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Method of ION-PLASMA Doping of Optical Fiber

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ABSTRACT*:* The work evaluates the possibilities of ion-beam and ion-plasma alloying of quartz glass and optical fibers. The introduction provides general theoretical information on ion doping methods, as well as radiation defects, the structure of defects, and the channeling effect arising from ion doping.

The main part describes ion-beam and ion-plasma methods of doping optical materials used in fiber-optic information transmission lines with various elements under vacuum conditions. To achieve this goal, the depositions of coatings in reactors at atmospheric and reduced pressure, as well as doping of optical materials and fibers in vacuum are considered. The basis of these methods is the introduction of dopants of rare-earth elements by low-energy ion bombardment with directed beams or ion-plasma treatment in a gas discharge.

However, the method of ion-beam doping is not without its disadvantages. The solution to this problem was achieved by using a standard “Vesuviy”-type ion implantator with a modernized ion source for ion-beam doping, and a “UVN-75R” universal vacuum post with special devices for ion-plasma doping.

In this regard, a method has been developed for the ion-plasma doping of optical fiber used in fiber-optic information transmission lines. As a result of using this method, an increase in productivity and a simplification of the design is achieved in comparison with the previous method.

KEYWORDS*:* optical fiber, vacuum, ion, doping, quartz, spectrum, erbium.

1. INTRODUCTION

Ion doping has significant advantages over traditional methods of manufacturing electronic devices: the possibility of introducing an impurity of any element into the material; low-temperature conditions of the process; relatively low annealing temperature (practically not affected by the temperatures at which the diffusion redistribution of the impurity in the material occurs); the possibility of using as an alloyed material any material for which conventional methods of doping are either unsuitable or difficult (for example, diamond, etc.); conducting local doping with "small" dimensions of the doped area. The ion doping process allows you to accurately dose the amount of impurity introduced, regulate the energy of the introduced ions, and control the depth of their penetration and distribution profile, i.e. the thickness of the doped layer [1-3].

The doping process includes the creation of vapors of the working material with a pressure of about 1 Pa, the supply of vapors to the ion source, partial ionization of vapors, ion extraction and formation of an ion flow, mass separation of ions, acceleration of ions to an energy of 20 keV - 2 MeV, processing of the surface of the material by the ion flow for a given time [4].

Since the introduction of ions is carried out in a vacuum (5.10^{-4}–5.10^{-5} Pa), the process virtually eliminates contamination of the alloyed material with foreign substances. The ion doping process is characterized by high productivity, reproducibility and stability, which leads to a reduction in the cost of products, manufactured using it. In some cases, the ion doping method provides a significant improvement in the parameters of devices and increases the yield of usable products.

A technological process that combines ion doping with non-thermal annealing of the doped layer as a result of laser, electronic or pulsed light irradiation has a perspective. It can be argued that this technological process of ion doping occupies a leading position in the research and production of electronic devices for various purposes.

The physical basis of the process of ion doping of electronic equipment materials consists in the distribution of doping ion runs in the substrate material, the formation and distribution of radiation defects, etc.

The concentration of the embedded impurity n at the depth R_p, with a Gaussian distribution of ranges, is determined by the relation

ISSN: 2350-0328
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Vol. 7, Issue 11 , November 2020

$$n = N_d / 2{,}5\Delta R_p \quad (1)$$

where N_d is the number of embedded ions per unit area.

Formula (1) gives, in the first approximation, the value of the maximum concentration of intercalated dopant ions. The maximum concentration $\Delta R_p << R_p$ at is determined by the ratio

$$N_{max} = N_d / \sqrt{\pi \cdot \Delta R_p}$$

The concentration of the dopant as a function of the depth of the doped layer can be determined by the relation

$$N(x) = N_{max} e^{-(x-R_p)^2 / \Delta R_p^2}$$

where x is the depth of the doped layer in the direction normal to the substrate surface.

Radiation defects. During deceleration, depending on the energy and mass of implanted ions, as well as on the mass of the substrate atom, the ion knocks out a certain number of atoms from the crystal lattice sites of the substrate. These atoms, in turn, displace other atoms, causing a cascade of collisions, which leads to the accumulation of vacancies, interstitial atoms and their components near the ion trajectory. With an increase in the dose of implanted ions, the disordered regions overlap, forming an amorphous layer of a certain thickness. The number of radiation defects and their distribution depend on the mass of the implanted ion, doping temperature, energy and dose.

