

# New octagonal shape double-clad Thulium-Ytterbium Co-doped fiber for generation of multi-wavelength and Q-switched lasers in 2 micron region

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We demonstrate a simple, compact and low cost multi-wavelength and Q-switched fiber lasers based on newly developed octagonal shape double-clad Thulium-Ytterbium co-doped fiber (TYDF). The fiber is fabricated using the modified chemical vapor deposition (MCVD) process in conjunction with the solution doping technique. At 5 m long of TYDF, the laser produces three lines at 1914.5 nm, 1934.7 nm and 1953.6 nm with peak powers of 6.3 dBm, -1.6 dBm and 1.7 dBm, respectively due to the nonlinear polarization rotation effect in the laser ring cavity. The Q-switched TYDF laser operating at 1983.4 nm is also successfully demonstrated by exploiting a multi-walled carbon nanotubes (MWCNTs) polymer composite based saturable absorber. The composite is prepared by mixing the MWCNTs homogeneous solution into a dilute polyvinyl alcohol polymer solution before it is left to dry at room temperature to produce thin film. Then the film is sandwiched between two FC/PC fiber connectors and integrated into the laser cavity for Q-switching pulse generation. By varying the 905 nm multimode pump power from 1570 to 1606 mW, the pulse repetition rate increases from 27.4 to 37.8 kHz and the pulse width fluctuates within 4.9  $\mu$ s to 3.8  $\mu$ s. The maximum pulse energy of 10.6 nJ is obtained at pump power of 1570 mW.

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## 1. Introduction

Recently, the development of Thulium doped optical fiber lasers (TDFs) operating at near 2  $\mu$ m has become an interesting topic for many researchers [1-3]. This is attributed to the possibility of achieving laser of high efficiency, high output power, and retina safe in addition to specific applications plausible for this wavelength, such as for remote sensing and biomedical applications [4]. However, there are still many issues to be addressed such as low quantum efficiency of generated laser in high-phonon energy glass host matrix such as silica-based glass fibers. Therefore, Thulium-doped fibers (TDFs) normally employ low phonon energy glass hosts such as Fluoride glass, in which the up-conversion intensity is reported to be quite high in ultraviolet region [5]. Nevertheless, since the fluoride host is a rather soft type of glass, it is very hard to draw optical fiber from the preform due to its lower melting temperature. Recently, the interest has shifted back to silica based host TDFs as the phonon energy of silica glass can be reduced by incorporating silica network modifiers like Aluminum (Al) and

Germanium (Ge). Thus TDFs with modified silica host have emerged as a promising gain medium for achieving an efficient TDFL [6].

Of late, a Ytterbium-sensitized Thulium-doped fiber laser was demonstrated for oscillation around 2 micron based on Ytterbium (Yb) to Thulium (Tm) energy transfer [7]. Yb<sup>3+</sup> has the advantage of possessing only two multiplets: the ground-state level <sup>2</sup>F<sub>7/2</sub> and the excited-state level <sup>2</sup>F<sub>5/2</sub>, resulting in highly efficient absorption in the range of 900 nm - 1000 nm [8]. This opens a possibility of developing an economic but stable low power fiber laser in the wavelength range of 1800 to 2100 nm using a cheap 980 nm diode pumping. This laser has tremendous application for sensing toxic gases due to their specific IR absorption. Most of the previous work dealing with TDF or Thulium Ytterbium co-doped fiber (TYDF) was confined to particular host compositions or Tm<sup>3+</sup>:Yb<sup>3+</sup> ratio for investigation of fluorescence or lasing mostly in visible and S-band regions. No systematic investigation was carried out on the influence of fiber core composition on lasing from <sup>3</sup>F<sub>4</sub> level through energy transfer from Yb<sup>3+</sup> → Tm<sup>3+</sup>.

