Quantum Electronics **47** (7) 669–674 (2017)

LASER MEDICINE

https://doi.org/10.1070/QEL16369

Laser heating dynamics and glow spectra of carbon-, titanium- and erbium-containing optothermal fibre converters for laser medicine*

A.V. Belikov, A.V. Skrypnik

Abstract. **Titanium- and erbium-containing optothermal fibre converters of laser radiation mounted at the distal end of quartz–quartz optical fibre are discussed for the first time. Technology of fabricating such converters is described. Carbon-containing converters are also considered. The laser heating dynamics of the converters and the glow spectra are studied by irradiating converters of each type by a 980±10 nm semiconductor laser with an average power up to 4 W. It is shown that alongside with broadband thermal radiation accompanying the laser heating of all three types of converters in the temperature range 600–1100 °C, only in the spectrum of the erbium-containing converter the intense bands with the maxima at wavelengths 493, 523, 544, 660, and 798 nm, corresponding to the erbium radiative transitions $^4F_{7/2} \rightarrow {}^4I_{15/2}$, $^2H_{11/2} \rightarrow {}^4I_{15/2}$, $^4S_{3/2} \rightarrow {}^4I_{15/2}$, $^4F_{9/2} \rightarrow {}^4I_{15/2}$ and $^4I_{9/2} \rightarrow {}^4I_{15/2}$, respectively, are present. Such converters can be used in laser medicine for tissue surgery as well as in procedures combining laser, thermal, biostimulation or photodynamic action.**

Keywords: *laser radiation, converter, erbium, titanium, carbon, fibre, glow, heating.*

1. Introduction

Laser radiation is widely used in materials processing and in medicine, including surgery [1, 2], biostimulation [3], photodynamic therapy [4], etc. The efficiency of most laser interventions depends on the absorption coefficient of chromophores, which are naturally present in a biotissue or introduced into it. In photodynamic therapy use is made of semiconductor lasers with the wavelengths 600–700 nm [4, 5], since the role of a chromophore is played by a photosensitiser, e. g., radachlorine that absorb light in this spectral region [6]. CO_2 and erbium lasers are considered as the most efficient ones in surgery [1, 2, 7–9], the absorption coefficients of their radiation by soft biotissues being determined by the strong absorption bands of water [10]. Recently contact laser surgery has made use of semiconductor lasers [1, 11–13] generating near-IR radiation with the wavelength 0.81–0.98 μm. The radiation of these lasers is weakly absorbed by soft biotissues. As a result, the efficiency of biotissue removal becomes low, and the size of the zone of damaged tissues surrounding the excision (subject to coagulation, denaturation, and necrosis) appears to be significant [9, 14–16].

To increase the efficiency of a semiconductor laser in contact laser surgery, a special optothermal fibre converter (OTFC) [17–22] is mounted at the distal end of optical fibre delivering laser radiation to biotissue. Most of such blackened tip converters contain carbon [19, 23–26]. In this case, the destruction of soft biotissue occurs mainly via the contact with a carbon-containing OTFC heated to high temperature (up to 900 °C [21]) by laser radiation. Note that broadband thermal radiation arising in the course of the converter heating can be also used for additional impact on biotissue [20]. ‘Hybrid probe’ tips are known that combine the effects of thermal and visible radiation on biotissue [27]. Thus, the development of new converters of laser radiation is one more possible way to expand the application field of semiconductor lasers in medicine.

In this connection, the development of converters that allow not only efficient excision of biotissue, but also selective, e. g., biostimulating or photodynamic impact on the chromophores that absorb light in the visible range of the spectrum, is rather promising.

In the present paper, we study converters of three types that differ in the substance converting laser radiation (erbium, titanium and carbon). Converters containing erbium and titanium are described for the first time. We study the laser heating dynamics of carbon-, titanium- and erbium-containing converters subjected to an incident 980 ± 10 nm semiconductor laser beam. The average powers of laser radiation and mean temperatures corresponding to thermal destruction of each converter are determined. The glow spectra arising under the exposure to semiconductor laser radiation having the wavelength 980 ± 10 nm are obtained for each converter type. The possible medical applications of the studied converters are discussed.

