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Optical losses and photo-induced absorption in chalcogenide glass fibers

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The paper presents a brief review of optical properties of infrared optical fibers based on chalcogenide glass materials. The most representative results on optical transmission of chalcogenide glasses used for fabrication of IR fibers as well as photo-induced absorption features in chalcogenide glass fibers are presented.

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Keywords: Chalcogenide glasses, IR transmission, Absorption bands, Photoinduced absorption, Localized states

1. Introduction

Chalcogenide glasses are semiconducting materials that contain two or more chalcogen elements - group VI in the periodic table, e.g. S, Se, Te - as alloy components. There is a constant interest in chalcogenide glasses as optical materials for IR spectral range for passive and active devices, non-linear elements, etc.

The chalcogenide glasses are very attractive materials for application in integrated and fiber optics due to a number of specific properties in comparison to other optical materials. The chalcogenide glasses exhibit excellent transmission in the infrared spectral region, usually up to ~ 12 μm [1-5]. Chalcogenide glasses exhibit high refractive index ($n \geq 2.4$), low phonon energy and record nonlinear coefficient, typically ×100 of the refractive index of silica [1-4]. These glasses have good solubility towards rare earth elements ions and high chemical and thermal stability [1, 2].

One of the main advantages of chalcogenide glasses as a host glass for rare-earth ions is the lower energy of phonons (~ 300-350 cm^{-1}) compared to phonon energy of fluoride (~ 550 cm^{-1}) or oxide glasses (~ 1100 cm^{-1}) [2]. Their low phonon energy and high refractive index provide chalcogenide glasses with low probability of multiphonon relaxation and high radiative transition probabilities of rare-earth ions levels. Chalcogenide glasses doped with rare-earth elements provide an opportunity to obtain fluorescence and possible laser action at longer wavelengths than available from oxide or fluoride vitreous hosts. Currently used silica-based solid-state lasers are operational only from the visible out to ~ 3 μm because intrinsic phonon bands extend from long wavelengths into the mid-infrared region. The rare-earth doped chalcogenide glasses offer excellent possibilities for use in constructing high efficient microsphere lasers, optical fiber amplifiers, photonic devices and other mid-infrared laser devices.

Along to thin film and bulk samples the infrared optical fibers based on chalcogenide glass materials have a great potential in development of optical communication and advanced photonic systems [1-5]. Besides mentioned advantages chalcogenide glasses exhibit a number of specific photo-induced effects. Both transparency as well as refractive index of chalcogenide glasses changes under illumination that gives for example the possibility to control the output optical signal, to write high quality gratings, to form waveguides and other optical elements [6, 7].

2. Chalcogenide glasses for fabrication of IR fibers

There are an extensive number of papers on optical properties of chalcogenide glasses and chalcogenide glass fibers technology [8-20]. Arsenic trisulfide (As_2S_3) glass is one of the most extensive studied chalcogenide materials. Arsenic trisulfide fibers have good optical transmission in 0.7-6 μm range, while for optical transmission at the longer wavelengths additional heavier elements like Te, Ge, or Se are added to As-S system [10]. Optical fibers made of high-purity chalcogenide glasses As-S, As-S-Se, As-Se and Ge-As-Se are employed for operation in the middle IR range. Chalcogenide glasses in the system As-S are suitable for transmission in the 1-7 mm range, while optical fibers based on As-Se and As-Ge-Se glass systems can be used in the spectral range 2-12 mm [1-5]. The multimode fibers made of arsenic sulfide and arsenic selenide glasses show low optical losses as ~ 23 dB/km and 79 dB/km at 2.4 μm and 4.5 μm, respectively [2]. Some estimations of the possible minimal losses in chalcogenide glass fibers have been done by Kanamory [9]. It is stipulated that the lowest optical losses in As_2S_3

glass fibers are at ~ 20 dB/km, which is quiet closed to obtained experimental figures obtained by the same authors [9].

