Skolkovo Institute of Science and Technology

as a manuscript

Aram A. Mkrtchyan

ALL-FIBER MODE LOCKED LASERS WITH AEROSOL SYNTHESIZED SINGLE-WALLED CARBON NANOTUBES

PhD Dissertation Summary

for the purpose of obtaining academic degree
Doctor of Philosophy in Physics

Moscow - 2022

This PhD dissertation was prepared at Skolkovo Institute of Science and Technology

Academic Supervisors:

Albert G. Nasibulin, Doctor of Science, Professor, Head of Laboratory of Nanomaterials at Skoltech

Yuriy G. Gladush, PhD in Physics

DISSERTATION TOPIC

Materials with nonlinear optical properties play a crucial role in photonic applications. Carbon nanotubes have proven to be an excellent nonlinear nanomaterial with large nonlinear response, controllable resonance spectral position and fast recovery time. In addition to high nonlinearity thin films of single-walled carbon nanotubes (SWCNTs) obtained by aerosol (floating catalyst) chemical vapor deposition method can be synthesized with a given thickness and position of resonance transitions without additional purification requirements and can be transferred to any surfaces by a simple dry transfer technique. All these features including the ability to control the SWCNT nonlinear absorption can make them extremely promising for high-tech applications.

Within present work we implemented aerosol synthesized SWCNTs as a saturable absorber (SWCNT-SA) on a side-polished fiber using dry transfer technique to generate various ultrashort pulse regimes in a fiber laser. In the second part we developed unique technique to control the nonlinear absorption of SWCNTs using electrochemical gating, which allows to manipulate the pulse states of the fiber laser and to reversibly switch between mode-lock and Q-switch regimes.

Despite many remarkable results demonstrated with SWCNT saturable absorbers in fiber lasers its degradation inside the laser cavity limits their widespread use. For this purpose we investigated thermal stability of the carbon nanotubes saturable absorber synthesized with different methods and utilized on fiber ferrule and side-polished fiber. The heating temperature and degradation mechanisms we revealed by measuring SWCNTs Raman spectrum G-peak position shift in an operating laser cavity.

It is worth noting that pulsed fiber lasers are actively developing in the submicron region. Traditionally Titanium:Sapphire laser sources are used at this region for high power ultrashort pulse generation with great output beam quality as well as for broad wavelength tuning. However, due to a number of significant drawbacks such as bulkiness, sensitivity to mechanical perturbations and extremely high cost their practical applications are significantly limited outside laboratories. Nevertheless, they remain the “golden standard” because of the absence of fiber based alternatives. Within the framework of the dissertation we developed first ultrafast neodymium doped polarization maintaining all-fiber lasers at 900 nm wavelength band using

artificial or semiconducting saturable absorbers.

Aims and objectives

1. To investigate polymer-free single-walled carbon nanotubes as a saturable absorber on a side-polished fiber deposited by the dry-transfer technique to generate various pulse regimes in a fiber laser.

2. To develop a technique for tuning the nonlinear optical absorption of SWCNTs by electrochemical gating and investigate the influence of tunable nonlinearity of SWCNT-SA on a pulse generation regime inside the polarization maintaining all-fiber laser.

3. To investigate the mechanism of the SWCNT-SA degradation by measuring the temperature of the SWCNT saturable absorber clamped between two connectors and on the side polish fiber.

4. To develop ultrafast neodymium-doped stable and robust all-fiber laser operating at 900 nm wavelength band.

5. To fabricate short pulse all-fiber laser with dispersion compensation at 900 nm wavelength band and investigate pulse regimes for different net dispersions.

KEY RESULTS

Key aspects to be defended:

1. We demonstrate an easy and robust method to implement aerosol CVD synthesized single-walled carbon nanotubes as a saturable absorber on a side polished fiber by the dry transfer technique. Q-switch, mode-lock as well as the 79^{th} order harmonic mode-locking regimes are obtained in a fiber laser with this approach [1].

2. We find out that the nonlinear optical absorption of SWCNT films can be significantly tuned by means of electrochemical gating. We show that under an applied voltage below 2 V the photobleaching of the material can be gradually reduced and even turned to

photoinduced absorption. Furthermore, we integrate a carbon nanotube electrochemical cell on a side-polished fiber to tune the modulation depth and implement it into a fully polarization-maintaining fiber laser. We demonstrate that the pulse generation regime can be reversibly switched between femtosecond mode-locking and microsecond Q-switching by changing the gate voltage [2].

