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Irradiation Effects in Optical Fibers

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1. Introduction

Intrinsic and extrinsic optical fiber-based sensors are promising devices to be used in very different and complex environments, by their very nature: capabilities to work under electromagnetic fields; possibility to carry multiplexed signals (time, wavelength multiplexing); small size and low mass; ability to handle multi-parameter measurements in distributed configuration; possibility to monitor sites far away from the controller. In the case of the optical fibers, the possibility to be incorporated into various types of sensors and actuators, free of additional hazards (i.e. fire, explosion), made them promising candidates to operate in adverse conditions as those required by space applications and terrestrial nuclear facilities (Alam et. al.ª, 2006; Alam et al.^b, 2006; Berghmans et al., 2008; Ott, 2002). In nuclear environments optical fibers found an application niche in optical communication links, embedded into various all-fiber or hybrid sensors or as light-guides for control and diagnostics (Alfeeli et al., 2007; Ahrens et al., 2001; Fernando et al., 2005; Fielder et al., 2005; Florous et al., 2007; Gan et al. 2008; Henschel et al., 2001; Kimurai et al. 2002; O'Keeffe et al. 2008; Reichle et al., 2007; Troska et al., 2003). For applications related to fusion installations the requirements are quite demanding because of the exposure to (Campbell, 2005; Griscom, 1998; Hodgson, 2006; ITER Physics Expert Group on Diagnostics, 1999; Shikama, 2003; Zabezhailov, 2005): ionising radiation, high temperature, and high electromagnetic disturbances.

One of the major drawbacks for optical fibers use under ionizing radiation is related to the development of colour centres, which affect dramatically the optical transmission in UV-visible-NIR spectral ranges (Griscom, 1998; Karlitschek, 1995). For this reason, optical fibers are by more than 30 years in the focus of colour centres research (Friebele, 1976; Kaiser, 1974).

Research on radiation induced colour centres in pure and doped bulk silica materials has a long history of over 50 years (Weeks, 1956), but it is still actual (Radiation effects, 2007; Devine et al., 2000; Pacchioni et al., 2000), as new materials and devices (optical fibers, waveguides, multiplexers, or fiber lasers) are continuously devised and evaluated. Apart from the diversity of the investigated materials and devices new challenges are presented by the various irradiation conditions to which such materials and devices are subjected.

The complexity of the colour centres dynamics lead to the use of complementary methods to individuate these centres (electron paramagnetic resonance: EPR, luminescence) besides the

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traditional optical absorption spectroscopy. New insights are also provided by in-situ experiments, when optical absorption changes are monitored during the irradiation, aiming to evaluate the irradiation effects disentangled from post irradiation relaxation and with the target to go deeper into the mechanisms of defects generation.

This chapter reports original results concerning the investigations carried out on optical fibers irradiated by gamma-ray, beta-ray, and neutron, using comparative measurements of optical absorption, EPR and luminescence. The optical measurements were performed mostly in the UV spectral range as the most effective colour centres are generated there. The EPR measurements were used to detect the presence of the intrinsic point defects E' centers (Devine et al., 2000; Pacchioni et al., 2000), as well as H related paramagnetic point defects typically observed in H loaded fibers (Li et al, 1996; Karlitschek et al., 1998). Finally, the photoluminescence measurements were applied to reveal the presence of oxygen deficient defects (ODC(II)) induced by irradiation (Skuja, 1998; Skuja et al.; 2005).

2. The physics of colour centres

Up to now, most of the investigations on the use of optical fibers in radiation environments were done in relation to their use for sensing and data transfer. One approach of interest is related to the use of optical fibers for periscopes (light transmitting pipe-lights) for plasma diagnostics in fusion installations. Within such a task, the spectral behaviour of optical fibers in the UV-visible spectral range is of interest (Brichard, 2005).

Many years of study on bulk silica and on preforms or silica fibers have enabled to evidence optical absorption effects from IR up to vacuum-UV induced by the devices manufacturing or by their subsequent use during high intensity light propagation or in irradiation environments (Devine et al., 2000; Pacchioni et al., 2000; Skuja, 1998). The relevant role of the absorption in affecting the optical efficiency of the devices has lead to isolate the contributions of various absorption bands. Many efforts have been done also to assign these bands to given point defects and to determine specific procedures that could enable to prevent the same defect formation and/or could let their conversion into optically inactive species. A short review of the main intrinsic absorption bands occurring in the UV spectral range will be given in the following.