Chalcogenide glass fibers that are usually fabricated in research laboratories and fabricated in industrial conditions may contain different amounts of basic impurities that affect the optical transmission in the IR. One of the major problems for reducing optical absorption in chalcogenide glasses is related to extrinsic absorption bands that are associated to the three main impurities, which are especially difficult to be removed during the technological process of purification. These impurities are mainly H_2, H_2O, and OH-bonding to the elemental cations [8]. For example the absorption bands between 4.0 µm and 4.6 µm are due to S-H or Se-H bonds and those at 2.78 µm and 6.3 µm are due to OH- (2.78 µm) and/or molecular water. In fact the lowest obtained losses at the important CO_2-laser wavelength 10.6 µm are still above 1 dB/m for the Se-based fibers [9].

The technological efforts of different groups that work on chalcogenide glasses have been focused toward the basic needs of optoelectronics applications: high transparency in IR, mechanical strength, environmental durability during storage and operation, stability of optical and mechanical properties, low costs of material and manufacturing, etc. [11].

While exhibiting a number of strong advantages as materials for IR, chalcogenide glasses are associated with some difficulties related to technological aspects. For example, different constituent elements used in these glasses are toxic. Chalcogenide glasses have a quite low T_g (some below 450 K), high coefficient dn/dT and high refractive index n [12]. A serious problem in fabrication of low-loss chalcogenide glasses remains a dramatic impact of impurities on the magnitude of optical losses [1-5].

High purity chalcogenide glasses designed for fabrication of low-loss optical fibers are usually prepared by preliminary purification of starting raw elements (better than six nines purity materials). According to [11] the average concentration of hydride- and oxide- impurity should be kept below 5×10^{-5} mol %, while the concentration of physical defects should be kept below 10^3 cm-^{3}. Actually the main impurities that dramatically affect the minimal losses in chalcogenide glasses are hydrogen, oxygen, as well as carbon. There are also other impurities that can be presented in chalcogenide glass materials – for example such elements like S, Se, P, Sb or transition metals [1].

Because of long optical path in fiber samples these impurities are easily revealed in the optical absorption spectra as the bands of selective absorption [13]. The specific position of different impurity absorption bands in vitreous arsenic sulfide and arsenic selenide is determined by the nature of impurity itself (Table 1) [1].

Table 1. Position of absorption bands for the main impurities in As-S and As-Se glasses [1].

Compound or functional group leading to absorption	Position of the maximum of Absorption band, µm
OH-	2.92-
S-H	4.01; 3.65; 3.11; 2.05
Se-H	7.8; 4.57; 4.12; 3.53; 2.32
Ge-H	4.95
As-H	5.02
P-H	4.35
H_2O	6.31; 2.86; 2.79
Ge-O	12.8; 7.9
P-O	8.3
CO_2	4.33; 4.31; 15.0
COS	4.95
CSe_2	7.8
CS_2	6.68; 4.65
Arsenic oxides	
Se-O	15.4; 12.7; 9.5; 8.9; 7.9; 7.5
Si-O	10.67; 11.06
Non-identified bands supposedly due to carbon presence	9.1-9.6 4.65; 5.17; 5.56; 6.0

According to [5], in order to achieve sufficient low optical losses in chalcogenide glass fibers the concentration of hydrides (OH, H_2) oxides and hidrocarbonates impurities must be kept in the range of 10^{-5}-10^{-6} mol %, while the concentration of physical defects should be kept in the range 10^2-10^3 cm^{-3}. At the same time the typical concentration of those impurities associated with the group of hydrogen, oxygen, nitrogen is usually in the range 10-100 ppm. at. [1].

3. Optical transmission loss in chalcogenide glass fibers

Considerable efforts have been concentrated for fabrication of low loss IR fibers from chalcogenide glasses. IR optical fibers can be fabricated from chalcogenide glasses in the systems As-S(Se), As-S-Se, As-Se-Te, Ge-As-Se, Ge-As-Se-Te, etc. [1-5].

Table 2. Minimum optical losses in some CHG fibers [1].

Minimum optical losses, dB/km					
Laser wavelength, µm	2.0 µm	2.9 µm	3.7 µm	5.6 µm	10.6 µm
As_2S_3	400	160	140	230	-
$As_2S_{1.5}Se_{1.5}$	1000	3800	400	200	-
As_2Se_3	160	160	80	85	650
$As_2Se_{1.5}Te_{1.5}$				1000	3500

Table 3. Minimum optical losses in some CHG fibers [1].

