MOCVD DEPOSITION OF ZINC AND BISMUTH CHALCOGENIDES FILMS ON THE SURFACE OF SILICA OPTICAL FIBRES

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Metal organic chemical vapour deposition (MOCVD) technology is adapted for the deposition of thin zinc and bismuth chalcogenides films on the surface of silica optical fibres with short tapered sections. Growth runs were carried out in a special tubular quartz reactor at atmospheric pressure of hydrogen at 425°C temperature using ZnEt₂, BiMe₃, Et₂Te and i-Pro₂Se as organometallic precursors. During the deposition of chalcogenides, the transmittance spectra of the fibre were recorded in regular short time intervals. In the transmittance spectra of the fibre with a tapered section coated by ZnSe and ZnTe, lossy mode resonances (LMR) were observed at a diameter of the tapered waist below 30 μ m. After the deposition of very thin Bi₂Te₃ and Bi₂Se₃ island films on the tapered waist with a diameter about 10 μ m optical fibres were built into erbium fibre ring lasers. A pulsed generation mode was achieved in some of lasers due to resonator Q-factor modulation. These results can be applied for the design of LMR fibre sensors and passively Q-switch pulsed fibre lasers.

Keywords: MOCVD, zinc and bismuth chalcogenides, tapered silica fibre, lossy mode resonance, Q-switch

1. Introduction

When transmitting optical radiation through optical fibres, it is important to isolate the light passing through the fibre core from the influence of the external environment. However, in some cases it is necessary to increase this interaction, in particular for sensory needs and the creation of laser elements based on saturable absorbers. One can solve this problem by creating a tapered part of the optical fibre and depositing thin films of semiconductor materials on its surface. Coating of the tapered fibre by a thin film of the material with a positive real part of the permittivity higher in magnitude than both the imaginary part of permittivity and the permittivity of the surrounding medium leads to the appearance of lossy modes propagating in the film along the waveguide. The number of lossy modes increases with increasing the film thickness. A coupling between a waveguide mode and a specific lossy mode of a thin film at a particular value of film thickness leads to attenuation dips in the transmission spectra (LMR) [1–3]. The coupling requires overlapping of mode fields and matching of mode phases, namely, equality of the real part of propagation constants of the lossy mode and the waveguide mode. Usually indium, tin and titanium oxides, their solid solutions and a number of polymers are used as coatings for LMR. This series of materials satisfies the conditions of LMR realization: they have the permittivity with sufficiently large real and insignificant, but not equal to zero, imaginary parts. However, their refractive index, a high value of which is one of the main factors for obtaining high sensitivity sensors based on LMR, does not exceed a value of 2.1 in a spectral range of 1.0-2.2 μ m. To the best of our knowledge, such materials as ZnSe and ZnTe with higher values of the real part of permittivity, which even at a wavelength of 2.2 μ m are about 2.44 and 2.71, respectively [4], have not been used for this purpose. Zinc selenide and zinc telluride crystallize in a sphalerite-type cubic lattice, the band gap of which at room temperature is 2.7 and 2.2 eV, respectively [5]. ZnSe has conductivity of an electronic type, and ZnTe has a hole-type conductivity. Due to the ability of in situ recording of the optical transmittance spectra during the deposition of semiconductor films by MOCVD on tapered silica fibres [6], it is possible to control the spectral position, shape, depth and width of the LMR line.

The saturation of optical absorption with increasing intensity of incident radiation in a wide spectral range, and a very fast relaxation, when removing the excitation discovered recently [7, 8], is a unique property of thin films of topological insulators such as Bi₂Se₂ and Bi₂Te₃, which can be widely used in quantum optics and lasers. Narrow-band chalcogenides of bismuth Bi₂Se, (0.3 eV) [9] and Bi₂Te₃ (data varies from 0.13 to 0.21 eV) [10] have rhombohedral crystal lattices. In these semiconductors, the quintuple blocks of Ch-Bi-Ch-Bi-Bi (Ch is selenium or tellurium) are interconnected by weak van der Waals bonds. In most cases, the deposited Bi₂Se₃ films have an electronic type of conductivity, and Bi2Te3 has a hole-type conductivity.

