; Bradley, D.K.; Callahan, D.A.; *et al.* Symmetric inertial confinement fusion implosions at ultra-high laser energies. *Science* **2010**, *327*, 1228–1231.

- 2. Galvanauskas, A. High power fiber lasers. Opt. Photon. News 2004, 15, 42–47.
- 3. Li, W.; Zhou, Q.; Zhang, L.; Wang, S.; Wang, M.; Yu, C.; Chen, D.; Hu, L. Watt-level Yb-doped silica glass fiber laser with a core made by sol-gel method. *Chin. Opt. Lett.* **2013**, *11*, 091601:1–091601:3.
- 4. Wei, T.; Li, J.; Zhu, J. Theoretical and experimental study of transient response of the Yb-doped fiber amplifier. *Chin. Opt. Lett.* **2012**, *10*, 040605:1–040605:4.
- 5. Zhu, R.; Wang, J.; Zhou, J.; Liu, J.; Chen, W. Single-frequency high-energy Yb-doped pulsed all-fiber laser. *Chin. Opt. Lett.* **2012**, *10*, 091402:1–091402:4.
- 6. Birks, T.A.; Knight, J.C.; Russell, P.St.J. Endlessly single-mode photonic crystal fiber. *Opt. Lett.* **1997**, 22, 961–963.
- 7. Jeong, Y.; Nilsson, J.; Sahu, J.K.; Soh, D.B.S.; Dupriez, P.; Codemard, C.A.; Baek, S.; Payne, D.N.; Horley, R.; Alvarez-Chavez, J.A.; *et al.* Single-mode plane-polarized ytterbium-doped large-core fiber laser with 633-W continuous-wave output power. *Opt. Lett.* **2005**, *30*, 955–957.
- 8. Poole, S.B.; Payne, D.N.; Mears, R.J.; Fermann, M.E.; Laming, R.I. Fabrication and characterization of low-loss optical fibers containing rare-earth ions. *J. Lightwave Technol.* **1986**, *4*, 870–876.
- 9. Dhar, A.; Paul, M.C.; Pal, M.; Mondal, A.K.; Sen, S.; Maiti, H.S.; Sen, R.J. Characterization of porous core layer for controlling rare earth incorporation in optical fiber. *Opt. Express* **2006**, *14*, 9006–9015.
- 10. Sen, S.; Rakhmatullin, R.; Gubaydullin, R.; Poppl, A. Direct spectroscopic observation of the atomic-scale mechanisms of clustering and homogenization of rare-earth dopant ions in vitreous silica. *Pysical. Rev. B* **2006**, *74*, 100201:1–100201:4.
- 11. Wang, S.; Li, Z.; Yu, C.; Wang, M.; Feng, S.; Zhou, Q.; Chen, D.; Hu, L. Fabrication and laser behaviors of Yb<sup>3+</sup>-doped silica large mode area photonic crystal fiber prepared by sol-gel method. *Opt. Mater.* **2013**, *35*, 1752–1755.
- 12. Liu, S.; Li, H.; Tang, Y.; Hu, L. Fabrication and spectroscopic properties of Yb<sup>3+</sup>-doped silica glasses by sol-gel method. *Chin. Opt. Lett.* **2012**, *10*, 081601:1–081601:4.
- 13. Chen, G.; Zhang, Q.; Yang, G.; Jiang, Z. Mid-infrared emission characteristic and energy transfer of Ho<sup>3+</sup>-doped tellurite glass sensitized by Tm<sup>3+</sup>. *J. Fluoresc.* **2007**, *17*, 301–307.
- 14. Charles, C.R.; Fournier, J.T. Coordination of Yb<sup>3+</sup> in some inorganic glasses from optical absorption and emission studies. *Chem. Phys. Lett.* **1969**, *3*, 517–519.
- 15. Melkumov, M.A.; Bufetov, I.A.; Bubnov, M.M.; Kravtsov, K.S.; Semjonov, S.L.; Shubin, A.V.; Dianov, E.M. Ytterbium lasers based on P<sub>2</sub>O<sub>5</sub>- and Al<sub>2</sub>O<sub>3</sub>-doped fibers. In Proceedings of the Europeen Conference on Oprical Communication, Stockholm, Sweden, 5–9 September 2004.
- 16. Kirchhof, J.; Unger, S.; Schwuchow, A.; Grimm, S.; Reichel, V. Materials for high-power fiber lasers. *J. Non-Cryst. Solids* **2006**, *352*, 2399–2403.

17. Li, Y.; Huang, J.; Li, Y.; Li, H.; He, Y.; Gu, S.; Chen, G.; Liu, L.; Xu, L. Optical properties and laser output of heavily Yb-doped fiber prepared by sol-gel method and DC-RTA technique. *J. Lightwave Technol.* **2008**, *26*, 3256–3260.

- 18. Andreas, L.; Gerhard, S.; Mario, S.; Thomas, K.; Volker, R.; Stephan, G.; Johannes, K.; Kraused, V.; Georg, R. A new material for high power laser fibers. *Proc. SPIE* **2008**, *6873*, 687311:1–687311:9.
- 19. Humbach, O.; Fabian, H.; Grzesik, U.; Haken, U.; Heitmann, W. Analysis of OH absorption bands in synthetic silica. *J. Non-Cryst. Solids* **1996**, *203*, 19–23.
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