

Article

## Heavily Tm<sup>3+</sup>-Doped Silicate Fiber for High-Gain Fiber Amplifiers

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**Abstract:** We report on investigation the potential of a 7 wt% ( $8.35 \times 10^{20}$  Tm<sup>3+</sup>/cm<sup>3</sup>) doped silicate fibers for high-gain fiber amplifiers. Such a high ion concentration significantly reduces the required fiber length of high-power 2 μm fiber laser systems and allows the high-repetition rate operation in 2 μm mode-locked fiber lasers. To evaluate the feasibility of extracting high gain-per-unit-length from this gain medium, we measure several key material properties of the silicate fiber, including the absorption/emission cross-sections, upper-state lifetime, fiber background loss, and photodarkening resistance. We show through numerical simulations that a signal gain-per-unit-length of 3.78 dB/cm at 1950 nm can be achieved in a watt-level core-pumped Tm<sup>3+</sup>-doped silicate fiber amplifier. In addition, an 18-dB 2013-nm amplifier is demonstrated in a 50-cm 7 wt% Tm<sup>3+</sup>-doped double-clad silicate fiber. Finally, we experimentally confirm that the reported silicate host exhibits no observable photodarkening.

**Keywords:** Thulium; silicate fiber; fiber laser; fiber amplifiers

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## 1. Introduction

Ultrafast laser sources in the 2- $\mu\text{m}$  eye-safe region are of interest for many applications, such as frequency comb metrology, environmental sensing, eye-safe LIDAR, space communication, medical surgery, particle accelerators, and light sources for efficient mid-IR generation. To produce 2- $\mu\text{m}$  laser emission in fibers,  $\text{Tm}^{3+}$  is a favorable ion because it offers high quantum efficiency, broad gain bandwidth, and strong absorption band around 800 nm, the wavelength available from commercial high-power diode lasers. Through the “two-for-one” cross-relaxation energy transfer, more than 70% slope efficiency has been reported in a 790-nm pumped  $\text{Tm}^{3+}$ -doped fiber laser [1]. In addition, the broad gain bandwidth from 1.8  $\mu\text{m}$  to 2.1  $\mu\text{m}$  allows a large wavelength tuning range and makes  $\text{Tm}^{3+}$ -doped fiber a suitable gain medium for generation of femtosecond pulses. In recent years, the output power from  $\text{Tm}^{3+}$ -doped fiber lasers and amplifiers have been scaled to well above the kilowatt level. One approach for further power scaling these light sources is to increase the  $\text{Tm}^{3+}$  concentration to reduce the required fiber length, and thus avoid detrimental nonlinear effects. More importantly, the high  $\text{Tm}^{3+}$  concentration enables the efficient cross-relaxation processes leading to high quantum efficiencies. The minimum  $\text{Tm}^{3+}$  concentration to generate efficient cross-relaxation process is known to be around 3wt%. Unfortunately, this doping level is hard to achieve in a silica fiber because excessive amounts of  $\text{Tm}^{3+}$  in silica fibers may cause concentration quenching and photodarkening. Therefore, most current  $\text{Tm}^{3+}$ -doped silica fibers employed in mode-locked fiber lasers have concentrations below 2 wt%, leading to the long required length of gain fibers. Such a meter-long cavity length limits the repetition rate of a mode-locked laser to multi-megahertz level.