To date, a large number of papers are devoted to Bi_2Se_3 and Bi_2Te_3 as saturable absorbers (SA) for creating pulsed fibre lasers. Such lasers can be divided into two types. The first type is lasers with passive mode locking, which are realized when it is necessary to obtain short-pulse ($\tau < 1$ ps) radiation. The second type is the lasers operating on the basis of passive Q-switching, which give high energy pulses with long enough duration (in the range of several microseconds). To integrate SA materials into a fibre laser system several schemes are implemented. The simplest approach is to apply a thin film of SA on the face of the fibre [11], however, this results in a relatively low damage threshold. Another approach is to place the SA material on the surface of the partially polished fibre (the so-called D-shape fibre) [12–13]. In this case the damage threshold increases significantly, since only the evanescent field interacts with SA, and an increase of the interaction length results in a strong nonlinear response. But an asymmetric design of such a device causes unwanted polarization losses, which can be avoided by reducing the interaction length or using the tapered fibres [14]. According to the method of fabrication, the latter are divided into fused fibre tapers [15] and chemically etched fibre tapers [16].

In this paper, we used the MOCVD technique to precipitate zinc and bismuth chalcogenides on chemically etched fibre tapers. During the deposition of the coating, the transmittance spectra of the tapers were recorded in regular short time intervals. As a result, we managed to implement first LMRs using thin ZnSe and ZnTe films and to fabricate a pulsed ring erbium laser with passive Q-switching (Q-switch) using Bi₂Te₃ films.

2. Materials and methods

2.1. Manufacturing of short fibre tapers

The fabrication of a silica taper with a chalcogenide coating was conducted in several stages. First, the protecting polymer coating was uncovered on the section of a silica fibre SMF28 of a length of 2-10 mm and then the fibre was mounted on the board with a fluoroplastic covering. For fibre etching we used a non-toxic solution $NH_4F + (NH_4)_2SO_4 + H_2O$ [17]. The bare section of the fibre was immersed for 3 h in 1 ml of the freshly prepared solution; the hydrophobic surface of the fluoroplastic did not allow the solution to extend. The selected molar ratios of NH₄F:(NH₄)₂SO₄:H₂O etchant at 22°C temperature provided an etching rate of 0.4 μ m/min and after 3 h the fibre diameter decreased to about 50 μ m. Then we removed the protective coating from 75 mm sections of the fibre on both sides of the taper. We immersed again a 20 mm part of the fibre with a taper in the centre into the etching solution

and continued etching to the required diameter of the tapered waist (10–40 μ m). An optical image of the taper prepared for precipitating chalcogenides is presented in Fig. 1.



Fig. 1. An optical image of one taper before the deposition of chalcogenide.

2.2. Deposition of chalcogenides

For the deposition of zinc and bismuth chalcogenides on the surface of tapers, a special tubular quartz reactor heated by a resistance furnace was manufactured. A sketch of the reactor is shown in Fig. 2.

Trimethylbismuth (BiMe₃), diethylzinc (ZnEt₂), diethyltellurium (Et₂Te) and diisopropylselenium (iPro₂Se) were used, respectively, as bismuth, zinc, tellurium and selenium organometallic sources, whose purity was certified by electronic grade. BiMe₃, ZnEt₂, Et₂Te and iPro₂Se bubblers were held at –15, 10, 25 and 27°C, respectively. The total hydrogen flow was equal to 0.8 and 0.5 l/min during the deposition of zinc and bismuth chalcogenides films, respectively. The films of ZnSe, ZnTe, Bi₂Te₃ and Bi₂Se₃ were deposited at the same temperature of 425°C. A relatively high temperature and a large excess of chalcogen during the deposition of bismuth chalcogenides by MOCVD (up to 40 in the case of Bi_2Se_3) are necessary in order to realize films with Bi_2Se_3 , Bi_2Te_3 stoichiometry and to avoid the deposition of numerous phases of the homologous series $nBi_2 \cdot mBi_2Se_3$ and $nBi_2 \cdot mBi_2Te_3$, where *n* and *m* are integers [17, 18].