Minimum optical losses, dB/km		
Laser wavelength,μm	α, dB/km	λ, μm
As_2S_3	23	2.4
$As_2S_{1.5}Se_{1.5}$	190	4.8
As_2Se_3	78	4.2

It is interesting to compare the parameters of infrared fibers obtained in the conditions of research laboratories and industrial companies. The most representative data obtained by M.F. Churbanov and coauthors [1] are presented in Table 2.

Single-mode and multimode fibers on the base of high purity chalcogenide glasses As-S, As-Se and Ge-As-Se have been fabricated by different research teams [2, 11, 15]. M.F. Churbanov and co-authors [2] showed that the minimum optical loses in single-mode fibers are much higher than in multimode fibers. The single-mode fibers from As-S glasses have obtained with the minimum optical losses from 200 up to 400 dB/km in the 1.3-3 μm region. The multimode fibers made form arsenic sulfide and from arsenic selenide have been obtained with the minimum optical losses equal to 23 dB/km and 79 dB/km at 2.4 and 4.5 μm respectively [2,15].

It is considered that for the basic optoelectronic and medical applications the level of achieved optical losses and mechanical properties of chalcogenide glasses As-S, As-Se and Ge-As-Se systems is quite satisfactory [2]. The spectral distribution of total optical losses in a multi-mode fibers made of high purity arsenic-sulfide glass with $As_{40}S_{60}$ core can be illustrated by Fig. 1 [2].

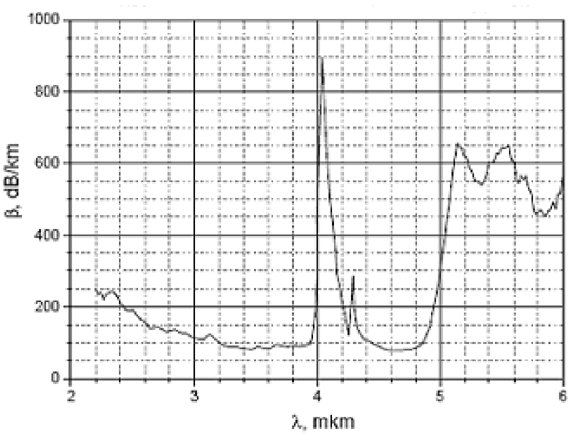

Fig.1. Spectral dependence of the total optical losses in a multimode optical fiber made of high purity As_2S_3 glass [2].

The NIR transmission spectra of $Ge_{20}Se_{80}$ and $Ge_{20}Se_{80-x}Te_x$ glasses are illustrated in Fig. 2 by the data from [5]. The absorption band at 4.5 μm is due to Se-H groups.

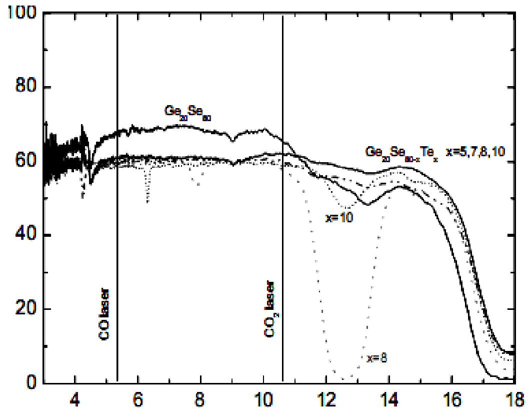

Fig. 2. NIR transmission spectra up to the long wavelength absorption edge are shown for $Ge_{20}Se_{80}$ and $Ge_{20}Se_{80-x}Te_x$ glassy samples. The values of x = 5, 7, 8, 10 are indicated in the figure [5].

Accordingly to authors [5] the pronounced absorption band at 12.5 μm observed on two samples (x = 8, x = 10), and superimposed on absorption band due to Ge-Se bonds, is caused by Ge-O bonds. This absorption band could be diminished or removed when baking of Ge, together with gettering is performed under vacuum prior to the start of the synthesis.

