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RESEARCH

Attenuation Nature of Optical Fibers with Oxygen-Deficit Silica Core after γ-Irradiation with 1 MGy Dose

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Abstract
The present work aims to investigate the nature of γ-irradiation induced attenuation in the near-infrared spectral region for single-mode optical fibers with a core made of oxygen-deficit silica glass and a fluorine-doped cladding. The optical fibers were fabricated by the MCVD method. After the irradiation with a dose of 1 MGy the optical fiber attenuation in the spectral range 1200–1600 nm was measured after 2 h, 3 and 10 months. The radiation induced attenuation in the 1600 nm region is higher and more stable than at 1300 nm. The silica glass phonon spectrum change is indicated by the measurements of the spectral dependence of absorption in the region of 1400–700 cm^{-1} when silica glass grains are irradiated with a dose of 1 MGy. The increased oxygen content in the glass enhances the destruction of its network. The nature of radiation-induced attenuation of fibers in the 1900 nm spectral region is justified by the change in the intensity of the silica glass fundamental band fourth overtone. The attenuation at γ-irradiation with an increasing wavelength in such single-mode fibers is also related to the penetration of optical radiation into the cladding, the radiation resistance of which is less than that of the core material.

Keywords Optical fiber · Silica glass · γ-irradiation · Attenuation · Phonon spectrum

1 Introduction

Civil nuclear industry, space, military applications and high energy physics experiments require special radiation-resistant optical fibers (OF) with minimized radiation-induced attenuation (RIA) [1]. The OF radiation resistance is highly relevant to plasma diagnostics in the development of the international thermonuclear experimental reactor [2, 3].

As known, doping silica glass reduces its radiation resistance [1]. Therefore, the most radiation-resistant OF are those with a core of pure or low fluorine-doped silica glass and a fluorine-doped cladding (Table 1). The probing radiation power in these OFs during RIA measurements did not exceed 40 μW to exclude photobleaching of radiation defects [4].

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The comparison of data (Table 1) testify that the deficit of oxygen in pure silica glass OF core provides the lowest optical losses both before and after γ-irradiation.

The work on fluorosilicate OF radiation resistance [4] indicates that γ-irradiation creates no defects that cause absorption bands in the wavelength region between 1300 and 1550 nm. Subsequent works [7, 8] found that the short-wavelength (SWL) RIA is due to Rayleigh scattering. The nature of RIA in the long-wavelength (LWL) region at wavelengths longer than 1500 nm [9] has not been completely determined.

Two main explanations for the nature of LWL RIA are proposed.

The first implies the formation of defects in the form of self-trapped holes (STH), which, however, are stable only at low temperatures [10, 11]. The existence of such radiation defects at room temperature has not been established.

The second more plausible explanation is results from the change in the glass network vibration spectra in the course of γ-irradiation [12–14]. The specificity of RIA changes for OF with an oxygen-deficit core in the process and after high doses of γ—irradiations has not been investigated.

Table 1 Attenuation of single-mode OFs at 1310 nm wavelength before (α_0) and after (RIA) γ-irradiation with 1000 Gy dose at comparable dose rate

Pure silica glass core	α_0 (dB/km)	RIA (dB/km)	Dose rate (Gy/s)
with low fluorine-doped [4]	0,41	3	1,4
with an oxygen excess [5]	0,35	10	0,75
with an oxygen-deficit [6]	0,30	0,62	1,0

The work aims to measure attenuation in single mode OF with an oxygen-deficit core after γ—irradiations with a dose of 1 MГр and the LWL RIA nature analysis.

2 Materials and Research Methods

The MCVD method on the automated complex OFC-12–729, 'Nextrom', was used to fabricate a single-mode OF preform on the basis of F300 silica glass tube with the outer diameter of 25 mm and wall thickness of 3 mm. The fluorosilicate cladding was fabricated by a two-step method [15]. The pure silica glass core layer was deposited by the conventional one-step method. The tube was compressed into a rod at a temperature of 2250 °C in three passes of the burner.

The principal distinctive feature of the OF preform fabrication is the creation of oxygen deficit in the core and its purification from the impurity of chlorine and OH groups, which significantly reduce the radiation resistance of OF [16]. For this purpose, after the core layer deposition, the tube was heated to a temperature of 2200 °C with a burner moving at a speed of 50 mm/min and its inner channel was purged with nitrogen containing no more than 10^{-6}% moisture (Fig. 1).