2. Materials and methods

In the present study, we used an Alta-ST semiconductor laser (Dental Photonics, Inc., USA) [21]. The output radiation at a wavelength of 980 ± 10 nm was delivered through a quartz–quartz optical fibre. The fibre core diameter amounted to 400 ± 5 μm, and the total fibre diameter was 440 ± 5 μm without the polymer coating and 475 ± 10 μm with the polymer coating. The average power at the quartz–quartz fibre output reached 25 W. The laser output was a train of 400 μs pulses with a repetition rate of 2 kHz, predetermined by the manufacturer as optimal for the correct operation of the system.

*Presented at the Fundamentals of Laser Assisted Micro- and Nanotechnologies (FLAMN-16) International Symposium (Pushkin, Leningrad oblast, 27 June to 1 July 2016).

A.V. Belikov, A.V. Skrypnik ITMO University, Kronverkskii prosp. 49, 197101 Saint Petersburg, Russia; e-mail: alesch_skrypnik@mail.ru

Received 24 March 2017
Kvantovaya Elektronika **47** (7) 669–674 (2017)
Translated by V.L. Derbov

The Alta-ST system has a unique built-in system for measuring the temperature of the distal fibre end, on which the converter is mounted. The system measures the intensity of thermal radiation resulting from the OTFC heating by laser radiation delivered to the converter via the optical fibre. The thermal radiation propagates through the same fibre but in the opposite direction to the laser radiation. At the fibre input, the thermal radiation is separated from the laser radiation by a spectrum splitter and detected by an FD10D IR detector (Thorlabs, USA), from which the signal is sent to an analogue-to-digital converter (ADC) and then is related to the temperature using a calibration curve. Calibration is performed by comparing the amplitude of the IR detector signal recorded at the ADC output and the temperature at the converter surface measured using a Fluke Ti 400FT thermal imaging camera (Fluke, USA) in the range 300 – 800 °C and using a Promin' pyrometer (Kamenets-Podolsk Instrumentation Plant, Ukraine) in the range 800 – 2500 °C. The minimal temperature that could be measured because of the limited sensitivity of the IR detector and the intrinsic noises of the receiving path amounted to 280 °C. The ADC allowed the temperature to be measured every 30 ms, with uncertainty not exceeding 5%. The measured converter temperature is displayed in real time in a window of the stLase-1.19 programme (Dental Photonics, Inc., USA), the system being connected to a USB port of a PC.

When the temperature value preset in the stLase-1.19 programme was achieved, the Alta-ST system changed the average output power in such a way that the current temperature of the distal fibre end differed from the preset value by less than ± 5%. The control programme of the system also allowed preliminarily specified temporal profiles of the average output power to be formed and the laser heating dynamics for the distal fibre end (or the converter mounted at this end) to be recorded, which is important for the converter formation.

To photograph the converters and to measure their geometric dimensions we used an AxioScope A1 microscope with the embedded Axio Vision rel.4.8.2 programme (Carl Zeiss GmbH, Germany), which allows the size of the object in the field of view of the microscope to be determined with the accuracy up to 1 μm. The average measured size, e. g., the diameter of the converter of each type, was estmated using the data of ten individual measurements. The confidence interval was determined using the StatGRaphics Plus 2.1 programme (Statistical Graphics Corp., USA).

The glow spectra arising in the converters exposed to the Alta-ST laser output delivered to the converter via the fibre, at the distal end of which the studied converter was mounted, were measured in the range 200 – 900 nm using an Ocean Optics USB2000 spectrometer (Ocean Optics, USA). In the process of spectra recording the input end of the receiving fibre of the spectrometer was set perpendicular to the axis of the optical fibre, at the distal end of which the converter was mounted, and the optical axis of the receiving fibre of the spectrometer passed through the centre of the converter. The distance between the plane of the spectrometer fibre receiving end and the axis of the optical fibre with the converter was constant for any type of the converter and amounted to 50.0 ± 1.0 mm. For all spectral measurements, the integrating constant of the receiving system was the same and equal to 100 ms. The spectrum was constructed by averaging the data of ten individual measurements.