D. Lezal and co-authors [5] used the method of purification and synthesis described in [11] to obtain chalcogenide sulfide and selenide glasses with good transparency in the IR (0.5–6 μm and 0.8–11 μm respectively) that could be used for high laser power delivery, e.g. Er:YAG, CO and CO_2 lasers [5]. As for the telluride glasses the typical absorption spectra of representative $Ge_{20}Se_{80}$ and $Ge_{20}Se_{80-x}Te_x$ glasses are shown in Fig. 2 [5].

I.D. Aggarwal and co-authors from the Naval Research Laboratory achieved a significant progress in fabrication of low loss chalcogenide glass fibers [15]. Available results of these authors [15] illustrate the transmission spectra for several glass materials (Fig. 3).

The advantage of application of chalcogenide glass fibers for infrared technique application is evident. Depending upon composition the sulfide, selenide and telluride based fibers transmit between 0.8-7 μm, 1-10 μm, and 2-12 μm respectively [15].

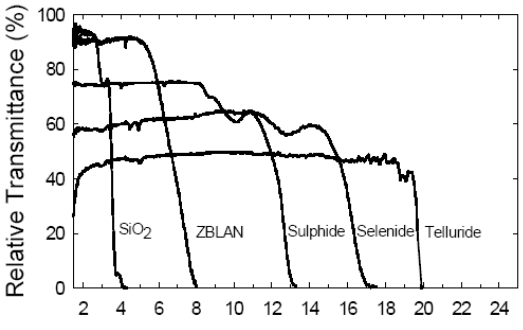

Fig. 3. Transmission spectra for several optical glasses (thickness of about 2-3 mm [15]

Aggarwal and co-authors [15] have fabricated low loss As-S fibers with the loss value about 0.1-0.2 dB/m in fiber length of about 500 m, while the lowest loss was about 0.023 dB/m. Purification and composition play an important role in making low loss fibers. Fig. 4 compare the losses obtained for a couple of chalcogenide glasses along with the lowest reported loses [16, 17].

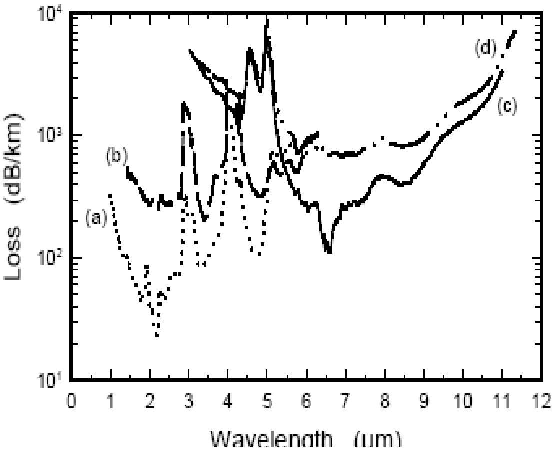

Fig. 4. Transmission loss spectra of (a) lowest loss sulfide fiber; (b) typical sulfide fiber; (c) lowest loss telluride fiber; and (d) typical telluride fiber [15].

The problem of fabrication of high quality IR fibers remains on the agenda both of research laboratories as well as of industrial companies. For example *Amorphous Materials, Inc.* [18] produces chalcogenide glass fibers obtained by utilization of unique process and draw tower design in fibers. Fibers have core diameter within the range 100-1000 μm and optical losses 0.2-0.4 dB/m at 5.25 μm and 9.27 μm, as well as 4-5 dB/m at 10.6 μm. Utilizing high purity materials the *Nippon Telegraph and Telephone Public Corp.* has obtained optical fibers based on As-S and As-Ge-S glass systems [19]. The minimal losses have been obtained in the uncoated fibers $As_{40}S_{60}$ that are equal to 35 dB/km at 2.44 μm, 182 dB/km at 2.12 μm and the fibers from systems $As_{38}Ge_5Se_{57}$ and $Ge_{20}Se_{80}$ optical losses were equal to 148 dB/km at 1.68 μm. The *High Tech Photonics* [20] use chalcogenide IR glasses (CIR) as the best As_2S_3-based material for fiber optics in the range 2-6 μm. Advanced purification technology of chalcogenide glasses provides the spectra free from the OH- absorption at 3 μm and this enables chalcogenide infrared fibers to be used for Er:YAG laser power delivery.

