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Nd^{3+}-doped silica glass and fiber prepared by modified sol-gel method

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Large-size Al^{3+}/Nd^{3+} co-doped silica glass with 5000 ppm Nd^{3+} and 50,000 ppm Al^{3+} doping concentrations was prepared by the modified sol-gel method combined with high-temperature melting and molding technology. Electron probe micro-analyzer tests indicated that high doping homogeneity was achieved with this sample preparation method. The spectral properties of the Nd^{3+} ions were evaluated. Nd^{3+}-doped silica fiber (NDF) with a core-to-clad ratio of 20/125 μm was drawn from the preform with the Al^{3+}/Nd^{3+} co-doped silica glass as the core. In the laser oscillation experiment, a maximum output power of 14.6 W at 1.06 μm with a slope efficiency of 39.6% was obtained from the NDF pumped by a commercial 808 nm laser diode. To the best of our knowledge, this is the highest laser power reported for an NDF operated at 1060 nm and prepared by a non-chemical vapor deposition method. In the master oscillator power amplifier experiment, a maximum power of 16.6 W corresponding to a slope efficiency of 30.5% at 1061 nm was also demonstrated. The laser performance of the NDF exhibited the great advantages and potential of the modified sol-gel method in fabricating Nd^{3+}-doped silica glass for a new type of NDFs like large mode area fibers and fibers with large diameter ratio of core/cladding.

Keywords: Nd^{3+}-doped silica; sol-gel; doping homogeneity.
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1. Introduction

Nd^{3+}-doped silica fiber (NDF) has good thermo-mechanical properties of silica glass and a perfect energy level structure of Nd^{3+} ions, which is widely used in communication, biomedical, military, material processing, high-power laser, and other fields[1,2]. The $^4F_{3/2} \rightarrow {}^4I_{11/2}$ transition of Nd^{3+} can generate a ~1060 nm laser with extremely low laser threshold and long fluorescence lifetime, which benefits the laser output and energy storage. Nd^{3+}-doped glass, crystal, and ceramics are well developed for this band[3–5]. Moreover, the application of Nd^{3+} in several other bands has also been widely considered by researchers. The $^4F_{3/2} \rightarrow {}^4I_{9/2}$ transition of NDF can generate a ~900 nm laser, which can be directly used in atmospheric detection, lidar, and Yb^{3+} ion pumping[6]. The deep blue lasers produced by its frequency doubling can be used in underwater communication, precision optics, atom cooling, and other fields[7,8]. The $^4F_{3/2} \rightarrow {}^4I_{13/2}$ transition of NDF can generate lasers in the E-band of wavelengths (1350–1450 nm), which plays an important role in the fields of remote sensing, surgery, optical fiber communication, information storage, and so on[9,10]. These new applications require NDFs with a larger core-to-clad ratio and higher doping concentrations.

However, the Nd^{3+} ion has high cation field strength and requires coordination with more oxygen to reduce the energy of the system. But, the rigid network of silica glass makes it difficult for oxygen ions to gather around the Nd^{3+} ion in sufficient numbers. The Nd^{3+} ion can only consume less oxygen ions by agglomeration. Consequently, the Nd^{3+} ion is very easy to agglomerate and even crystallize in silica glass when the Nd^{3+} ions doping concentration is more than 1000 ppm (parts per million), resulting in deterioration of optical quality. Co-doping Al^{3+} is one of the good methods to eliminate agglomeration to some extent[11,12]. In the 1990s, the application of NDF to ~1060 nm lasers was widely pursued using a modified chemical vapor deposition (MCVD) method[13,14]. IPHT (Institut für Physikalische Hochtechnologie, Jena, Germany) fabricated

$Al^{3+}/P^{3+}/Ge^{4+}$ co-doped NDF with Nd^{3+} contents of 1300 ppm by the MCVD method. Using laser diode (LD) pumping, researchers have obtained several tens of watts of the ~1060 nm laser output in this NDF with a slope efficiency of 46%[15]. Such a high slope efficiency is enabled by the ultralow optical loss of fibers prepared with the MCVD method. Nevertheless, the MCVD process combined with solution doping is difficult to fabricate large-diameter fiber core glass with heavy RE^{3+} contents while maintaining high homogeneity[16]. As a result, it is difficult to meet the needs of the development of a new type of optical fiber towards a large core-to-clad ratio and large mode area (LMA).