The structure of the defects. The simplest defects are created when the lattice atom is displaced into the internode (Frenkel defects) with the formation of a vacancy and an internode atom (see Fig. 1).

The dose of amorphization is determined from the ratio

$$N_*, d = 2E_d N/(dE/dx)_n,$$

where N is the density of the substrate atoms; $(dE/dx)_n$is the energy loss in a nuclear collision per unit length; E_d is the effective threshold energy of the lattice atom displacement.

The number of displaced atoms per incident ion in a primary collision is determined from the ratio

$$N_{d,p} = E/2E_d.$$

For silicon $E_d \approx 14$, for other semiconductor materials $E_d \approx (8 \div 30)$ eV.

The total number of atoms displaced by a single ion is determined from the ratio

$$N_d = E_n/2E_d,$$

where E_n is the total energy lost by a particle in primary and secondary collisions with the nuclei of atoms.

The effect of channeling. Since the elements of the single-crystal structure of the substrate are arranged symmetrically, the alloying ions move along the crystallographic directions with small indexes (for example, <110>, <111>, <100> for a diamond-type lattice), they penetrate deeper into the crystal than when moving in other directions or when moving in an amorphous body. In this example, the doping ion moves as if in a channel in which there are no nuclear collisions and all the braking processes are caused only by electronic collisions. In this case, the mileage of the alloying ion is proportional to the speed. The angle at which the ion enters the channel is called critical (see Fig. 2). In order for the ions to move, they must enter the channel at angles of some critical ψ_c value.

The critical angle depends on the type of substrate material, the energy of the alloying ion E, and the crystallographic direction. Minimal channeling is observed at angles of 7-10°. To eliminate the effect of channeling, the substrate is tilted to the axis of the crystal at an angle of no more than 7°. Completely eliminate the channeling effect is not possible either by doping at an elevated temperature, or by disorienting between the axis of the crystal and the direction of the ion beam. This can only be achieved by pre-amorphizing the doped layer.

ISSN: 2350-0328
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Vol. 7, Issue 11 , November 2020

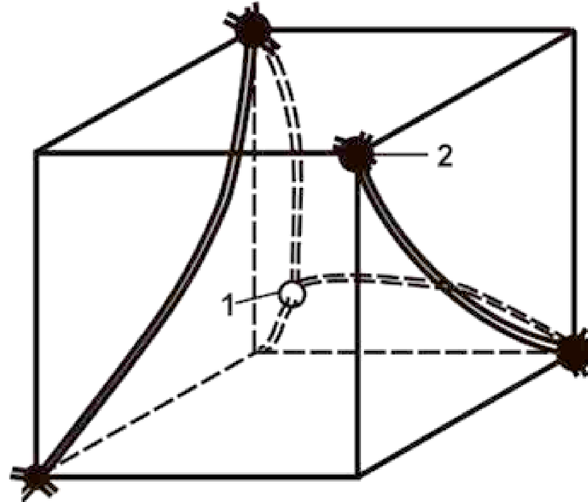

Fig. 1. **Frankel Defect: 1-vacancy, 2-interstitial atom**

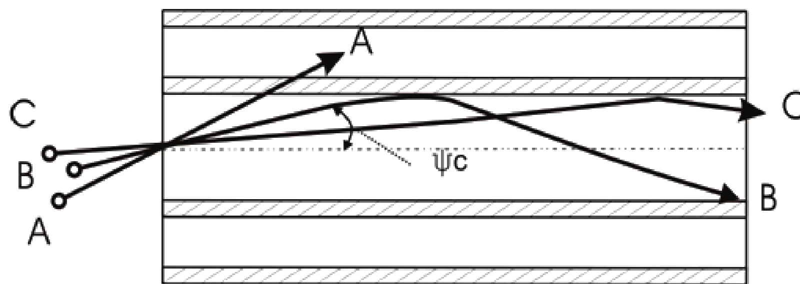

Fig. 2.**Scheme of the channeling effect: ψ_c– critical angle of channeling, A – non-channeled ion, B,C – channeled ions**

II.MAIN PART

Evaluation of the possibilities of ion doping of quartz glass and light guides shows the following:

1. For ion doping with rare earth elements, it is necessary to design and manufacture a special ion source using vapors of rare earth materials (REM). There are no standard sources for such elements. To solve the problem of REM evaporation from the crucible or the presence of a vaporized erbium compound, it is necessary to modify the standard sources of plasma-type ions in “Vesuviy” series installations.
2. Standard technological devices for ion doping are designed for group or individual processing of the front surfaces of products such as washers (silicon plates with a diameter of up to 150 mm) with a total processing area of up to 1-1.5 m^2 per cycle. Special equipment is required for ion irradiation of internal parts of products, such as quartz tubes. In this case, the doping will occur at an acute angle to the surface.
3. Estimation of the depth of penetration of erbium ions in the near-surface layer of quartz glass (without taking into account possible diffusion) by calculation methods [3] shows that at energies of erbium ions up to 100 keV, the maximum penetration will not exceed 140 nm, and at energies up to 300 keV, respectively, 380 nm, for 1 MeV will be about 1.1 microns. The average run of the ions will be about two times lower than the values shown. Taking into account the real capabilities of the process equipment (ion energy of more than 80 keV), estimates of the run of erbium ions in materials such as silicon, aluminum and tantalum oxides will exceed 50-70 nm at normal incidence on the surface. The dose of irradiation with erbium ions is practically limited to values of the order of 10^{16}-10^{17} ions/cm^2,

ISSN: 2350-0328
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Vol. 7, Issue 11 , November 2020

which makes it possible to obtain very high concentrations of impurities (over 10^{21} cm^{-3}). With repeated use of ionized ions, the depth of runs increases accordingly, but the processing intensity decreases.

4. There is also the option of the erbium doping of the surface layer of glass which comprises applying a thin coating of surface erbium or arista rain material (the coating thickness is 10-100 nm) and ion bombardment of the surface by a stream of argon, krypton or xenon doses of about 10^{16} cm^{-2} for embedding the cover in the surface layer of glass. There is no need to use special sources of erbiumions.

A. Deposition of coatings in atmospheric and low-pressure reactors

Taking into account the technological features of obtaining a product (a quartz tube doped with erbium on the inner surface), the main task is to obtain an erbium-doped layer on the surface and in the near-surface zone of silicon dioxide. In such a system (extended tube), doped coatings can be obtained, for example, by pyrolytic deposition in atmospheric pressure reactors.

In this case, the inert carrier gas is passed, for example, through a liquid source containing the desired proportion of ethyl silicate and erbium containing material. Spent vapors are passed through the liquid to prevent reverse diffusion of oxygen from the atmosphere.

Deposition of dielectric layers by pyrolysis methods is carried out at relatively low temperatures (300-700°(C)in standard heating installations, such as monosilane and oxygen. When using gas lines of ammonia, mixtures of monosilane with argon and oxygen, it is possible to precipitate silicon oxynitrides (up to 10% of silicon nitride in silicon dioxide).

Various methods of obtaining thin layers of silicon dioxide by plasma discharge are also known, in particular [5-6]:

- the precipitation of the inorganic compounds in the interaction of monosilane or its derivatives with oxidizing agents,
- deposition of layers by decomposition of organosilicon compounds in an inert or oxidizing atmosphere,
- plasma anodizing of silicon coating in a dry or wet atmosphere.

Monosilane is usually used as the starting material, and nitrous oxide, oxygen, nitrogen oxide, and carbon dioxide (mainly nitrous oxide) are used as oxidants.

Various designs of reactors are used for deposition of silicon dioxide layers, in particular "cold wall" reactors that provide the following parameters of the coating process. Temperature 200-300°C, pressure 14-133 Pa, power 250 W, power density 0.1 W/cm2, working gas flow 1-3 l/min, deposition rate 30-60 nm/min, impurity concentration up to 4%.

B. Doping of optical materials and fibers in vacuum

The development of technology for the production of optical fibers with low attenuation and high information throughput has led to their widespread use in communication and information processing systems. The use of single-mode fibers makes it possible to create transcontinental fiber-optic information transmission systems (FOTS), while the creation of optical amplifiers on active optical fibers doped with rare earth element ions (REM) allows solving the problem of reducing intermediate repeaters and developing high-power laser radiation sources compatible with FOTS.