The refractive index difference between the core and cladding measured on a refractometer P-101 is 0.0095. The diameters of the preform, cladding and core measured on the same instrument are 16.3, 9.3 and 0.91 mm.

The fiber with a diameter of 125 µm was drawn from the preform at the temperature of the graphite heater ≈ 2150 °C with simultaneous application of a two-layer epoxyacrylate UV-curable coating with a thickness of 65 µm.

OF attenuation before and after its radiation treatment were measured by the cutback method using an optical spectrum analyzer of "Yokogawa AQ6370C" brand in the range of 1200–1600 nm with a resolution of 0.2 nm. To improve the estimation accuracy of OF small optical losses, attenuation measurements were performed on a 7 km long fiber segment. The LP_{11} mode cutoff wavelength was determined on the same instrument by comparing the transmission spectra of the straight and bent fiber sections. Attenuation (α_0) and LP_{11} mode cutoff wavelength (λ_c) of single-mode OF are presented in Table 2.

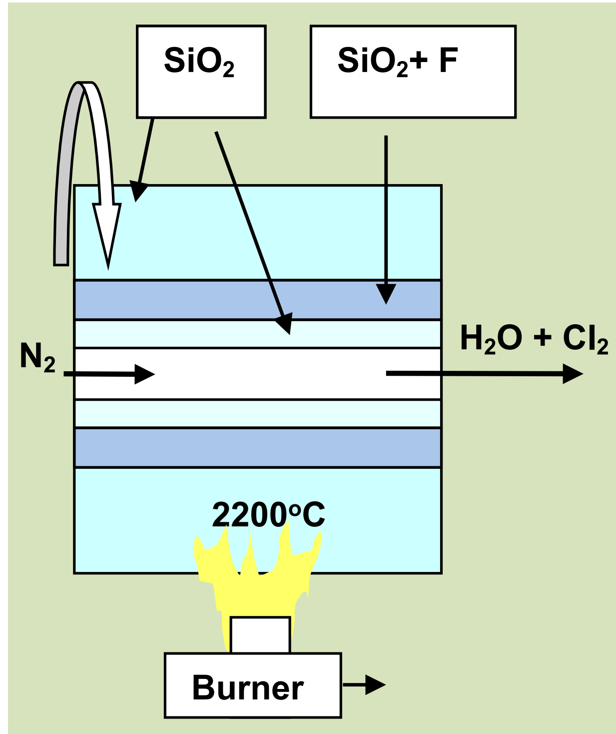

Fig. 1 High-temperature process for purification of silica core glass from impurity chlorine and OH groups

Table 2 OF parameters

λ_c, nm	α_0 (dB/ km) ($\lambda = 1310$ nm)	α_0 (dB /km) ($\lambda = 1550$ nm)
1150	0.3	0.18

The spectral dependence of OF attenuation revealed a low content of OH groups (Fig. 2), which is due to oxygen deficit in the core. The attenuation at a wavelength of

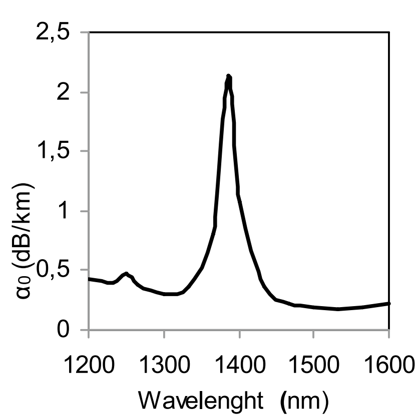

Fig. 2 Spectral dependence of OF attenuation (α_0) before its irradiation

1.38 μm, equal to 2 dB/km, corresponds to the content of this impurity 10^{-7} wt%.

Irradiation of 200 m long OF sections was carried out using a ^{60}Co γ source at a temperature of ≈ 25 °C, dose rate of 5.5 Gy/s and total dose of 1 MGy. The spectral dependence of attenuation in the fiber (α) was measured after its irradiation after 2 h, 3 and 10 months. The probing signal power did not exceed 1 μW. The spectral dependence of RIA was determined by the difference between the attenuation spectra of irradiated and non-irradiated fiber.