3. Carbon-containing optothermal fibre converter

The converter of this type was formed using two-stage technology described in Refs [18, 28, 29]. It was a spherical 3D carbon-containing OTFC [28]. In the process of fabricating this converter, the distal end of the optical fibre was put in contact with the planar surface of the target made of activated coal. The semiconductor laser radiation with an average power up to 2.0 ± 0.1 W propagated along the optical fibre during 0.50 ± 0.01 s and affected the surface of the target containing carbon. As a result, the target was destroyed and the destruction products settled on the surface of the fibre distal end (step 1). Then the distal fibre end was kept free in air, and a semiconductor laser pulse with a maximal average power of 11.0 ± 0.1 W and duration of 1.00 ± 0.01 s propagating twice through the fibre with a pause of 0.50 ± 0.01 s, affected the converter by forming a modified layer (step 2). The characteristic appearance of the carbon-containing OTFC is presented in Fig. 1.

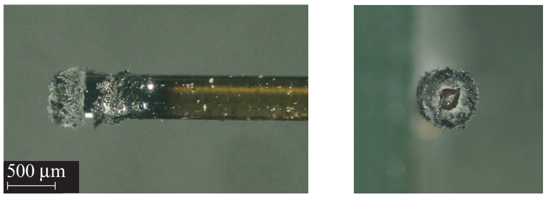

Figure 1. Characteristic appearance of a carbon-containing OTFC.

The diameter of the carbon-containing OTFC is 680 ± 20 μm, its length being 340 ± 20 μm [28]. Its side and end surfaces are coated with a carbon film. In the centre, at the axis of the fibre, there is a transparent 'window' with a diameter of 150 ± 20 μm. The design of the carbon-containing OTFC is described in detail in Refs [28, 30], where it is shown that alongside with the carbon film the converter contains silica, in which the light-absorbing microscopic inclusions are located.

4. Titanium-containing optothermal fibre converter

The converter of this type was formed using the three-stage technology. In the process of fabricating a titanium-containing OTFC, the distal fibre end was immersed by 2 – 3 mm into a cell with TiO_2 powder having a mean particle size of 0.1 – 0.2 μm (ZAO 'Novosibirsk Nanomaterials', Russia). The radiation of the semiconductor laser with a maximal average power up to 12.0 ± 0.1 W propagated through the optical fibre during 0.50 ± 0.01 s, affecting the powder (step 1). As a result, the products of the powder ablation settled on the surface of the distal fibre end. Then, the distal fibre end stayed immersed into the powder, and a specially shaped semiconductor laser pulse with a maximal average power of 11.0 ± 0.1 W and duration 1.00 ± 0.01 s propagating twice through the fibre with a pause of 0.50 ± 0.01 s affected the converter by forming a modified layer (step 2). The modified layer consisted of the mixture of TiO_2 powder and silica weakly bound to the fibre. Then, the laser radiation with an average power of 3.5 ± 0.1 W was incident during 30 s on the converter consisting of the modified layer placed in air (step 3). The oscillograms demonstrating the behaviour of the average output power and the converter temperature in the course of the converter fabrication are presented in Fig. 2. The characteristic appearance of the titanium-containing OTFC is presented in Fig. 3. Its diameter amounted to 780 ± 20 μm and its length was 700 ± 20 μm.