High-energy (E=3.0-5.0 MeV) vacuum methods of optical fiber doping were used as a priority for research purposes. However, it is advisable to develop methods for doping optical fibers with various impurities in a vacuum due to the purity of the ion doping process and the ability to control the technological parameters of doping in order to vary the dose and depth of introduction of impurity additives. The closest in technical essence to the developed method of doping an optical fiber is a method of doping an optical material, particularly a silicate glass with erbium ions with energies in the range 3.0-5.0 MeV, with doses of $0.6\text{-}1.2 \times 10^{16}$ions/$cm^2$ at a temperature of 300°C [7], as a result of this implantation, the concentration of erbium can reach up to 0.2% at depths of ~1.0 to 1.2 microns and the method of doping with erbium ions with energy of 3.5 MeV, the dose and 3×10^{16}ions/cm2 of single crystal $LiNbO_3$ with subsequent annealing at 1060°C with a concentration of up to 0.18% [8].

With these methods, large concentrations of evenly distributed impurities can be obtained, the amount of doped impurities can be predicted and accurately controlled, and the distribution profiles of the introduced impurities can be adjusted. The main disadvantages of this method are the following:

1. This method requires a high-energy ion accelerator with ion energies of several MeV.
2. Such accelerators have very small ion beam sizes, so it is almost impossible to use them for processing large surfaces, even if the ion beam is scanned.

ISSN: 2350-0328

International Journal of Advanced Research in Science, Engineering and Technology

Vol. 7, Issue 11 , November 2020

3. The density of the ion current in such accelerators is within tenths of a microampere per square centimeter, so a very long processing time is required to obtain a sufficiently high doping concentration.

4. Since high-voltage ion accelerators are quite expensive and cumbersome experimental installations, this method is also very expensive, non-technological, difficult to control and can be used for purely research purposes, for example, to study the effects of doping REM optical materials, in particular, the phenomenon of photoluminescence, the formation of radiation defects, spontaneous emission, etc.

Based on the above, the task was set to create ion-beam and ion-plasma methods for doping optical materials used in fiber-optic data transmission lines by various elements in a vacuum. The methods are based on the introduction of alloying admixtures of REM by low-energy ion bombardment with directed beams or ion-plasma treatment in a gas discharge [9].

The problem is solved by using a standard “Vesuviy”-type ion implantator with an upgraded ion source for ion-beam doping, and a universal vacuum post "UVN-75R" with special devices for ion-plasma doping.

When ion-beam doping of materials, the introduction of rare-earth element ions to a given concentration is performed by ion implantation using ion beams with an ion energy of 75-150keV and an ion current density of 2-5mkA/cm^2. To dope extended samples, they are placed in a rotating container, and ion implantation is performed by scanning with an ion beam. The “Vesuviy” implantator is currently widely used for processing semiconductor materials and products and can simultaneously process an area of up to 0.15 m^2 at the energy of bombarding ions in the range of 50-150 keV (with an accuracy of 0.1 %), ion currents of 0.2-0.5 mA, the degree of mass separation of the ion flow M/ΔM–100, and the accuracy of measuring the implantation dose is 3-4 % [10-12]. The main node of “Vesuviy” is an ion source that allows ion processing of products, with an output aperture of 3x30 mm, at a distance of 2.5 m by an ion beam with dimensions of 10x40mm, while it is possible to scan the ion beam vertically at a speed of 3 kHz within 0.5 m, with an ion current density of up to 1-2 mkA/cm^2, at such a current, the dose, for example, within 10^{15}-10^{17}ions/cm^2 is collected in 20-25 minutes at any of the above energies (50-150 keV), the starting treatment pressure is – 2x10^{-6} mmHg, and when processing 3-4x10^{-6} mmHg, the total power consumption is within 100 kVA.

However, the method of ion-beam doping is not without disadvantages.

1. To implement this method, you must use a “Vesuviy”-type implantator, which is quite expensive and requires a large area to accommodate.

2. For processing extended samples, a scanning system must be used, since the spot of the ion beam is small (10x40mm).

3. The density of the ion current during scanning is small (1-2mkA/cm^2), respectively, to obtain the necessary doping concentration, a relatively long processing time is required.

In this regard, the task was to create an ion-plasma method for doping optical fiber used in fiber-optic data transmission lines. As a result of using this method, you can increase productivity and simplify the design compared to the previous method.

