177 fs erbium-doped fiber laser mode locked with a cellulose polymer film containing single-wall carbon nanotubes

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A mode-locked soliton erbium-doped fiber laser generating 177 fs pulses is demonstrated. The laser pumped by a 85 mW, 980 nm laser diode emits 7 mW at 1.56 μm at a pulse repetition rate of 50 MHz. Passive mode locking is achieved with a saturable absorber made of a high-optical quality film based on cellulose derivative with dispersed carbon single-wall nanotubes. The film is prepared with the original technique by using carbon nanotubes synthesized by the arc-discharge method. © *2008 American Institute of Physics*. [DOI: 10.1063/1.2918450]

Ultrafast fiber lasers attract great attention due to their simple design, high stability, low alignment sensitivity, and low cost. It was shown[1–3] that passive mode locking in erbium-doped glass solid state lasers can be achieved by using carbon single-wall nanotubes (CSWNTs) as a saturable absorber. Different types of media containing CSWNTs have been developed since the first experiment: CSWNTs films evaporated onto quartz slabs or SEmiconductor Saturable Absorber Mirror (SESAM),[1,4] aqueous suspensions of CSWNTs,[2,5,6] CSWNTs embedded in different polymers (PvA, polyimide, carboxymetilcellulose).[7,8]

The optical parameters of nanotube-containing media considerably depend on their synthesis technique. An important problem today is the development of the manufacturing technology of the optical quality CSWNTs for the operation of mode-locked lasers. In this paper, we propose a new method for preparing CSWNT-containing films for use in a low-threshold femtosecond Er fiber laser. A principally new type of polymers, cellulose derivatives, in particular, carboximethyl cellulose (CMC), has been used for the film formation. This substance has two advantages: it is a surfactant with a high nanotube-dispersion activity[9] and a polymerlike medium for the matrix formation. Thus, the number of components in the final material is reduced to two: CSWNTs and polymer. Other films usually contain three components: CSWNTs, surfactant, and polymer. CSWNTs were synthesized by arc-discharge method in He atmosphere with $Ni-Y_2O_3$ catalyst.[10] CSWNT-containing CMC films of optical quality were prepared by pouring the CSWNTs suspension in the 1% aqueous solution of sodium salt of CMC onto immobile substrate, followed by a slow evaporation of the solvent in a vessel. The film thickness was varied from 4 to 100 μm.

A homemade arc-discharge CSWNTs were used as a starting material.[10] The raw soot was not acceptable for optical applications. It contained about 20% of the CSWNTs and contaminants: amorphous carbon and catalyst particles. The process of formation of polymeric films incorporating CSWNTs simultaneously served as a purification process.

Stable suspensions of individual CSWNTs in 1 wt % aqueous solution of CMC (medium viscosity, Sigma) have been prepared by ultrasonication (1 h, 200 W) followed by ultracentrifuging (acceleration 150 000 g, 1 h) (Ultracentrifuge Optima Max-E, Beckman-Coulter). The upper part of the solution was taken after ultracentrifuging. The "polymer+CSWNTs" films have been formed by the solution cast onto a flat substrate, followed by a slow drying. The concentration of nanotubes in the film varied from 0.01 to 0.001 wt % depending on their initial concentration in the suspension.

UV-visible-near infrared (NIR) optical transmission spectra have been registered with the spectrophotometer Lambda-950 (Perkin Elmer). The Raman spectra have been observed with the micro-Raman spectrometer Jobin-Yvon S-3000.