An installation of small-diameter tapers into the reactor and their sealing is a technically difficult task that we managed to solve. The outer pigtails of the fibre were connected to the transmittance spectrum recording circuit schematically shown in Fig. 2. During the film deposition, the transmittance spectra of the taper were recorded in a range of 1–1.65 μ m wavelength in regular time intervals using a NIRQUEST-512 spectrometer controlled by the OceanOpticsView software. In these experiments a halogen lamp served as a light source. The transmittance coefficient was determined normalizing the spectrum of transmitting light to that of an uncoated taper (prior to any film deposition).

2.3. Methods of post-growth study of coatings

To determine the stoichiometry of the deposited films, we used an energy dispersive X-ray spectrometer (EDX) X-MaxN, which was fitted with an electron microscope. Analysis was performed with the program INCA, *Oxford Instruments*. To standardize and optimize the profiles of the emission lines of characteristic radiation the following standards were used: crystals ZnS (Zn-L α), Bi₂Se₃ (Bi-M α and Se-L α) and PbTe (Te-L α). The measurement of the standards and the analysis of



Fig. 2. A sketch of the reactor with *in situ* transmittance registration equipment used in this work for the deposition of zinc and bismuth chalcogenides on tapered fibres.

the samples were performed under identical conditions at an accelerating voltage of 10 kV and an electron probe current of 1.4 nA.

The surfaces of as-grown films were studied using a scanning electron microscope Jeol JSM-6480LV with a tungsten thermionic cathode or on an atomic force microscope SmartSPM, AIST-NT7. Film thicknesses were defined by AFM microscopy using a scratch. Raman scattering spectroscopy was carried out at room temperature using a Micro-Raman spectrometer XPloRA (Horiba Scientific) excited with a solid state green laser $(\lambda = 532 \text{ nm})$ in the backscattering configuration. The signal was collected through a 100x objective lens and dispersed by the 1800 g/mm grating under a triple subtractive mode with a spectra resolution of ~1 cm⁻¹. The lowest available frequency was 50 cm⁻¹. The laser power and accumulation time of recording were varied from 0.3 to 3 mW and from 20 to 200 s, respectively.

3. Results

3.1. Lossy mode resonances at the deposition of ZnSe and ZnTe films on the tapered fibre

We carried out series of experiments on the deposition of ZnTe and ZnSe films on the tapered fibres with different diameters from 10 to 40 μ m. The curves in Fig. 3(a) show a change in the transmittance of the tapered fibre (the diameter of the taper (*d*) is 15.5 μ m and the length (*l*) 2.5 mm) at two different wavelengths (1400 and 1565 nm) versus the ZnSe film deposition duration. The change of the ZnSe film thickness during its deposition is also shown on the top of a horizontal scale.

The current film thickness was calculated by multiplying the average deposition rate by the precipitation process time. The deposition rate was determined using the final film thickness according to the AFM data and the duration of its deposition. Three resonance dips in the transmittance, which correspond to different orders of LMR, are clearly seen in both curves. The thicknesses of the ZnSe coatings at which resonance dips at a wavelength of 1400 nm occur are 35, 85, and 185 nm, respectively. It is seen in Fig. 3(a) that in a certain range of the film thickness near LMRs of the first and second order, the transmit-



Fig. 3. Optical transmittance of tapered fibres versus the deposition duration of ZnSe and ZnTe films on their surfaces. Curves 1 and 2 correspond to wavelengths of 1400 and 1565 nm. The taper parameters are $d = 15.5 \,\mu\text{m}$, $l = 2.5 \,\text{mm}$ and $d = 23.2 \,\mu\text{m}$, $l = 3.8 \,\text{mm}$ for (a) and (b), respectively.

tance is close to zero. This suggests that the diameter of the taper ($d = 15.5 \ \mu m$) is smaller than it is necessary to observe narrow resonances of these orders. Figure 3(b) shows a change in the transmittance of the tapered fibre with $d = 23.2 \ \mu m$ and $l = 3.8 \ mm$ with the ZnTe film deposition duration at two wavelengths. The transmittance at the deepest point of the first resonance dip is 0.75 and 0.5 for 1400 nm and 1565 nm, respectively. Figure 4 shows the transformation of transmittance spectra for a short ZnTe film deposition duration when the first resonance is developing.