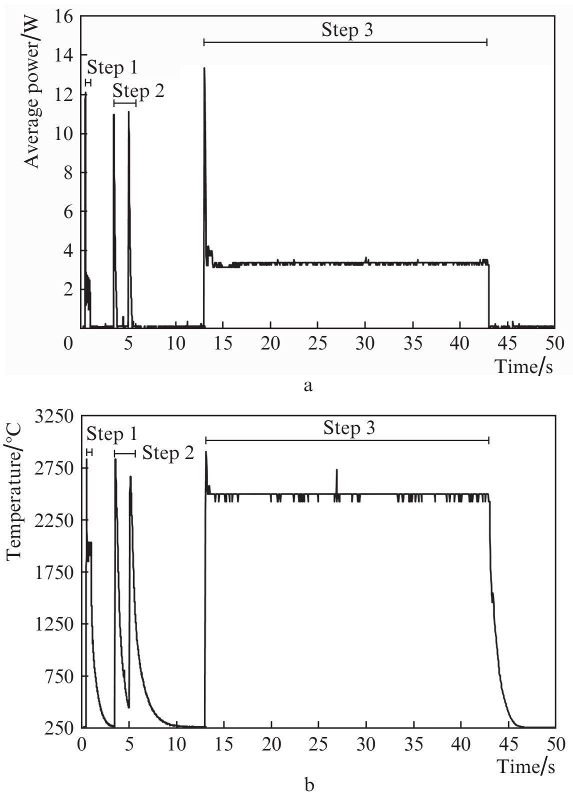

Figure 2. Typical oscillograms of (a) the average output laser power and (b) the converter temperature in the process of the formation of a titanium-containing OTFC.

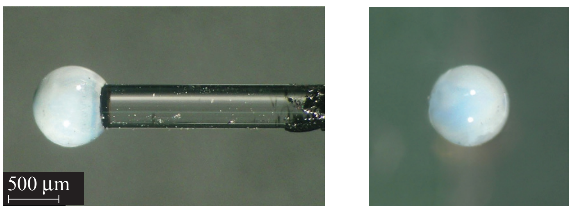

Figure 3. Characteristic appearance of a titanium-containing OTFC.

The titanium-containing converter consisted of titanium oxide TiO_2 sintered with the silica as a result of thermal processing (step 3), which is confirmed by its white colour [31]. We should also note the high mechanical strength of the binding between the converter and the fibre, provided by the sintering procedure (step 3).

5. Erbium-containing optothermal fibre converter

The converter of this type was formed using the three-stage technology. In the process of its fabrication the distal fibre end was immersed by 2–3 mm into a cell with a powder of erbium oxide Er_2O_3 having a mean particle size of 0.5–1.0 μm (ECOM, Japan). The radiation of the semiconductor laser with a maximal average power up to 13.0 ± 0.1 W propagated through the optical fibre during 0.50 ± 0.01 s and affected the powder (step 1). As a result, the powder ablation products settled at the surface of the distal fibre end. Then, the distal fibre end stayed immersed into the powder. A specially shaped semiconductor laser pulse having a maximal average power of 11.0 ± 0.1 W and duration of 1.00 ± 0.01 s propagating twice through the fibre with a pause of 0.50 ± 0.01 s affected the converter by forming a modified layer (step 2). This layer was a mixture of the Er_2O_3 powder and silica, weakly bound with the fibre. Then, the converter consisting of the modified layer placed in air was exposed to laser radiation with an average power of 5.0 ± 0.5 W during 15 s (step 3). The oscillograms demonstrating the behaviour of the average output power and the converter temperature in the process of its formation are presented in Fig. 4. The characteristic appearance of the erbium-containing OTFC is presented in Fig. 5. Its diameter was 890 ± 20 μm and the length was 820 ± 20 μm.