Non-chemical vapor deposition (Non-CVD) methods have a significant advantage: they can be used to prepare large-size silica glass; hence, these methods have been adopted to prepare RE^{3+}-doped silica glass and fiber[17–19]. Fujimoto *et al.* used the zeolite method to prepare Nd^{3+}-doped silica glass and fibers. The prepared fiber could provide mode-locked laser output with a high repetition frequency through short-cavity amplification[20]. The maximum laser power they reported was 27.2 mW, with a slope efficiency of 19.6%. Wu *et al.*[21,22] also used sol-gel methods to fabricate the NDF. However, the laser output power in the aforementioned studies was limited to the order of milliwatts.

The modified sol-gel method combined with high-temperature melting and molding technology has been demonstrated as an innovative technique to prepare large-size RE^{3+}-doped silica glass with heavy doping and high homogeneity. A series of achievements have been achieved in LMA photonic crystal fibers (PCFs) prepared by this method[23–25]. In this Letter, large-size Al^{3+}/Nd^{3+} co-doped silica glass was prepared using this method. To test the laser performance, a 20/125 NDF was drawn at 1800°C–2000°C using this core glass, preliminarily. A maximum output power of 14.6 W was obtained with a slope efficiency of 39.6% at ~1060 nm from the NDF under an 808 nm LD pump. An amplifier with a maximum power of 16.6 W corresponding to a slope efficiency of 30.5% at 1061 nm was also achieved. The properties of silica glass, fiber fabrication process, and its optical properties will be demonstrated.

2. Experiment

In the sol-gel preparation process, tetraethoxysilane (TEOS), C_2H_5OH, $AlCl_3 \cdot 6H_2O$, and $NdCl_3 \cdot 6H_2O$ were used as precursors. Deionized water was added to sustain the hydrolysis reaction. The preparation process of Nd^{3+}-doped silica glass by the sol-gel method is the same as that of Yb^{3+}-doped silica glass, which was described in details in Refs. [23,26]. The centimeter-scale Nd-doped monolithic glass can be further ground and polished directly into a preform core of the NDF. The mean doping concentrations of Nd^{3+} and Al^{3+} were 5000 and 50,000 ppm, respectively. The results of inductively coupled plasma-optical emission spectrometry analysis show that the Nd^{3+} and Al^{3+} contents in the silica glasses were close to the theoretical values.

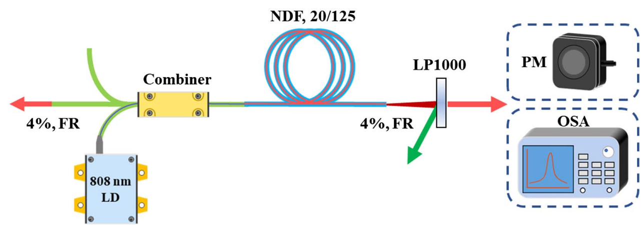

Fig. 1. Experimental laser setup. FR, Fresnel reflection; PM, power meter; OSA, optical spectrum analyzer.

The glass rod was cut and polished into 2-mm-thick sheets to study their physical and spectral properties. To investigate the doping homogeneity, the distributions of Al^{3+} and Nd^{3+} were characterized using an electron probe micro-analyzer (EPMA, Shimadzu, 1720H). A spectrophotometer (Lambda 900 UV-VIS-NIR, Perkin-Elmer) was used to record the absorption spectra of the glass sheets in the range of 200–1000 nm. The fluorescence lifetime and fluorescence spectrum of the glass were measured using a time-resolved spectrometer (Edinburgh Instruments, FLS920) with an excitation of an 808 nm LD. Fourier transform infrared (FT-IR) spectra were measured using a Nexus FT-IR spectrometer (Thermo Nicolet).

The rod-in-tube method was used to prepare the preform of the NDF. First, the Al^{3+}/Nd^{3+} co-doped bulk silica glass was processed into a round rod with a size of $\phi 2.8 \times 30$ mm. It was then inserted into a matched pure silica tube to prepare a fiber preform. Finally, the fiber preform was drawn to 20/125 NDF at 2000°C in a drawing tower. The refractive index profile of the 20/125 NDF was measured using an interferometer fiber analyzer (IFA-100, Interfiber Analysis, LLC). The core transmission loss of the NDF was measured using a broadband light source and the cutback method.

Figure 1 shows a schematic of the experimental laser setup. A multimode LD with an output power of 40 W at 808 nm was used as the pump source. The 2 + 1 combiner had double-clad input/output fibers with core/clad diameters of 20/125 μm and an numerical aperture (NA) of 0.08/0.46, which matched well with the 20/125 NDF. The two fiber end-faces were cleaved at 0° to provide a Fresnel reflection (FR) of ~4%, thereby forming the laser cavity. To separate the signal power from the residual pump power, a 1000 nm long-pass filter (LP1000) was used. A power meter (PM, Thorlabs S442C) and an optical spectrum analyzer (Yokogawa AQ6370D) were utilized to record the laser output power and laser spectrum, respectively. In the experiment, the NDF was bent into 15 cm diameter rings and fixed on a metal plate for cooling.

