

Article

In Situ Preparation of Thin-Film Q-Switches Based on Vanadium Dioxide for Pulsed Fiber Lasers

Dmitriy P. Sudas ^{1,2,*}, Sergei M. Popov ²  and Petr I. Kuznetsov ²
¹ World-Class Research Center, Peter the Great St. Petersburg Polytechnical University, Polytechnicheskaya ul. 29, 195251 St. Petersburg, Russia

² Kotel'nikov Institute of Radioengineering and Electronics, Russian Academy of Sciences, Fryazino Branch, Sq. Vvedenskogo 1, Fryazino, 141190 Moscow, Russia; popov@fireras.ru (S.M.P.); pik218@mail.ru (P.I.K.)

* Correspondence: dmitriisudas@mail.ru

Abstract: In the presented work, erbium fiber lasers operating in the pulsed mode with a nonlinear element containing a vanadium oxide saturable absorber are demonstrated. The structure of the saturable absorber is based on a segment of thinned silica fiber coated with a thin-film vanadium oxide by the method of metalorganic chemical vapor deposition. A fiber laser scheme is demonstrated that allows controlling the transmission of the internal cavity of the resonator during laser generation and deposition of a thin film. We have demonstrated a method for obtaining and annealing nanocoatings with laser generation control. We controlled the laser output parameters directly during the synthesis of the saturable absorber material. Vanadium oxides obtained in the work demonstrated the Mott–Paierls phase transition practically at room temperature. In this work, the optical characteristics of the output radiation of a fiber laser with a saturable absorber were measured. At temperatures above 70 °C, the coatings demonstrate a passive Q-switch with a repetition rate of 38 kHz and a pulse duration of 3.8 μs. At temperatures below the phase transition, a short-term mode-locking mode occurs. The transmission jump at a wavelength of about 1350 nm during structural rearrangement was 24%. For comparison, VO₂ nanopowder in a polydimethylsiloxane elastomer matrix was used as a saturable absorber material. The nanopowder modulator made it possible to obtain pulses with a frequency of 27 kHz and a duration of about 7.2 μs.

Keywords: pulsed fiber laser; Q-switch; thin film; nanosheet VO₂; MOCVD



Received: 31 October 2024

Revised: 27 January 2025

Accepted: 31 January 2025

Published: 3 February 2025

Citation: Sudas, D.P.; Popov, S.M.; Kuznetsov, P.I. In Situ Preparation of Thin-Film Q-Switches Based on Vanadium Dioxide for Pulsed Fiber Lasers. *Photonics* **2025**, *12*, 133. <https://doi.org/10.3390/photonics12020133>

Copyright: © 2025 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Fiber laser systems are actively used in science and technology due to a wide range of design solutions and output parameters [1–5]. The pulsed generation mode is actively used in areas where it is necessary to study ultrafast processes occurring in various materials, in medicine, and in applications where high peak powers are required [6,7]. The conversion of continuous laser generation to pulsed generation is carried out by different methods, depending on the required pulse repetition rates and their duration. By embedding a nonlinear element in the laser circuit that demonstrates the saturation absorption (saturable absorber—SA) effect [8], i.e., enlightenment under the action of ever-increasing optical power, it is possible to achieve a change in the resonator quality factor. This switching of the quality factor is accompanied by an alternation of pulse generation during enlightenment and its absence during material switching. The parameters of pulse generation for such SAs depend on the optical properties of the material used and are limited by the recovery time after saturation and the intensity of this saturation. Saturable absorbers tend to lose

their properties over time, unlike artificial SAs [9,10] or MAX-phase [11,12]. It is possible to extend the life of the material by reducing the density of the optical power interacting with it. When using optical fibers, the saturable absorber material is applied to the cleaned side surface [13]. To ensure the interaction of electromagnetic radiation with a thin-film material, it is necessary to thin the reflective cladding either by mechanical grinding or chemical etching. The second type of thinning is simpler and allows for higher optical quality of the surface.

Many materials that demonstrate the saturation absorption effect can also belong to other classes, such as topological insulators [14] with an insulating volume and conductive surface states. Among the large number of oxides used in devices, it is worth noting the materials that demonstrate a phase transition [15–17]. When such materials are exposed to external influences, a change in the electronic or crystalline structure occurs. This transition leads to a significant change in optical and electrical properties and allows the use of such materials as filters or modulators.

Vanadium oxides exhibit the correlation properties of vanadium—a significant dependence of the energy position of the electron bands on the degree of filling of these bands with electrons. This dependence is expressed in the presence of the hysteresis-free Mott transition [18], which is an electronic phase transition of the second order. In addition, this transition initiates the implementation of a more inertial structural Peierls transition [19], which has thermal hysteresis and is called a first-order transition. A significant part of vanadium oxides demonstrates both of these transitions and, upon reaching certain critical conditions (temperature, electric field, or optical power), their monoclinic lattice transforms into a more ordered tetragonal or rhombohedral one. The only exception is the higher oxide V_2O_5 , which has only an electronic transition [20]. This phase change is called a metal-to-insulator transition (MIT).

Depending on the stoichiometric ratio of oxygen and vanadium, the critical transition temperature will differ from cryogenic ($-133\text{ }^{\circ}\text{C}$) to elevated ($250\text{ }^{\circ}\text{C}$) [21]. One of the most promising for use is vanadium dioxide VO_2 with an almost room temperature MIT of about $60\text{--}70\text{ }^{\circ}\text{C}$ [22] and accompanied by a drop in electrical resistance by 105 times. However, a well-known fact is the dependence of the hysteresis loop width of the electrical properties of the material on the size of the crystallites forming this material. Each of the mentioned vanadium oxides included in the Magnelli series has a significantly different transition temperature, and the properties of these transitions depend on the phase composition, surface morphology of the grown oxide, substrate, and impurities contained in the material. Therefore, the method of metal–organic chemical vapor deposition (MOCVD) is suitable for the synthesis of these vanadium oxides [23]. This technology allows for the growth of coatings with a high degree of homogeneity on complex surfaces, such as optical fiber. It is possible to obtain materials of different stoichiometry due to the possibility of varying the precipitation parameters over a wide range. Since the optical constants of the grown materials and the thickness must strictly correspond to the parameters necessary to maintain a stable pulse mode, it is advisable to carry out synthesis in the process of creating this coating.

The paper demonstrates a method for creating Q-switch fiber lasers with the selection of parameters for the deposition of vanadium oxide coatings directly in the process of laser generation.

2. Materials and Methods

For simplicity, the thinned section of optical fiber with the applied saturable absorber nanomaterial will be called an SA. The SA was based on standard single-mode fiber SMF-28e with a core diameter of $8.2\text{ }\mu\text{m}$. A 2 mm long segment was thinned to a diameter of

8.5 μm to ensure contact between the radiation propagating along the optical path and the material of the grown coating. The length of the thinned section was determined by preliminary mechanical cleaning of the protective polymer. The used polishing composition of ammonium fluoride NH_4F ensured a diameter thinning rate of about 0.5 μm per minute at room temperature. An aqueous solution of ammonium sulfate $(\text{NH}_4)_2\text{SO}_4$ was also used to improve the quality of the silica surface and accelerate the removal of etching products. The geometric parameters of the thinned segment of the optical fiber (taper) were controlled using an optical microscope NU-2 (Carl Zeiss AG, Jena, Germany). Upon reaching the required diameter, the etching process was stopped, and the surface was washed multiple times with bidistilled water.