The ubiquitous and most easily induced absorption features in the UV range is the band at about 213 nm (5.8 eV), attributed to the E' centers (Pacchioni et al., 2000; Skuja, 1998). This band is characterized by an oscillator strength, *f*, of about 0.2, and can be well described by a Gaussian profile with full width at half maximum (FWHM) of about (0.7±0.1) eV. The point defect responsible for this absorption is characterized by a microscopic structure consisting in an unpaired electron in an approximately sp³ hybrid orbital of a dangling bond of a threefold coordinated silicon atom: $O=Si^{\circ}$, where the symbol = represents single bonds to three oxygen atoms and the symbol \bullet stays for the unpaired electron (Pacchioni et al., 2000).

The electronic excitation process associated to this absorption band has been strongly debated and, recently, support to its attribution to an electronic transition from the defect related bonding states to the unpaired electron level has been given (Agnello et al., 2008).

It is noteworthy to observe that this defect is also paramagnetic and is characterized by an EPR signal (Weeks 1956) well correlated to the optical absorption (Weeks et al., 1963) with principal g-values (Weil et al., 1994, Pacchioni et al., 2000): g_1 =2.0003, g_2 =2.0006, g_3 =2.0018.

Another relevant absorption band, diffusely observed in the UV range in various forms of irradiated silica, is the one peaked at about 258 nm (4.8 eV) (Pacchioni et al., 2000; Skuja, 1998). This band can be described by a gaussian profile with FWHM of 1.05 eV and oscillator strength f=0.03 (Cannas et al., 2006). Excitation within this absorption originates a well known red luminescence band at about 650 nm (1.9 eV) that has an additional excitation channel at 620 nm (2.0 eV). This overall optical activity has been attributed to the Nonbridging Oxygen Hole Centers (NBOHC) (Pacchioni et al., 2000). The microscopic model associated to this defect consists in a non-bridging oxygen atom: O=Si-O[•], having an unpaired electron in a non-bonding oxygen orbital (Pacchioni et al., 2000). The electronic transitions responsible for the absorption and emission of this defect are still debated but, usually, they are related to electron promotion from bonding states of the O=Si-O[•] moiety to the non bonding states of the dangling O and, in particular, to that of the unpaired electron (Bakos et al., 2004). Due to the presence of an unpaired electron in the oxygen orbital, this defect is responsible also for an EPR signal (Pacchioni et al., 2000; Skuja, 1998). By studying the dependence of this signal as a function of temperature the following main g values have been determined: g1=1.9999, g2=2.0095, g3=2.078 (Pacchioni et al., 2000). However, the identification of NBOHC by the EPR technique is complex due to the overlap with other signals and to the resonance line being spread over a wide magnetic field (about 80 G at 9.8 GHz microwave frequency, X-band) that prevents its detection at low defect concentrations (typically below 10^{16} centers/cm³).

Other defects responsible for the absorption increase around 258 nm (4.8 eV) are interstitial ozone molecule (O₃) and the peroxy radical: $O=Si-O-O^{\bullet}$ (Skuja et al.; 2005). Both these defects should be observed as product of irradiation in oxygen rich materials or as a consequence of oxygen displacements processes induced by irradiation. Their detection is however difficult due to the overlap with the NBOHC band and, in particular, the attribution of an absorption band to the peroxy radical in this spectral range is still a matter of debate (Skuja et al.; 2005).

A defect of particular relevance for the UV optical absorption in SiO_2 is also the oxygen deficient center: ODC(II) (Pacchioni et al., 2000; Skuja et al.; 2005; Skuja, 1998). This defect is usually associated to an absorption band peaked at 248 nm (5.0 eV) and gives rise to two emission bands at 460 nm (2.7 eV) and 280 nm (4.4 eV) (Skuja, 1998). The ODC(II) can be induced during fiber fabrication but it is also relevant in relation to the photosensitivity of silica and in connection to fiber photon writing processes (Pacchioni et al. 2000; Karlitschek et al., 1998). In this respect, the connection of the ODC(II) with another defect usually named H(I) center is of particular importance due to the use of hydrogen for radiation hardening of fibers. This latter defect is paramagnetic and is characterized by a doublet of EPR lines split by 74 G (Skuja, 1998). The structural model for the H(I), confirmed by many experimental investigations, consists in a silicon atom bonded with two oxygen and one hydrogen and having an unpaired electron on silicon: $O=Si^{-}$ H (Pacchioni et al., 2000; Skuja, 1998).