Simultaneously with the fiber, the coarse grains of F-300 grade silica glass and reduced glass fused from artificial quartz crystals in a hydrogen atmosphere were irradiated. The glass was ground to grains of less than 300 μm. The absorbance of the grains before and immediately after the irradiation was measured on a Bruker Tensor 37 spectrometer in the spectral range 1400—700 cm^{-1} with a resolution of 2 cm^{-1}.

3 Results

The investigation of our fabricated OFs shows that their RIA grows with an increasing wavelength (Fig. 3). In the spectral range of 1200–1600 nm no absorption bands due to any radiation defects have been revealed. The rate of relaxation processes of optical loss recovery in the fiber increases with a decreasing wavelength. 10 months after the irradiation, the OF RIA completely disappears at the wavelength of 1300 nm, while in the spectral region of 1600 nm it remains at the level of 5 dB/km. Therefore, the LWL RIA is more stable than the SWL RIA.

The RIA peak constancy in the 1380 nm region after the relaxation of radiation defects for 10 months should be noted, indicating the high stability of the radiation-induced OH groups. However, during the relaxation process, a shape change of this peak and a shift of its center to the long-wavelength region are observed. This fact is determined by two processes:

– increase with wavelength of the light propagating fraction in the cladding;
– more effective RIA relaxation of the core material compared to the fluorosilicate cladding.

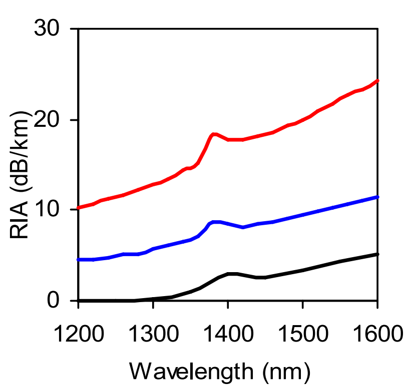

Fig. 3 OF RIA after γ-irradiation with a dose of 1 MGy after 2 h (the red line), 3 months (the blue line) and 10 months (the black line)

Figures 4 and 5 shows the absorption spectral dependence of F 300 silica glass and reduced oxygen-deficit glass. The data is normalized by the absorption maximum. The combination of vibrations of different structural elements in the glass network determines the complex shape of the absorption band. The band in the region of 800 cm^{-1} is related to the specificity of oxygen atoms vibrations [17].

The increased oxygen content in silica glass causes the formation of radiation defects in the form of non-bridging oxygen. The authors of [14] believe that such defects lead to the destruction of the glass network. For this reason, the spectra of irradiated silica glass with different oxygen contents differ significantly (Figs. 4, 5).

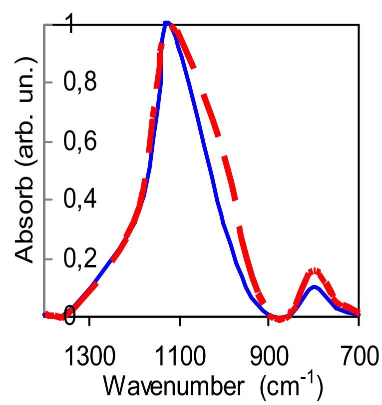

Fig. 4 Spectral dependence of silica glass F 300 absorbance before (the blue solid line) and after (the red dashed line) γ-irradiation with a dose of 1 MGy

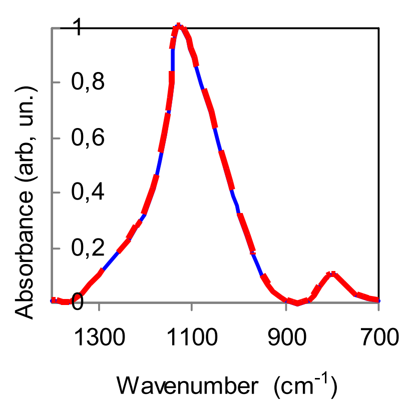

Fig. 5 Spectral dependence of reduced silica glass absorbance before (the blue solid line) and after (the red dashed line) γ-irradiation with a dose of 1 MGy

4 Discussion

Large OF γ-irradiation doses (more than 0.5 MGy) indicate the superior radiation resistance of our OF fiber compared to the recently published characteristics of the analog [18] (Table 3). The RIAs for the compared fibers do not differ significantly, while the irradiation dose of our OF is almost twice as high.