A prepared section of the optical fiber with a thinned segment was placed in a quartz reactor for subsequent deposition of nanolayers. The scheme of the MOCVD method and in situ control of the transmission spectrum is shown in Figure 1. In situ control means control on-site in real time. This means the ability to evaluate the process in real time and make adjustments to it as needed.

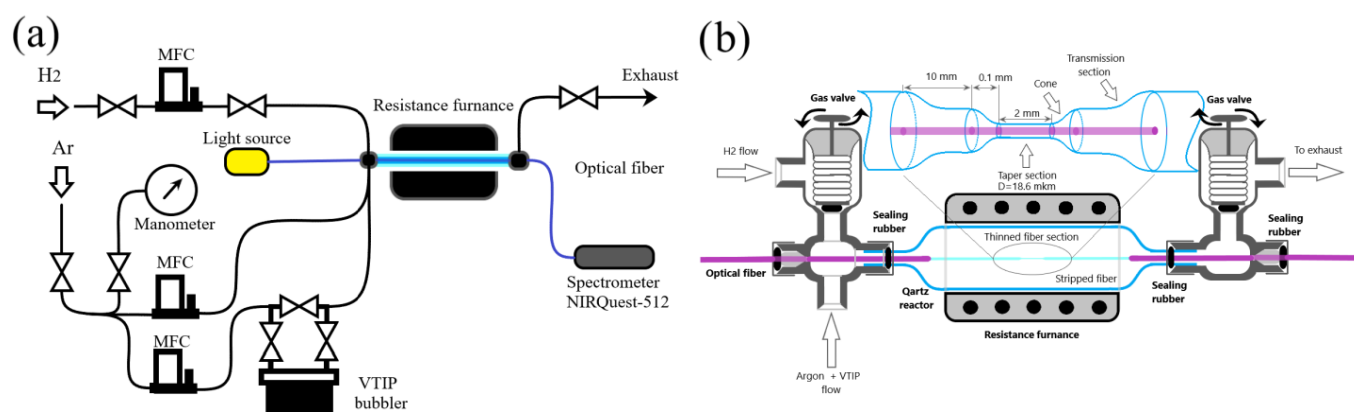


Figure 1. Scheme of vanadium oxide coating deposition by MOCVD and in situ control of optical path transmission spectrum (a). Enlarged part of quartz reactor with thinned segment of optical fiber placed (b). The inset shows an approximate image of thinned segment of optical fiber.

The nanolayer deposition technique was based on the use of vanadyl triisopropoxide (VTIP) as a vanadium source, which was placed in a stainless-steel bubbler and fed to the reaction zone by a flow of dried argon. An additional Ar line was used to control the position of the deposition zone inside the quartz reactor. The total velocity of the vapor–gas mixture, set by mass flow controllers (MFCs), was 50 cm/s during deposition. The vanadium organic storage was thermostatic at 40 $^{\circ}\text{C}$, and the gas lines and mixing units were heated to 50 $^{\circ}\text{C}$. Deposition was performed at temperatures from 180 to 290 $^{\circ}\text{C}$ by heating a specially made quartz reactor with a resistance furnace. Some coating samples were additionally annealed in a reducing environment to reduce the ratio between oxygen and vanadium in the coatings. For this purpose, the pre-deposited films were heated from 365 to 465 $^{\circ}\text{C}$ and maintained in a hydrogen atmosphere. The H_2 flow was 100 cm^3/min and was 10 times greater than argon. In addition to the optical fiber, we placed plane-parallel plates of sapphire or silicon in the reactor. The tightness of the system was checked with a manometer before each synthesis process. The resistive furnace allowed temperature control in an inert atmosphere with an accuracy of up to 1.0 $^{\circ}\text{C}$, and in a reducing hydrogen environment up to 0.5 $^{\circ}\text{C}$.

A halogen lamp (HL-2000 OceanInsight Inc., Orlando, FL, USA) was used as a radiation source, and a spectrometer (NIRQuest-512 OceanInsight Inc., Orlando, FL, USA) with an operating range of 900 to 1700 nm and a resolution of 0.1 nm served as a receiver. The coating deposition rate was calculated from its thickness determined by a scratch on an

interference microscope. The main method of analysis of the obtained coatings was the study of Raman scattering spectra obtained using the Raman-luminescent spectrometer “InSpektr” ($\lambda = 532$ nm, power = 30 mW, spot size = 1 μm). Photographs of the surface were obtained using a scanning electron microscope (JSM-6480LV Jeol Ltd., Tokyo, Japan). Transmission spectra of quartz and sapphire plates were obtained using a spectrometric complex (Specord UV VIS Carl Zeiss AG, Jena, Germany).

The fiber laser circuit used for pulse mode testing is shown in Figure 2.

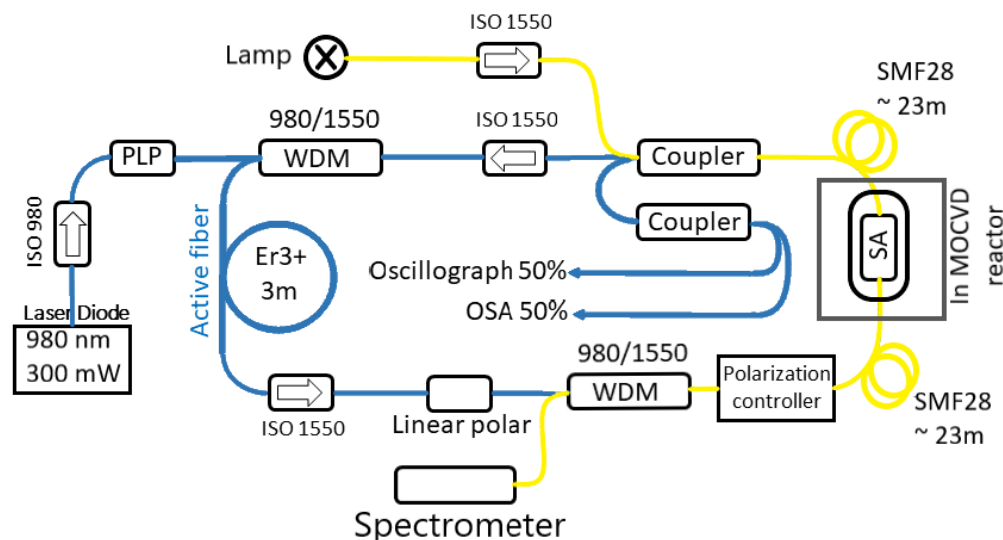


Figure 2. Schematic diagram of the erbium fiber ring laser used in the work. The part involved in monitoring the transmission spectrum of the thinned fiber segment during the coating process is highlighted in yellow.