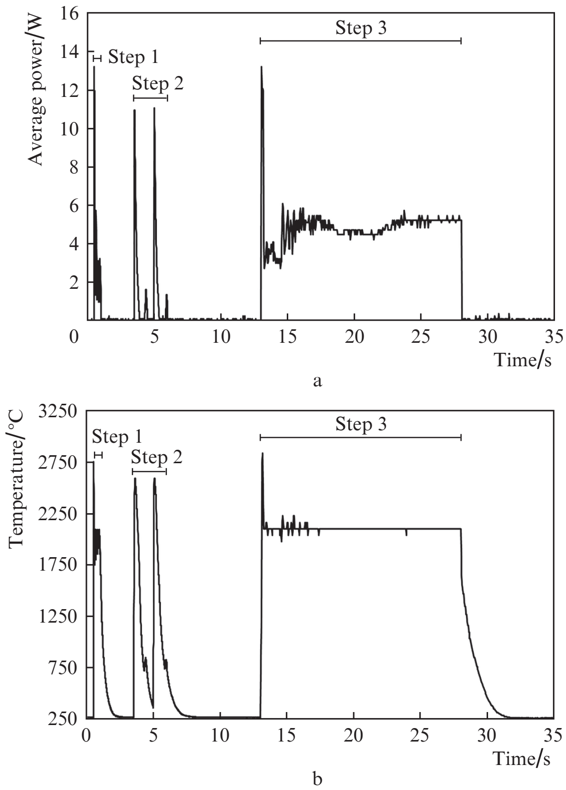

Figure 4. Typical oscillograms of (a) the average output laser power and (b) the converter temperature in the process of the formation of an erbium-containing OTFC.

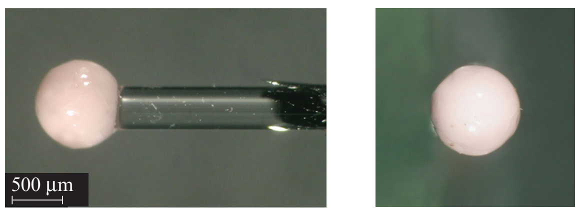

Figure 5. Characteristic appearance of an erbium-containing OTFC.

The erbium-containing converter consisted of erbium oxide Er_2O_3 sintered with silica as a result of thermal processing (step 3), which was confirmed by its pink colour [32]. Note also the high mechanical strength of the binding between the converter and the fibre, provided by the sintering procedure (step 3).

6. Laser heating dynamics of carbon-, titanium- and erbium-containing converters

Typical dependences characterising the heating dynamics of the converters placed in air at different incident average output powers of the 980 ± 10 nm semiconductor laser are presented in Fig. 6.

Under the action of the laser output with an average power of 0.30 ± 0.05 W, the temperature of erbium-, carbon- and titanium-containing converters is stabilised at a level of 400 ± 50 °C, 600 ± 50 °C and 800 ± 50 °C, respectively. At this power, the converters were not destroyed.

