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Modified thin-film fabrication method using vacuum thermal evaporation and vacuum synthesis: application to preparation of Er-doped fiber amplifiers

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ABSTRACT: This paper considers capabilities of vacuum modification optical materials with application rare-earth elements and manufacturing procedures of optical materials with high concentration erbium active ions. Recently, the problem of increasing concentrations erbium active ions in rare-earth elements doped optical amplifiers without decreasing of their quantum efficiency, gets a major actually. Achievement in the field of ionic-plasma vacuum processing techniques allows practical possibility to establish new generation effective optical amplifiers with extremely high (> 10^{20} sm^{-3}) concentration of rare-earth elements. On basis the technology of vacuum thermal deposition and vacuum synthesis, Er_2O_3 - SiOx - Al_2O_3 multicomponent planar light guide has been manufactured. The multicomponent planar light guide is created on local segment of an optical fiber core. The erbium ion concentration spans from 10^{19} to $8 \bullet 10^{20}$ cm^{-3} in generated optical structure. In experimental thermal deposition processing vacuum chamber parameters did not change. From given experimental results we can consider that, using different impurities SiOx and Al_2O_3 allow reaching high concentration free erbium ions. Spectral characteristics of obtained optical structure with active rare-earth elements provide photoluminescence in the range ~1550 nm. The proposed method allows actualizing local modification of the single-mode optic fibers for the purpose of fabrication of short-distant fiber optical amplifiers, sensors and modulators.

KEYWORDS: fiber amplifiers, planar light guide, optic fibers, rare-earth elements, vacuum thermal deposition, multicomponent films.

1 INTRODUCTION

The problem of obtaining short-distant erbium fiber amplifiers stimulates designing the materials with extremely high contents active ions of erbium. In the pure film of rare-earth oxide Er_2O_3 the concentration of Er ions reaches values ~10^{22} cm^{-3} [1]. That value exceeds the concentration Er in the standard systems alloyed by erbium and allows reaching the extremely high quantity of radiating centers. For realization short-distant amplifiers on basis active fibers it is necessary increasing the concentration of active ions in the glass without considerable deterioration of magnifying properties of the active medium. The purpose of work is modification a section of optical fiber by erbium obtaining multicomponent films Er_2O_3 - SiO_x - Al_2O_3, using the method of vacuum thermal evaporation from erbium and a silicon monoxide. The active section of fiber will have essentially smaller length, than in common fiber-optical amplifiers due to higher contents active ions of erbium in the modified field of the lightguide. In this case we are using the low temperature vacuum methods of synthesis glasses without any heating the glass of fiber.

For creating high-effective light emitting structures it is necessary, that concentration of erbium exceeded 10^{18} cm^{-3}. It is known, that erbium ions clustering is a main factor responsible for deterioration of amplification properties erbium doped optical guides [1], [2].

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At major concentration active ions (> 10^{18} cm^{-3}) it is possible generating clusters, consisting of two and more erbium ions. That becomes to the considerable decrease the quantum efficiency of luminescence the laser transition ($^4I_{13/2} \rightarrow {}^4I_{15/2}$) because of effect up-conversion.

This effect is called by a pairwise interaction closely located ions. There are simultaneously locating in an excited state $^4I_{13/2}$. One of the ions without radiation relaxing to the ground state $^4I_{15/2}$ and another one drives higher energy level $^4I_{11/2}$ [3], [4]. Thus, the part of erbium ions absorbs the radiation of amplified signal and reduce efficiency the fiber amplifier.

In cases of high concentration erbium ions there are existing two methods for struggle against effect of up-conversion. The first method consists in addition of Al_2O_3 in array SiO_2. Impurity of Al_2O_3 changes the structure of glass and reduces the rate of migration excited ions Er^{3+} and positive influences erbium miscibility [5]. The second method uses the low-temperature (without melting) vacuum synthesis of glass at which activator ions has flown down uniformly in array of glass and erbium clusters are not formed. It is obvious, that the efficiency is given by integrating of these two procedures.

2 DESCRIPTION OF EXPERIMENTAL METHOD

In this work the technical implementation of a method preparation optic fiber to alloying by rare-earth elements was shown. This method consists in abrasive polishing on preset thickness the optic fiber with big radius of curvature (R = 180 mm), the control of polishing thickness operates from recording the laser radiation which where entered into the fiber. Polishing stops, when the local section of a fiber is a baring to the core. This moment is defined from the intensity degree of laser radiation at the fiber output.