All fiber components and connectors in the ring were made using SMF-28e single-mode fiber. A 980 nm wavelength single-mode pigtailed laser diode of up to 300 mW output power was used as a pump source in the experiments. Silica fiber of our own production with an aluminosilicate core activated by erbium ions was used as the active medium. The absorption at 980 nm wavelength was 9.2 dB/m, and the total length was 3 m. The length was chosen to provide peak laser generation in the region of 1550–1560 nm. In this case, the MOCVD setup was located in a clean room, and the measurement stand was outside it in another room. Fiber components were also not shortened to facilitate manipulations with the circuit in different experiments. Based on this, the length of the ring resonator without an SA was measured accurately and was equal to 18.6 m. The total length together with the delivery fiber paths was about 66 m. Unfortunately, the conditions of use of the setup do not allow us to significantly reduce this length. The value of group velocity dispersion was equal to the standard value characteristic of SMF28e. Polarization-independent isolators (ISO 1550) provided the direction of radiation along the resonator. An isolator (ISO 980) and pump laser protector (PLP) were used to protect the pump laser diode. Both of these components prevent reflected power from reaching the pump diode. The radiation output from the ring was provided by an optical output coupler with a 70×30 ratio. An additional 50×50 ratio coupler was welded to the larger arm of this output coupler for simultaneous monitoring of the pulse shape and the generation spectrum. The spectrum and average power of laser radiation were recorded with a Keysight Agilent 86140B (Keysight Technologies, Inc., Santa Rosa, CA, USA) spectrum analyzer, and the shape, amplitude, and pulse repetition rate with a Picometrix-AP-300 (Picometrix LLC, Ann Arbor, MI, USA) photodetector connected to a Keysight MSOX3102T (Keysight Technologies, Inc., Santa Rosa, CA, USA) oscilloscope. The upper recording frequencies for these devices are 1.5 and 1 GHz, respectively. To control the transmission

of the thinned light guide during the deposition process, an additional WDM and an isolator were introduced into the ring, preventing part of the laser radiation from entering the spectrometer.

To obtain the laser generation parameters, samples of SA were created based on commercially available vanadium dioxide 99.8% (Merck KGaA, Darmstadt, Germany) dissolved in polydimethylsiloxane elastomer (PDMSe). To confirm the data obtained from the polymer samples, several groups of such samples were used for comparison. An important factor was the time after the final hardening of the material. The mass concentration of the powder was 3 wt%. The samples were kept at room temperature for a week to completely harden the polymer. Temperature studies were performed by placing a sample with a polymer composite on a Peltier element.

3. Results and Discussion

Standard methods of X-ray diffraction studies did not yield results due to the high degree of coating amorphism. Energy-dispersive analysis is also not applicable, since the vanadium and oxygen lines partially overlap and an accurate quantitative assessment is not possible. In this case, a comparison of the energy spectrum of the obtained coatings with the corresponding reference V_2O_5 was carried out. All peaks coincided with an accuracy of up to 2%. The method chosen in the work is the study of Raman spectra of vanadium oxide coatings obtained on the sapphire surface. Information on the quality of the optical surface and Raman spectrum data are shown in Figure 3. In addition to this evaluation method, transmission spectra of plane-parallel plates with a deposited coating before and after annealing were used. As a result of changing the optical constants of the material, the cutoff position shifted from 516 nm, characteristic of V_2O_5 , towards 1600 nm (Figure 3c). This value corresponds to a band gap of about 0.7 eV, characteristic of vanadium dioxide.

As can be seen from the presented data, the coating is uniform, but the composition requires explanation. It is clearly seen that the obtained coatings (Figure 3b black line) consist of a mixture of four oxides with characteristic peaks in the Raman spectra: V_2O_5 (145, 405, 688, 992 cm^{-1}), V_6O_{13} (161, 845, 880, 936, 1033 cm^{-1}), VO_2 (193, 223 cm^{-1}), V_3O_5 (294, 487 cm^{-1}) [21,24]. The peaks related to V_3O_5 are partially overlapped by the sapphire lines. All coatings are characterized by a dominant amount of vanadium pentoxide in the material, which is not surprising since the oxidation state of the VTIP precursor molecules is 5 and it is energetically more efficient to obtain this oxide during the reaction. The content of VO_2 and V_3O_5 is trace, so their effect on the properties of the coating is small. Large V_6O_{13} is formed during the reaction of thermal rearrangement of isopropoxide groups [25]. To study the most interesting vanadium oxide VO_2 for applied science, it is necessary to reduce the amount of oxygen in the resulting coatings by compensating for oxidation processes during the synthesis of the coating. The use of a reducing hydrogen environment leads to the formation of radicals with a lower oxidation state than VTIP in greater quantities. This leads to an increase in the content of VO_2 and V_3O_5 in the coating, but does not allow the complete removal of higher oxides. The most effective technique is annealing in a hydrogen atmosphere of the already obtained coatings.

After the deposition process was complete, we turned off the VTIP supply and maintained the material at the synthesis temperature. This was necessary to complete the gas transport processes inside the reactor. Depending on the initial composition and thickness, the annealing temperature ranged from 365 to 450 °C, and at the preliminary stage of efficiency evaluation on plane-parallel plates, the annealing time was 10 min (Figure 3b red line). Raman scattering data also show a significant amount of vanadium dioxide in the film, accompanied by characteristic peaks: 193, 260, 334, 389, 613 cm^{-1} [24]. Some amount of V_3O_5 is also present, in contrast to the reduced V_6O_{13} and V_2O_5 .