Fabricating highly  $\text{Tm}^{3+}$ -doped fibers has been an active subject in the field of 2- $\mu\text{m}$  fiber lasers. Several glass hosts, such as germanate, tellurite, and silicate, have been intensely studied. In 2007, Wu *et al.* successfully demonstrated a 106-W germanate fiber laser with 5 wt% of the  $\text{Tm}_2\text{O}_3$  concentration [2]. Although this result makes the germanate fiber a promising gain medium for high-power 2- $\mu\text{m}$  generation, the germanate fiber tends to have low photodarkening resistance and low compatibility with passive silica fibers, which makes it difficult to build an all-fiber laser system. On the other hand, an efficient 2- $\mu\text{m}$  tellurite fiber laser has been reported; however, the presented  $\text{Tm}^{3+}$  concentration is relatively low and the fiber background loss is too high [3]. A good alternative approach reported here is using multi-component silicate glass as the fiber host. Compared with silica glass, the multi-component silicate glass has less-defined glass network, which provides much higher  $\text{Tm}_2\text{O}_3$  solubility. It is believed the multi-component silicate fibers can be doped with more than 10 wt% of  $\text{Tm}_2\text{O}_3$ . Such high solubility allows for considerably shorter fiber lengths, which are beneficial for the mitigation of nonlinear effects, high pulse repetition rates, and efficient two-for-one cross-relaxation processes. Furthermore, different from germanate and tellurite glasses, the main glass network of the silicate fiber is  $\text{SiO}_2$ , which leads to much stronger mechanical strength and better compatibility with conventional passive silica fibers.

The first cw and Q-switched  $\text{Tm}^{3+}$ -doped silicate fiber lasers were successfully built by Geng *et al.* in 2009 [4,5]. Following this research, an all-fiber passively mode-locked laser system based on highly  $\text{Tm}^{3+}$ -doped silicate fiber was also demonstrated [6]. All of these promising results were achieved in  $\text{Tm}^{3+}$ -doped silicate fibers with the concentration less than 5wt%. We report in this paper our latest

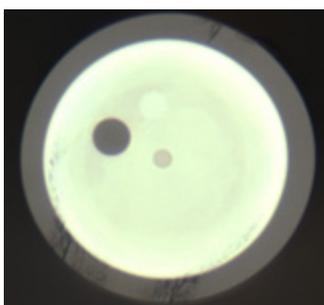
progress in silicate fiber fabrication as well as the simulation and testing of  $\text{Tm}^{3+}$ -doped silicate fiber amplifier with one of the highest published concentrations ( $8.35 \times 10^{20}/\text{cm}^3$ ) for a  $\text{Tm}^{3+}$ -doped fiber.

## 2. Material Properties of $\text{Tm}^{3+}$ Doped Silicate Fiber

The preforms of the  $\text{Tm}^{3+}$ -doped used in this paper were made of alkaline-earth silicate glasses. The glass included  $\text{SiO}_2$ ,  $\text{Al}_2\text{O}_3$ ,  $\text{BaO}$ ,  $\text{ZnO}$ , and  $\text{La}_2\text{O}_3$ .  $\text{Al}_2\text{O}_3$  was added to ensure high mechanical strength and good chemical durability. Transition metals such as Fe and Cu, as well as alkali ions, were eliminated to further enhance the glass properties.

A photograph of the cleaved fiber end is shown in Figure 1. The fiber has a double-clad structure with a  $152 \mu\text{m}$  circular outer cladding, a  $125 \mu\text{m}$  circular inner cladding with a NA of 0.617, and a  $10 \mu\text{m}$  circular core with a NA of 0.149 that is single mode at wavelengths above 1550 nm. A low-index rod was inserted inside the inner cladding to provide cladding mode mixing and improve spatial overlap between the pump modes and doped core. It has been shown in [6] that the added low-index rod increases the pump absorption in the doped core. Since it is easier to manipulate the refractive index of this multi-component glass than silica, the outer cladding of double-clad silicate fibers can also be made of silicate glass. This is important because the thermal properties of the glass outer cladding are far superior to the properties of the polymers widely used as outer claddings in double-clad silica fibers. The fiber was jacketed with low-index polymer for the protection from rugged environments. The fiber parameters are listed in Table 1.