The radiation resistance of OFs without oxygen deficit in the core in the paper published in 1989 [13] is also significantly inferior to our OFs. Thus, at an irradiation dose of 1 MGy and a dose rate of 2.77 Gy s^{-1}, the RIA of such fibers at a wavelength of 1300 nm is 80 dB/km, while for our OFs at the same γ-irradiation dose it is significantly lower (12.5 dB/km) according to Table 3.

The results of our studies on the RIA change after γ-irradiation are well approximated (Fig. 6) by the formula proposed in [13]:

$$lnRIA(t) = lnRIA_0 - t/\tau, \quad (1)$$

where RIA_0 is the attenuation up to the moment of stopping OF irradiation, t is the time after irradiation, τ is the parameter of RIA relaxation time depending on the wavelength.

The results extrapolating this relationship to 3.5 years with τ equal to 2300 and 2650 h for both 1300 and 1600 nm wavelengths give a residual attenuation of less than 0.1 dB/km. For a similar OF but without oxygen deficit in the core, the RIA after such relaxation time in this spectral range decreased only by 40–50% [18].

The comparison of spectra under irradiation (Figs. 4 and 5) indicates an increase in radiation induced absorption for the glass with increasing the oxygen content. This, in turn, affects the RIA fourth overtone of the fundamental vibration band of the glass network vibration. This overtone band with an attenuation of ≈ 40 dB/km is revealed in the 1900 nm region on the spectral dependence of the attenuation of OF with a silica glass core and fluorine cladding [19]. In work [18], the overtone band absorption intensity for similar OF does not differ significantly (≈ 30 dB/km).

Earlier in [20, 21] it was already noted that γ-irradiation leads to a change in the fundamental absorption band by vibrations of silica glass network atoms. The destruction of the glassy state matrix of SiO_2 is provoked by the formation of non-bridging oxygen [14]. As our studies show (Figs. 4 and 5), with the reduction of silica glass the change of its fundamental band under γ-irradiation does not occur. However, in the absence of oxygen deficit in silica glass, γ-irradiation changes its fundamental spectrum, especially in the long-wavelength region (Fig. 4). Therefore, the LWL RIA is most likely caused by a change in the spectrum of the fourth overtone of the silica glass vibration spectrum in the 1900 nm region, rather than STH.

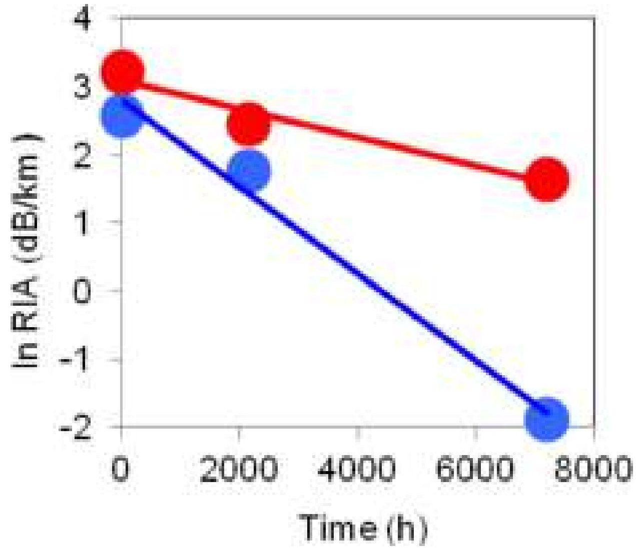

Fig. 6 Linear approximation of RIA relaxation in ln RIA-time coordinates based on experimental data (Fig. 3) for wavelengths of 1.3 μm (the blue line) and 1.6 μm (the red line)

The position of this band in the optical spectrum can change when the fundamental spectrum of doping silica glass is shifted. Indeed, the experiments [9] qualitatively confirm the corresponding RIA shift of this band to the long-wavelength region when doping silica glass with GeO_2.

The main results on the study of RIA in the long-wavelength region for OF with pure silica glass core and fluorosilicate cladding are performed for single-mode fibers. In such OF with increasing wavelength, the growth of RIA can occur due to an increase in the fraction of light penetrating into the cladding, which has low radiation-optical resistance. Taking into account this circumstance, multimode fibers in which radiation propagates only through the silica glass core were used in [14]. In such fibers, a broad RIA band of the 2000 nm region is also observed, indicating that this phenomenon is due to the core material property.