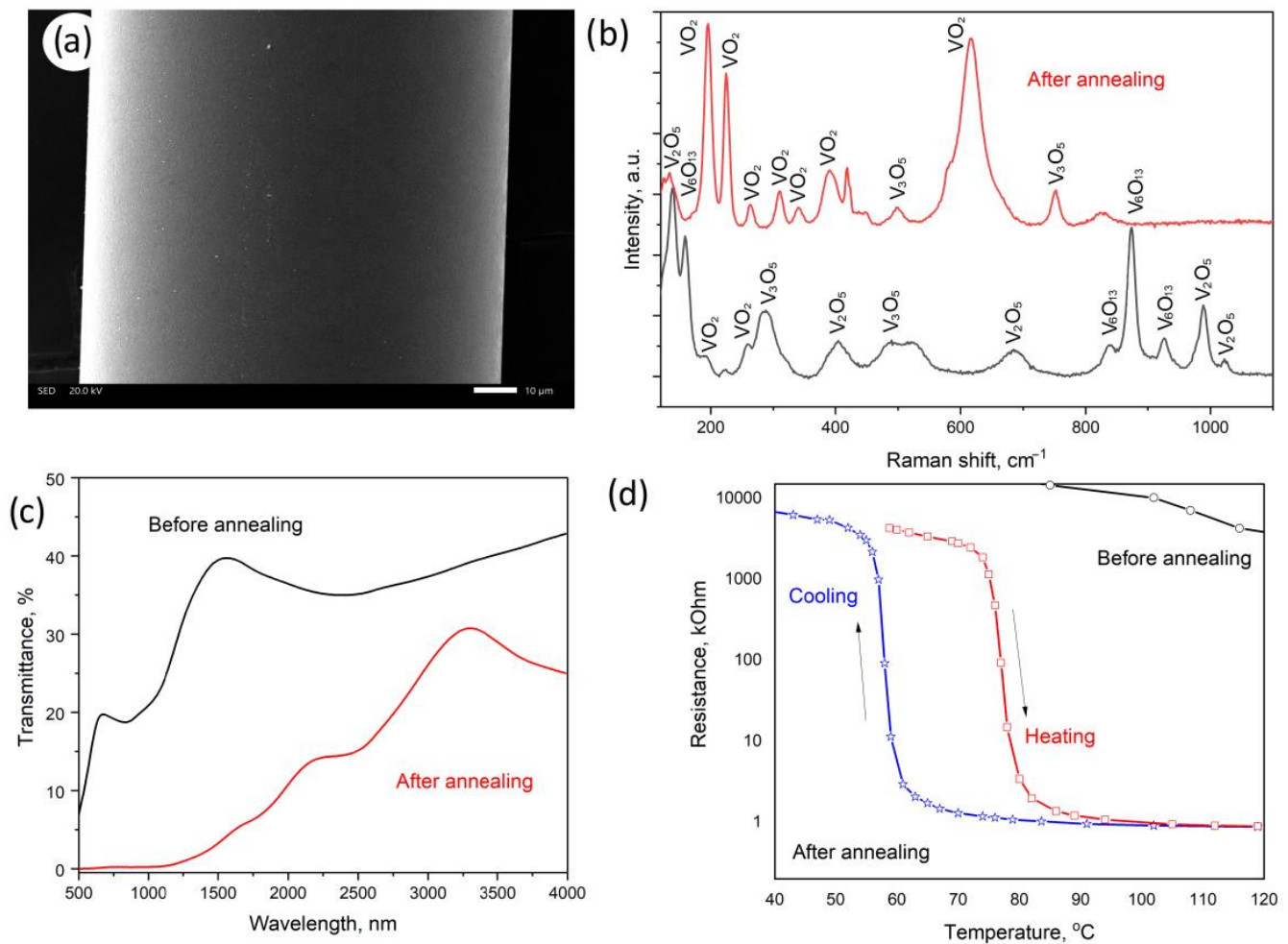


Figure 3. Characterization of the coatings obtained by the MOCVD method. SEM image of the surface of the optical fiber (a), Raman spectrum of 150 nm thick coatings on the sapphire surface before and after annealing in hydrogen (b), transmission spectra before and after annealing of the vanadium oxide coating on a quartz plate (c), data on the electrical resistance of the nanocoating on a thinned segment of the fiber at different temperatures (d).

We studied the behavior of the electrical resistance of the coating material at different temperatures. For this purpose, silver electrodes were applied to the nanocoating on a thinned segment of the fiber at a distance of 2 mm from each other, and the resistance was measured at different temperatures (Figure 3d). The material before annealing had a high resistance over the entire measured range, which is typical for vanadium pentoxide with a phase transition at higher temperatures. After annealing the coating, a phase transition began to be observed with a hysteresis loop centered at about 68–69 °C, which exactly matches vanadium dioxide. The conductivity jump was 10,000 times.

By inserting a part of the optical fiber with a thinned segment into the laser cavity, the transmission spectrum was checked depending on the thickness of the grown coating. Data on the parameters of laser generation before coating application and the transmission spectrum are shown in Figure 4.

In continuous mode, the wavelength of laser generation is 1562 nm and the maximum output power is 17 mW (Figure 4a). The wavelength corresponds to the fact that the energy conversion in the active fiber occurs quite efficiently, since for erbium fiber, the better the fiber is pumped, the more long-wavelength the generation shifts. The low value of the output power is due to the use of a set of elements in the laboratory circuit that introduce additional losses but allow the circuit to be easily adapted to different generation modes.

Such elements primarily include isolators and a pump laser protector that prevent reflected power from entering the pump diode.

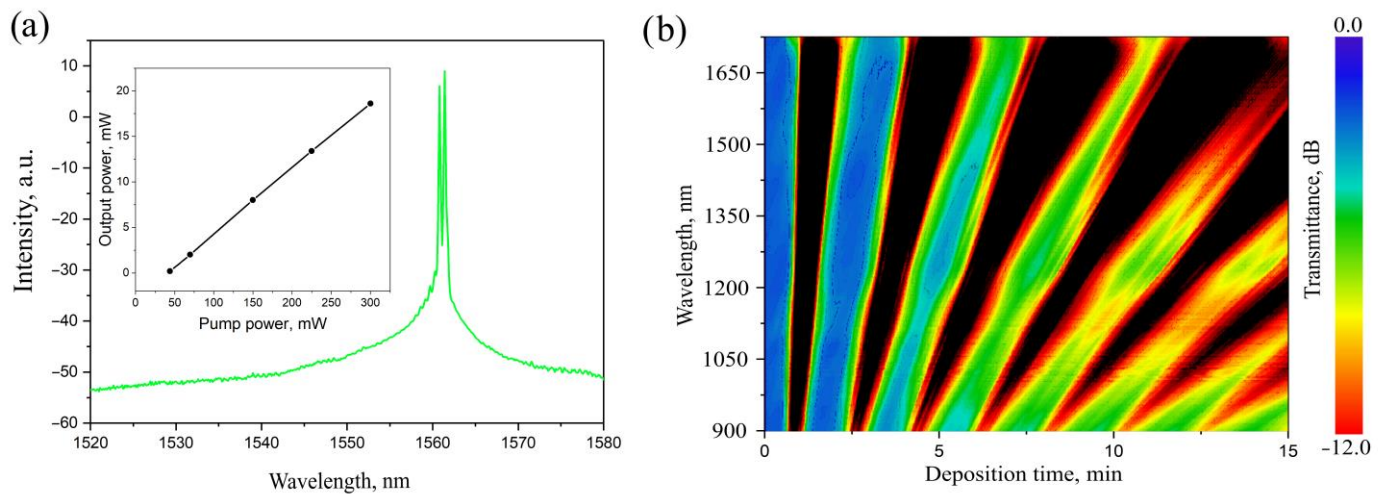


Figure 4. Investigation of optical parameters of a ring laser circuit with a Q-switch inside a quartz reactor. The laser generation spectrum obtained at a pump power of 150 mW is shown in (a). The inset shows the dependence of the output optical power on the pump power. A spectral scan of the deposition process is shown in (b). For clarity, the full transmission spectrum in the range from 900 to 1700 nm is shown.

When examining the spectral sweep over a long deposition process, significant dips in transmission are visible as the film thickness increases (Figure 4b). This effect is called the lossy mode resonance (LMR) [26] and is caused by mode coupling between the coating cladding modes and the fundamental mode of the fiber core. At a certain thickness, some of the energy flows from the core into the coating and is dissipated into the environment. The greater the coating thickness, the longer the wavelength of this resonance. Its appearance is explained by the high optical transparency of V_2O_5 obtained at the deposition stage. However, pulsed generation is not observed in the entire range of the experiment and for coating thicknesses up to 900 nm.

To check the occurrence of a pulsed mode during the production of VO_2 , the optical characteristics of the laser were studied as the resulting coating was annealed. The annealing results are shown in Figure 5.