Over the last few years, Q-switched fiber lasers are being intensely studied due to their applications in many areas, but because of the limitation of saturable absorbers (SAs) at 2  $\mu\text{m}$  wavelength, little research has been carried out on Q-switching near a wavelength of 2  $\mu\text{m}$ . The use of single-walled carbon nanotubes (SWCNT) material as a saturable absorber (SA) has been widely investigated in Q-switched fiber lasers due to their inherent advantages, including good compatibility with optical fibers, low saturation intensity, fast recovery time, and wide operating bandwidth, while the other types of crystal and semiconductor based SAs cannot be used for an all fiber laser structure due to their relatively big volume [9-10]. Recently, a new member of carbon nanotubes family called multi-walled carbon nanotubes (MWCNTs) [11-12] have also captured much attention for nonlinear optics applications as an alternative to SWCNTs. They possess similar characteristics to the SWCNTs but have lower production cost, which is 50% - 80% cheaper than the SWCNT material [13]. Compared to SWCNTs, the MWCNTs have higher mechanical strength, photon absorption per nanotube and better thermal stability due to its higher mass density [14].

In this paper, the performance of the newly developed octagonal shape double-clad TYDF is investigated. The proposed laser operates in the wavelength region near 2000 nm based on energy transfer from  $\text{Yb}^{3+} \rightarrow \text{Tm}^{3+}$  using a multimode pump which operates at around 905 nm. A Q-switched TYDF laser (TYDFL) is also demonstrated using a new developed MWCNTs-based SA as the Q-switcher in the all-fiber ring laser. The SA is constructed by sandwiching a MWCNT- polyvinyl alcohol (MWCNT-PVA) film between two fiber connectors.

## 2. Working principle

Fig. 1 shows the energy diagram to explain the working principle of the TYFL. Since, the active fiber has ytterbium ions as sensitizer, it employs up-conversion process to generate laser at 2  $\mu\text{m}$  region. The pumping can be carried out using any laser diodes operating within wavelength region from 905 nm to 980 nm. The advantages are these laser diodes available in high power, easily obtained and relatively cheaper compared to other wavelength range. Furthermore, the pump propagates inside inner cladding of the fiber so that overlapping factor of the pump and ions increases. As the pump photons are absorbed by the ytterbium ions, it excites the ion from ground state  $^2F_{7/2}$  to  $^2F_{5/2}$ . As the ion relaxes to ground state, energy transfer process happens to neighboring thulium ion. When the thulium ions in ground state ( $^3H_6$ ) absorb the donated photons it got elevated to  $^3H_5$  level before it irradiatively relaxes to  $^3F_4$ . Thulium ions that populate this state level drops to ground state again generating the 2  $\mu\text{m}$  laser.

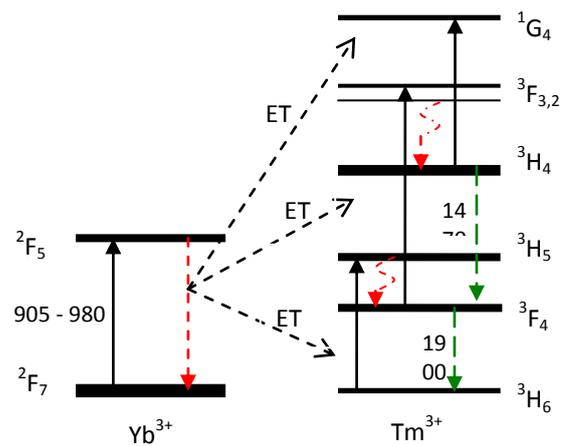


Fig. 1. Energy diagram levels for both Ytterbium and Thulium ions in TYDF showing an energy transfer.