For deposition of silicon dioxide using the method of electron beam evaporation, that strongly complicates the equipment. Therefore, the technology of silicon dioxide deposition with evaporation silicon monoxide SiO has been applied. The silicon monoxide is easily enough volatilized from a resistive heater boat, unlike silicon dioxide SiO_2. When oxygen is bleeding in working space (at pressure $2\text{-}3\cdot10^{-3}$ Pa), molecules of a volatilized silicon monoxide steam oxidize to silicon oxide (SiO_x), where $1<x<2$.

For obtaining multicomponent Er_2O_3 - SiO_x - Al_2O_3 films on the fragment of optical fiber was applied the technology of vacuum thermal co-deposition with bleeding in oxygen in vacuum chamber. There were using two tantalum boats resistive heating for co-evaporating metal-erbium, a monoxide of silicon and aluminum. The main parameter at the choosing erbium-doped materials suitable for effective light amplification in 1540-1550 nm bandwidth, is ability isolating Er^{3+} ions from each other for attenuation a concentration quenching. As is known, additional doping in quartz glass Al_2O_3 or P_2O_5, increases a concentration limit of entering oxides of rare earth elements [6].

In the current work it was co-doping erbium oxide (Er_2O_3) with using aluminum oxide (Al_2O_3). Al_2O_3 provides magnify refraction index of evaporated film. In our case, it is the positive factor, as it is necessary for "pulling out" optical modes from a fiber core for interaction with erbium ions in the evaporated film. Decreasing the clustering of erbium by co-doping of aluminum oxide promotes reduction in tens times overall dimensions of fiber amplifiers. The additive 2,5 mol.% Al_2O_3 to SiO_2 allows to increase concentration erbium oxide in dielectric layer from 0,1 to 1 mol.% [7]. It has been shown [8], that by using the method of vacuum controllable deposition with reactive gas (oxygen) it is possible to receive amorphous film structures with the big concentration of erbium in which ions Er^{3+} appear uniformly distributed in a glass array.

Application of technology vacuum deposition materials do not allow to formation clusters of erbium, caused by small mutual solubility of oxides of erbium and silicon in melts. Clustering repressing at low-temperature vacuum synthesis occurs at the expense of statistically uniform hit of the molecules, making an optical guide, from a gas phase on rather cold substrate in conditions when migration of particles in material volume is complicated. It is possible to name such glasses structurally unstable. Their annealing with further melting conducts to catastrophic increase the up-conversion, and at the big concentration of erbium to fibration of phases with radical falling quantum efficiency of excitation the main laser transition in Er^{3+} [9].

At sedimentation of multicomponent films in vacuum the control of thickness of evaporated materials and concentration of alloying dopants is very important. In this experiment the radio-frequency method has been applied. This method is grounding on measuring the frequency vibration of a quartz crystal of evaporated material on it [10], [11]. As bottom of quartz measurer serves the plate of the quartz resonator defining frequency of the stable self-oscillator. The generated frequency depends on mass of the quartz resonator, it is possible to define thickness of the film which have deposited on a quartz plate.

Accuracy of a method measuring mass of deposited layer with application the quartz resonator in many respects is defined by stability of generator frequency included in the schema quartz resonator. For example, for a crystal with

resonance frequency f = 5 MHz, mass m_g = 100 mg sensitivity ($\Delta m_c / \Delta f$) is equal 0,02 mg/Hz. Registering a frequency drift of the quartz resonator on 1 Hz, it is possible to check a mass gain in a microgram. The range of gauged depths for serial devices lays in limits from 10 to 10000 nm with accuracy 5-10% [12]. The method of the quartz resonator allows adjusting a procedure of manufacturing of films in advance given depth.

The most powerful sources of an error at measuring are the methodical (multiplicative) error and an error from temperature action. Existence of a methodical error is caused by that the sensor and a metallic surface with fragment of fiber optic are in various points of vacuum chamber. In this operation the methodical error is compensated by means of optimization of a standing of sensors and the account of their angular arrangement concerning evaporators (see figure 1).

Two boats of resistive heating have been applied to controllable evaporation of deposited components of a film. Between boats there is a screen from a foil, arranged along a line (AB), pairing a centre point between evaporators and the middle of a processed optical fiber. The foil does not allow the evaporable material to get from one heater on the sensor, which checking evaporation from the second resistive heating boat.

Quartz plates of thickness gauges are arranged under angle in 30º from a line (AB). It is known, that density of a deposited film inversely proportional to a cosine of drift angle from a line (AB) and to a quadrate of distance from a radiant of the evaporated material. The processed surface of a fiber optic and two quartz plates of thickness gauges are arranged on identical distance from boats of evaporators. Therefore, if to consider a cosine of drift angle from a line (AB) it is possible to check precisely enough a relation of the evaporated components on a fragment of an optical fiber.