In the presented section of the transmission value at a wavelength of 1350 nm, it is possible to distinguish two fragments: the heating to the annealing temperature (black line) and the annealing (red line). A slight increase in transmittance during heating is due to thermal expansion during heating. As soon as the recrystallization process occurs, the generation mode caused by the restoration of the surface becomes weakly pulsed and stabilizes over time. The completion of the processes occurring in the volume of the material during the annealing process was determined by the stabilization of the transmission. The primary linear nature of the increase in transmission is explained by the annealing of the surface layer of the material. Further restructuring of the composition in depth leads to a jump-like increase in transmission. During this period, the main recovery process occurs. The parameters of the Q-switch in the heated state at a temperature of 100 °C are shown in Figure 6.

The maximum average laser generation power obtained in the circuit with a heated SA was 10.5 mW. The pulse repetition rate and duration in the Q-switch were 38 kHz and 3.8 μ s, respectively. When the temperature was reduced below the critical value, the material of the saturable absorber was converted from the metallic phase to the semiconductor phase, and a jump in reflection occurred over the entire IR wavelength range. The SA in the

reactor was cooled smoothly according to the program in an argon atmosphere to room temperature, and the change in the generation parameters was observed. The results of these measurements are shown in Figure 7.

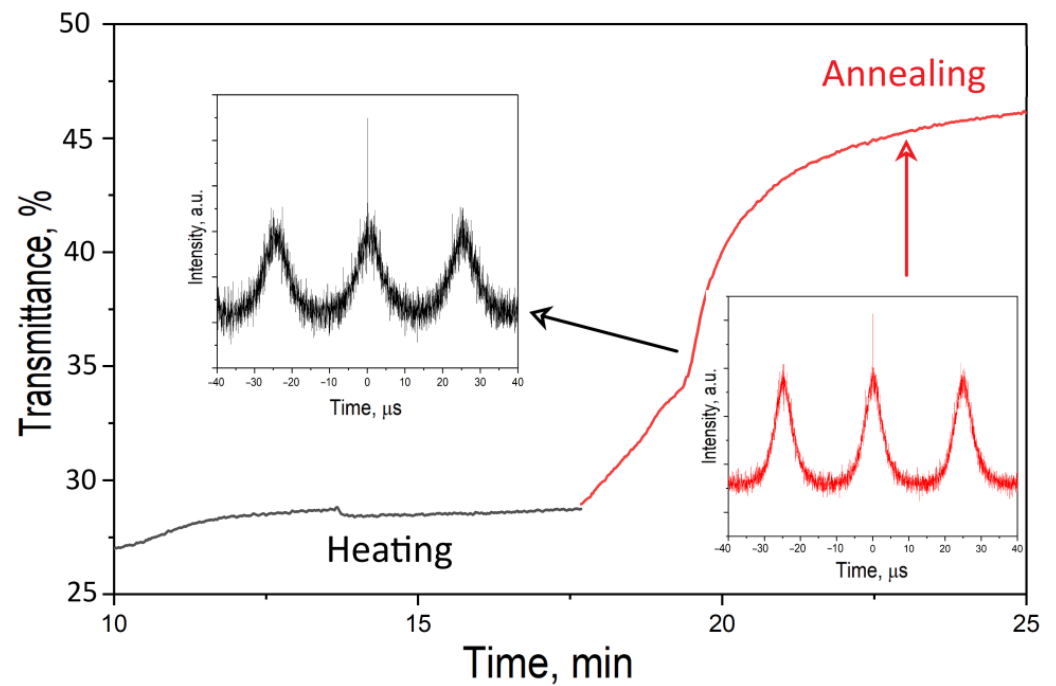


Figure 5. Investigation of optical parameters of laser ring scheme during annealing of vanadium pentoxide coatings by the cross-section of the transmission value at a wavelength of 1350 nm. The insets show the data of the photodetector.

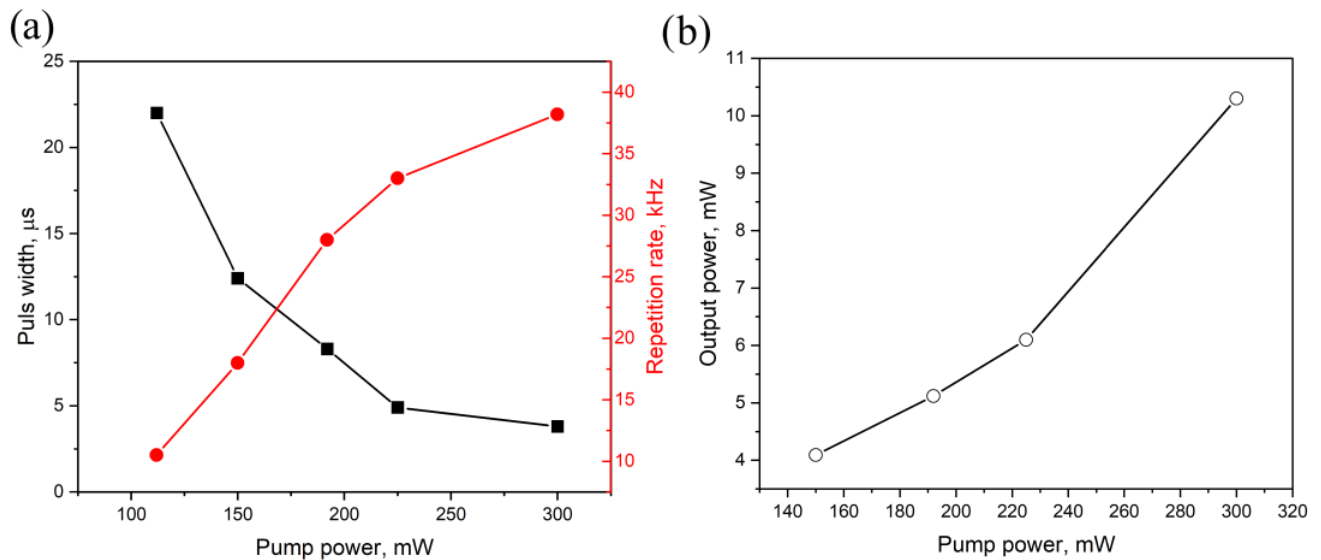


Figure 6. Study of the characteristics of laser pulse generation in the heated state of the SA. Dependences of the pulse repetition frequency and their duration on the pump power (a); dependence of the output power on the pump power (b).

The transmission drop during modulator cooling was 24% in the predominant range of the observed spectrum and at a wavelength of 1350 nm. As cooling proceeds, an increase in losses in the resonator is observed, accompanied by a drop in transmission (Figure 7). An increase in resonator losses is accompanied by a shift of the generation peak to a shorter-wavelength region of the spectrum. At the moment of time corresponding to a temperature

of about 50 °C, two peaks at about 1550 and 1530 nm appear simultaneously. Due to the high gain of the erbium active fiber in the region closer to 1530 nm, the incompletely “pumped” active medium allows maintaining generation only at this wavelength. At the moment of phase reconstruction of the saturable absorber material, a short-term mode-locking occurs. This change in the pulse regime is accompanied by a characteristic broadening of the generation spectrum and a sharp change in the pulse shape. In this case, the nature of the pulses shown in Figure 7c shows that the pulse repetition rate is the same, unlike the amplitude. Such an effect can be observed due to the residual influence of the Q-switch mode. The non-uniformity of the material on the cones of the thinned fiber segment contributes to the overall behavior of the pulsed mode, and by adjusting the geometry of the thinned section, it is possible to achieve a more stable mode. In addition, the output optical coupler is selected in such a way as to provide the best laser generation characteristics in the Q-switch and is not optimal in the mode-locking. A further increase in losses in the resonator leads to the disappearance of the pulsed generation and a transition to a continuous wave. The pulse duration in the mode-locking was 3.2 ns at a frequency of 4.5 MHz.