The Raman spectrum of CSWNT-containing film is shown in Fig. 1. There are two characteristic features con-

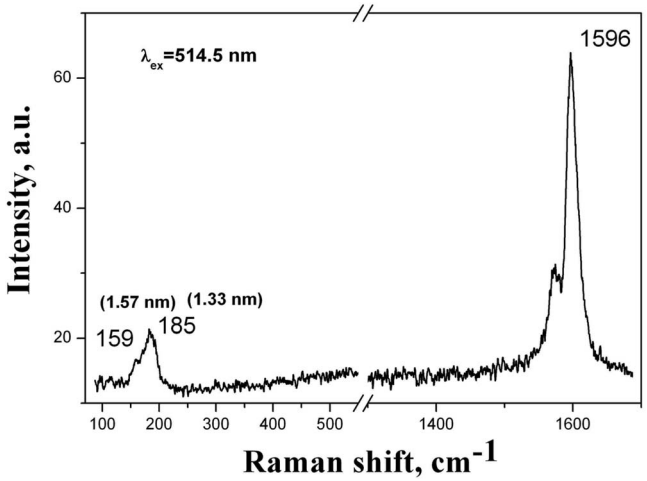

FIG. 1. The Raman spectrum of CMC film incorporating arc-produced CSWNTs. For the breathing Raman modes at 100–200 cm^{-1} the peak frequencies and the corresponding nanotube diameters are indicated.

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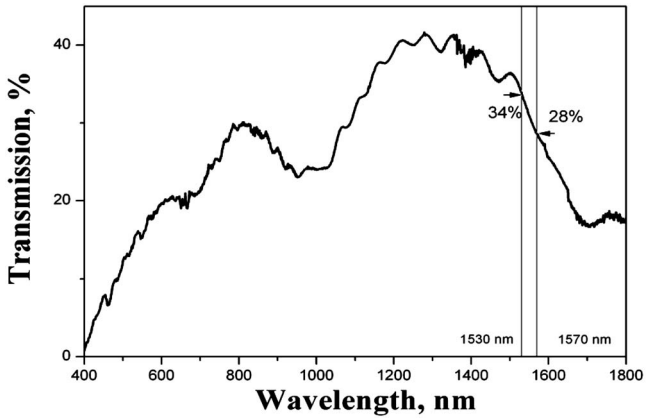

FIG. 2. UV-visible-NIR transmission spectrum of CMC film incorporated arc-produced CSWNTs. The numbers show the exact transmission in the working spectral range of Er^{3+} fiber laser.

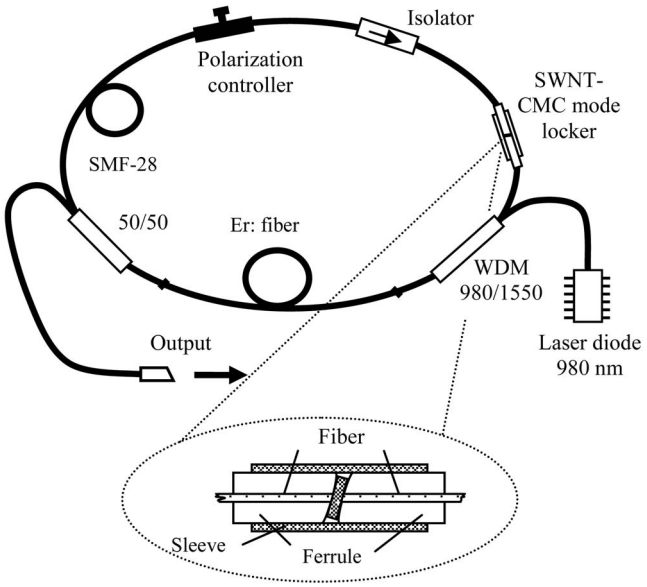

FIG. 3. A diagram of the mode-locked fiber ring laser incorporating a saturable absorber in the form of "CSWNT-CMC" composite film.

firming the presence of CSWNTS: a split tangential mode at 1596 cm^{-1} and a number of breathing modes at 150–190 cm^{-1}. The CSWNT diameters estimated on the base of these frequencies are 1.3–1.6 nm.[11] Due to a compressive stress arising in the polymeric films, a position of the tangential Raman mode (1596 cm^{-1}) is relatively upshifted to the position typical of CSWNT suspensions (1592 cm^{-1}).