The existence of a conversion between ODC(II) and H(I) has been evidenced by studies on surface defects on silica and has been used to support the model of the ODC(II) as consisting in a two-fold coordinated silicon: O=Si^{••} (Radzig, 1998, Skuja, 1998).

Despite the almost clear identification of many absorption features in the UV spectral range, the processes of defect formation and defect stability are still matter of investigation, both experimentally and theoretically (Griscom, 2001; Mashkov et al. 1996; Kajihara et al., 2008). In particular, it has been shown that the defect formation processes feature a complex

dynamics related to the presence of precursor of the defects and also affected by the inhomogeneity intrinsic to the amorphous nature of the material. Furthermore, the stability of irradiation induced defects is affected by the presence of species present in the material and, in particular, it has been evidenced that H related species (H, H₂, H₂O) usually affects the formation and thermal removal of many defects as NBOHC, E' and ODC(II) (Griscom et al., 1998; Devine et al., 2000; Pacchioni et al. 2000; Nuccio et al. 2009; Messina et al. 2009).

3. Experiment

The goals of the here reported investigations were: to further improve the optical absorption measurements in the UV-visible spectral range in fibers; to evaluate the possibility to use alternative methods for identifying colour centres in fibers (i.e. EPR, luminescence); to check through an inter-laboratory comparison the capabilities of two laboratories located in Italy and Romania in performing EPR tests on irradiated optical fibers.

The investigated optical fibers were commercially available products. The characteristics of some of the investigated optical fibers are summarised in Table I.

Characteristics	Core	Cladding	Maximum	
Optical fiber sample nickname	diameter (µm)	diameter (µm)	operating temperature (°C)	Jacket type
S42-1	1000	1035 (TECS)	-	Tefzel
S43-1	400	440 (doped silica)	150	Tefzel
S44-1	600	660 (doped silica)	300	Polyimide
S45-1	600	660 (doped silica)	300	Polyimide
S46-1	600	660 (doped silica)	300	Polyimide
S47-1	400	500	-	Tefzel

Table I. The irradiated optical fibers.

The optical fibers are either solarization resistant optical fibers, optical fibers with enhanced UV transmission or radiation hardened optical fibers developed for UV applications.

The length of the samples for the optical absorption measurements was 120 mm. For the case of EPR and luminescence tests five pieces from the same optical fibers samples, without jacket and having a length of 10 mm each, were measured in the same time.

3.1 Set-ups

The general set-up for the off-line optical absorbance measurements is similar to that previously used (Sporea, 2005; Sporea, 2007), but, for the purpose of this investigation, it has a better S/N ratio (1,000:1 full signal), 16 bits A/D conversion resolution, a dynamic range of 25,000:1, a greater quantum efficiency in the UV range (65 % at 250 nm), spectral resolution 1.2 nm, a sensitivity of 0.065 counts/e-, and a minimum OD detection level of 0.4 for the optical set-up used (this value is determined by two factors: first, the core of the

connecting optical fibers and the core of the samples are different, and second, the sample optical fibers have no fixed connectors, hence, a biasing level which limits the set-up lowest detectable OD). Such a detecting scheme makes possible a better tracking of the colour centre development in the UV spectral range and enables a higher range of absorption levels to be detected (O.D. of 4.4). For the reported optical absorption curves the signal was averaged over three detected acquisitions with a value of 2 for the box car parameter. Irradiation and off-line measurements were carried out at room temperature.

Depending on the case, various set-ups were used for on-line measurements. During the gamma, beta and neutron irradiations tests were carried out both at room temperature and with the optical fiber heating. For these purposes specially designed set-ups were built (Figure 1), to make possible the simultaneous irradiation at room temperature (upper plate) and with the heating of the optical fibers (lower plate). In the case of neutron and beta irradiation a small oven was constructed which made possible the irradiation of the optical fibers in a linear geometry. For on-line measurements during gamma irradiation a circular geometry was used, the optical fiber samples (items 4 and 7 in Fig. 1 b) were coiled with a diameter of about 130 mm, and the gamma source was placed at the coil centre (item 6 in Fig. 1 b).