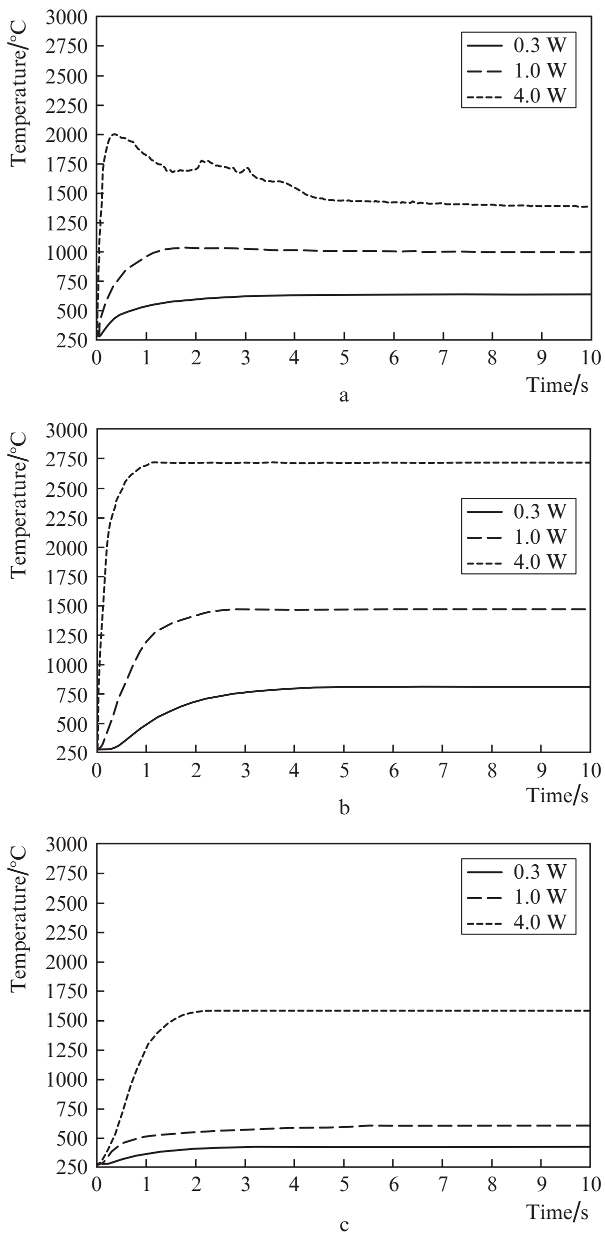

Figure 6. Dynamics of laser heating of (a) carbon-, (b) titanium- and (c) erbium-containing converters at different incident average powers of a 980 ± 10 nm semiconductor laser.

Under the action of the laser output with an average power of 1.0 ± 0.1 W, the temperature of erbium-, carbon- and titanium-containing converters is stabilised at a level of 600 ± 50 °C, 1000 ± 50 °C and 1450 ± 50 °C, respectively. At this power, the converters were not destroyed. At an average output power of 4.0 ± 0.1 W the temperature reached 2700 ± 50 and 1600 ± 50 °C for the titanium-containing and erbium-containing converters, respectively. For the carbon-containing converter the heating dynamics was different: during the first 4–5 s of laser impact its temperature was unstable, which can be explained by the partial destruction of the converter, namely, the loss of the surface carbon film, which was also reported in Refs [28, 30].

The destruction of the converters in air under the action of laser radiation is accompanied by deformation (change of shape) of the converter and leads to the fall of the temperature recorded in the experiment. It is observed at the average power of laser radiation exceeding 10.0 ± 0.1 W for the titanium- and erbium-containing converters, and above 4.0 ± 0.1 W for the carbon-containing converter.

Thus, under the same conditions the minimal heating efficiency is demonstrated by the erbium-containing OTFC, and the maximal efficiency by the titanium-containing OTFC. Both demonstrate higher resistance against the destructive action of laser radiation. The observed differences are obviously related to the difference in absorption properties of the converter materials.

7. Glow spectra of the carbon-, titanium- and erbium-containing converters

The typical glow spectra of the converters heated by laser radiation to temperatures 600–1100 °C, recorded in the range 200–900 nm, are presented in Fig. 7.

For the carbon- and titanium-containing converters the glow spectra in the region 200–900 nm do not contain clearly expressed bands (Figs 7a and 7b). The detected glow of these converters can be associated with the thermal radiation that arises under heating to temperatures of 600–1100 °C. If we assume the converter to be a black body, then, according to Refs [33, 34] the maximum of its glow spectrum will shift with increasing temperature towards the short-wavelength region. This circumstance can explain the growth of the glow intensity in the range 500–900 nm with increasing temperature of the carbon- and titanium-containing converters.

The glow spectrum of the erbium-containing converter, recorded in the region 200–900 nm, is essentially different from that of other two considered converters and includes clearly expressed bands with the maxima at the wavelengths 493, 523, 544, 660, and 798 nm, corresponding to the erbium radiative transitions $^4F_{7/2} \rightarrow {}^4I_{15/2}$, $^2H_{11/2} \rightarrow {}^4I_{15/2}$, $^4S_{3/2} \rightarrow {}^4I_{15/2}$, $^4F_{9/2} \rightarrow {}^4I_{15/2}$ and $^4I_{9/2} \rightarrow {}^4I_{15/2}$, respectively, related to up-conversion [35].