As crystal of quartz are sensitive to temperature changes, in this procedure the cooling system for data sensors is used. Compensation the influence of a thermal radiation from the evaporator on a resonator frequency drift in the evaporation process allows to measure thickness of the deposited layer with accuracy about ~ 5%. The device for vacuum adjustable evaporation is simultaneous from two resistive heating boats is shown on figure 1.

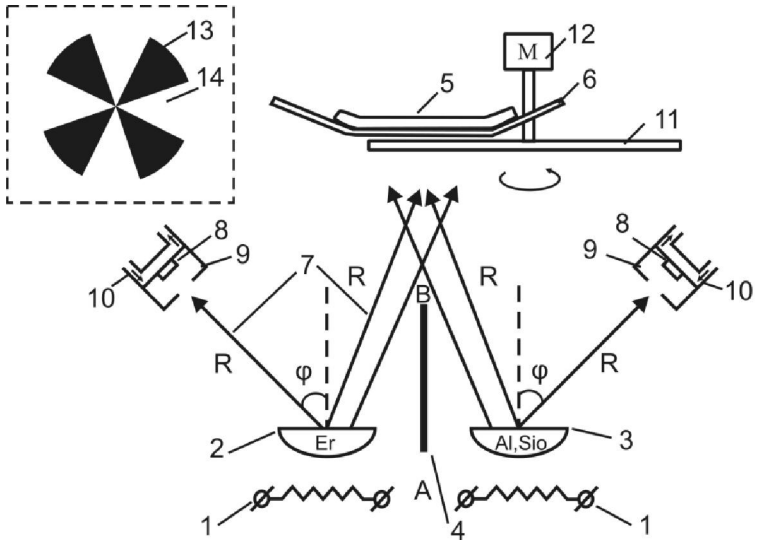

Fig. 1. Equipment for vacuum controllable deposition of multicomponent films

1 – the resistance tantalum radiator; 2 – the container with metal erbium Er; 3 – the container with metallic aluminum Al and with monoxide of silicon SiO; 4 – the shield parting compartments (AB); 5 – a metal background; 6 – an optical fiber fragment with a open core section; 7 – directions of evaporation steams of materials from containers; 8 – quartz plate; 9 – covering diaphragm; 10 – a water radiator; 11 – a disk - an obturator with four gashes; 12 – the motor with a gyration frequency control; 13 – a disk general view - an obturator; 14 – the gauged triangular gash on a disk.

Evaporation of erbium and silicon monoxide were spent on vacuum installation by means of heating of different tantalum boats at pressure of working gas (oxygen) 0,25-0,35 Pa (about $3 \cdot 10^{-3}$ mm Hg) and temperature 1300-1600 ºC.

In the first container (2) has been seated metal erbium in additive 5 mol.%, from total quantity of evaporated substances. In the second container (3) the powder of silicon monoxide and aluminum bronze are placed simultaneously, in a prospective ratio for process vapor deposition a films. Obtaining of films SiO_x - Er_2O_3 different composition was carried out at changing the rate of evaporation erbium, whereas the rate of evaporation silicon monoxide and aluminum was supported at constant value. The rate of evaporation erbium, silicon monoxide and aluminum was fixed by means of two quartz resonators (8). Speed of deposition of a film varied from 5 to 20 nm/sec. The disk-obturator (11) rotated with frequency 3000 sec^{-1}. This method has allowed receiving planar optical fiber with deposited components in the form of a wedge with good indexes of geometry.

The method of controllable vacuum deposition has allowed providing the precise control composition of glass on planar light guide, and also possibility to create high concentrations of alloying additions (18 mol.% Al_2O_3 and up to 1 mol.% Er_2O_3) [13].

In our experiments of vacuum deposition the temperature of planar light guide carrier was supported with accuracy ±1 % by means of the radiator and system of a feedback on the basis thermoelectric couple. The stabilized temperature of a planar light guide carrier ensures equal requirements for deposition silicon, aluminum and erbium oxides on a surface planar light guide. It means that glass on a bearing area is shaped directly of a deposited phase, passing the melting stage. The planar wave guide made by an offered technique (5) represents a thin film on a planar light guide carrier which on the end is narrowed (see Figure 2). Laser radiation (4) propagates on a core (1) of single-mode optical fiber. The cladding of an optic fiber (3) is removed by means of absorption polishing from a local fragment of a fiber. The refractivity index of the deposited multicomponent film reaches 1,54-1,56. It allows to "pump out" effectively energy from a core of (1) single-mode fiber. For the cuneiform geometry of a film (7) the significant part of radiation (8) is returned in a core (1) after interaction with the rare-earth components. The rare-earth film optically have been pumped by external radiation (10) (λ = 980 nm), providing signal gain. The electromagnetic wave (6), falling on narrowed edge (7), continues to propagate in zigzag trajectory. In result, in some point, the angle becomes less critical and optical radiation (9) starts over again to get to a core of an optical fiber (1).