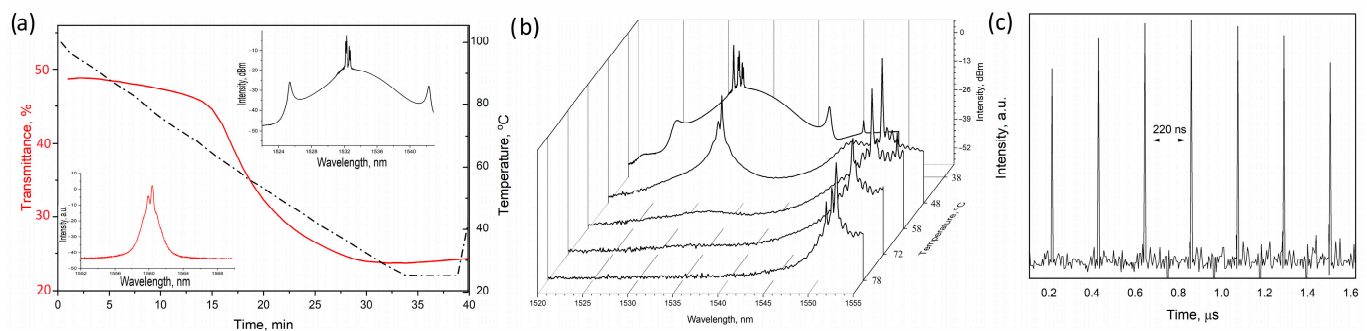


Figure 7. Study of laser pulse generation characteristics during cooling of Q-switch by transmission at a wavelength of 1350 nm (a). The insets show the generation spectrum in Q-switch (bottom) and mode-lock (top). The dotted line indicates the temperature in the reactor. Laser generation spectra during deposition of saturable absorber material (b), pulse shape during short-term occurrence of mode-locking (c).

The absorption saturation effect parameters for the used vanadium dioxide were estimated using plane-parallel plates with a 300 nm thick coating. A standard Z-scan system with an open aperture was used for this purpose. A PERI-OEM femtosecond laser (Avesta-Project Ltd., Moscow, Russia) with a pulse duration of 80 fs and an output power of 200 mW was used as a radiation source. Two PD300R-IR units from (Ophir Optronics Solutions Ltd., Jerusalem, Israel) and a set of attenuators and filters were used as power meters. An electric translator was used to change the plate coordinate. The result of measuring the transmittance dependence of the plate with the material is shown in Figure 8.

The frequency of the laser pulses used is 100 kHz. The laser beam radius in the waist was calculated using the formula $r_0 = 2\lambda f / \pi D$. Here, $\lambda = 1560$ nm—wavelength; $f = 10$ cm—focal length of a lens; D —diameter of the incident laser beam at the input of the focusing lens. The size of the laser beam at the lens input was determined from the spatial distribution of its intensity, which was measured by scanning the laser beam cross-section with a fiber connected to a fiber power meter. The measured spatial distribution of the laser beam intensity was approximated by a Gaussian function. From the approximation, the diameter of the input laser beam was determined to be $D = 3.8$ mm, and from the previous formula, its radius was calculated, which was approximately $r_0 = 26.4$ μm . The saturation intensity was determined using the standard dependence:

$$T(I) = \alpha_{\text{sat}} / (1 + I/I_{\text{sat}}) + \alpha_{\text{non-sat}} \quad (1)$$

where α_{sat} —modulation depth (saturable loss); $\alpha_{\text{non-sat}}$ —unsaturable losses; I_{sat} —saturation intensity (the required intensity to reduce absorption by $0.5 \alpha_{\text{sat}}$). The modulation depth was about 7% at a saturation intensity of 3.72 MW/cm^2 . The value mentioned in the text is an average between the results obtained from several similar samples. Since the purpose of this study was to demonstrate the method of fixing the pulse mode directly during the application process, a complete study of the material was not provided. Determining the recovery time after saturation [27,28] is complicated by the fact that VO_2 is a Mott insulator. Note that the Mott electronic transition determines the ultrafast response of vanadium dioxide to the optical transfer of electrons from the valence band to the conduction band in femtosecond spectroscopy experiments. The structural transition occurs over a longer time of the order of several picoseconds. The restructuring time during the electronic transition is in the region of less than 100 fs [29]. Therefore, for a complete assessment of intraband electronic transitions, it is necessary to use other excitation sources with longer pulses and different repetition rates. The measurement can only be carried out in the metallic phase of the material, since the incident optical power causes phase transformation before saturation occurs.

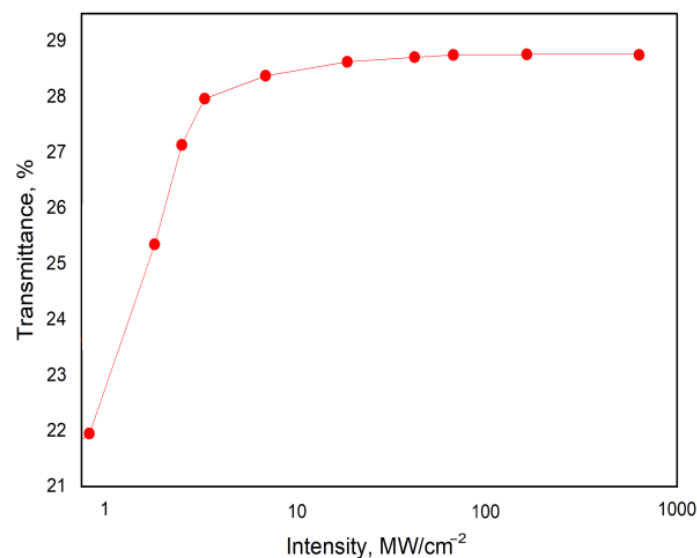


Figure 8. Study of the modulation depth of a vanadium dioxide film on a flat quartz plate.

For comparison, we also used vanadium oxide nanopowder in a polymer. The optical characteristics of the fiber laser with a powder SA are shown in Figure 8. Unlike the modulator based on a thin film, the temperature of which was changed in the reactor, this sample was mechanically adjacent to the Peltier element.