6. Photo-induced absorption

Because of long optical path of probing light in fiber optic samples, optical fibers are convenient specimens for investigation not only the weak absorption bands in chalcogenide glass materials with very low concentration of impurities but also to study variations of the optical transmission under external perturbations, for example the photoinduced absorption (PA). Photoinduced light absorption (PA) in unclad chalcogenide glass fibres firstly was described by *Andriesh and co-authors* [21]. When illuminating the lateral surface of the fiber with bandgap light the intensity of the probing light at the output of the fibre decreases because of photo-induced absorption effect. Typical behaviour of PA kinetics after switching on/off the excitation light is illustrated in Fig. 5 on the example of As-Ge-Se glass fibers [23].

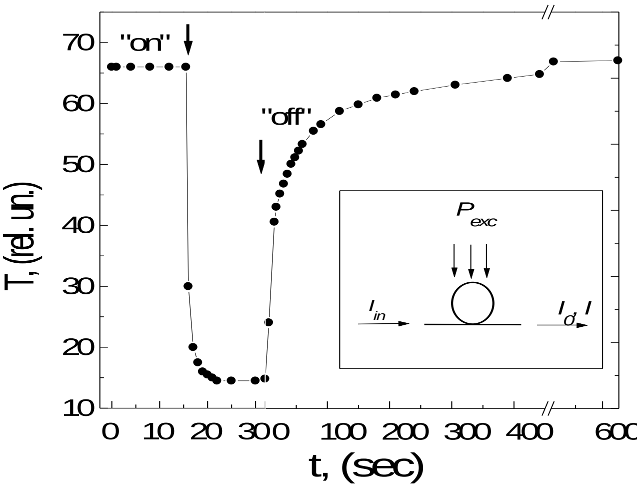

Fig. 5. PA kinetics in As-Ge-Se glass after switching on/off the excitation light 0.96 μm. The insert illustrates the set-up for PA measurement in chalcogenide glasses fibers [21].

Detailed investigation of PA has been carried out on unclad optical fibres made of different compositions - As_2S_3, As-S-Se and As-Ge-Se glasses [21-24]. During these measurements in order not to affect the PA coefficient the intensity of probing light usually was selected to be much less than the intensity of the lateral illumination. Typical intensity of excitation light intensity ranged from the 10^{-6} to 10^{-2} W/cm^2 and the exposure time ranged from tenths of a second to hours.

The magnitude of PA depends on the conditions of exposing by light (Fig. 6) [21]. Optical losses in As-S glass fibers have been measured at room temperature under different conditions: in the dark, under lateral illumination with band-gap light, with non-monochromatic probing light and under irradiation with a neutron flux (Fig. 6).

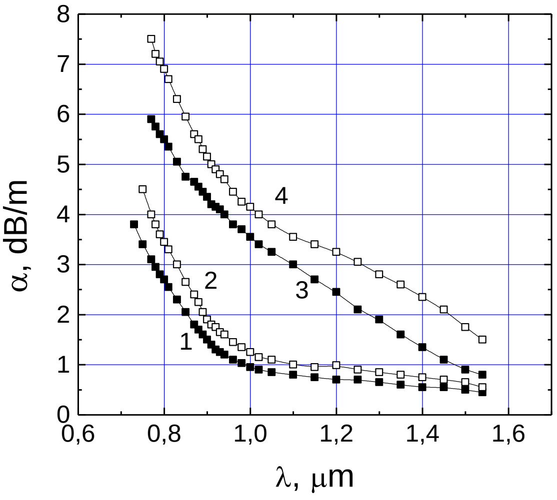

Fig. 6. Optical absorption in As-S glass fibers measured under different conditions at room temperature: 1 – in the dark; 2 – under lateral illumination with band-gap light; 3 – the measurements have been carried out with non-monochromatic probing light; 4- under irradiation with neutron flux [7, 21].