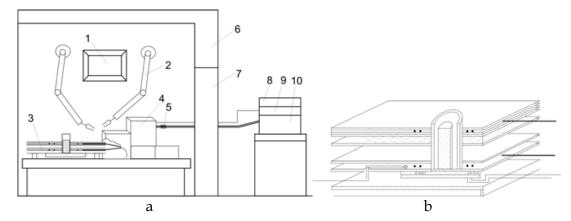


Fig. 1. The set-up for on-line measurements of gamma-irradiated optical fibers at room temperature and under heating conditions: a – the sketch of the irradiation hot-cell (1 – hot – cell window; 2 – remote manipulators; 3 – set-up for the optical fibers positioning; 4 – shielding; 5 – connecting optical fibers; 6 – hot-cell room; 7- hot cell access door; 8 – optical fiber mini spectrometer; 9 – broad band stabilized light source; 10 – laptop); b – detail of the set-up for the optical fiber positioning and heating.

The design of the special set-up made possible the irradiation at room temperature of one sample while a second sample was heated up to 240 °C. The possibility to have the sample heated during the irradiation enables the study of the colour centres dynamics under temperature stress.

3.2 Irradiations and measurements

The ⁶⁰Co gamma-ray off-line irradiations have been performed at room temperature by using the SVST-Co-60/B, tote-box type, multipurpose irradiation facility of the National Institute for Physics and Nuclear Engineering – Horia Hulubei (IFIN-HH). The SVST-Co-60/B irradiator has today an activity of 267000 Ci, and the irradiations were performed in the irradiation chamber, at a dose rate ~ 700 Gy/h.

For the absorbed dose measurements two dosimetric systems are used: ALANINE / EPR dosimetry system, calibrated for 20 Gy – 10 kGy range, and ECB dosimetric system, calibrated for 1 kGy – 100 kGy ranges. The measured absorbed doses for both the systems are traceable to RISO High Dose Reference Laboratory – Denmark, and their performances were tested by international dosimetric intercomparisons. The uncertainty of absorbed dose measurement is around 3%.

The on-line irradiations were performed in a hot-cell at the ⁶⁰Co gamma-ray irradiation facility of the "Horia Hulubei" National Institute of R&D for Physics and Nuclear Engineering–IFIN-HH. The dose rate can be modified by changing the distance of the optical fiber sample from the gamma source (i.e. a dose rate of 5.3 kGy/ h at 6 cm distance form the source).

The beta irradiation conditions were the following: the mean electron energy: 6 MeV, the electron beam current: 1 μ A, the pulse repetition rate: 100 Hz, the pulse duration: 3.5 μ s, the beam diameter: 10 cm, the spot uniformity: +/- 5 %. In order to have Bremsstrahlung, the electron beam collided on a tungsten target, and gamma-rays were generated. To separate the electron flux from the gamma-rays two Al foils, one having a thickness of 3 mm, and one of 4 mm were placed at the distances of 10 mm and respectively at 120 mm away from the target.

The neutron irradiation was performed at the Cyclotron accelerator facility of the National Institute of R&D for Physics and Nuclear Engineering "Horia Hulubei", Magurele, based on the reaction 9Be + d \rightarrow n + X, using a deuteron beam (13 MeV) and a thick beryllium target of 165 mg.cm⁻². To obtain the desired neutrons fluencies the samples are located downstream at the distances from 10 to 40 cm from the Be target. The neutrons flux above 1 MeV is estimated with a relative error of about 20 %. The maximum neutron flux achievable in our setup, at a distance of 10 cm from the target, is 2.10⁹ n.cm⁻².s⁻¹.µA⁻¹, corresponding to a deuteron beam intensity of 10 µA. In practice, a neutron fluence of 10¹³ n.cm⁻² can be obtained in about 1-6 days of irradiation, depending on the position of the samples in respect to the Be target. It is estimated that the neutron flux was uniform over 10 mm length of the optical fiber sample, with a spatial uniformity of 3%.