In contrast to the film implementation of the Q-switch, the transmission jump at a wavelength of 1350 nm was 28%, but was accompanied by some small oscillations arising due to the movement of the softening polymer mass. At the same time, in contrast to the clearly expressed phase transition, accompanied by a significant change in the optical characteristics of lasing, in this case, there is only an increase in losses due to a change in the refractive index of the polymer composite. An increase in the refractive index of PDMS due to heating leads to a more efficient output of the optical field from the fiber and leads to an increase in losses. Because of this, the Q-switch mode characteristics do not change, but only the threshold of its formation shifts (Figure 9b,c). The maximum average power of lasing obtained in the ring with a heated SA was 8.8 mW. The pulse repetition rate and their

duration in the Q-switch mode were 27 kHz and 7.2 μ s, respectively. Using a polymer as a matrix for a saturable absorber is not effective since it allows achieving rather weak pulse mode parameters in comparison with a film implementation. In addition, the property of a polymer composite changing its optical characteristics depending on temperature does not allow a reliable assessment of the contribution of the SA material.

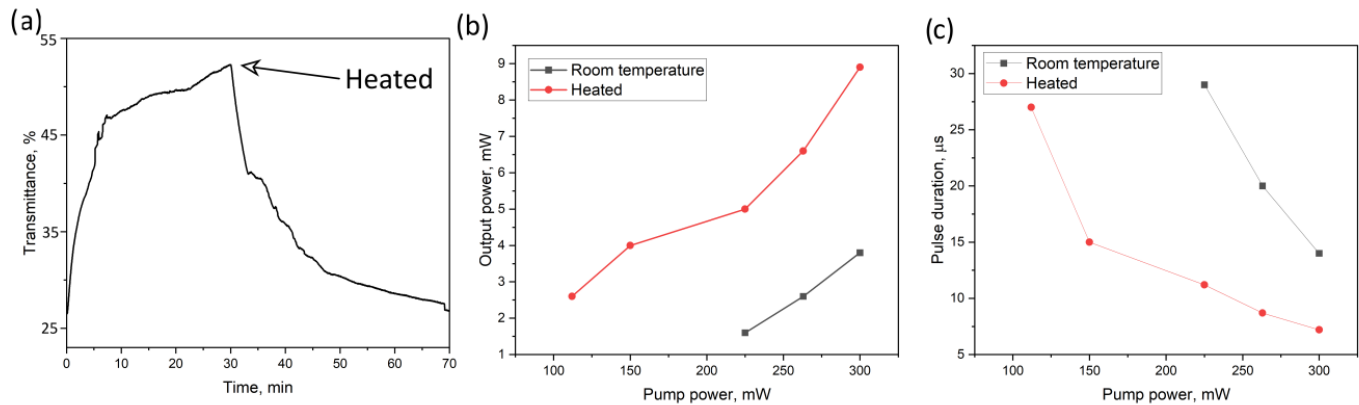


Figure 9. Study of laser pulse generation characteristics during cooling of a nanopowder-based Q-switch by transmission at a wavelength of 1350 nm (a); dependence of output power on pump power before and after heating (b); dependence of pulse duration on pump power before and after heating (c).

From the obtained data, it is evident that the phase transition in the nanopowder is practically not manifested due to the low degree of homogeneity of the composition and also demonstrates fewer outstanding parameters of pulsed laser generation.

4. Conclusions

The presented work presents erbium fiber lasers operating in a pulsed mode with a nonlinear element containing a vanadium oxide saturable absorber. The developed scheme of the fiber laser is shown, allowing one to estimate the transmission of the internal cavity of the resonator during laser generation and deposition of a thin film of vanadium oxides. A technique for producing and annealing nanocoatings with in situ control of the occurrence of a pulsed laser generation mode directly in the process of synthesis of the saturable absorber material is described. Vanadium oxides obtained in the work demonstrated a structural phase transition at almost 70 °C. In the present article, the optical characteristics of the fiber laser with a nonlinear element before and after the phase transition were studied. At temperatures above 70 °C, the coatings demonstrate a passive Q-switch with a repetition rate of 38 kHz and a pulse duration of 3.8 μ s. At temperatures below the phase transition, a short-term mode-locking occurs. The transmission jump at a wavelength of about 1350 nm during structural rearrangement was 24%. For comparison, VO₂ nanopowder in a polydimethylsiloxane elastomer matrix was used as a saturable absorber material. The nanopowder-based modulator made it possible to obtain pulses with a frequency of 27 kHz and a duration of about 7.2 μ s. The measurement of the absorption saturation parameters showed that the modulation depth was about 7% at a saturation intensity equal to 3.72 MW/cm². The demonstrated method for estimating the parameters of a Q-switch allows for more efficient production of thin-film saturable absorbers.

Author Contributions: Conceptualization, D.P.S. and P.I.K.; methodology, D.P.S.; software, S.M.P.; validation, P.I.K., S.M.P. and D.P.S.; formal analysis, D.P.S.; investigation, D.P.S.; resources, S.M.P.; data curation, P.I.K.; writing—original draft preparation, D.P.S.; writing—review and editing, P.I.K.; visualization, D.P.S.; supervision, P.I.K.; project administration, P.I.K.; funding acquisition, P.I.K. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by the Ministry of Science and Higher Education of the Russian Federation as part of the World-class Research Center program: Advanced Digital Technologies (contract No. 075-15-2022-311 dated 20 April 2022) (Sudas D.P.), and the work was partially carried out according to the State Task of the Kotelnikov Institute of Radio Engineering and Electronics RAS (Popov S.M. and Kuznetsov P.I.).

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Data are contained within the article.

Acknowledgments: The authors thank V.O. Yapaskurt for SEM photographs.

Conflicts of Interest: The authors declare no conflicts of interest.