3. Results and Discussion

3.1. Homogeneity characterization of the Nd^{3+}-doped silica glass

Figure 2(a) shows a photograph of a polished large-size core-glass block and rod. The original size of the glass block before

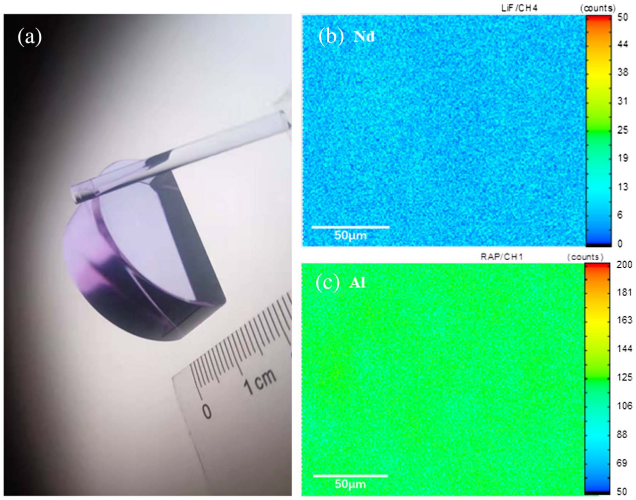

Fig. 2. (a) Image of Al^{3+}/Nd^{3+}-doped silica core-glass block and rod. EPMA mapping of the elements in the silica glass rod: (b) Nd and (c) Al.

cutting out the glass rod was $\phi 30 \times 12$ mm. There were no bubbles or stripes in either the glass block or the rod. The uniform distributions of Nd^{3+} and Al^{3+} in the glass rod are displayed in Figs. 2(b) and 2(c), respectively. Thus, large Al^{3+}/Nd^{3+} co-doped silica glass rods with high optical quality and high Nd^{3+} and Al^{3+} doping levels can be prepared by the sol-gel method. It can be used to fabricate a new type of NDFs like LMA fibers and fibers with large diameter ratio of core/cladding.

3.2. Spectroscopic properties of Nd^{3+}-doped silica glass

Figure 3(a) labels the absorption peaks of Nd^{3+} from the ground state to the excited state in the wavelength range of 200–1000 nm. The peak absorption cross section at the $^2H_{9/2} \rightarrow {}^4F_{5/2}$ level was 1.09 pm^2, located at 804 nm. The main

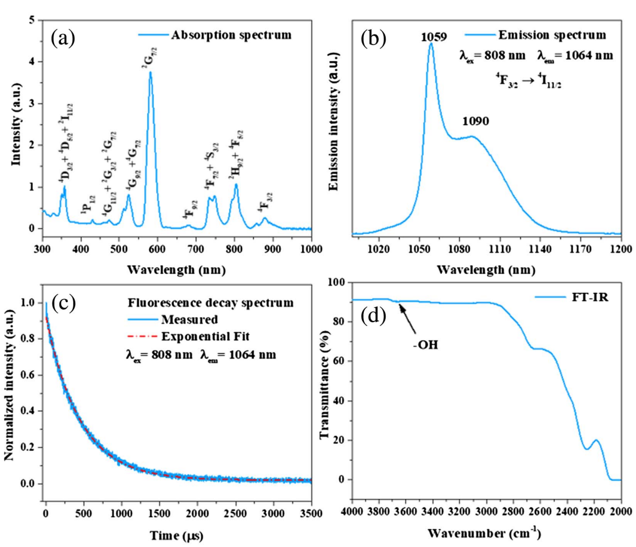

Fig. 3. Nd^{3+} spectrum: (a) absorption, (b) fluorescence, and (c) fluorescence decay. (d) FT-IR spectrum of an Al^{3+}/Nd^{3+} co-doped silica glass chip.