The EPR measurements were performed in Romania at the IRASM irradiation facility (National Institute for Physics and Nuclear Engineering – "Horia Hulubei") using an X band Magnettech MiniScope MS 200 spectrometer provided with a rectangular TE_{102} resonant cavity and 100 kHz modulation frequency.

Both non-irradiated and irradiated samples have been introduced into fused quartz tubes (1 cm sample length, 5 mm outer tube diameter) and fixed into the resonant cavity always in the same position. The mass of measured samples varied between 20 and 130 mg. The EPR spectra have been registered under the experimental conditions, detailed in the following figures, and with the magnetic field sweep rate of 18 G/min. The g-factors of the observed EPR signals have been determined by using a ZnS: Mn^{2+} standard. Similar measurements were done at the University of Palermo on a Bruker EMX-micro working in X-band (about 9.8 GHz) with a rectangular TE_{102} resonant cavity and at 100 kHz magnetic field modulation using an EPR tube, 10 mm outer diameter, for sample mount. For specific colour centres detection, a preliminary calibration of the two instruments (the Magnettech MiniScope MS 200 spectrometer in Romania and the Bruker EMX-micro in Italy) was run using a gamma ray irradiated sample of high purity silica to compare the sensitivity of the two instruments. Based on this calibration, additional parallel measurements were performed in the two laboratories and with the equipment of Palermo a concentration estimate has been carried out.

The luminescence effects were investigated at this stage only in Palermo with a Jasco FP6500 spectrofluorometer using a 150 W Xenon lamp. Excitation light impinged on the fiber samples perpendicularly to their axis using an artwork mount at 45° back-scattering configuration, and emission light was collected at 90° with respect to the excitation direction.

4. Results

Figures 2-4 illustrate the optical absorbance of the irradiated optical fibers as they were subjected to various total gamma irradiation doses. These spectra evidence that irradiations mainly induce the formation of bands below 300 nm that could be associated to E'-Si centres and Non-Bridging oxygen hole centres (Skuja, 1998), whose amplitude increase on increasing the irradiation dose. At higher wavelength negligible absorption is found. It can be observed that the fibers S44, S45 and S46 are more radiation resistant than the others.

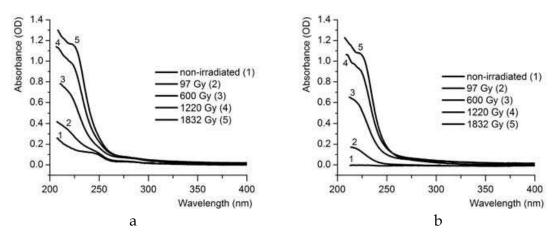


Fig. 2. Optical absorption spectra in optical density units (OD) for: a - sample S42-1; b - sample S43-1.

Optical fibers S44-1, S45-1 and S46-1 are hydrogen loaded type optical fibers, so they exhibit a higher radiation hardening than the other samples. Optical fibers S42-1 and S43-1 are optical fibers with an enhanced UV response and have a high OH content. The optical fiber S47-1 was designed for UV laser beam delivery applications with high fluorine content.

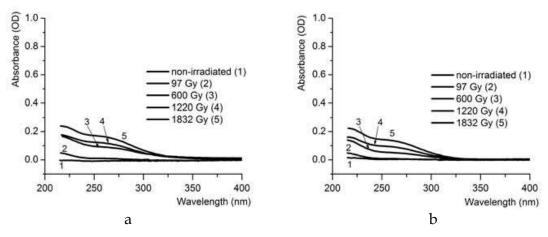


Fig. 3. Optical absorption spectra in optical density units (OD) for: a - sample S44-1; b - sample S45-1.

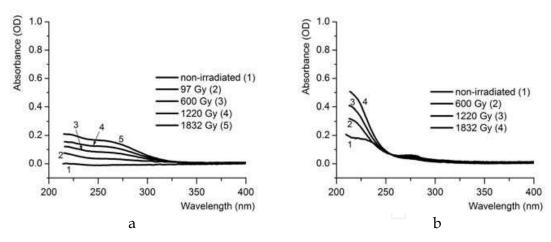


Fig. 4. Optical absorption spectra in optical density units (OD) for: a -sample S46-1; b - sample S47-1.

