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Polarization insensitive in-fiber mode-locker based on carbon nanotube with N-methyl-2-pyrrolidone solvent filled fiber microchamber

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We report an in-fiber laser mode locker based on carbon nanotube with n-methyl-2-pyrrolidone solvent filled in-fiber microchamber. Symmetrically femtosecond laser fabricated in-fiber microchamber with randomly oriented nanotubes assures polarization insensitive oscillation of mode locking. The proposed and demonstrated passively mode locked fiber laser shows higher energy soliton output. The laser has an output power of ~29 mW (corresponding to 11 nJ energy). It shows stable soliton output with a repetition rate of ~2.3 MHz and pulse width of ~3.37 ps. © 2012 American Institute of Physics.

Mode locked ultrafast erbium doped fiber lasers (EDFL) have shown many advantages over conventional solid state light sources in a wide range of applications such as optical communication, metrology, sensing, medical application, and other fields of science and technology. A range of approaches have been used to achieve mode locking in EDFLs. Currently, most popular techniques include nonlinear polarization rotation (NPR) facilitated effective saturable absorber and semiconductor saturable absorber mirrors (SESAMS), although other methods of mode-locking are actively studied. In practical terms, different types of saturable absorbers can be optimal for different applications depending on specific practical requirements.

Recently, carbon nanotube (CNT) based saturable absorbers have attracted a great deal of attention due to their inherent nonlinear optical properties, such as fast recovery time (<1 ps), low cost, and wide band absorption. The properties of CNT saturable absorber can be significantly tailored by the CNT bundle engineering. So far, various mode locked laser implemented by CNT have been demonstrated including fiber lasers, waveguide lasers, and semiconductor lasers.

Most of the methods using CNT as a saturable absorber involve in directly depositing CNT or fabricating CNT polymer composites. However, CNT in liquid solution as a mode locker has been less studied. So far, only dimethylformamide (DMF) solution and poly-methyl-methacrylate (PMMA) have been investigated for fiber laser mode locking. Il’chev et al. demonstrated CNT in heavy water for erbium glass laser mode locking. Although Martinez et al. tend to achieve a robust design with an in-fiber microfluidic device, CNT in DMF solvent may show undesired properties resulting in agglomeration of CNTs which may lead to unstable mode locking. In Ref. 16, specialty fiber, i.e., hollow core fiber has been demonstrated for accommodating CNT to achieve in-fiber mode locking regime. Nevertheless, the introduction of specialty fiber may create issues such as low robustness and high cost packaging.

In this letter, we propose and demonstrate a fiber laser mode locked by CNT dissolved in n-methyl-2-pyrrolidone (NMP) solvent filled in-fiber microchamber. We use NMP because it is a high boiling point organic solvent, which allows a stable dispersion of CNTs at small concentrations even with no surfactants and polymers. CNT can also be assembled in bundles of different sizes by CNT salting-out technique in NMP. That will play a key role in achievement of saturable absorber with predefined ultrafast recovery time, low saturation intensities, and non-saturable losses.

This type of low loss saturable absorber shows stable generation of soliton pulse while maintaining the capability of heat dissipation hence lead to stable and high energy pulse generation. One important property of this type of mode locker is polarization insensitive. Previously, tapered fiber based polarization insensitive mode locker has been reported for polarization insensitive mode locking. However, the in-fiber micro-chamber based saturable absorber has more advantages which could preserve the mechanical strength, geometry, and polarization insensitivity.

Efficient absorption of CNTs at specific wavelength is determined by the band gap of the specific chiralities of semiconducting single wall CNTs. Additionally, the presence of metallic tubes in the single wall carbon nanotubes (SWNTs) bundles is important for realization of the ultrafast absorption recovery (τ < 1 ps), because in bundles containing both metallic and semiconducting SWNTs, the non-radiative absorption recovery of CNTs is significantly faster than that of individual CNTs. The fast non-radiative absorption recovery of CNTs is important for achieving stable operation of passively mode locked fiber lasers. Furthermore, CNTs can be used as a saturable absorber and SESAM simultaneously.

![Absorbance vs Wavelength](image)

**FIG. 1.** Absorption spectrum of the CNT-NMP saturable absorber measured by a wide band spectrometer.
relaxation achieves by charge tunneling from semiconducting on metallic tubes. For the CNT saturable absorber preparation, we used the purified HiPco SWNT purchased from Unidym (Lot # P0261). According to the datasheet provided by Unidym, the purified HiPco samples contain at least 85% of SWNT with less than 15% of impurities such as catalyst particles, amorphous C, and multi wall CNT. The ratio of semiconducting to metallic SWNTs is about 2:1. The SWNTs were ultra-sonicated by Nanoruptor Processor (Diagenode SA) during 1 h at 170 W in NMP with the presence of Triton X-100 non-ionic surfactant. To remove residual bundles, the dispersion was placed in to MLS 50 rotor and centrifuged at 30 kRPM during 2 h with Beckman Optima Max-XP ultracentrifuge. The resulting solution shows excellent homogeneity with no visible SWNTs aggregates. The absorption spectrum of CNT solution subtracted on absorption of pure NMP is shown in Figure 1. It shows the typical multi peak structure between 1000 and 1600 nm, which corresponds to the absorption of semiconducting single wall CNTs with diameter distribution between 0.8 and 1.3 nm. No obvious agglomeration of CNTs has been observed even when the solution has been kept in the lab for months.