emission peak of the fluorescence spectrum [see Fig. 3(b)] was located at 1059 nm, while the secondary emission peak was located at 1090 nm. The effective linewidth of the emission peak was 44.5 nm, which was obtained by dividing the integral area of the peak by the maximum peak. Emission cross sections (σ_{emi}) were calculated based on the Judd–Ofelt theory[27–29]. The maximum value of σ_{emi} at 1059 nm was 1.77 pm^2, which was higher than that of the σ_{emi} of Nd^{3+}-doped silica glass prepared using other methods with similar compositions (1.4–1.5 pm^2)[30]. As shown in Fig. 3(c), the fluorescence decay spectrum exhibited a typical exponential decay. The fluorescence lifetime (460 μs) was also longer than that reported in other similar studies (<400 μs)[11,30]. A high σ_{emi} and long fluorescence lifetime improve the laser behavior of Nd^{3+}-doped glass. The longer fluorescent lifetime of Nd^{3+} ions indicates that the agglomeration of Nd^{3+} in silica glass was considerably suppressed by co-doping with Al^{3+}. According to Fig. 3(d), the hydroxyl content in silica glass was calculated as 1.8 ppm. The calculation method is described elsewhere[31]. Low hydroxyl content is also a contributor to long fluorescence lifetime.

3.3. Basic optical parameters and laser performance of NDF

Figure 4(a) shows the refractive index profile of the 20/125 NDF. The refractive index difference (Δn) between the core and silica cladding was $\sim 2.83 \times 10^{-3}$, and the calculated core NA was 0.09. The inset in Fig. 4(a) shows the fiber cross section. It has a round cladding. Figure 4(b) depicts the core transmission loss of the NDF. The typical 0.75 dB/m transmission loss of the fiber core at 1200 nm was mainly caused by impurities introduced during the preparation process. The cladding pump absorption coefficient of the NDF at 808 nm was ~2 dB/m.

Figure 5(a) depicts the output power of three NDFs with fiber lengths (L) of 3, 4, and 5 m. The output power was the sum of the forward and reverse output powers. In the case of $L = 5$ m, a maximum laser output power of 14.6 W was achieved when the 808 nm pump power was 38.3 W, and the corresponding slope efficiency was 39.6%. Under the same pump power, for $L = 4$ and 3 m, laser powers of 14 and 13 W, respectively, were obtained, with slope efficiencies of 38.2% and 35.1%. A smaller fiber length corresponds to a smaller absorbed pump power, which reduced the output power or slope efficiency in our experiment. However, a fiber length exceeding 5 m did not significantly improve the slope efficiency because of the

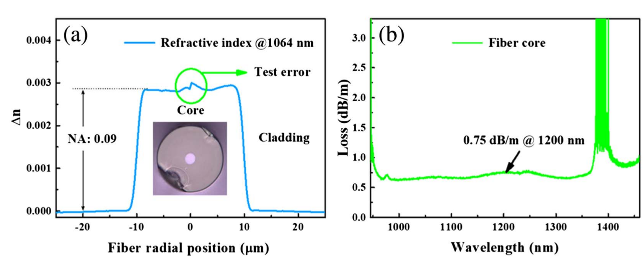

Fig. 4. (a) Refractive index profile and (b) transmission loss of the NDF. The inset shows the cross section of NDF.

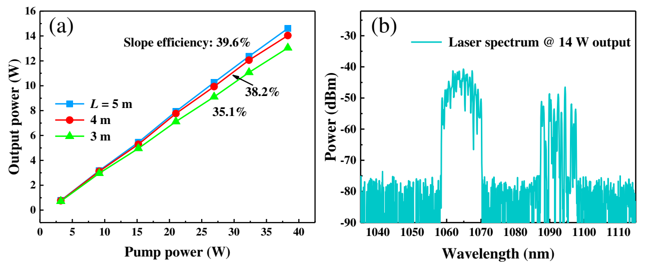

Fig. 5. (a) Laser output power versus pump power and (b) laser spectrum at 14 W output.

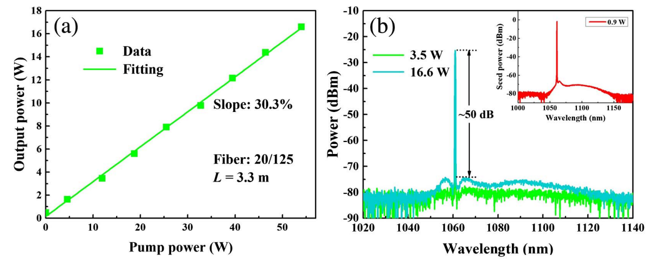

Fig. 6. (a) Slope efficiency and (b) laser spectrum of the NDF amplifier.

relatively high transmission loss of the NDF [0.7 dB/m, see Fig. 4(a)].

The absorbed 808 nm pump power was further calculated according to the residual pump power. For $L = 5$, 4, and 3 m, the absorbed pump powers were 29.7, 27.8, and 24.7 W, respectively; thus, the corresponding slope efficiencies were 51.2%, 52.5%, and 54.9%, respectively. Therefore, through optimized fiber structure tailoring (such as increasing the ratio of core/cladding, adopting octagonal cladding) and/or by reducing the fiber transmission loss, the laser efficiency of the NDF can be expected to be improved further.