**Figure 1.** Photograph of the cleaved  $\text{Tm}^{3+}$ -doped silicate fiber.



**Table 1.** Fiber parameters.

	Diameter ( $\mu\text{m}$ )	Refractive index	NA
Active core	10	1.64	0.149
Low-index rod	20	1.506	
Inner cladding	125	1.623	0.617
Outer cladding	152	1.513	

The silicate fiber was manufactured at Advalue Photonics by the rod-in-tube technique. This technology is commonly used for producing fibers made of multi-component glasses such as fluorozirconate and silicate-based fibers with multi-component glass core compositions. The main reason is that some of the chemicals required to fabricate silicate fibers cannot be volatilized. A core glass rod and two cladding tubes were fabricated and assembled to form the fiber preform. The core

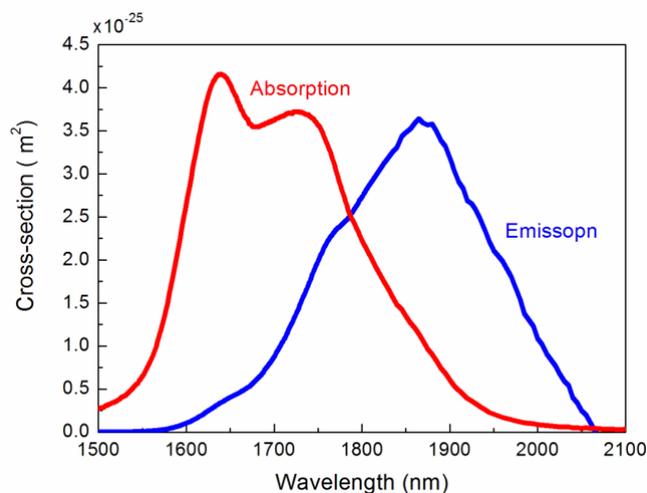
rod was cored out of a larger bulk sample with a diamond-embedded core drill, and subsequently polished. Both the inside and outside surfaces of the glass tubes for the inner and outer claddings were polished to a high surface quality. The inside diameter of the inner-cladding tube was matched to the diameter of the core rod, and the inside diameter of the outer-cladding tube is matched to the outer diameter of the inner-cladding tube. Both core and cladding glasses were carefully prepared to ensure that their chemical and thermo-mechanical characteristics are compatible, especially their softening temperature and thermal expansion coefficient. The fiber was drawn in a special furnace optimized for non-silica glasses. One important advantage of fabricating double-clad fibers by using the rod-in-tube method is that one can control the refractive indices of the fiber core and inner-cladding glasses precisely and independently.

The propagation loss was measured using the cut-back method in a 150-cm-long silicate fiber doped with 7 wt% of  $\text{Tm}_2\text{O}_3$ . A 250-mW, 976-nm probe laser was coupled into the fiber core under fixed launching conditions, and the residual output power was repeatedly measured after gradually cutting back the fiber sample. The wavelength of the probe laser was chosen to be outside of the  $\text{Tm}^{3+}$  absorption band. The propagation loss and a launched laser power were inferred from an exponential fit to the measured residual power as a function of the fiber length. The measured propagation loss was 0.7 dB/m.

The glass host composition affects the solubility of the rare-earth dopant which, in turn, may affect the fluorescence lifetime, absorption, emission, and excited-state absorption cross-sections of the dopant transitions. To obtain a reliable theoretical prediction of the performance of the silicate fiber laser sources, we need to know the spectroscopic properties accurately, especially the emission and absorption cross-sections. These properties were measured in bulk silicate glass samples with the same chemical composition and  $\text{Tm}^{3+}$ -concentration as the  $\text{Tm}^{3+}$ -doped silicate fiber core.

The absorption cross-sections were obtained from the measured white-light absorption spectrum of the sample. The spectral shape of the emission was obtained from the measured fluorescence spectrum of a thin silicate glass plate, pumped at 793 nm near one of its edges. The emitted fluorescence was collected from the thin side of the sample. Possible re-absorption of the signal, which might have distorted the measured fluorescence spectrum, was avoided by keeping the sample thin enough. Figure 2 shows the measured cross-section spectra for absorption and emission of  $\text{Tm}^{3+}$  in the silicate glass host. The absorption and emission peaks are located at 1640 nm and 1865 nm, respectively. The maximum absorption cross-section is measured to be  $4.16 \times 10^{-25} \text{ m}^2$ . The absolute scaling of the emission cross-section is determined by applying the McCumber method to the measured peak absorption cross-section [7]. The calculated peak emission cross-section is  $3.59 \times 10^{-25} \text{ m}^2$ . Although this method has been shown to be rather inaccurate when precise cross-section values are required [8], it provides a convenient estimate for the calibration of the measured fluorescence spectrum. As shown in Figure 2, the spectra of  $\text{Tm}^{3+}$  ions in silicate glass are fairly similar to the spectra in silica glass [9], except for the larger emission cross-sections in the long-wavelength region.