The fabrication of the in-fiber microchamber was carried out through femtosecond laser micromachining followed by selective chemical etching of the modified area. A commercial femtosecond laser (SpectraPhysics) emitting at 800 nm with 1 kHz repetition rate, 150 fs pulse duration, and 150 nJ pulse energy was employed for the micromachining. The laser beam was tightly focused onto the fiber via a 100 x objective lens. The fiber with the laser processed region was then chemically etched in 5% HF acid to facilitate forming of the microchamber. Figure 2 shows the microscopic image of the femtosecond machined microchamber in a standard telecom fiber (SMF28). The image shows a size of ~28 µm by ~17 µm rectangular chamber across the whole fiber area. We use a commercial tool kit (LUNA technologies) to examine the polarization sensitivity and transmission loss of the device at the desired wavelength region. Figure 3 shows the measured insertion loss (IL) and polarization dependent loss (PDL) of the microchamber with and without the CNT NMP solvent. The microchamber exposed in the air shows ~3 dB insertion loss. The oscillation appeared across the spectrum range is the Fabry-Perot effect produced by the two edges of the microchamber. When the CNT NMP solvent is filled in the microchamber, we can clearly see that the insertion loss has decreased to ~0.5 dB with alleviation of Fabry-Perot effect. This is due to the refractive index matching of the induction of the solvent. It further indicates a low loss saturable absorber. In Figure 3, it also presents the PDL of the

![Figure 2](image2.png)

FIG. 2. (Color online) Microscopic pictures of the femtosecond machined in-fiber microchamber in a standard SMF 28 fiber at two orthogonal directions (a) top view and (b) side view of the microchamber.

![Figure 3](image3.png)

FIG. 3. (Color online) Measured IL and PDL of the in-fiber microchamber when it exposed to the air and filled with CNT NMP solvent.

![Figure 4](image4.png)

FIG. 4. (Color online) Schematic illustration of the proposed CNT mode locked EDFL.
device which is less than $\sim 0.5$ dB across the whole wavelength range from 1520 nm to 1600 nm. It proves that the saturable absorber is polarization insensitive.

Figure 4 illustrates the schematic configuration of the CNT mode locked EDFL. The EDFL constitutes $\sim 1$ m of highly concentrated Erbium-doped fiber (EDF Er80-8/125 from Liekki) as the gain medium. Two fiber pigtailed isolators (OIS) are employed before and after the gain fiber to ensure single direction oscillation of the laser with elimination of other unwanted feedback in the laser cavity. The laser is pumped through a grating stabilized 980 nm laser diode (LD) using a 980 nm/1550 nm wavelength division multiplexing (WDM). A set of commercial diode laser driver and controller (Thorlabs) is employed to stabilize the operation of the pump. A standard fused fiber coupler where 50% of light is coupled out the laser cavity. An in-line polarization controller was incorporated in the laser cavity for mode locking optimization purpose. The involvement of polarization controller does not affect the laser to be self-started. The fiber microchamber is then spliced into the laser cavity which forms an all fiber laser cavity. The total length of the laser cavity is $\sim 88$ m. The whole cavity has an average anomalous dispersion of about $\sim 17.48$ ps/nm/km which will result in soliton output.

Figure 5 shows a typical output optical spectrum of the EDFL centered at $\sim 1564$ nm with a spectral bandwidth at full width half maximum (FWHM) of $\sim 0.24$ nm. The Kelly side bands indicate fundamental soliton shape of the output pulses. The output pulses have then been directly fed through a commercial autocorrelator without any pre-amplification. The measured autocorrelation trace corresponding to the pulse duration of $\sim 3.37$ ps is shown in Figure 6. A typical pulse train is shown in Figure 7 with $\sim 428$ ns interval between the two adjacent pulses thus giving a repetition rate of $\sim 2.3$ MHz. The EDFL is pumped at $\sim 300$ mW which allows $\sim 29$ mW output power corresponding to energy of $\sim 12.4$ nJ. Note that this is much higher level than the fiber laser mode locked by a solid format CNT.$^{23}$ The results further confirms that CNT solvent is feasible for high energy mode locked fiber laser.$^{15}$ We also found that CNTs dispersed in NMP solvent are not apt to agglomerate where samples are still able to mode lock fiber lasers after few months. We have switched on the mode locker fiber laser for more than 24 hours at the laboratory condition, with no obvious fluctuations observed for both the optical spectrum and pulse duration. However, one major issue for such kind of saturable absorber is the evaporation of NMP and moisture adsorption of the solvent. Therefore, in case the saturable absorber has to be exposed in air for long time, i.e., weeks, months, the evaporation and moisture adsorption would be able to affect the thermodynamic equilibrium in the solvent results in CNT aggregation. Hence, this may induce significant scattering loss in the laser cavity which would be detrimental to the laser mode locking. We believe by proper packaging, a highly stable high energy mode locked fiber laser by CNT in NMP solvent will be applied in many applications in the future.

In conclusion, we have proposed and demonstrated a CNT in NMP solvent filled in-fiber microchamber mode locked EDFL. The polarization insensitive saturable absorber without any optimization and control presents high stability over long time. This indicates significant potential of applying liquid type saturable absorber for laser mode locking. The application of CNT in NMP solvent in an in-fiber microchamber also maintains the all-fiber format of the laser configuration. The laser with relatively simple design...
generates soliton pulses with ~3.37 ps temporal width and an output power of ~29 mW (pulse energy of ~11 nJ) with a repetition rate of ~2.3 MHz at 1564 nm.