3. We identify the process behind the degradation of the polyvinyl alcohol (PVA) composite and polymer free carbon nanotube saturable absorbers by measuring the heating temperature of the sample in accordance with the G-band position of Raman spectrum. We demonstrate that the samples on fiber ferrule undergo different degradation mechanisms, while the parameters of the pulse generation depend weakly on the sample preparation technique. We show that with an incident power of 20 mW the temperature can exceed 100ºC in a 30% absorbing sample. Under illumination PVA polymer composites demonstrate the gradual deterioration of the optical properties governed by thermal decomposition of the polymer matrix. In contrast, polymer-free SWCNT-SA shows no change in optical properties under illumination below the threshold defined by the 1 kW peak power, which corresponds to 25 mW average power. In case of SWCNTs on side-polished fiber the average power for the sample degradation exceeds 3 W. The developed techniques allow to optimize the parameters of the saturable absorber and maximize the stability for a required pulse regime [1,3].

4. We demonstrate polarization-maintaining neodymium doped all-fiber laser at a 905 nm wavelength with rectangular shape dissipative soliton resonance mode-lock pulses having 1 nJ energy, 30 pm spectral and 80 ÷ 430 ps temporal widths. The laser works in nonlinear amplifying loop mirror (NALM) cavity configuration with large net-normal dispersion. To suppress dominant parasitic emission of Nd atoms at 1064 nm band we utilize an active fiber – 920/1064 wavelength division multiplexer – active fiber sandwich-like sequence in the NALM loop. Excellent agreement with numerical simulation is achieved with proper selection of the net-dispersion and nonlinearity parameter of the cavity. The simulation is used to recover pulse width for the pulses out of autocorrelation window [4].

5. We describe dispersion managed ultrashort pulse generation at a 920 nm wavelength in a Nd-doped polarization maintaining all-fiber laser. Linear laser scheme is developed with

chirped fiber Bragg grating as a semi-transparent output coupling mirror and SESAM as a second fully reflecting mirror. Chirped fiber Bragg grating also serves as a fiber dispersion compensator and select the laser emission wavelength. Self-starting pulse generation regimes observed with 10 ÷ 317 pJ energy, 50 ÷ 2 ps width, and 17 ÷ 51 MHz repetition rate at the 0.24 ps^2 ÷ -0.03 ps^2 net dispersion range. Besides, harmonic mode-locking up to the 12^{th} order with 0.43 GHz repetition rate is achieved by selecting 0.01 ps^2 net cavity dispersion and by adjustment of the pump power. [5].

The work was supported by RFBR (project № 20-32-90233) and Council on grants of the President of the Russian Federation (grant number НШ-1330.2022.1.3).

Author's personal contribution to the aspects to be defended

All the experimental results are obtained by author or with his direct participation. He developed most of the fiber laser setups, including the fabrication of the electrochemical cell with carbon nanotubes in inert atmosphere. He conducted all the measurements of the laser generation regimes including SWCNTs nonlinear absorption tuning measurement under electrochemical gating. Author also carried out numerical simulations used to optimize the laser resonator design and to investigate intracavity pulse evolution for 900 nm wavelength band lasers. This work was performed by the author in the Laboratory of Nanomaterials, Skoltech in the period from 2018 to 2022.

PUBLICATIONS AND APPROBATION OF RESEARCH

First-tier publications

1. **A. A. Mkrtchyan,** Y. G. Gladush, D. Galiakhmetova, V. Yakovlev, V. T. Ahtyamov, A. G. Nasibulin. Dry-transfer technique for polymer-free single-walled carbon nanotube saturable absorber on a side polished fiber // Optical Materials Express, V. 9, Issue 4, P. 1551-1561, 2019.

2. Y. Gladush, **A. A. Mkrtchyan**, D. S. Kopylova, A. Ivanenko, B. Nyushkov, S. Kobtsev, A. Kokhanovskiy, A. Khegai, M. Melkumov, M. Burdanova, M. Staniforth, J. Lloyd-

Hughes, and A. G. Nasibulin. Ionic Liquid Gated Carbon Nanotube Saturable Absorber for Switchable Pulse Generation // Nano Letters, V.19, Issue 9, P. 5836 – 5843, 2019.

3. D. Galiakhmetova, Y. Gladush, **A. Mkrtchyan**, F. S. Fedorov, E. M. Khabushev, D. V. Krasnikov, R. Chinnambedu-Murugesan, E. Manuylovich, V. Dvoyrin, A. Rozhin, M. Rümmeli, S. Alyatkin, P. Lagoudakis, and A. G. Nasibulin. Direct measurement of carbon nanotube temperature between fiber ferrules as a universal tool for saturable absorber stability investigation // Carbon, V. 184, P. 941 – 948, 2021.

4. **A. A. Mkrtchyan**, Y. G. Gladush, M. A. Melkumov, A. M. Khegai, K. A. Sitnik, P. G. Lagoudakis, and A. G. Nasibulin. Nd-doped polarization maintaining all-fiber laser with dissipative soliton resonance mode-locking at 905 nm // Journal of Lightwave Technology, V. 39, Issue 17, P. 5582 – 5588, 2021.