The other mechanism of LWL RIA is due to the increase in the concentration of OH groups during the irradiation of silica glass. As known, an increase in the content of this impurity causes a significant increase in the attenuation in OF based on silica glass in the region of 1400–1600 nm [22]. Obviously, the formation of OH groups leads to the same destruction of the

Table 3 Effect of oxygen deficit in the core of a single-mode OF with fluorosilicate cladding on its RIA after γ-irradiation with a dose greater than 0.5 MGy

OF type	Dose (MGy)	Dose rate (Gy/s)	RIA (dB/km) (λ = 1300 nm)	RIA (dB/km) (λ = 1550 nm)
Our OF with oxygen deficit	1	5.5	12.5	23
OF [18] without oxygen deficit	0.59	7.6	14.1	23.3

glass mesh as non-bridging oxygen. It can be assumed that the LWL of RIA is due only to the short-wavelength absorption edge of OH groups with a maximum in the region of 2.73 µm. However, the replacement of hydrogen by deuterium shifts this band but does not significantly change the RIA in the 1.6 µm region when the glass is γ-irradiated with a dose of 1.88 MGy [23]. This fact indicates the destructive effect of OH groups on the silica glass network.

In oxygen-deficit silica glass, γ-irradiation decreases the content of OH groups [24], which may provide a reduction in LWL RIA.

When analyzing the RIA of single-mode fluorosilicate OFs, an increase with wavelength of 20–40% of the radiation penetration into the cladding should be considered. Its fluorine content is known [25] to reduce the radiation resistance of silica glass. In our OF, fluorination of porous cladding glass was carried out in an atmosphere of fluorine-containing gas (SiF_4) diluted with inert gas [26], rather than oxygen, as in the technology of similar fibers. Therefore, such conditions for the formation of the fluorine-doped cladding ensured oxygen deficit in it, thus contributing to the RIA reduction of our single-mode OFs.

It should be noted that the excess of oxygen in silica glass can change its phonon spectrum and, as a consequence, increase the intensity of the combination absorption band of OH groups at a wavelength of 1250 nm. The ratio of the hydroxyl absorption bands intensities at wavelengths of 1380 and 1250 nm in silica glass OF with excess oxygen is ≈ 3 [5, 22]; while in our OF with oxygen-deficit silica core (Fig. 2) as in synthetic silica glass [27], this ratio is ≈ 25. Thus, this ratio can be an indicator of the oxygen content of silica glass.

The oxygen deficit in silica glass is also important for reducing optical losses in the region of its special transparency at λ ≈ 1550 nm. The silica glass is doped with nitrogen at a very low partial oxygen pressure (≈ 10^{-7} Pa) [28], which provides a large oxygen deficit. Therefore, the attenuation in nitrogen-doped OFs with increasing wavelength starts at 1700 nm and reaches 2 dB/km at 1800 nm [29].

In OF with silica glass core without oxygen deficit, the attenuation rise with increasing wavelength starts at 1600 nm and exceeds 6 dB/km in the 1800 nm region [5, 18].

Observing the high radiation resistance of the fibers studied by us, it should be noted that their RIA is due to the material of the solid core. Therefore, fibers of a new type with a hollow core may be more promising in this respect [30].

5 Conclusion

Based on the study of the effect of γ-irradiation on optical losses of single-mode OFs with pure silica glass core, the following conclusions regarding the novelty and significance of the work can be drawn:

1. Creating oxygen deficit in silica glass contributes to the reduction of both its optical loss and RIA at high γ-irradiation doses in the region of its special transparency from 1300 to 1600 nm.
2. The main cause of RIA at λ above 1300 nm occurs due to the change in the vibration spectrum of the silica glass matrix, which determines its attenuation in the 1900 nm region for the fourth overtone of the fundamental absorption band.
3. The destruction of the glass network under γ-irradiation results from the formation of non-bridged oxygen and OH groups.
4. Oxygen deficit in the silica glass core OF significantly reduces the duration of RIA relaxation after irradiation.

The work results indicate the need to investigate the oxygen deficit degree in silica glass influence on its attenuation and radiation resistance in the 1300–1600 nm spectral regions. This is of high importance for the development of OF technology with extremely low attenuation at a wavelength of 1550 nm for long-distance communication lines.

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Data Availability No datasets were generated or analysed during the current study.

Declarations

Ethical Approval Not applicable.

Conflict of Interest The authors declare no competing interests.

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