Fig. 4. Transmittance spectra transformation for a short ZnTe film deposition duration when the first resonance is developing. The tapered waist is 3.8 mm in length and the tapered waist diameter is $23.2 \,\mu\text{m}$.

The minimum of the transmittance in the resonance dip decreases sharply from 0.93 to 0.53 with 13 s deposition time, and the minimum of the resonance dip shifts from 1210 to 1570 nm. The width of the resonance dip increases from 65 to 136 nm. According to the post-growth calculation, the film deposition rate equals 0.56 nm/s and the increment of the coating thickness during this duration was only 7.2 nm. The study of the optical transmittance spectra of a series of tapers with tapered sections of 5 mm in length and different diameters showed that a diameter of about 21 μ m provided a narrow resonance dip at a wavelength of 1565 nm, corresponding to the first-order LMR, with transmittance not equal to zero.

Figure 5 shows the AFM images of square $2 \times 2 \,\mu m^2$ ZnSe and ZnTe film surfaces with a thickness of 80 and 260 nm, respectively. In both images there are a lot of grains with a marked triangular shape. The roughness of the coatings is characterized by rms values of 2.9 and 10.6 nm, respectively. These values are more than two orders of magnitude smaller than the wavelengths of the excited resonances and, in our opinion, the roughness does not affect the shape of resonances.

In the Raman spectrum of the ZnSe film there is a peak around 253 cm⁻¹ due to the LO mode. In the Raman spectrum of the ZnTe film there is a peak around 200 cm⁻¹ and three intense LOphonon replicas against the background of a wide PL emission. These spectra are not shown in this paper. The EDX spectra show that the examined



x: 2.0 µm



x: 2.0 µm

Fig. 5. AFM images of (a) the surface of the ZnSe film with a thickness of 80 nm and (b) the surface of the ZnTe film with a thickness of 240 nm.

samples contain not only Zn, Se or Te, but also Si, O and sometimes Ge elements. The last three elements come from silica fibre.

The in situ transmittance spectra allowed us not only to control the deposition of ZnSe and ZnTe films on the tapered fibre, but also to find the temperature regimes of their complete decomposition by hydrogen, in order to reuse the same tapered fibre in our research. At 530°C temperature a sufficiently rapid removal of both ZnSe and ZnTe takes place.

3.2. Deposition of Bi_2Te_3 and Bi_2Se_3 films on the chemical tapered fibre

During the deposition of Bi₂Se₃ and Bi₂Te₃ films, the quintuple block is bounded to the substrate by weak van der Waals bonds. Therefore, to form thin continuous films, it is necessary to neutralize the free bonds on the substrate surface. For this, a number of approaches are used; in particular, a two-stage process of low-temperature nucleation [19] and deposition of buffer layers of various selenides, including ZnSe [17] and ZnTe [18]. As we noted above in Section 2, the low temperature MOCVD deposition of Bi₂Se₃ and Bi₂Te₃ films leads to the formation of numerous phases with other stoichiometries. We did not deposit buffer layers of zinc chalcogenides to suppress the free bonds of the fibre surface, since their deposition leads to appearing of lossy modes and the manifestation of resonance phenomena. In addition, the island films can be used as SA in pulsed ring fibre lasers, which is one of the objectives of this study. As expected, the AFM investigation confirmed an island character of bismuth chalcogenide coatings deposited on the tapered fibre.

The EDX analysis of the composition of thick coatings confirmed the Bi_2Te_3 and Bi_2Se_3 stoichiometry of the deposited films. For thin island films, this stoichiometry was confirmed by Raman spectroscopy. The Raman spectra of the Bi_2Te_3 and Bi_2Se_3 islands showed peaks at wavenumbers of 58, 99, 132 cm⁻¹ and 71, 131, 173 cm⁻¹, respectively, which are slightly lower than those of active lattice vibrations A^1_{1g} , E^2_{g} and A^2_{1g} , in a few quintuple Bi, Te₃ and Bi, Se₃ materials [20].

