Highly efficient thulium-doped high-power laser fibers fabricated by MCVD

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Abstract: We report a hybrid process by combining both vapor-phase and solution-doping techniques of rare-earth doped preform fabrication in conjunction with the MCVD technique, in order to fabricate highly efficient Tm-doped laser fibers. The proposed fabrication route takes advantage of co-doping silica with high alumina content through the vapor-phase doping process, which is otherwise difficult to achieve using conventional solution doping technique. In addition, by employing the solution doping method, high-purity thulium halide precursors that have low vapor pressures up to several hundred degree Celsius. These high-purity thulium halide precursors can be used to dope the fiber core region with a high thulium concentration that is optimized for an efficient two-for-one cross-relaxation process for 79xnm diode pumped thulium-doped fiber laser. Fibers fabricated using the hybrid approach show more homogeneous and flat-top dopant profiles, compared with the conventional approach, where both aluminum and thulium are incorporated in the core through solution doping. This will ensure that more doped region will take part in the cross-relaxation process. Superior laser performance with a slope efficiency of >70% in the two-micron band has been demonstrated when diode pumped at ~790nm.

1. Introduction

Thulium (Tm)-doped fiber lasers (TDFLs) operating in the “eye-safe” region from 1700nm to 2100nm are under considerable research over the past decade owing to their power scaling ability to kW level [1,2]. Areas such as medical applications, LIDAR systems, material processing (i.e. welding, cutting and marking) of plastics, free-space propagation for satellite communications, and non-linear frequency conversion to the mid-IR, have found significant advantages from high power 2µm sources [3–5]. Tm-doped fibers pumped with 79xnm high power diodes, enable a cross-relaxation process to achieve two excited ions into the 3F 4 manifold for one pump photon (two-for-one cross-relaxation), when the fiber core composition including the thulium concentration is optimized [6–8]. This has the advantage of reaching Tm-doped fiber laser quantum efficiency up to 200% in the two-micron band.

High power Tm-doped fibers are commonly fabricated by modified chemical vapor deposition (MCVD) technique in conjunction with the well-established solution doping method to introduce both Al 2O 3 and Tm 2O 3 into the fiber core. The importance of aluminium as a co-dopant in silica matrix is that it reduces the clustering between adjacent rare earth ions, allowing incorporation of higher concentration of rare earths within the silica network [9,10].

Previous reports have shown that thulium concentration exceeding 2wt% is required in order to make the two-for-one cross-relaxation process effective for 79xnm pumped TDFL allowing the laser efficiency to reach well above the stokes limit [9,11,12]. The core of the fiber should be co-doped with several molar percent of alumina to allow incorporation of the high concentrations of thulium ions. However, the alumina concentration over 4mol% is
difficult to achieve using conventional solution doping process [13]. Thus, high thulium concentration in the fiber without an adequate level of alumina concentration may cause adverse effects such as reduced lifetime and unwanted energy transfer upconversion (ETU) process [14,15].

In this paper, we demonstrate a novel fabrication route to increase the alumina content in the core by using a vapor phase technique. The thulium ions are incorporated in the core by conventional solution doping route within an optimized aluminosilicate glass host composition. With our proposed rare earth (RE)-doped preform fabrication route (called hereafter hybrid gas phase-solution doping technique), not only high dopant concentrations of Al2O3 and Tm2O3 can be reached, but also a more flat-top dopant distribution of both thulium and aluminium within the core over conventional MCVD-solution doping process is possible. Another important aspect of this fabrication route is the flexibility of using high-purity thulium halide precursors despite their low vapor pressures up to several hundred degree Celsius. Fabrication of RE-doped fiber based on MCVD and all-vapor phase doping method using RE-organometallics precursors has not been reported (to the best of our knowledge) for a heavily Tm-doped aluminosilicate core ensuring an efficient cross-relaxation process. The reaction of halides such as AlCl3 with organometallics in vapor phase may limit the amount of RE incorporation in the core. Fibers fabricated using the hybrid gas phase-solution doping process yield a laser efficiency of ~73% at ~2070nm. When tested over different fiber lengths and thulium concentrations (3.5 – 5.6wt%), the free-running laser efficiency of 70 ± 3% has been reached covering the wavelength range of 1980 – 2080nm.

2. Experimental and results

We present here the performance of several Tm-doped fibers with thulium concentrations varying from 2wt% – 6wt% fabricated in-house. Six fibers were fabricated by MCVD in combination with a hybrid gas phase-solution doping technique with different thulium and aluminium concentrations in the core, and one fiber was fabricated with our optimized MCVD and conventional solution doping process offering a maximum cross-relaxation process with this fabrication method.