## 3. Fabrication and characterization of TYDF

A double-clad low RI coated octagonal shaped TYDF was fabricated by the modified chemical vapor deposition (MCVD) process in conjunction with the solution doping technique. A pure silica glass tube of outer/inner diameter 20/17 mm was used for deposition of two porous unstinted  $\text{SiO}_2$  soot layers to make a preform while maintaining a suitable deposition temperature at around  $1550 \pm 10^\circ\text{C}$ . An alcoholic solution containing doping elements i.e. Tm, Yb, Y, Al in terms of their chlorides of Alfa standard, was used to soak the porous layer for about 30 min to achieve efficient doping. Then, dehydration and oxidation were performed at the temperature around  $900\text{-}1000^\circ\text{C}$ . Sintering of the un-sintered layers was also done by slowly increasing the temperature from  $1500$  to  $2000^\circ\text{C}$  using the conventional MCVD technique. Upon completion of sintering as well as oxidation, the tube was slowly collapsed to convert it into optical preform. The fabricated optical preform consists of  $\text{Al}_2\text{O}_3$ ,  $\text{Y}_2\text{O}_3$ ,  $\text{Tm}_2\text{O}_3$  and  $\text{Yb}_2\text{O}_3$  dopants with average weight percentage of 5.5, 3.30, 0.70 and 4.0, respectively measured from electron microprobe analyses (EPMA). The presence of  $\text{Y}_2\text{O}_3$  helps to decrease the phonon energy of alumino-silica glass, which assists in preventing the clustering of Yb and Tm ions into the core glass matrix and thus increases the probability of radiative emission.

The fabricated circular preform is converted to octagonal shaped through grinding followed by polishing method. Such octagonal shaped low RI coated fiber is then drawn at temperature of  $2050^\circ\text{C}$  with outer cladding diameter of 125  $\mu\text{m}$  from such geometrically modified preform. As opposed to the conventional single mode fiber where the pump light is coupled directly into the core, the pump light travels down the fiber in the first cladding and get absorbed by the dopants, in this case the Yb ions when it overlaps with the core. Such octagonal geometry of the cladding improves the pump absorption efficiency. The doping levels of  $\text{Tm}^{3+}$  and  $\text{Yb}^{3+}$  ions of the fabricated

TYDF are measured to be around  $4.85 \times 10^{19}$  ions/cc and  $27.3 \times 10^{19}$  ions/cc, respectively using an electron probe micro-analyser (EPMA). The  $\text{Tm}^{3+}$  and  $\text{Yb}^{3+}$  cladding absorptions of the fiber are measured to be 0.325 and 3.3 dB/m at 790 nm and 976 nm respectively. The cladding absorption loss curve of the octagonal shaped double-clad TYDF is measured and the spectrum is given in Fig. 2. The cladding absorption of such fiber at 790 nm and 976 nm are found to be 0.325 and 3.3 dB/m respectively. Inset of Fig. 2 shows the microscopic view of fiber cross-section. It is shown the double-clad fiber has a core and inner-cladding diameters of 5.96  $\mu\text{m}$  and 123.86  $\mu\text{m}$ , respectively. NA of the fabricated TYDF is measured to be 0.23.

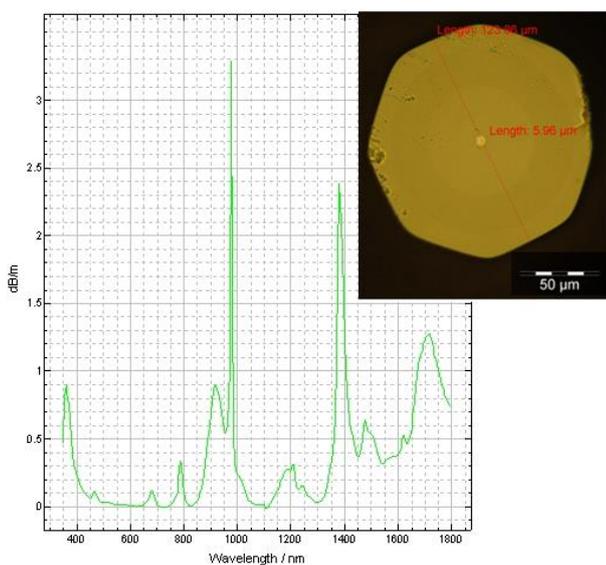


Fig. 2. Spectral attenuation curve of the newly developed octagonal shaped low RI coated fiber. Inset shows microscopic cross-sectional view of an octagonal shaped low RI coated fiber.