3.2.1. In situ transmittance spectra at the deposition of Bi_2Se_3 and Bi_2Te_3 coating on the tapered fibre

Two series of transmittance spectra recorded during the deposition of the Bi_2Se_3 coating on the tapered fibres with a waist length of 5.2 mm and diameters of 8.6 and 13.2 μ m are shown in Fig. 6(a-b), respectively. Since the concept of thickness is not applicable to noncontinuous films with islands, we used the duration of the deposition process as a parameter characterizing the volume of deposited material on the taper surface, the current value of which is indicated by numbers on the top right of the curves. Both growth processes were carried out at 425°C temperature and a Se/Bi ratio in the vapour phase of 40. In the transmittance spectra of the fibre with a taper diameter of 8.6 μ m (Fig. 6(a)), one can see some chaotic beats, but the transmittance is almost constant in a range of 1170-1650 nm. After 8 s of the deposition, when the transmittance was less than 10%, the deposition process was stopped. The transmittance spectra of Bi₂Se₃ deposited on the taper with a diameter of 13.2 μ m have a different character (Fig. 6(b)). In a range of 4-11 s the transmittance spectra show a monotonous decrease of transmittance with increasing wavelength, the rate of which increases with time. Starting from the 13th second of the deposition, the transmittance does not change, and as can be seen from Fig. 6(b), the spectra corresponding to the deposition times from 13 to 15 s completely coincide.

Figure 6(c, d) shows a series of transmittance spectra in the formation of Bi₂Te₃ coating on the tapered fibres with taper waist diameters of 10 μ m (c) and 13.2 μ m (d), with slightly different waist lengths, 4.7 and 5 mm, respectively. In the taper with a diameter of 10 μ m, the transmittance drops sharply to almost zero during the first 2 s of deposition. The drop in transmittance in the long-wavelength part of the spectrum takes place only on the curves lower than those shown in Fig. 6(c). According to AFM, this coating is not continuous, the average height of the islands is 2.8 nm, and therefore the average rate of their deposition is 1.4 nm/s. When Bi₂Te₃ is deposited on the fibre taper with a diameter of 14.3 μ m, an increase in the transmittance in the shortwavelength region of the spectrum is observed; however, unlike the deposition of Bi₂Se₃ films on a similar taper (Fig. 6(b)), at the final deposition stage the transmittance spectra did not coincide. The *in situ* control of the transmittance of tapers with Bi, Te, and Bi, Se, coatings at 425°C deposition temperature showed a steady increase in transmission in the entire spectral range when we stopped the supply of reagents. This is due to the decomposition of bismuth chalcogenides by hydrogen. If it is necessary to reuse the tapered fibre for research purposes, bismuth chalcogenides can be completely removed in hydrogen at 480°C.



Fig. 6. Transmittance spectra during the deposition of Bi_2Se_3 (a, b) and Bi_2Te_3 (c, d) on the tapered fibre. Waist lengths and diameters of tapers are equal to: (a) 5.3 mm, 8.6 μ m; (b) 5.2 mm, 13.2 μ m; (c) 4.7 mm, 10.0 μ m; (d) 5.0 mm, 14.3 μ m. The numbers in the top of curves show the film deposition duration in seconds.

3.2.2. Q-switched fibre lasers with Bi_2Te_3 coating as a saturable absorber

Thin coatings of Bi₂Te₃ and Bi₂Se₃ were deposited on series of tapers with a diameter from 10.5 to 15 μ m to test as passive Q-switchers in a ring cavity of erbium fibre lasers. According to the AFM study, most of realized coatings are island films, on which bismuth chalcogenide islands occupy a small part of the taper surface. Figure 7(a) shows an AFM image of the Bi, Te, film on the taper with a waist length of 2.5 mm and a waist diameter of 14.8 μ m with 70.3% transmittance at a wavelength of 1.56 μ m. Islands of an average height of 5 nm occupy no more than 20% of the surface of the taper. An increase in the density of the islands leads to a sharp decrease in transmittance. Thus, for the taper with a waist length of 3.0 mm and a diameter of 12.3 μ m, and the surface, almost completely covered by the islands with an average height of 11 nm (Fig. 7(b)), the transmission was only 2.3%.