References

1. Katta, N.; Estrada, A.D.; McErloy, A.B.; Milner, T.E. Fiber-laser platform for precision brain surgery. *Biomed. Opt. Express* **2022**, *13*, 1985–1994. [\[CrossRef\]](#) [\[PubMed\]](#)
2. Kar, T.; Deshmukh, S.S.; Goswami, A. Investigation of fiber laser micro-channel depth on silicon wafer. *Mater. Today Proc.* **2022**, *60*, 2105–2110. [\[CrossRef\]](#)
3. Benoit, P.; Le Méhauté, S.; Le Gouët, J.; Canat, G. All-fiber laser source at 1645 nm for lidar measurement of methane concentration and wind velocity. *Opt. Lett.* **2021**, *46*, 126–129. [\[CrossRef\]](#)
4. Kbashi, H.J.; Sharma, V.; Sergeyev, S. Dual-wavelength fiber-laser-based transmission of millimeter waves for 5G-supported Radio-over-Fiber (RoF) links. *Opt. Fiber Technol.* **2021**, *65*, 102588. [\[CrossRef\]](#)
5. Franczyk, M.; Stefaniuk, T.; Anuszkiewicz, A.; Kasztelan, R.; Pysz, D.; Filipkowski, A.; Osuch, T.; Buczynski, R. Nanostructured active and photosensitive silica glass for fiber lasers with built-in Bragg gratings. *Opt. Express* **2021**, *29*, 10659–10675. [\[CrossRef\]](#)
6. Bogusławski, J.; Kowalczyk, M.; Iwanowski, P.; Hruban, A.; Didusko, R.; Piotrowski, K.; Dybko, K.; Wojciechowski, T.; Aleszkiewicz, M.; Sotor, J. Exploiting nonlinear properties of pure and Sn-doped Bi₂Te₂Se for passive Q-switching of all-polarization maintaining ytterbium- and erbium-doped fiber lasers. *Sci. Rep.* **2017**, *7*, 7428. [\[CrossRef\]](#) [\[PubMed\]](#)
7. Gaida, C.; Gebhardt, M.; Heuermann, T.; Stutzki, F.; Jauregui, C.; Limpert, J. Ultrafast thulium fiber laser system emitting more than 1 kW of average power. *Opt. Lett.* **2018**, *43*, 5853–5856. [\[CrossRef\]](#) [\[PubMed\]](#)
8. Marini, A.; Cox, J.D.; García de Abajo, F.J. Theory of graphene saturable absorption. *Phys. Rev. B* **2017**, *95*, 125408. [\[CrossRef\]](#)
9. Yoshida, E.; Kimura, Y.; Kimura, Y.; Nakazawa, M.; Nakazawa, M. Femtosecond Erbium-doped fiber laser with nonlinear polarization rotation and its soliton compression. *Jpn. J. Appl. Phys.* **1994**, *33*, 5779. [\[CrossRef\]](#)
10. Liu, X.; Li, Q.; Pan, D.; Ye, F.; Malomed, B.; Fu, H. A robust and novel linear fiber laser mode-locked by nonlinear polarization evolution in all-polarization maintaining fibers. *J. Light. Technol.* **2021**, *39*, 7509–7516. [\[CrossRef\]](#)
11. Najm, M.M.; Almukhtar, A.A.; Hmood, J.K.; Najm, S.M.; Nizamani, B.; Abdullah, M.N.; Harun, S.W. Passive Q-switched fiber laser with MAX phase molybdenum titanium aluminum carbide film. *Opt. Quantum Electron.* **2024**, *56*, 121. [\[CrossRef\]](#)
12. Ahmad, H.; Kamely, A.A.; Yusoff, N.; Bayang, L.; Samion, M.Z. Generation of Q-switched Pulses in Thulium-doped and Thulium/Holmium-co-doped Fiber Lasers using MAX phase (Ti₃AlC₂). *Sci. Rep.* **2020**, *10*, 9233. [\[CrossRef\]](#) [\[PubMed\]](#)
13. Kassani, S.H.; Khazaeinezhad, R.; Jeong, H.; Nazari, T.; Yeom, D.-I.; Oh, K. All-fiber Er-doped Q-Switched laser based on Tungsten Disulfide saturable absorber. *Opt. Mater. Express* **2015**, *5*, 373–379. [\[CrossRef\]](#)
14. He, X.; Zhang, H.; Lin, W.; Wei, R.; Qiu, J.; Zhang, M.; Hu, B. PVP-assisted solvothermal synthesis of high-yielded Bi₂Te₃ hexagonal nanoplates: Application in passively Q-switched fiber laser. *Sci. Rep.* **2015**, *5*, 15868. [\[CrossRef\]](#) [\[PubMed\]](#)
15. Ran, Y.; Pei, Y.; Zhou, Z.; Wang, H.; Sun, Y.; Wang, Z.; Hao, M.; Zhao, J.; Chen, J.; Yan, X. A review of Mott insulator in memristors: The materials, characteristics, applications for future computing systems and neuromorphic computing. *Nano Res.* **2023**, *16*, 1165–1182. [\[CrossRef\]](#)
16. Ruzmetov, D.; Ramanathan, S. Metal-Insulator Transition in Thin Film Vanadium Dioxide. In *Thin Film Metal-Oxides*; Ramanathan, S., Ed.; Springer: Boston, MA, USA, 2010. [\[CrossRef\]](#)
17. Karanikolopoulos, D.; Gagaoudakis, E.; Droulias, S.; Louloudakis, D.; Mouratis, K.; Polychronaki, M.; Katsoprinakis, G.E.; Aperathitis, E.; Vernardou, D.; Binas, V.; et al. Influence of Mg doping on the ultrafast electron dynamics of VO₂ films. *Appl. Phys. A* **2021**, *127*, 751. [\[CrossRef\]](#)
18. Mott, N.F. The basis of the electron theory of metals, with special reference to the transition metals. *Proc. Phys. Soc.* **1949**, *62*, 416. [\[CrossRef\]](#)
19. Peierls, R. *More Surprises in Theoretical Physics*, Princeton; Princeton University Press: Princeton, NJ, USA, 1992. [\[CrossRef\]](#)
20. Goodenough, J.B. Anomalous Properties of the Vanadium Oxides. *Annu. Rev. Mater. Res.* **1971**, *1*, 101–138. [\[CrossRef\]](#)

21. Shvets, P.; Dikaya, O.; Maksimova, K.; Goikhman, A. A review of Raman spectroscopy of vanadium oxides. *J. Raman Spectrosc.* **2019**, *50*, 1226–1244. [[CrossRef](#)]
22. Shao, Z.; Cao, X.; Luo, H.; Jin, P. Recent progress in the phase-transition mechanism and modulation of vanadium dioxide materials. *NPG Asia Mater.* **2018**, *10*, 581–605. [[CrossRef](#)]
23. Greenberg, C.B. Undoped and doped VO₂ films grown from VO(OC₃H₇)₃. *Thin Solid Films* **1983**, *110*, 73–82. [[CrossRef](#)]
24. Chen, X.-B.; Shin, J.-H.; Kim, H.-T.; Lim, Y.-S. Raman analyses of co-phasing and hysteresis behaviors in V₂O₃ thin film. *J. Raman Spectrosc.* **2012**, *43*, 2025–2028. [[CrossRef](#)]
25. Högerl, M.P.; Serena Goh, L.M.; Abou-Hamad, E.; Barman, S.; Dachwald, O.; Pasha, F.A.; Pelletier, J.; Köhler, K.; D’Elia, V.; Cavallo, L.; et al. SOMC grafting of vanadium oxytriisopropoxide (VO(OiPr)₃) on dehydroxylated silica; analysis of surface complexes and thermal restructuring mechanism. *RSC Adv.* **2018**, *8*, 20801–20808. [[CrossRef](#)]
26. Savelyev, E.A.; Sudas, D.P.; Kuztestov, P.I. Using lossy mode resonance for in situ measurement of the refractive index of a layer deposited on an optical fiber lateral surface. *Opt. Lett.* **2022**, *47*, 361–364. [[CrossRef](#)] [[PubMed](#)]
27. Zang, W.-P.; Tian, J.-G.; Liu, Z.-B.; Zhou, W.-Y.; Song, F.; Zhang, C.-P.; Xu, J.-J. Accurate determination of nonlinear refraction and nonlinear absorption by a single Z-scan method. *J. Opt. Soc. Am. B* **2004**, *21*, 349–356. [[CrossRef](#)]
28. Kryukov, P.G.; Letokhov, V.S. Propagation of a Light pulse in a Resonantly amplifying (absorbing) medium. *Sov. Phys. Uspekhi* **1970**, *12*, 641–672. [[CrossRef](#)]
29. Lantz, G. Ultrafast Electron Dynamics in Mott Materials. Strongly Correlated Electrons. Université Paris Sud—Paris XI 2015, English. NNT: 2015PA112014. Fftel-01148820. Available online: <https://theses.hal.science/tel-01148820> (accessed on 18 June 2024).

Disclaimer/Publisher’s Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.