#### 4. Lasing characteristic of the TYDF laser

The lasing characteristic of TYDFL is investigated based on ring configuration and multimode pumping. The experimental setup for the ring TYDFL is shown in Fig. 3, which the resonator consists of a piece of TYDF as the gain medium, a multimode combiner (MMC) and 10 dB output coupler. A multimode 905 nm laser diode is used as the optical pump source which is coupled into the TYDF via a MMC. The output of the TYDFL is tapped out using the 10 dB coupler which allows 90% of the light to oscillate in the laser cavity. The output spectrum and power of the laser are measured by an optical spectrum analyzer (OSA) and power meter, respectively. The performance of the laser is investigated for three different TYDF lengths; 5m, 10m and 15m. This laser configuration contains no adjustable parts and can only be controlled externally by the amount of pump power.

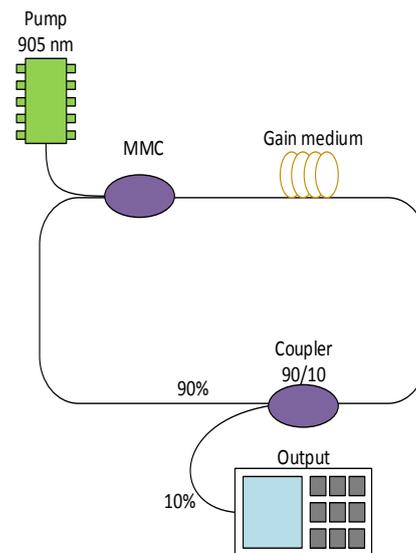


Fig. 3. Configuration of the proposed 2 micron fiber laser based on the fabricated TYDF and multimode pumping.

By cladding pumping of the double-clad TYDF, an amplified spontaneous emission (ASE) is generated in 1950 nm region, which oscillates in the ring cavity to generate laser. The laser starts to lase at different threshold pump powers depending on the fiber length and the output laser power increases almost linearly with the increment of the pump power. Fig. 4 shows the output power trend of the laser against the pump power at three different gain medium lengths. As shown in the figure, the threshold pump powers are obtained at 1000 mW, 2044 mW and 1000 mW with the use of 5m, 10m and 15m lengths of TYDF respectively, in the ring laser. The output power of all TYDF lasers (TYDFLs) linearly increases with the pump power with a linearity of more than 90%. The maximum output power of 21.9 mW is achieved at pump power of 3.5 W with the longest length of 15 m. It is observed that the use of 15m long TYDF length not only gives the maximum output power but also the minimum threshold among all the three tested lengths. The slope efficiency of 0.3 %, 0.9 % and 0.8 % is obtained with the use of 5m, 10m and 15m long TYDF respectively, as the gain medium. This shows that the use of 10 – 15 m long TYDF produces so much higher efficiency compared to that of the use of 5m length. This is attributed to the number of Thulium ions, which is higher with longer fiber and thus increases the population inversion in the fiber. This increases the stimulated emission and gain in the cavity, which in turn produces a higher output power at 2 micron region. Although the efficiency with 15m length is slightly less than the 10m length but the use of 15 m long TYDF generates a 2 micron laser at lower threshold pump power. This indicates that the optimum length of the ring TYDFL is within 10 to 15 m.