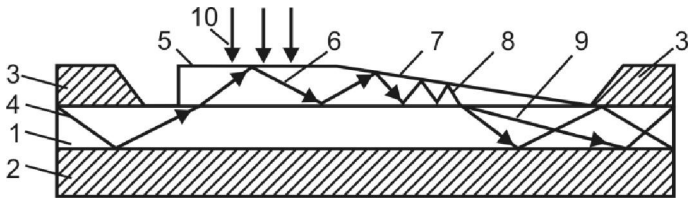

Fig. 2. A thin-film planar lightguide with the narrowed edge, generated on a fragment of a core of an optical fiber

1 – an optical fiber core; 2 – an optical fiber cladding; 3 – an optical fiber cladding, with a remove fragment; 4 – the radiation in a fiber; 5 - the generated planar optical fiber; 6 – the path of radiation in a planar optical fiber; 7 – cuneiform narrowing; 8 – the path of radiation in cuneiform narrowing; 9 - the radiation which has come back to a core; 10 - the radiation of optical pump (λ = 980 nm).

Efficiency of the matching can reach 65-75 %. Thickness of a film is picked up in such a manner that allows to define distribution of a path of incident beam, therefore efficiency of radiation returned in the fiber can be big enough and provide gain of a signal by erbium on 5-10 dB from a site in 2-5 cm.

3 EXPERIMENTAL RESULTS AND DISCUSSION

The experiment results on modified optic fibers have shown the photoluminescence on wavelength 1540-1550 nm. A spectrum is characterized by the basic peak on wavelength ~1540 nm. Measurements were spent at indoor temperature (T=300 K) and without any preliminary annealing. It points possibility of activation erbium ions (Er^{3+}) without using high-temperature operations. The observable result can be explained as higher concentration of optically active ions Er. The form of the received spectrums will be coordinates with literary data [14], [15].

The spectral characteristic of multicomponent film Er_2O_3 - SiO_x - Al_2O_3 are shown lower in figure 3.

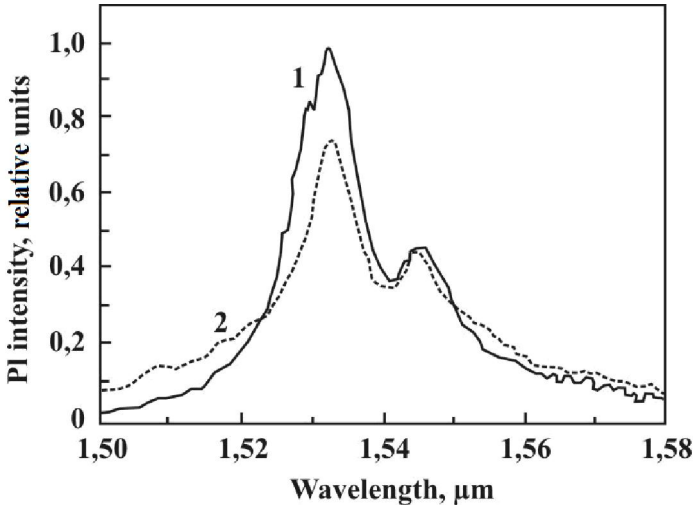

Fig. 3. Spectral characteristic of a film (Er_2O_3 - SiO_x - Al_2O_3) on the fragment of optical fiber

1 - optical medium alloyed 10 mol.% Al_2O_3 and 3000 ppm Er_2O_3, 2 - optical medium alloyed 1,5 mol.% Al_2O_3 and 3000 ppm Er_2O_3.

In experimental equipment as the signal source semiconductor laser with the peak output power 5 mW and λ=1540 nm was used. In the capacity of the pumping source the output power up to 250 mW semiconductor laser with fiber yield and λ=980 nm was applied.

Quantitative limiting concentration Er_2O_3 in glass SiO_2-Al_2O_3 defined by measuring results the efficiency conversion of a pumping radiation in a signal (hereinafter efficiency of amplification). The higher amplification of samples (rather to light guide length) in comparison with the light guides alloyed by MCVD technology, shows, that Al_2O_3 at vacuum deposition allows to reduce clustering of ions Er in a quartz glass. The given method allows precisely controlling the concentration of dopants in glass, and creates demanded refractive index [7], [13].