The glow of the erbium-containing converter in the visible region can be used for tissue biostimulation simultaneous with excision, which, according to Ref. [36], makes it possible to reduce the terms of its post-operation recovery. The closeness of this converter glow band having a maximum at 660 nm to the absorption band of radachlorine [6] offers potential applications of the erbium-containing OTFC for photodynamic therapy in oncology, dermatology, etc. The titanium-

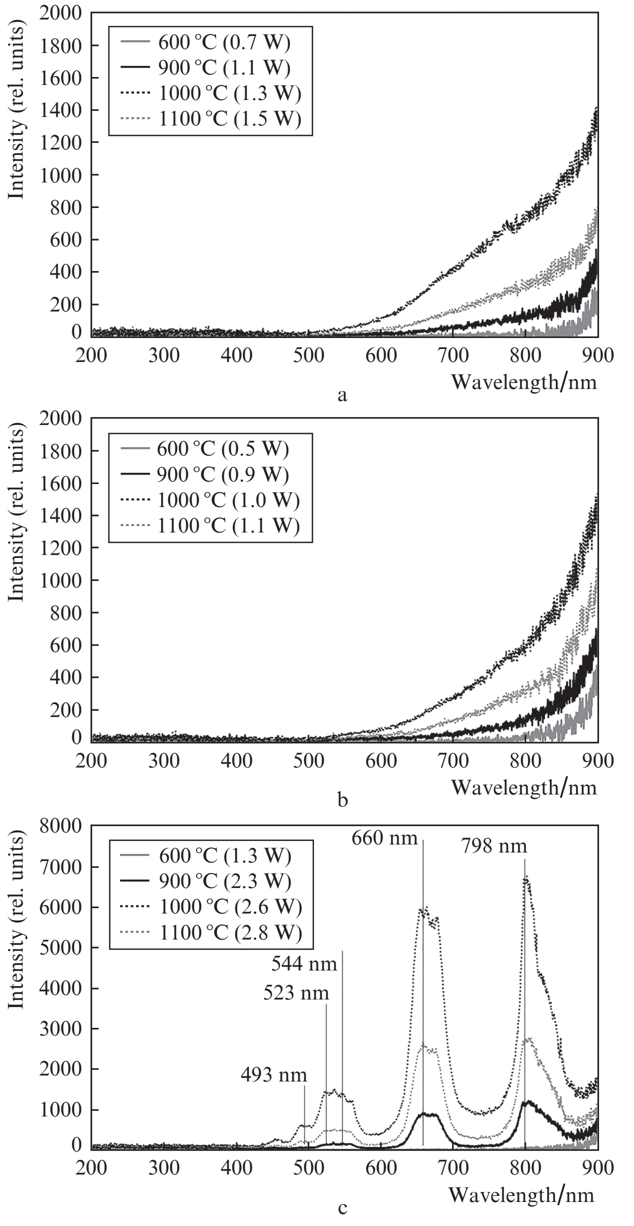

Figure 7. Glow spectra of (a) carbon-, (b) titanium- and (c) erbium-containing OTFCs at different temperatures of the converters and incident average powers of a 980 ± 10 nm semiconductor laser.

and erbium-containing converters can be also used for the excision of tissues in contact laser surgery as an alternative to the carbon-containing converters. Due to high efficiency of converting the laser energy into heat, the titanium-containing converter can increase the efficiency of tissue excision at a comparable laser output power.

8. Conclusions

We have studied carbon-, titanium- and erbium-containing optothermal fibre converters placed at the distal end of the quartz–quartz optical fibre. The carbon-containing OTFC was formed using the two-stage technology, while the titanium- and erbium-containing converters were formed using the three-stage technology. It is shown that the titanium-containing converter is heated most efficiently by 980 nm laser radiation. It is experimentally revealed that the titanium- and erbium-containing converters are more resistant to the destructive action of laser radiation than the carbon-containing converters. It is found that under the action of laser radiation with a wavelength of 980 nm all converters radiate in the range 200–900 nm. Apart from thermal radiation, only the glow spectrum of the erbium-containing converter exhibits clearly expressed bands, the presence of which will allow one to expand the field of its biomedical application. For example, the band with a maximum at 660 nm can be used for biostimulation and photodynamic therapy. The titanium- and erbium-containing converters can be considered as an alternative to the existing carbon-containing OTFC in contact laser surgery.

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