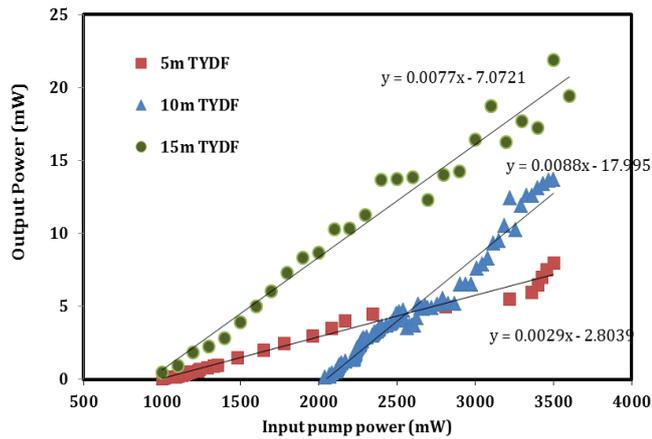


Fig. 4. The output power of the ring TYDFL against the pump power at three different TYDF lengths.

The output spectra of the ring TYDFL are also investigated for three different TYDF lengths. The result is shown in Fig. 5 when the multimode 905 nm pump power is fixed at 3.5 W. It is found that the output spectrum shifts to longer wavelength with the increase of TYDF length. This is attributed to the emitted photons, which are re-absorbed by the Thulium ion at the fiber length to emit ASE photons at a longer wavelength. The ASE oscillates in the cavity to generate laser. At 5 m long of TYDF, the laser produces three lines at 1914.5 nm, 1934.7 nm and 1953.6 nm with peak powers of 6.3 dBm, -1.6 dBm and 1.7 dBm, respectively due to the nonlinear polarization rotation (NPR) effect in the cavity. The laser operates at a single wavelength of 1961.4 nm with a peak power of 4.8 dBm as the TYDF length is increased to 10 m. Further increase of TYDF length to 15 m resulted in dual-wavelength output at 1562.7 nm and 1990.3 nm. These results indicate that the optimum TYDF length for the proposed ring cavity is about 10 m where the side modes are significantly suppressed to allow single wavelength operation. It is also observed that operating wavelength shifts to a longer wavelength with increasing pump power.

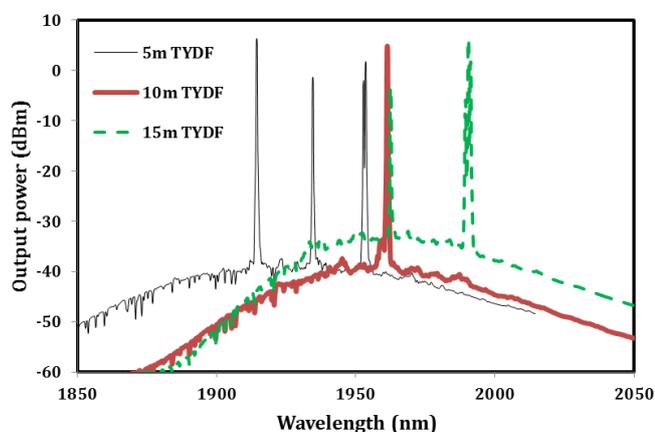


Fig. 5. The output spectra of the proposed TYDFL at three different TYDF lengths.

## 5. Q-switching performance of the TYDFL

In this work, the key part of Q-switching generation is the fabrication of saturable absorber incorporating dispersed MWCNTs. To match the TYDFL operating at 2  $\mu\text{m}$  region, the choosing of MWCNTs with suitable mean diameter and distributed diameter range is a critical step. In this work, we used SWCNTs with the purity of 99%, distributed diameter of 10-20 nm and length of 1-2  $\mu\text{m}$ . The host material was PVA, which is a water-soluble synthetic polymer with monomer formula  $\text{C}_2\text{H}_4\text{O}$ . It has excellent film forming, emulsifying, and adhesive properties. It also has high tensile strength, flexibility, high oxygen and aroma barrier, although these properties are dependent on humidity.