The basis of the proposed method is the introduction of alloying admixtures of REM by ion-plasma treatment in a gas discharge. In this case, optical fiber doping is performed by ion-plasma deposition by coating REM and simultaneously bombarding the gas discharge with ions. Gas discharge parameters – discharge voltage 5-7kV, discharge current up to 5A. This method can be performed by igniting a discharge either in an inert gas or in REM vapors. The latter method is preferable, because of the higher purity of the deposited alloying element.

An ion source with a cold cathode is used for ion-plasma treatment of REM coatings obtained by the method of "ion mixing" (ion mixing), which is fixed on the transition flange to the vacuum chamber of the post.

Figure 3 shows the spectrum of a sample of silicon doped with erbium, on the x-axis is the energy of the analysing electron beam (keV) and the ordinate the intensity of the characteristic radiation. As can be seen from the given spectrum, there are no foreign impurities in the sample and only characteristic peaks of erbium are present. The silicon peak is also visible in the near-surface layer.

III. STUDY OF THE PHYSICOCHEMICAL CHARACTERRISTICS OF MODIFIED OPTICAL MATERIALS.

For long-distance fiber-optic systems for transmitting information, it is effective to use optical quantum amplifiers based on active fibers. The same fibers can be used as input and output radiation power amplifiers, lasers and bandpass filters, converters.

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 7, Issue 11 , November 2020

One of the varieties of optical amplifiers doped with rare earth elements are erbium amplifiers EDFA (optical amplifiers doped with erbium), operating at a wavelength of 1.55 μm and having low optical loss (up to 0.2 dB / km) and high efficiency compared to conventional amplifiers. They have high noise immunity and throughput, the transmission line distance reaches tens of thousands of kilometers as a result of using EDFA.

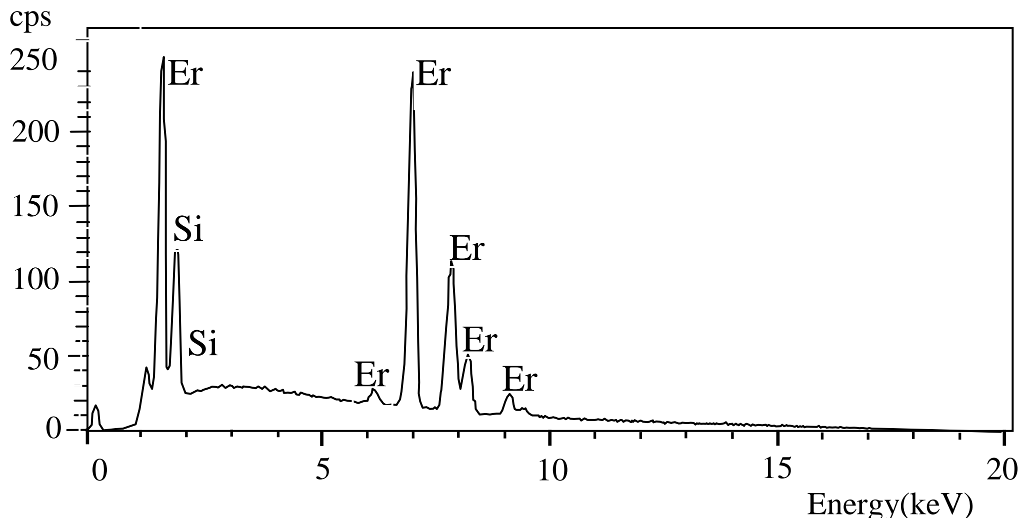

Fig.3. **Spectrum of an erbium-doped quartz sample**

For doping of optical materials with rare earth metals, traditional thermal diffusion chemical methods are currently used, for example, the method of chemical vapor deposition - MCVD. With all such methods, the alloying level is usually 10-1000 ppm (parts per million), i.e. several tens of units per million of surrounding atoms, and with these doping methods it is rather difficult to obtain high concentrations of impurities uniformly distributed over the volume; it is impossible to predict and accurately control the amount and control the distribution profiles of the introduced impurities. In addition, a precipitation effect is possible, i.e. as a result of heating, the introduced impurities can form local accumulations with a high concentration. Most of these methods are multistage, i.e. consist of many operations.