Value of losses modified planar light guides has 100 dB/km, in this case at 10 cm length their influence on efficiency of the laser makes no more than 0,001 %.

It is established, that vacuum synthesis at moderate temperatures in a vacuum bench (150-200°C) allows to receive planar light guides with concentrations up to $8 \cdot 10^{20}$ cm^{-3}.

The following stage of experiment includes an experimental research modified optic lightguide on temperature operations. Figure 4 represents effects of heating the samples in a muffle furnace and some variations in a photoluminescence of ions Er.

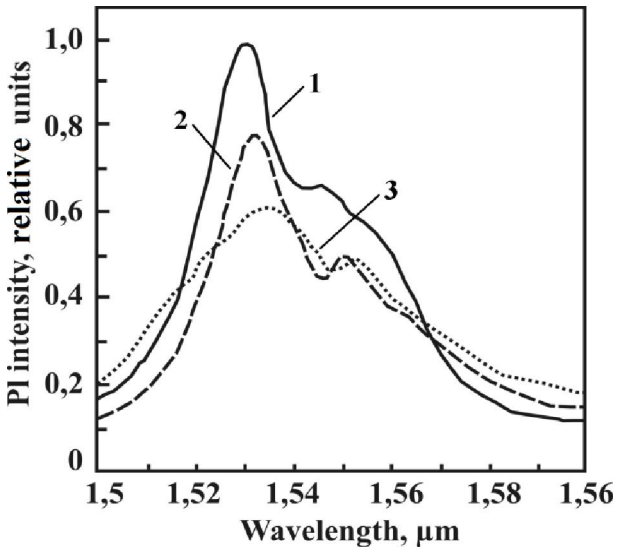

Fig. 4. Spectral characteristic of a film (Er_2O_3 - SiO_x - Al_2O_3) on the fragment of optical fiber in heating

1 - without heating, 2 - with heating at 400 °C, 3 - with heating at 700 °C.

As seen in Figure 4 the glass emolliating calls erbium clustering that has led to wide a luminescence spectrum. On basis the spent measurements the dependence of quantum efficiency of samples modified optical fibers on erbium ions concentration has built (Table 1).

Table 1. Results of measuring quantum efficiency active planar light guide from erbium ions concentration with various impurity densities Al_2O_3

Er_2O_3 ppm / Al_2O_3 mol.%	Amplification efficiency in relative unit								
	10	**50**	**100**	**250**	**500**	**1000**	**3000**	**5000**	**10000**
0	1	0.77	0.61	0.38	0.07	–	–	–	–
1.5	1	0.94	0.92	0.79	0.60	0.29	0.02	–	–
2.5	1	0.98	0.96	0.92	0.80	0.69	0.30	0.13	–
10	1	0.98	0.96	0.94	0.85	0.73	0.34	0.15	0.03
15	1	1	0.98	0.96	0.91	0.81	0.43	0.23	0.10
18	1	1	1	0.98	0.96	0.88	0.56	0.32	0.18

In the table results of measuring the efficiency of amplification samples modified optical fibers, in relative units are given. For relative units received the highest amplification at the maximum doping by aluminum oxide and at optimum doping by erbium oxide [15]. A condition of deposition of films was identical, at various concentrations of aluminum oxide and erbium oxide, with the control of composition of films. In spite of the fact that at high concentration of aluminum oxide the relative efficiency of amplification decreases, it is possible to reduce length of the planar fiber amplifier to 5-10 cm at the expense of the high considerable quantity of erbium ions radiating centers.

4 CONCLUSION

Possibility of direct vapor deposition rare-earth oxides allow to magnify to the maximum limit concentration of active ions erbium. In rare-earth oxide concentration Er^{3+} reaches value ~10^{22} cm^{-3}, that in many respects surpasses value of concentration of the erbium concentration received by other doping technique. Ability of vacuum methods uniformly to precipitate and in co-evaporate any oxides of rare-earth and other elements in the form of free ions, possibility the control of thickness of a deposited film allows to magnify to a limit of concentration of active ions rare-earth elements, thereby to reach giant quantity of the radiating centers.

The magnifying of concentration of erbium allows reducing a length of an active section in erbium in rare-earth elements doped optical amplifiers. Possibility of obtaining active waveguides with extremely high contents of erbium stimulates development new short - distant optical amplifiers on the basis modified optical materials. Besides, the properties of erbium oxide (Er_2O_3) as, a tall transparency in wide area of an IR-range, the big index of refraction and photoluminescence properties, do its perspective for application in photonics, integrated optics and telecommunication.

The yielded device can be used for gain and processing of optical signals, for example, in integral-optical switches, modulators, amplifiers and control sensors.

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