Tm-doped preforms were fabricated in an MCVD apparatus using Suprasil F300 silica substrate tubes from Heraeus with a nominal OD/ID of 20/16mm. First, a few cladding layers of silicon oxide (SiO2) were deposited inside the tube. Next, an aluminosilicate soot layer was introduced by mixing SiCl4 and AlCl3 with oxygen at an elevated temperature. The AlCl3 evaporator mass flow was set in the range of 30 – 60mg/min and helium was used as a carrier gas to transport the AlCl3 vapor to the deposition zone through a separate heated delivery line that was maintained at about 200°C. Depending on the aluminum concentration in the gas mixture, the soot deposition temperature was adjusted to maintain an adequate porosity of the soot body to allow uniform incorporation of thulium ions during the solution doping stage. Subsequently, the preform was removed from the MCVD lathe and it was soaked for a period of one hour in an aqueous solution of thulium chloride (TmCl3.xH2O). Owing to the wet nature of the solution doping technique, an effective dehydration process was performed. Finally, the substrate tube was reassembled on the MCVD lathe for oxidation and sintering of the core layer. The tube was then collapsed into a solid rod in the usual manner.

The preforms fabricated using the two different routes were post-processed to modify their core to cladding ratio. The circular cladding in the preform was milled to a near octagonal shape to ensure an efficient pump absorption in the cladding pump geometry. The preforms were drawn into 200µm diameter fibers and coated with a low index polymer. The core diameter in all fibers was about 8µm.

The resultant fibers were characterized by an IFA-100 Multi-wavelength Optical Fiber Analyzer for the refractive index profile and by Electron Dispersive X-Ray Spectroscopy (EDX) to identify the core composition and dopant distribution. Additionally, cladding
absorption measurements were carried out using a white light source and an optical spectrum analyzer. The characteristics of five thulium-doped fibers are shown in Table 1. Here, HGS and SD refer to hybrid gas phase-solution doping and conventional solution doping respectively.

Table 1. Thulium-doped fibers characteristics.

<table>
<thead>
<tr>
<th>Fiber</th>
<th>NA</th>
<th>Core Abs at 790nm [dB/m]</th>
<th>Tm$_2$O$_3$ [wt%]</th>
<th>Al$_2$O$_3$ [wt%]</th>
<th>Lifetime [µs]</th>
</tr>
</thead>
<tbody>
<tr>
<td>SD-01</td>
<td>0.20</td>
<td>1400</td>
<td>5.1</td>
<td>6.8</td>
<td>385</td>
</tr>
<tr>
<td>HGS-01</td>
<td>0.23</td>
<td>1150</td>
<td>4.3</td>
<td>10.6</td>
<td>503</td>
</tr>
<tr>
<td>HGS-02</td>
<td>0.27</td>
<td>1600</td>
<td>6.4</td>
<td>11.6</td>
<td>524</td>
</tr>
<tr>
<td>HGS-03</td>
<td>0.22</td>
<td>1100</td>
<td>4.0</td>
<td>9.3</td>
<td>487</td>
</tr>
<tr>
<td>HGS-05</td>
<td>0.22</td>
<td>800</td>
<td>2.8</td>
<td>9.6</td>
<td>454</td>
</tr>
</tbody>
</table>

We compared the dopant distribution of HGS-01 and SD-01 fibers. The core composition was 10.6 wt% of Al$_2$O$_3$ and 4.3 wt% of Tm$_2$O$_3$ for HGS-01 and 6.8 wt% of Al$_2$O$_3$ and 5.1 wt% of Tm$_2$O$_3$ for SD-01. The results show that with the hybrid gas phase-solution doping technique, the aluminum incorporation in the core can be significantly increased. The normalized thulium distribution in the core region overlapped with the fiber refractive index is shown in Fig. 1.

The thulium and aluminum dopants distribution follow the refractive index profile in both fibers. However, in fiber HGS-01, a flat-top dopant profile has been achieved, which will ensure more thulium doped region to take part in cross-relaxation process [7].

![Thulium distribution overlapped with fiber refractive index profile (FRIP).](image)

The fluorescence lifetime of $^{3}F_4$ energy level manifold in thulium was measured under 790nm pulsed pump light with a modulation frequency of 10Hz and 10% duty cycle. An InGaAs photo-detector was used to capture the 2µm fluorescence signal. The lifetime of the excited level was recorded on an oscilloscope. Owing to the high aluminum concentration and better distribution within the core, fibers fabricated by hybrid gas phase-solution doping technique have shown a noticeable enhancement in the fluorescence lifetime compared to SD-01, which was fabricated with conventional solution doping process, as shown above in Table 1.

The laser performance of thulium-doped fibers was measured in a 4% – 4% laser cavity configuration. The fibers were cladding pumped by a 790nm fiber coupled multimode laser diode through a combination of collimating lenses. The pump launch efficiency into the fibers was estimated at ~90%. Dichroic mirrors were used to separate the pump from signal wavelengths, at both pump launch and pump throughput ends of the fiber. The slope efficiency was calculated with the total output power from both ends of the fiber.

The output power versus the absorbed pump power of HGS-01 and SD-01 fibers are presented in Fig. 2. The output power of ~22W was limited by the available pump power.
Fiber length was adjusted to obtain the maximum output power in a free-running laser cavity, which was 10m and 12m for HGS-01 and SD-01 respectively. The output spectrum of HGS-01 fiber at its maximum output power is shown in the inset of Fig. 2. A monochromator was used to measure the 2µm emission band. The slope efficiency ($\eta$) of HGS-01 fiber was ~70% compared to ~63% of SD-01 fiber. The fiber HGS-01 fabricated by the hybrid gas phase-solution doping technique is showing improved two-for-one cross-relaxation process compared with the fiber fabricated by conventional solution doping due to more uniform thulium doped profile in the core as shown in Fig. 1.