Recently, a number of works have appeared [9-10] devoted to the study of the surface properties of fiber-optic materials modified with ions of rare-earth elements at high energies of ion implantation, such as ions of neodymium (Nd^{3+}),erbium (Er^{3+}), ytterbium (Yb^{3+}), cerium (Ce^{3+}), praseodymium (Pr^{3+}), holmium (Ho^{3+}), and thulium (Tn^{3+}). Of the entire series of rare earth elements, trivalent atoms of erbium (Er^{3+}) are the most effective, since the use of an erbium-doped optical fiber at an operating wavelength of 1.55 μm makes it possible to obtain a high gain at low pump powers.

Experiments on the thermal vacuum deposition of erbium on quartz substrates from molybdenum evaporators at temperatures up to 2300K were carried out using the developed basic technology of ion doping with erbium of optical materials. The deposition of erbium on the samples was carried out in a vacuum installation at a vacuum of the order of $6.6{\cdot}10^{-3}$ Pa, followed by heat treatment in a muffle furnace in an air atmosphere.

The study of the composition and structure of erbium coatings with a thickness of 50 nm to 500 nm was carried out by the methods of secondary ion mass spectrometry and electronic probe analysis on the JEOL installation in the energy dispersive analysis mode.

The thickness of the analyzed layer reaches 3-5 microns at a primary electron energy of 20 keV. Figure 4 (a, b) show the energy spectra, micrographs, and quantitative data on the yield of photons in pulses from erbium coatings on a quartz substrate.

Analysis of the spectra shows that with an increase in the coating thickness in the range of 50-500 nm, the composition of the coating practically does not change. Heat treatment (Fig. 4, b) at temperatures up to 5000C also does not affect the total erbium content, which indicates the stability of the composition when using the vacuum technology of forming coatings of rare earth elements on quartz glass.

ISSN: 2350-0328
International Journal of Advanced Research in Science, Engineering and Technology
Vol. 7, Issue 11 , November 2020

Vacuum deposition of coatings does not change the surface topography of the SiO_2 substrate and defects visible on the surface are determined by the initial processing (polishing) of the surface and preliminary cleaning prior to coating deposition

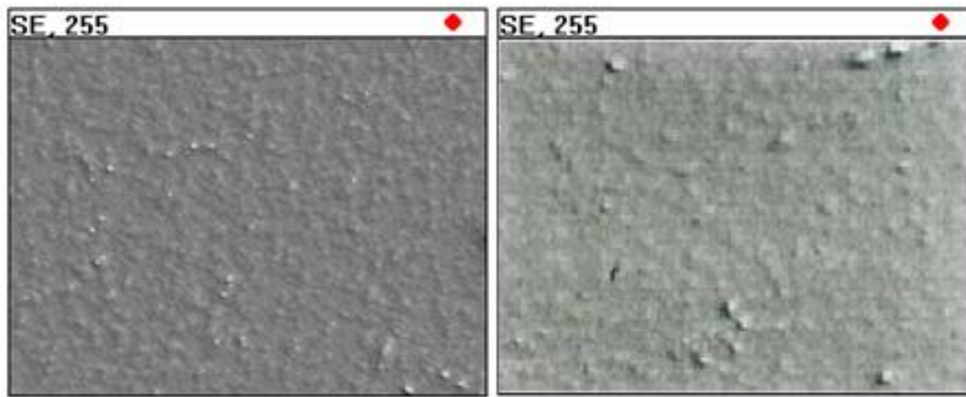

Fig.4. **Microphotograph of erbium coating:** a)before heat treatment at magnification x255, b)after heat treatment in x255 magnification

Cleaning the surface of the substrate in a peroxide-ammonia mixture and concentrated sulfuric acid immediately before vacuum deposition of coatings reduces the number of "punctures" in the coating (porosity) and increases the adhesion strength up to 2 times.

The study of the technology of ion doping of optical fibers with erbium, which allows one to obtain high gains and high concentrations of introduced erbium at low energies of ion implantation, was carried out on a modernized installation of ion implantation "Vesuviy-2" at energies up to 100 keV and doses in the range of 10^{15}-10^{16} at /cm^2 using method of ion mixing and subsequent annealing [11-12].

IV. CONCLUSION

Thus, it is shown that with low-energy ion implantation, intensive doping of silicon with erbium occurs over the surface and depth of samples with a large uniformity and high concentration, there is no precipitation effect, and the depth and concentration of doping can be easily adjusted by the parameters of ion bombardment. The method is technological, and with the help of a special container, you can simultaneously process samples with large dimensions, such as extended optical cables.

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