**Figure 2.** Measured absorption and emission cross-section spectra of the  $\text{Tm}^{3+}$ -doped silicate fiber.



The fluorescence lifetime of the 7-wt%  $\text{Tm}^{3+}$ -doped silicate glass was measured to be 0.635 ms. This value is similar to the  $\text{Tm}^{3+}$  lifetime in low  $\text{Tm}^{3+}$ -doped silicate glass fibers. The measured fluorescence relaxation curve showed a single exponential decay, thus demonstrating negligible concentration quenching in spite of the high  $\text{Tm}^{3+}$  concentrations.

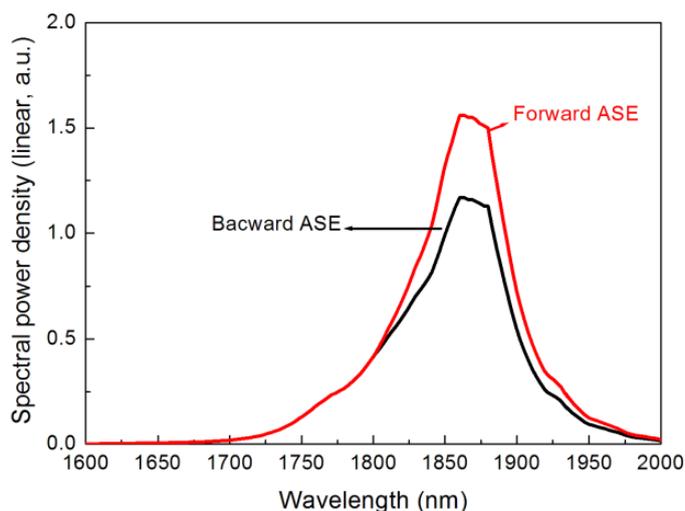
### 3. Theoretical Predictions of $\text{Tm}^{3+}$ -Doped Silicate Fiber Laser Sources

#### 3.1. 1575-nm Core-Pumped Amplified Spontaneous Emission (ASE)

Various theoretical models of rare-earth-ion doped fiber laser sources have been presented. The simulation code employed in this paper is LIEKKI™ Application Designer v4.0 [10]. This code solves the coupled laser rate equations numerically to predict the output performance of the fiber source, such as the amount of pump power and the length of fiber required to achieve a certain output power level, the population inversion at every point along the fiber, the forward and backward ASE output powers and spectra, the gain spectrum, excess noise, *etc.*, in either a core-pumped or a cladding-pumped configuration. Comparison of the code prediction to the measured properties of  $\text{Tm}^{3+}$ -doped superfluorescent fiber sources [9] and  $\text{Tm}^{3+}$ -doped silicate fiber laser [4] has shown that this code is quite accurate.

We firstly used the code to predict forward and backward ASE output performance of the 7 wt%  $\text{Tm}^{3+}$ -doped silicate fiber, and compare the simulation results of the 1.25 wt%  $\text{Tm}$ -doped silica fibers, which is reported in [9]. For a fair comparison, the silicate and silica fiber ASE sources have exactly the same fiber structure, and are pumped with exactly the same way. Figure 3 shows the forward and backward ASE intensity spectra of a 5-cm 9/125  $\text{Tm}^{3+}$ -doped silicate fiber, core pumped with a 900-mW 1575-nm laser. As expected, ASE wavelengths of the silicate fibers are red-shifted in comparison with the silica fibers. Full-width half-maximum (FWHM) of the forward and backward ASE are 110 nm and 96 nm, respectively. The broad ASE bandwidth confirms that  $\text{Tm}^{3+}$ -doped silicate fibers are suitable gain medium for generation of ultra-short pulses.