At first, the MWCNTs were dispersed in water with assistance of surfactant. The surfactant solution was prepared by dissolving 4 g of sodium dodecyl sulphate (SDS) in 400 ml deionized water. Then, 250 mg MWCNT was added to the solution and the homogenous dispersion of MWCNTs was achieved after the mixed solution was sonicated for 60 minutes at 50 W. The solution was then centrifuged at 1000 rpm to remove large particles of undispersed MWCNTs to obtain a stable dispersed suspension. MWCNTs-PVA composite was prepared by adding the dispersed MWCNTs suspension into a PVA solution by one to four ratio. PVA solution was prepared by dissolving 1 g of PVA ( $M_w = 89 \times 10^3 \text{ g/mol}$ ) in 120 ml of deionized water. The homogeneous MWCNTs-PVA composite was obtained by sonification process for more than one hour. The CNT-PVA composite was casted onto a glass petri dish and left to dry at room temperature for about one week to produce thin film with thickness around 10  $\mu\text{m}$ . The SA is fabricated by cutting a small part of the prepared film ( $2 \times 2 \text{ mm}^2$ ) and sandwiching it between two FC/PC fiber connectors, after depositing index-matching gel onto the fiber ends. The insertion loss of the SA is measured to be around 3 dB at 1550 nm.

Raman spectroscopy was performed on the MWCNT-PVA film using laser excitation at 352 nm to confirm the presence of MWCNT. Fig. 6 shows the Raman spectrum, where obviously indicates two well-defined peaks at  $1585 \text{ cm}^{-1}$  and  $1433 \text{ cm}^{-1}$ , which corresponds to the G and D band, respectively. The D band was reported to be upshifted to  $1275 \text{ cm}^{-1}$  in MWCNTs embedded in poly-methyl methacrylate (PMMA) composite, while in this work we observed a downshift of D-band ( $1433 \text{ cm}^{-1}$ ) for MWCNTs-PVA film. In addition, other distinguishable features at  $2900 \text{ cm}^{-1}$  and a small peak at  $848 \text{ cm}^{-1}$  were also observed as depicted in Fig. 6. However, G' peak that usually occurs at  $2700 \text{ cm}^{-1}$  is absence. One important feature, a low energy radial breathing mode (RBB) at Raman shift of around  $100 \text{ cm}^{-1}$  to  $400 \text{ cm}^{-1}$ , which is usually observed in SWCNTs film is not seen in our spectrum. This is attributed to the MWCNTs has many layers of graphene wrapped around the core tube, which restricts the breathing mode.

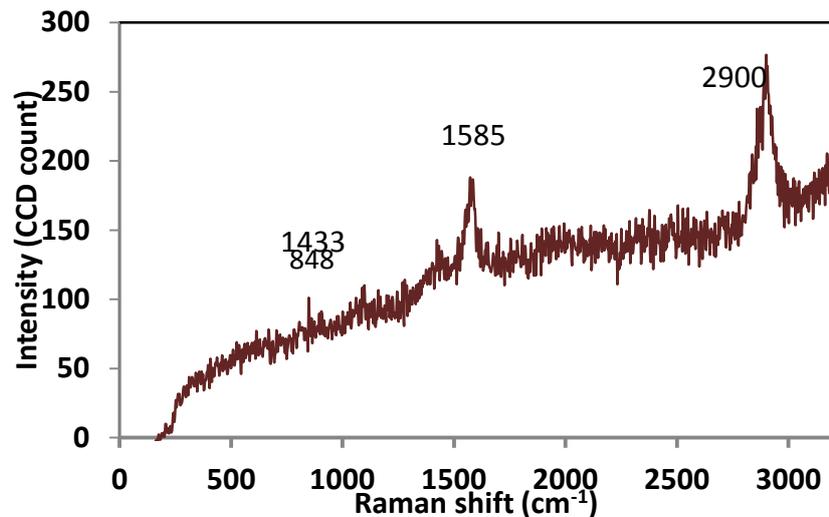


Fig. 6. Raman spectrum from the fabricated MWCNTs-PVA film.