The most important is experiment with action of neutron irradiation. Neutron radiation generates new defects that mean increasing the concentration of gap states and respectively contributes to the increasing of PA, which is observed in experiments.

The spectral distribution of steady-state PA in chalcogenide glass fibres exhibits an exponential character (Fig. 7).

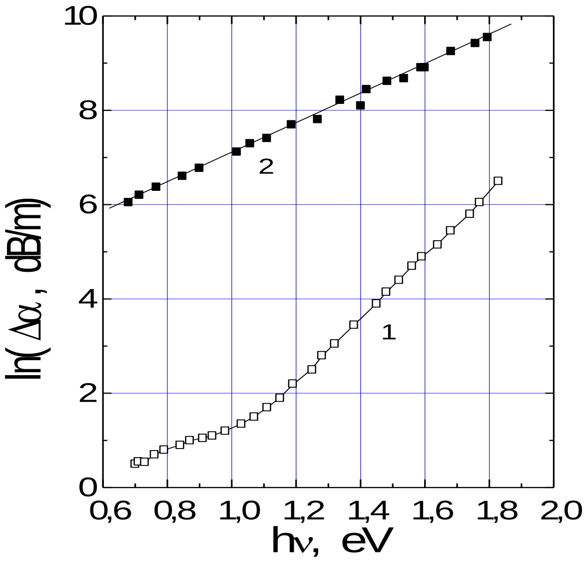

Fig. 7. Spectral distribution of optical absorption in chalcogenide fibers As_2S_3. The temperature T=300 K (1); 77 K (2).

The photo-induced absorption in chalcogenide glass fibers can be explained by the model of carriers multiple trapping in localized states [22, 23] (Fig. 8).

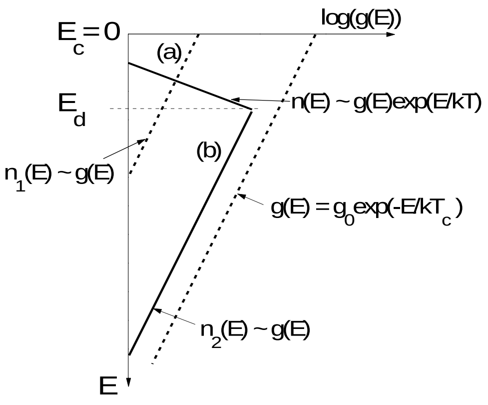

Fig. 8. The sketch of the localized states and of excess

This model treats reversible photo-induced absorption in chalcogenide glass fibers as a pure electronic process. The steady state coefficient of photo induced absorption is determined mainly by the competition of different processes: excess carrier generation, subsequent capturing, thermal activation and recombination [22, 23]. The capture coefficient is supposed to be constant.

Increase of the PA coefficient with decreasing the temperature (Fig. 7) is explained in terms of thermal activation process of localised carriers on the traps in the gap [22, 23]. When temperature decreases the probability of thermal activation of trapped carriers decreases too. This fact leads to an increase of the density of carriers that are captured on localised states in the gap, and respectively to an increase of the PA coefficient. Really it was experimentally shown that the steady state PA coefficient exhibits very strong temperature dependence. While decreasing the temperature from the 330 to 77 K the PA coefficient increases by several orders of magnitude (Fig. 7). On the other hand if the temperature and the capture coefficient are constant, PA is proportional to the density of states. That means that spectral distribution of the PA can reflect the spectral distribution of localized states. From this point of view one can state that there are regions with increased densities of states in the middle of the optical gap of studied glasses (Fig. 7) that was earlier confirmed by many experiments [7].

As it was established earlier, there are different groups of localized states in the forbidden gap of chalcogenide glasses [7]. This fact should be observed when studying the kinetics of PA in the conditions of variation of illumination regime. For example [22-24] during the process of band-gap light illumination of As_2S_3 glass fibers at 77 K one can observe the non-monotonous character of *photodarking* and *photobleaching* kinetics, as well as transient enhancement of PA at sub-bandgap illumination. The mathematical solutions for the PA kinetics in chalcogenide glass fibers have been obtained [22, 23]. Computer simulation of PA kinetics correlates very well with experimental results (Fig. 9) [25].