**Figure 3.** The forward and backward Amplified Spontaneous Emission (ASE) intensity spectra of a 5-cm 9/125 Tm<sup>3+</sup>-doped silicate fiber, core pumped by a 900-mW 1575-nm laser.

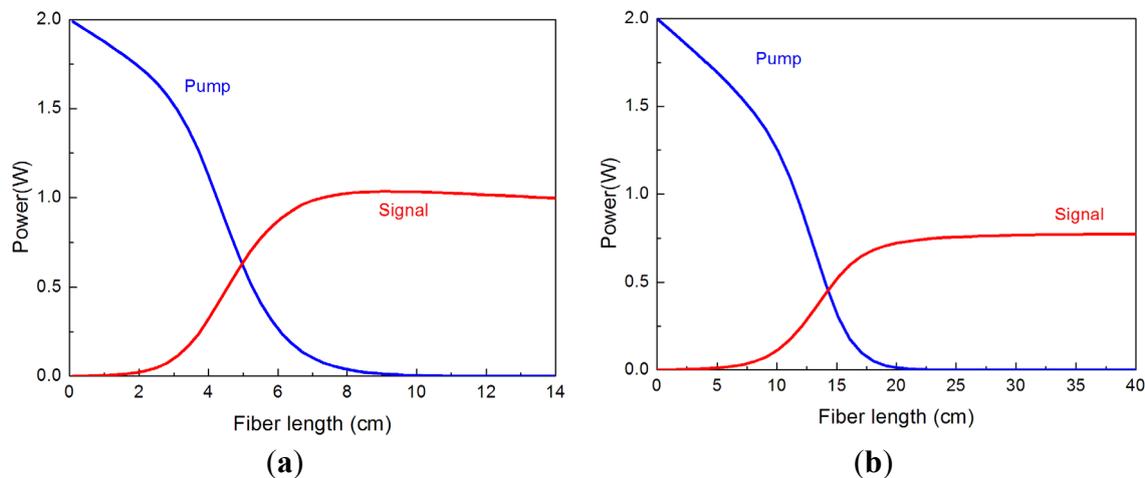


### 3.2. Single-Mode Tm<sup>3+</sup>-Doped Fiber Amplifier with High Gain Per-Unit-Length

To evaluate the highest gain per unit length from a single-mode Tm<sup>3+</sup>-doped silicate fiber, we used the code to compare the predicted performance of two heavily Tm<sup>3+</sup>-doped fiber master-oscillator power amplifiers (MOPAs), one made with a 7 wt% Tm<sup>3+</sup>-doped silicate fiber and the other one with a 4 wt% Tm<sup>3+</sup>-doped silica fiber. For a fair comparison, two amplifiers had exactly the same fiber structure, and they were seeded and pumped exactly the same way. Both fibers were taken to have a single-mode core diameter of 10  $\mu\text{m}$  and an inner-cladding diameter of 125  $\mu\text{m}$ . To reflect actual differences between the two types of fibers, namely their propagation loss, upper-state lifetime, and Tm<sup>3+</sup> concentration, each of these three parameters was given its actual measured value. For the silica fiber, we used the best current values reported for commercial fibers, *i.e.*, a loss of 15 dB/km [11], an upper-state lifetime of 0.42 ms [12], and a concentration of  $2.67 \times 10^{20}$  Tm<sup>3+</sup>/cm<sup>3</sup> ( $\sim 4$  wt% Tm<sub>2</sub>O<sub>3</sub> [13]). The corresponding values for the silicate fiber were 0.7 dB/m, 0.635 ms,  $8.35 \times 10^{20}$  Tm<sup>3+</sup>/cm<sup>3</sup> and, respectively. The fiber length was a free parameter. Each amplifier was seeded with 1-mW 1950-nm laser and core-pumped with a launched pump power of 2 W at 1575 nm. The simulator used the actual measured absorption and emission cross-section spectra of these two fibers as input.