UV-visible-NIR optical transmission spectrum of the same film is shown in Fig. 2. It is seen that the transmission coefficient is 28%–34% in the operational spectral range of Er^{3+}-fiber laser.

Z-scan setup[7] was used to study a saturable absorption in the films. The films were irradiated by 80 fs pulses from an erbium-doped laser (Avesta EFO-80) with a pulse repetition rate of 70 MHz and an average output power of 10 mW. The incident laser beam was focused by a lens (achromatic doublet) with a focal distance of 20 mm. The transmitted radiation was transformed into a parallel beam by a lens with a focal distance of 30 mm and was detected with a wide-aperture germanium photodiode. The sample was placed in the laser-beam waist perpendicular to the Z axis directed along the laser beam and was moved along the Z axis. This led to the variation of the beam power density on the sample and provided a possibility to measure the dependence of the transmission coefficient of the sample on the laser power density.[12]

This procedure has allowed to select a sample with a highest value of saturable losses. Its transmission coefficient was 30% under a low intensity laser irradiation. Its transmission increased up to 15% due to the absorption saturation under a maximum intensity of the laser beam. The calculated value of the energy density E was $\sim 50\ \mu J/cm^2$. With subtraction of Fresnel reflection losses ($\sim 10\%$), the initial transmission of an immersion-bleached film can be $\sim 40\%$.

A saturable absorber, fabricated for laser, was CMC film containing homogeneously distributed CSWNTs. A small piece of such film was inserted between two ends of a single-mode fiber and was firmly fixed in place by these ends (Fig. 3). To avoid the parasitic reflections quenching self-mode locking, the fiber ends were cut at the angle of $\sim 7°$. The absorber was designed on the base of a standard FC/APC connector. Splice losses were minimized by using an index matching fluid. The fiber-to-fiber insertion loss for the pure CMC film (5 μm thickness) was about 4%.

We used an erbium-doped fiber ring laser in the experimental setup (Fig. 3). The erbium-doped fiber with length of 1.25 m was pumped through a wavelength division multiplexing (WDM) coupler by a 980 nm laser diode along the propagation direction of laser radiation in the fiber ring. The optical losses in the erbium-doped fiber at 1.53 μm were 54 dB/m, the fiber core diameter was 3.5 μm and the dispersion was $\beta_2 = +0.035\ ps^2/m$. To provide unidirectional lasing and to minimize the polarization-mode dispersion, a two-stage polarization-independent isolator with the polarization-mode dispersion less than 100 fs was used in the scheme. The radiation emerging from the active fiber propagated through a second 50/50 coupler. This coupler provided the 50% coupling output of the laser, while the remaining radiation was incident on the saturable absorber. This coupler was placed directly behind the active fiber to reduce the influence of the nonlinear effects during the propagation of a pulse in the cavity fiber with the negative dispersion, resulting in the narrowing of the pulse spectrum.[12] To compensate the dispersion of the active fiber, the fibers with the opposite dispersion sign were added into the cavity: a SMF-28 fiber of length 1.56 m ($\beta_2 = -0.022\ ps^2/m$) and a HI1060FLEX fiber of length 0.41 m ($\beta_2 = -0.007\ ps^2/m$). Thus, the total dispersion in the cavity was $\beta_{tot} = 0.0065\ ps^2/m$ (which can be considered zero within the calculation error). Because the absorption is saturated at comparatively low energies, the saturable absorber was located so that to reduce the incident power, i.e., behind all elements of the cavity, in front of WDM coupler.

The laser was self-started and mode locked irrespective of a polarization state in the cavity. As the pump power was increased, the lasing spectrum broadened and a dependence on the polarization state became stronger despite the minimization of polarization-dependent quantities in the cavity elements, until the appearance of multiple-pulse mode locking or Q-switched mode locking. To control the polarization state, a polarization controller was added into the cavity, which was used to optimize the spectral width.