A set of the tapers with transmittance in a range of 70–90% at a wavelength of 1.56 μ m were built in the ring cavity of erbium fibre lasers and tested as passive Q-switchers. As a pump source of the lasers a fibre-coupled laser diode with a wavelength of 980 nm and an output power up to 300 mW was used. In the tested five tapers with the Bi₂Se₃ coating Q-swiching was not observed. The laser pulse generation was observed only on one taper with a waist diameter of 10.5 μ m and a length of 4 mm covered by a very thin Bi, Te, coating. The taper transmittance was about 78% at a wavelength of 1.56 μ m. This laser starts to operate in the CW mode at a pump power of 30 mW. At pump powers from 34 to 40 mW, pulses with a kHz repetition rate appear (see Fig. 7, upper), which manifest the effect of the deposited SA layer.



Fig. 7. AFM images of Bi_2Se_3 (a) and Bi_2Te_3 (b) surfaces on the tapered waist of the fibre. Waist diameters of tapers are equal to (a) 8.6 μ m and (b) 12.3 μ m.

The Q-switching in lasers with the thicker tapers arose after pouring the tapers with Bi_2Te_3 films by polymer coating (polydimethylsiloxane elastomer, the refractive index of which is 1.40 at 20°C temperature). For example, for the taper with a waist diameter of 13.0 μ m and l = 4 mm, the transmittance fell from 90 to 48% after pouring the polymer. The laser started to operate in the CW mode at a pump power of 40 mW and the Q-switching was realised at pump powers of 48 to 107 mW. The pulse train at 100 mW pump power is shown in Fig. 8 (bottom curve). The emergence of Q-switching can be explained by an in-

crease of the energy density of the evanescent field of the laser radiation passing through the core of the fibre, and, as a consequence, the strengthening of its interaction with the SA film.

4. Conclusions

We have deposited zinc and bismuth chalcogenides on the tapered section of the silica fibre by MOCVD for the first time. For fabrication of the fibre tapers chemical wet etching based on a nontoxic solution of $NH_4F + (NH_4)_2SO_4 + H_2O$ was applied. The film deposition on the tapered



Fig. 8. The pulse train of the tapered fibre: with Bi_2Te_3 coating (upper) and double Bi_2Te_3 polydimethylsiloxane elastomer coating (below). The parameters of tapers are given in the text.

fibres was carried out in a special tubular quartz reactor with registration of the transmittance spectra in real time.

The experimental results show that in the transmittance spectra of the fibre tapers of a diameter lower than 30 μ m, covered by thin (30–200 nm) ZnSe and ZnTe films with high refractive indexes, narrow dips associated with the LMR phenomenon appear under certain conditions.

Ultrathin bismuth chalcogenide layers with a transmittance of 70-90% at a wavelength of 1.56 μ m were deposited on the tapered fibres of diameters from 10.5 to 15 μ m. The Bi₂Se₃ and Bi, Te, stoichiometry of the coatings deposited at 425°C is confirmed by EDX and Raman spectroscopy. The fibre tapers with deposited bismuth chalcogenide films were tested as SA in the erbium fibre ring laser. Pulse generation was obtained using a fibre taper with a diameter of 10.5 nm covered by an island Bi, Te, coating. However, a narrow range of the power of pump laser radiation, for which this generation was observed, points out that the diameter of the taper has to be further reduced. It was found that a polymer coating on top of the deposited films makes it possible to realize Q-switchers on the tapered fibre up to 15 μ m in diameter. It is of interest to examine the possibility of using two-layer Bi, Te, -ZnTe and Bi, Te, -ZnSe heterostructures as SA. The MOCVD method allows us to deposit these heterostructures in a single technological cycle.

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SiO₂ ŠVIESOLAIDŽIŲ DENGIMAS CINKO IR BISMUTO CHALKOGENIDŲ PLĖVELĖMIS, AUGINTOMIS METALORGANINIO CHEMINIO GARŲ NUSODINIMO BŪDU

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