Figures 4a,b show the theoretical prediction of the output power *versus* fiber length for the silicate and silica fiber MOPAs, respectively. As the results shown in Figure 4a, the silicate fiber MOPA will produce 1.05 W of signal output power at its optimum length, 8 cm. It is corresponding to a 30.2-dB signal gain and gain-per-unit-length of 3.78 dB/cm at 1950 nm. On the other hand, Figure 4b shows that, at the same pump power level, the silica fiber MOPA needs a 25-cm fiber length to produce the maximum signal output power, 0.77 W. The relatively lower signal output power and efficiency of the silica fiber MOPA are caused by the relatively shorter <sup>3</sup>F<sub>4</sub> lifetime. The corresponding signal gain per unit length of silica fiber MOPA is 1.15 dB/cm at 1950 nm.

**Figure 4.** Theoretical predictions of the 1950-nm output single and residual pump powers dependence on fiber length for (a) a silicate fiber master-oscillator power amplifier (MOPA) and (b) a silica fiber MOPA. The red and blue solid-curves show the output signal and residual pump power as a function of the fiber length, respectively.



#### 4. Experimental Demonstration of 793-nm Cladding-Pumped Tm<sup>3+</sup>-Doped Silicate Fiber Amplifier

To provide an experimental validation of the silicate fiber amplifier operation, we developed a 793-nm cladding-pumped Tm<sup>3+</sup>-doped silicate fiber MOPA. The experimental setup is shown in Figure 5. It is a ring-cavity 2013-nm Tm<sup>3+</sup>-doped silicate fiber laser, followed by a Tm<sup>3+</sup>-doped silicate fiber amplifier. The aforementioned 7 wt% Tm<sup>3+</sup>-doped silicate fibers were employed in both the fiber oscillator and amplifier. Without the use of wavelength selection devices, the laser was naturally operated at 2013 nm with the maximum output power of 17.8 mW and slope efficiency of 15.9 %. The 2013-nm seed laser was then amplified by another 50-cm 7 wt% Tm<sup>3+</sup>-doped silicate fiber with the signal input power of 472.5 μW.

**Figure 5.** Experimental Tm<sup>3+</sup>-doped silicate fiber amplifier.

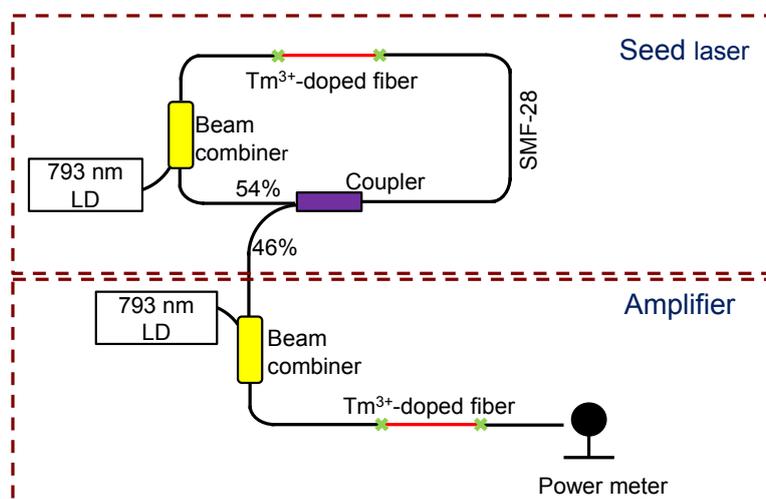
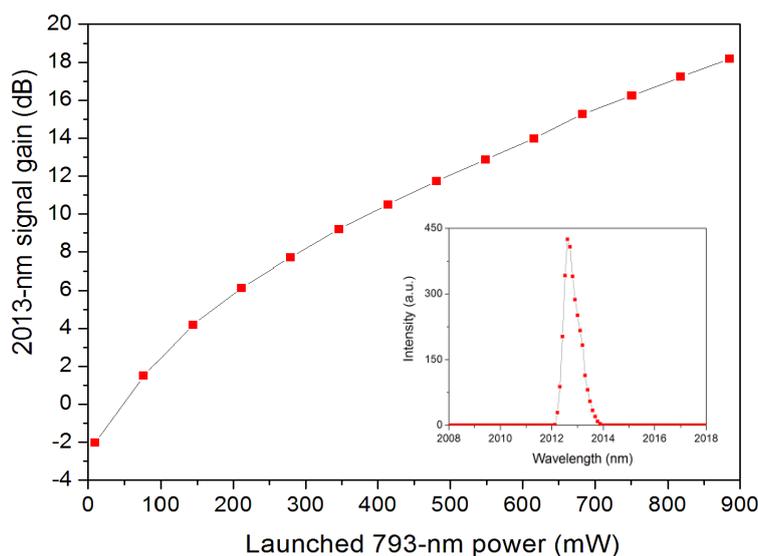