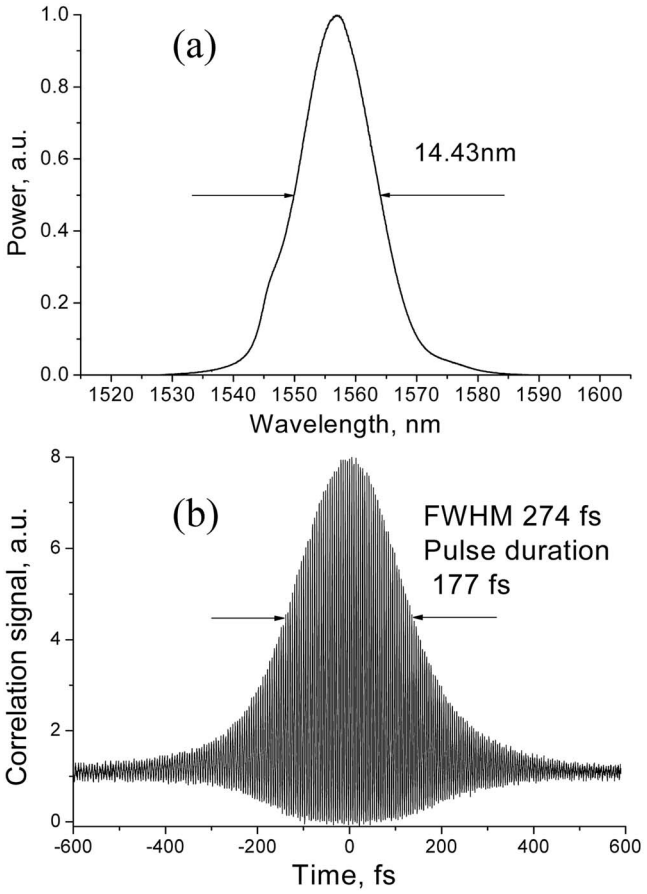

FIG. 4. (a) Laser output spectrum and (b) interferometric autocorrelation function.

We obtained stable lasing with an average output power of 7 mW upon pumping by 85 mW. The pulse energy and repetition rate were 0.14 nJ and 50 MHz, respectively. The output spectrum of the pulses of full width at half maximum 14.43 nm is shown in Fig. 4(a). To compensate the output-pulse chirp, we selected a proper length (~0.32 m) of the output fiber and replaced the isolator at the laser output by a fiber with the angle polished end. The measured correlation width was 274 fs. This corresponded to the pulse width of 177 fs (assuming sech^2 pulses) and the time-bandwidth product of 0.3158. The expected value for sech^2 pulses was 0.3148. Thus, our laser emitted the transform-limited soliton-like pulses.

The output laser pulse train had no transient variations. No time degradation of the film was observed. Taking into account the fiber diameter, the pulse duration and the repetition rate the damage threshold estimation appeared to be $>1\times10^9$ W/cm^2. The cw mode locking in fiber lasers is preceded by a brief period of Q-switching and Q-switched mode locking. The peak intensity in these two regimes is 2-3 orders of magnitude higher than in the mode locking one. But even in Q-switching regimes no damage of the film has been observed.

In summary, this work demonstrates for the first time an operation of the ultrashort-pulse (177 fs) erbium-doped fiber laser equipped with a saturable absorber based on carboxymethyl cellulose film with embedded CSWNTs grown by arc-discharge technique. The developed procedure of the film fabrication provides a rather simple and low-cost way of manufacturing of the saturable absorber devices for fiber lasers. The estimated damage threshold of such films is high enough ($>1\times10^9$ W/cm^2). Lasers equipped with the CSWNT saturable absorbers operate at low pump thresholds (25 mW). They are compact and have excellent operational parameters (a high mechanical stability and a weak dependence on external perturbations). Such lasers are demanded for optical communication, ultrafast process analysis, control of the fast response of optoelectronic devices, multiphoton microscopy, etc.

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