Figure 5(b) depicts the laser spectrum for $L = 4$ m at the forward output port when the output power reached 14 W. The spectrum consisted of two broad laser peaks at 1060–1070 nm and 1090–1100 nm. These peaks corresponded to the fluorescence peaks of Nd^{3+} [Fig. 3(b)]. Therefore, for an NDF-based amplifier, achieving a gain bandwidth >40 nm is possible. The laser spectra for $L = 5$ and 3 m exhibited no significant difference from the spectrum for $L = 4$ m.

Table 1 lists the laser performance at ~1060 nm of different Nd^{3+}-doped fibers in Refs. [15,20,21] for comparison. It can be found that the NDF prepared by the MCVD method has higher output power and slope efficiency due to the very low optical loss. Compared with other non-CVD methods, the NDF prepared in this work shows higher output power. It should be pointed out that the NDF prepared in this work was preliminary and has a high transmission loss without the purification technology. But, the loss can be significantly suppressed by the purification technology that we have developed and have applied in Yb^{3+}-doped silica fiber (YDF). The purification technology can reduce the optical loss of YDF prepared by the modified sol-gel method to 50 dB/km, and it is as low as the losses of fibers prepared by MCVD[26]. Moreover, the modified sol-gel method is also beneficial to adjust the refractive index through co-doping F^- or P^{5+} to reduce the NA of the core and thus to improve the beam quality of the LMA fiber[19].

Further, a monolithic all-fiber master oscillator power amplifier (MOPA) was built based on the setup in Fig. 1 to investigate the laser performance of the 20/125 NDF. The seed laser used in the MOPA is a fiber laser consisting of a 5 m commercial PM-NDF-5/125 fiber and two fiber Bragg gratings with a wavelength at 1061 nm, and it has an output power of 1 W at 1061 nm. The seed laser was connected at the left side of the 2 + 1 combiner. To reduce the insertion loss due to mode field mismatch, a 5/125 to 20/125 mode-field adapter was spliced between them. For high-power amplifiers, short fiber lengths are desirable. So, a 3.3-m-long 20/125 NDF was used in MOPA. In addition, the NDF was cleaved with an angle of ~8° as the output port of the MOPA.

The output power of the MOPA is plotted versus the launched pump power in Fig. 6(a). A maximum output of 16.6 W is achieved when the pump power is increased to 54 W, and the corresponding slope efficiency is 30.3%. It should be noted that an 808 nm LD with an output power of 60 W was used here as the pumping source for the MOPA. Figure 6(b) shows the laser spectra of MOPA at the maximum output power and seed power (depicted in the inset). The peak-to-peak contrast between the emission at 1061 nm and the amplified spontaneous emission around 1060 nm reaches ~50 dB, so the output power can be further improved. Comparing the spectra of the amplifier and seed laser, it can be found that there is a spectral depression near the 1061 nm laser peak. However, the reason for this remains unclear.

Table 1. The Laser Performance of Different NDFs from the Literatures and This Work.

Preparation Method	Nd^{3+} Content (ppm)	Power (W)	Slope Efficiency	Reference
Modified sol-gel	5000	14.6	39.6%	This work
Sol-gel	4000	0.025	42%	[21]
Zeolite	10,700	0.027	19.6%	[20]
MCVD	1300	30	46%	[15]

4. Conclusion

In this study, large-size Al^{3+}/Nd^{3+} co-doped silica glass with high Nd^{3+} and Al^{3+} doping concentrations and high doping homogeneity was prepared using a modified sol-gel method combined with high-temperature melting and molding technology. The large emission cross section (1.77 pm^2) and long fluorescence lifetime (460 μs) indicated superior spectral properties and excellent Nd^{3+} ions dispersity. Based on this core glass, an

NDF with core and cladding diameters of 20 and 125 μm, respectively, was drawn. A maximum output power of 14.6 W was achieved with 38.3 W pumping at 808 nm in the laser oscillation experiment. To the best of our knowledge, this is the highest laser output power reported for NDF operated at ∼1060 nm among the non-CVD methods. Besides, a maximum power of 16.6 W corresponding to a slope efficiency of 30.5% at 1061 nm was demonstrated in the MOPA experiment. By further optimizing fiber structure (such as increasing the ratio of core/cladding, adjusting the shape of cladding and core) and/or reducing the fiber loss, it is expected to increase the laser power and efficiency of NDF.

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