Figure 6 shows the measured signal gain as a function of launched pump power. A maximum gain of 18.2 dB with respect to the launched signal power was reached at an absorbed pump power of

460 mW (885 mW launched). The corresponding 2013-nm signal gain-per-unit-length of this amplifier was 0.54 dB/cm. Because of the 2 times larger emission cross-section at 1950 nm, the signal gain at 1950 nm could be much higher than that at 2013 nm under the same pump power. As shown in Figure 6, there is no sign of signal gain saturation, and the maximum signal gain is limited by the available pump power. The 793-nm cladding absorption coefficient of this silicate fiber was measured as  $\sim 5.18$  dB/m. It means a fiber length of 2 m would be needed to provide sufficient pump absorption and further increase the signal gain. The measured output spectrum of the fiber amplifier measured at 18.2 mW is shown in the inset of Figure 6.

**Figure 6.** Experimental  $\text{Tm}^{3+}$ -doped silicate fiber amplifier.



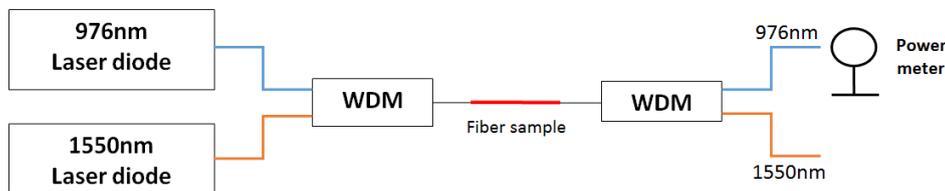
## 5. Photodarkening Measurement of $\text{Tm}^{3+}$ -Doped Silicate Fiber

A measurement method providing an application-independent quantification of photodarkening in fibers has been proposed by Koponen *et al.* [14]. In this method, a visible probe laser is used to measure the transmission of the pumped active fiber. The photodarkening-induced loss of silica or silicate fibers at the probe wavelength is then estimated from the measured reduction in transmission. UV-induced color centers in the silica or silicate fibers have an absorption peak at 500 nm and the tail of this absorption line extends to the NIR [15]. In this experiment, we aim to measure the application-independent photodarkening resistance of the  $\text{Tm}^{3+}$ -doped silicate fibers, namely, the maximum  $\text{Tm}^{3+}$  concentrations allowed in the fibers before the onset of photodarkening.

The experimental setup for the photodarkening test in silicate fibers is shown in Figure 7. The employed fiber sample was a 7-cm  $\text{Tm}^{3+}$ -doped silicate fiber with a 10- $\mu\text{m}$  core and 125- $\mu\text{m}$  cladding. To get a significant population inversion, the fiber sample was core-pumped by a 1550-nm laser with a 400-mW output power. A 976-nm probe laser was also coupled into the fiber through a WDM fiber coupler to periodically measure the photodarkening-induced reduction in fiber transmission. The 976-nm laser was chosen to separate the attenuation effects coming from original  $\text{Tm}^{3+}$ -absorption and photodarkening. Both the 1550-nm pump and 976-nm probe lasers were temperature/wavelength stabilized with long-term power fluctuations of less than 1%. The test fiber was spliced to the WDMs