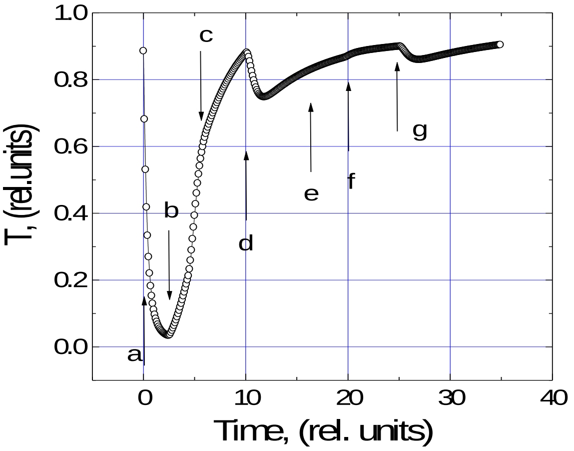

Fig. 9. Computer simulation of the time dependence of optical transmission of As_2S_3 glass fibers at consecutive illumination with bandgap and sub-bandgap light: (a) the bandgap light is switched on; (b) the bandgap light is switched off; (c), (e), (f) the light with photon energy $h\nu_{sb} = 1.96$ eV is switched on; (d), (g) the light $h\nu_{sb}$ is switched off [25].

These peculiarities of the non-monotonous character of *photodarking* and *photobleaching* kinetics can be explained by redistribution of trapped carriers on localised states in the gap [23].

It is to note that for a wide range of excitation light intensity (10^{-6}-10^{-2} W/cm^2) the PA coefficient exhibits a power-law dependence versus the intensity of the excitation light $\Delta\alpha \sim (P_{exc})^n$, where the parameter n is close to 0.5. For excitation light intensity up to ~ 10^{-2} W/cm^2 no irreversible changes of optical transmission was observed. The qualitative analysis of experimental results in terms of the multiple trapping enabled us to evaluate some parameters of localized states distribution and careers transport. For example: the characteristic energy of localized states distribution E_0 was found to be ~ 0.26 eV. It was possible to evaluate the temperature dependence of hole drift mobility in As_2S_3 [24, 26]. The hole drift mobility in As_2S_3 in the temperature interval 300-77 K changes in the range 10^{-8} to 10^{-12} cm^2/Vsec [24, 26].

7. Conclusions

High-purity chalcogenide glasses of the systems As-S, As-S-Se, As-Se and Ge-As-Se are very promising materials for fabrication of infrared fibers for various optical and photonic applications in the spectral range 0.6 to 15 μm.

Chalcogenide glasses have many advantages for fabrication of IR fibers in comparison with other materials but they are subjected to a significant impact of impurities that leads to additional losses in optical fibers. The methods of glass preparation should include procedures for preliminary purification of initial raw materials (> 6 nines purity) and the average concentration of hydride and oxide impurities must be kept below ~ 5×10^{-5} mol %, while the concentration of physical defects - below ~ 10^3 cm^{-3} [11].

Available technologies in research laboratories allow fabrication of IR fibers from chalcogenide glasses with low losses 0.023 dB/m at 2.4 μm [2, 15]. Industrial companies reported the minimal losses as 0.2-0.4 dB/m [19, 20]. The achieved optical losses and mechanical characteristics of chalcogenide glass fibers based on As-S, As-Se and Ge-As-Se systems are quite satisfactory for use in laser power delivery (e.g. Er:YAG, CO and CO_2 lasers), fiber optic sensors or medical applications [2, 5, 11,15].

Infrared optical fibers based on chalcogenide glasses exhibit a number of phenomena, including the photo-induced absorption. PA in chalcogenide glass fibers can be explained by the model of excess carriers multiple trapping in localized states in the gap. The qualitative analysis of PA experimental results in terms of the multiple trapping model enabled to evaluate some parameters of localized states distribution and careers transport.

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