5. **A. A. Mkrtchyan**, M.S. Mischevsky, Y. G. Gladush, M. A. Melkumov, A. M. Khegai, A. G. Nasibulin. Dispersion managed mode-locking in all-fiber polarization maintaining Nd-doped laser at 920 nm // Optics letters (in print).

Other publications

6. D. M. Zhigunov, D. A. Shilkin, N. G. Kokareva, V. O. Bessonov, S. A. Dyakov, D. A. Chermoshentsev, **A. A. Mkrtchyan**, Y. G. Gladush, A. A. Fedyanin, and A. G. Nasibulin. Single-walled carbon nanotube membranes as non-reflective substrates for nanophotonic applications // Nanotechnology V. 32, Issue 9, 2021, Number 095206.

7. M. G. Burdanova, G. M. Katyba, R. Kashtiban, G. A. Komandin, E. Butler-Caddle, M. Staniforth, **A. A. Mkrtchyan**, D. V. Krasnikov, Y. G. Gladush, J. Sloan, A. G. Nasibulin, and J. Lloyd-Hughes. Ultrafast, high modulation depth terahertz modulators based on carbon nanotube thin films // Carbon V. 173, P. 245–252, 2021.

Reports at conferences

1. **Poster presentation**, V International Conference on Quantum Technologies, (ICQT 2019),“Multiregime pulse fiber laser based on electrochemically gated carbon nanotube saturable absorber” Moscow, Russia, July 15-19, 2019;

2. **Invited talk**, All-Russian Conference on Fiber Optics (RFO-2019), "Multiregime pulse fiber laser based on electrochemically gated single walled carbon nanotube saturable absorber", Perm, Russia, October 8-11, 2019;
3. **Oral talk**, 14th International Conference on Advanced Carbon NanoStructures (ACNS'2019), "Multiregime pulse fiber laser based on electrochemically gated single walled carbon nanotube saturable absorber", Saint-Petersburg, Russia, July 1-5, 2019;
4. **Oral talk,** 19th International conference Laser Optics (ICLO 2020), "Multiregime pulse fiber laser based on electrochemically gated single walled carbon nanotube saturable absorber", online, November 2 - 6, 2020**;**
5. **Poster presentation,** VI International Conference on Quantum Technologies (ICQT-2021), "Neodymium-doped polarization maintaining all-fiber laser with dissipative soliton resonance mode-locking at 905 nm", online, July 12-16, 2021;
6. **Oral talk,** Russian Fiber Optics conference (RFO-2021), "Neodymium-doped polarization maintaining all-fiber laser with dissipative soliton resonance mode-locking at 905 nm", Perm, Russia, October 2021;
7. **Poster presentation**, V Summer School on Nonlinear photonics (SSNLP-2021), "Neodymium-doped polarization maintaining all-fiber laser with dissipative soliton resonance mode-locking at 905 nm", NSU, Novosibirsk, Russia, August 9 – 14, 2021;
8. **Poster presentation,** Conference on Lasers & Electro-Optics (CLEO/EUROPE-EQEC 2021), "Neodymium-doped polarization maintaining all-fiber laser with dissipative soliton resonance mode-locking at 905 nm", online, June 21 – 25, 2021;
9. **Oral talk,** 20th International conference Laser Optics (ICLO 2022), "900 nm wavelength band all-fiber polarization maintaining mode-locked laser", Saint-Petersburg, Russia, June 20-24, 2022.

CONTENTS

This dissertation contains an introduction, three main chapters and a conclusion. The total length of dissertation is 130 pages with 44 figures and two tables. The bibliography contains 190 items.

- Chapter 2 is dedicated to the fabrication of saturable absorber based on aerosol synthesized single-walled carbon nanotubes deposited on a side-polished fiber by dry

transfer technique. Using this method Q-switched, mode-locked pulses as well as the harmonic mode-lock regime with the 79th order are successfully demonstrated in all-fiber laser. In the second part of Chapter 2 we developed original electrochemical cell to control the modulation depth of the SWCNT-SA using Fermi level gating, which allowed to generate and reversible switch laser pulse state between mode-lock and Q-switch regimes by gate voltage below 2 V.

- In Chapter 3 we investigated degradation mechanisms of the polyvinyl alcohol (PVA)/SWCNT composite and aerosol synthesized SWCNT-SAs sandwiched between fiber ferrules and deposited on a side-polished fiber. The heating temperature and degradation mechanisms we revealed by measuring SWCNTs Raman spectrum G-peak position shift in an operating laser cavity.
- In Chapter 4 we developed first ultrafast neodymium doped polarization maintaining all-fiber lasers in 900 nm wavelength range using artificial or semiconducting saturable absorbers.