The schematic of the proposed Q-switched TYDFL is shown in Fig. 7. It was constructed using a simple ring cavity, in which a 15 m long the fabricated TYDF was used for the active medium and the homemade SA was used as a Q-switcher. The double-clad TYDF was forward pumped by a 905 nm multimode laser diode via a multimode combiner (MMC). The laser output was obtained via a 10 dB output coupler located between the SA and MMC, which channeled out about 10% of the oscillating light from the ring cavity. The cavity length is measured to be approximately 20 m. The optical spectrum analyzer (OSA, Yokogawa, AQ6370B) is used for the spectral analysis of the Q-switched TYFL with a spectral resolution of 0.05 nm whereas the oscilloscope (OSC, Tektronix, TDS 3052C) is used to observe the output pulse train of the Q-switched operation via a photo-detector (EOT, ET-5010-F). All components used in our setup were polarization independent, i.e. they support any light polarization. No polarization controller (PC) was included in the laser cavity as we had observed earlier that a PC did not improve our pulse stability. There was no significant pulse jitter observed through the oscilloscope during the experiment. The lasing threshold was approximately 1570 mW.

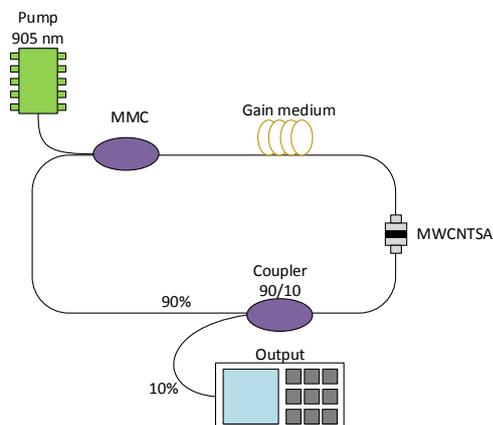


Fig. 7. Configuration of the proposed Q-switched TYDFL

Fig. 8 shows the optical spectrum of the Q-switched TYDFL at the pump power threshold of 1570 mW respectively. The corresponding output power at its threshold is obtained at 0.29 mW. The 3-dB bandwidth of Q-switched fiber laser spectrum is measured to be  $\sim 0.4$  nm at the center wavelength of 1983.4 nm and signal to noise ratio of about 32 dB, as shown in Fig. 8. There is no chirp appeared on the optical spectrum within the peak wavelength range, indicating that the cavity perturbation is suppressed. Without the SA, the TYDFL produces dual-wavelength output at 1562.7 nm and 1990.3, as shown in Fig. 5, due to NPR effect and cavity perturbations. By incorporation of the SA in the cavity, the Q-switching laser dominates the lasing mode, and thus suppresses the side mode.

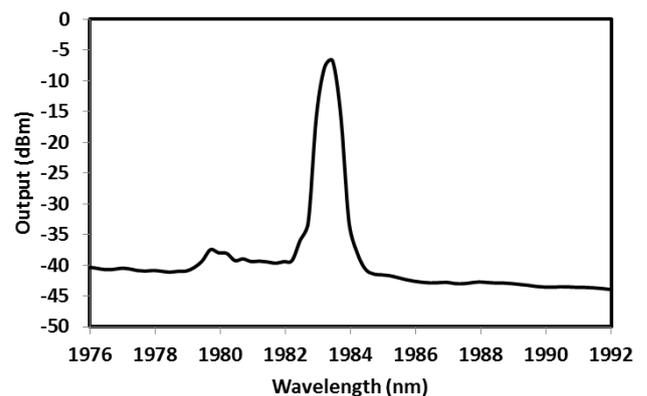


Fig. 8. Output spectrum of the Q-switched laser

Fig. 9 shows the typical oscilloscope trace of the Q-switched pulse train at pump power of 1570 mW. It is observed that the pulses were sequenced in a train uniformly at the pulse separation of 36.6  $\mu$ s, which corresponds to repetition rate of 27.4 kHz. The pulse width of the pulse train is measured to be around 4.9  $\mu$ s. It comes to our attention that the repetition rate and pulse width of the Q-switched pulses is sensitive to the pump power. It is