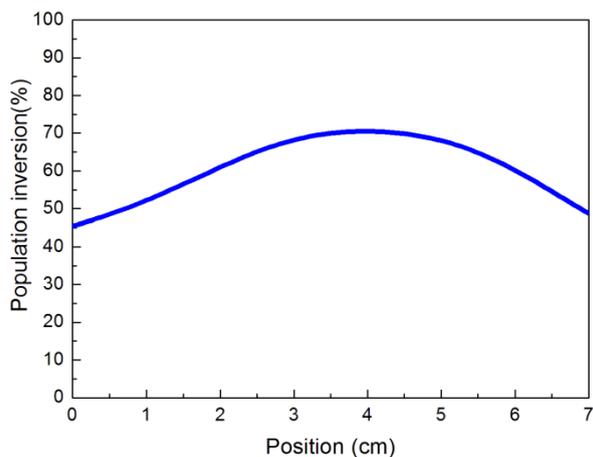
and the whole optical system was well isolated from possible mechanical shocks in the lab environment and thus assured stability during the measurements.

**Figure 7.** Photodarkening measurement setup.

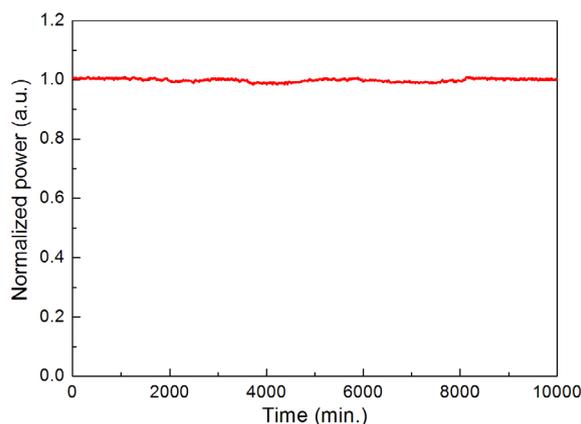


The population inversion along the fiber sample is simulated and shown in Figure 8. The result indicates >45% average population inversion along the whole fiber sample. Figure 9 shows the normalized 976-nm transmission of the silicate fiber samples measured as a function of exposure time. After pumping for over 10,000 min, no sign of photodarkening was observed. Because no onset of photodarkening can be obtained in this fiber sample under such high population inversion conditions, the fiber should be immune to photodarkening when used in most laser systems.

**Figure 8.** The population inversion along the 7-cm  $Tm^{3+}$ -doped silicate fiber.



**Figure 9.** Measured normalized transmission of the silicate fiber at 976 nm as a function of pump exposure time.



## 6. Conclusions

In this paper, we present the first study of a single-mode 7 wt% Tm<sup>3+</sup>-doped silicate fiber for high-gain fiber amplifiers. To the best of our knowledge, 7 wt% is the record high Tm<sup>3+</sup> ion concentration in fibers. Such a high ion concentration could be beneficial for ultra-short 2- $\mu$ m fiber amplifiers and high-repetition-rate 2- $\mu$ m mode-locked fiber lasers. The studied essential material properties include absorption/emission cross-sections, upper-state lifetime, fiber background loss, and photodarkening resistance. These results allow us to precisely evaluate the potential of the silicate fiber for high-gain fiber amplifiers. Our simulation results indicate that a signal gain-per-unit-length of 3.78 dB/cm can be reached in a watt-level core-pumped Tm<sup>3+</sup>-doped silicate fiber amplifier. As a step in this direction, we report a 2013-nm fiber MOPA with the 7 wt% Tm<sup>3+</sup>-doped double-clad fiber. A 2013-nm signal gain of 18 dB was achieved in a 50-cm gain fiber with the limitation of the available pump power. We also present experimental evidence that, even under intense core pumping, silicate fibers can support high Tm<sup>3+</sup> doping levels without the onset of photodarkening. Both results put the heavily Tm<sup>3+</sup>-doped silicate fibers at an advantage for high-gain fiber amplifiers.

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## Conflicts of Interest

The authors declare no conflict of interest.

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