![Laser spectrum at maximum output power from HGS-01](image)

Fig. 2. Laser characteristics of HGS-01 and SD-01 fibers. (Inset: laser spectrum at maximum output power from HGS-01)

We evaluated the performance of Tm-doped fibers fabricated by the hybrid gas phase solution doping technique with different thulium and aluminum concentrations. The length of the Tm-doped fiber was adjusted in a free-running laser configuration to obtain a maximum laser efficiency. The results are summarized in Table 2.

**Table 2. Performance of thulium-doped fibers fabricated using hybrid process.**

<table>
<thead>
<tr>
<th>Fiber</th>
<th>Tm [wt%]</th>
<th>Al [wt%]</th>
<th>Length [m]</th>
<th>Emission [nm]</th>
<th>$\eta$ [%]</th>
<th>Max Output Power [W]</th>
</tr>
</thead>
<tbody>
<tr>
<td>HGS-01</td>
<td>3.8</td>
<td>5.6</td>
<td>11</td>
<td>2025</td>
<td>71.1</td>
<td>22.6</td>
</tr>
<tr>
<td>HGS-02</td>
<td>5.6</td>
<td>6.1</td>
<td>8</td>
<td>2036</td>
<td>72.4</td>
<td>24.4</td>
</tr>
<tr>
<td>HGS-03</td>
<td>3.5</td>
<td>4.9</td>
<td>12</td>
<td>2022</td>
<td>70.1</td>
<td>20.9</td>
</tr>
<tr>
<td>HGS-04</td>
<td>3.0</td>
<td>7.8</td>
<td>9</td>
<td>2013</td>
<td>62.6</td>
<td>16.2</td>
</tr>
<tr>
<td>HGS-05</td>
<td>2.5</td>
<td>5.1</td>
<td>10</td>
<td>2009</td>
<td>60.4</td>
<td>15.8</td>
</tr>
<tr>
<td>HGS-06</td>
<td>2.0</td>
<td>7.2</td>
<td>12</td>
<td>2004</td>
<td>58.5</td>
<td>15.2</td>
</tr>
</tbody>
</table>

An increase in thulium concentration from 2wt% to 5.6wt% have reached an improvement in the laser efficiency by ~18.5%. Fibers HGS-01, HGS-02 and HGS-03 show slope efficiency of >70% as shown in Fig. 3. Our study shows that a thulium concentration of ~3.5wt% is enough to maintain a good TDFL efficiency in the two-micron wavelength band when diode pumped at ~790nm.
We also measured the efficiency of TDFLs at their free-running emission by varying the fiber length for different thulium concentrations. The aim is to establish the spectral region where the TDFL would perform efficiently depending on the thulium concentrations. This has been applied to two Tm-doped fibers fabricated using the hybrid gas phase-solution doping process with thulium concentrations of 3.5wt% (HSG-03) and 5.6wt% (HSG-02).

Figure 4 shows the normalized emission spectrum at different wavelengths with the corresponding fiber length and the laser efficiency. The laser efficiency maintained 70 ± 3% covering the wavelength region from 1980nm to 2080nm. We have presented the wavelength bands where the difference in free-running laser efficiency with respect to pump absorbed and pump launch is <3%. It is clear from Fig. 4 that the laser tends to perform better at longer wavelengths for thulium concentrations >5wt%, whereas a lower thulium concentration of ~3.5wt% is needed when considering efficient operation at shorter wavelengths.

3. Conclusion
We have successfully demonstrated a set of highly efficient thulium doped fibers by combining solution doping and gas phase techniques in conjunction with the MCVD process. The proposed fabrication route allows high Al₂O₃ incorporation into the silica matrix and a more uniform distribution of thulium ions across the fiber core region, leading to an efficient two-for-one cross-relaxation process in Tm-doped fibers with ~790nm pumping and therefore, shows high slope efficiencies of >70%, which is >90% of the theoretical limit in the two-micron band.
Our study shows that a thulium concentration of about 3.5 wt% with a more uniform dopant profile is sufficient to maintain high slope efficiencies in TDFLs without going for much higher thulium concentrations [16]. With a lower thulium concentration, the thermal load density in fiber will be reduced, and higher output power can be achieved in the two-micron band without thermal damage.

Furthermore, the fiber technology presented in this paper can be easily adaptable to the manufacturing environments. Moreover, the proposed technology makes it possible to fabricate a large-mode-area Tm-doped fiber with a pedestal design, which comprised of alumino-silicate layers deposited via the gas phase process, surrounding the core. Such pedestal layers, compared to germanium and/or phosphorous-doped silica layers, will significantly reduce the thermal mismatch with the silica cladding, offering much greater flexibility when there is a need for depositing a large pedestal [17] and fabrication of a polarization-maintained fiber in a pedestal geometry [18].

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**References**