observed that the Q-switching pulse generation starts at the threshold pump power of 1570 mW and disappears as the pump power is increased above 1620 mW. The dependence of the repetition rate and pulse width on the input pump power is shown in Fig. 10. As shown in the figure, the repetition rate increases almost linearly with the pump power. The result coincides well with the inherent characteristic of the Q switched fiber laser as reported before [10]. The pulse repetition rate can be varied from 27.4 to 37.8 kHz as the pump power increases from 1570 and 1620 mW. On the other hand, the pulse width decreases from 4.9 to 3.8  $\mu$ s as the pump power increases from 1570 to 1606 mW. However, with further increase in pump power, an anomalous behavior is observed where the pulse width increases to 5.1  $\mu$ s at 1620 mW as shown in Fig. 3. The pulse energy at 1570 mW is measured to be around 10.6 nJ.

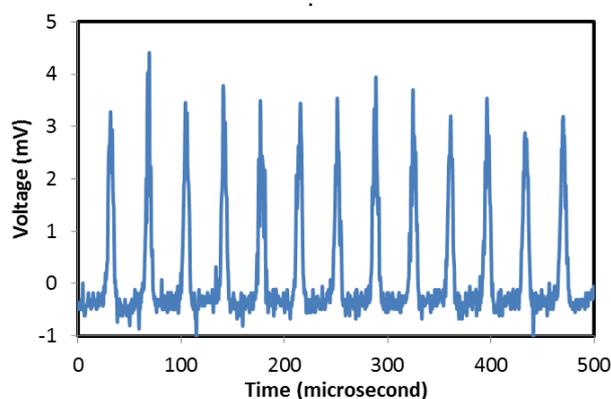


Fig. 9. Typical pulse train of the Q-switched TYDFL at pump power of 1570 nm.

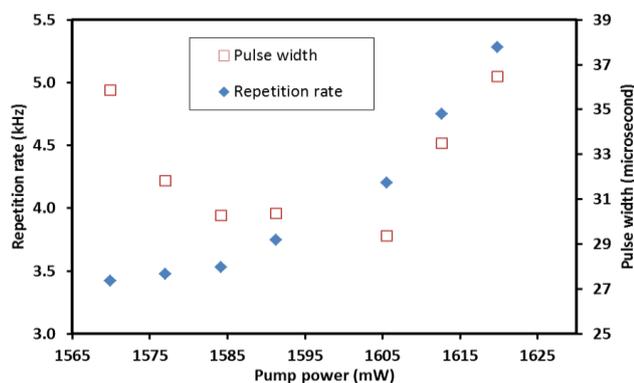


Fig. 10. Repetition rate and pulse width as functions of pump power.

## 6. Conclusion

We have demonstrated multi-wavelength and Q-switched fiber lasers based on newly developed octagonal shape double-clad TYDF operating at 2 micron region. The MCVD process in conjunction with the solution doping technique was used to fabricate the fiber. The multi-wavelength laser operating at 1914.5 nm, 1934.7 nm

and 1953.6 nm has been realized using a ring configuration due to the NPR effect in the cavity. By incorporating the homemade MWCNTs SA in the ring cavity, a Q-switching pulse train operating at 1983.4 nm was successfully demonstrated. By varying the 905 nm multimode pump power from 1570 to 1606 mW, the pulse repetition rate increases from 27.4 to 37.8 kHz and the pulse width fluctuates within 4.9  $\mu$ s to 3.8  $\mu$ s. The maximum pulse energy of 10.6 nJ is obtained at pump power of 1570 mW. A higher performance Q switching is expected to be achieved in both fiber lasers with the optimization of the SA and laser cavity. Besides showing good Q-switching performance, the saturable absorber is easy to fabricate and cheap.

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