Decomposition of the spectra as a function of energy obtained from the data of fibers S43, S44 and S45 (Figs. 2, 3) has been carried out using Gaussian bands. This decomposition enabled to further explore the presence of specific point defects in the irradiated fibers.

In Fig. 5 an example of the decomposition result is reported for the fiber S45 at the various irradiation doses. The found bands are characterized by peak position (4.8 ± 0.1) eV and FWHM (1.0 ± 0.1) eV, and by (5.7 ± 0.1) eV and FWHM (0.8 ± 0.1) eV, in reasonable agreement with the bands attributed to the NBOHC and to the E' center (Pacchioni et al., 2000; Skuja, 1998), respectively.

It is found also that more than 70% of the absorption spectra is attributed to the absorption bands of the E' center and of the NBOHC. A further band peaked at 198 nm (6.25 eV), whose origin is unknown, is necessary to describe the absorption below 210 nm (at energy larger than 5.9 eV). Usually this band contributes less than 25% to the overall decomposition. It was also found that these Gaussian amplitudes increase for all investigated dose evidencing that the induced absorption is related to the processes of these defects generation.

Similar results are found for the other investigated fibers. As can be observed the spectra of the S43 sample (Fig. 2) feature a low contribution at about 258 nm (4.8 eV) with respect to the component at 213 nm (5.8 eV), at variance to the samples S44, S45 and S46 (Fig. 3, 4). These aspects are confirmed by the spectra decomposition. Since the ratio between the composing bands, and in particular those related to the E' center and to the NBOHC, is not constant and depends on the fiber, it can be guessed that the microscopic process of defects generation depends on the specific preparation method and could be affected by hydrogen loading (Devine et al., 2000, Karlitschek et al., 1998).

In Fig. 6 the change of the optical attenuation for an optical fiber with enhanced UV transmission, 400 μ m core diameter, and Tefzel jacketing (sample similar to S43-1) under neutron irradiation at a total fluence of $1.18 \times 10^{13} \text{ n/cm}^2$ is given as an example.

It is observed that also in this case the absorption increases non-linearly with the fluence for wavelengths below 400 nm. Due to the instrumental reduced sensitivity below 235 nm no specific feature can be associated to point defects in this case.

A solarization resistant 600 µm core diameter, Polyimide jacketed optical fiber (sample similar to S46-1) was subjected to beta irradiation and separately to Bremsstrahlung (Fig. 7a), at room temperature. During an additional experiment the same type of optical fiber

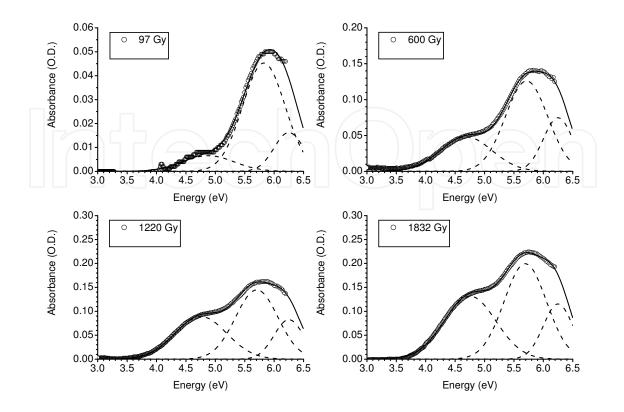


Fig. 5. Spectral decomposition using Gaussian bands of the spectra of sample S45-1 irradiated at various doses; the circles represent experimental points, the dashed lines the component Gaussian bands (peaked at about 4.8 eV, 5.8 eV and 6.25 eV) and the full line the sum of dashed lines.

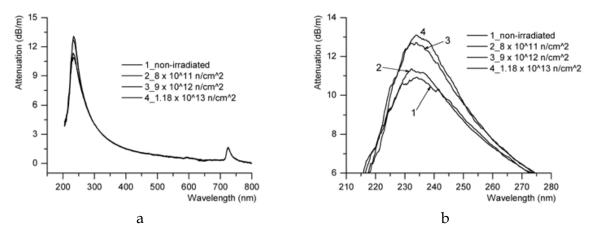


Fig. 6. The optical absorption spectra in the UV-visible range for an optical fiber with enhanced UV transmission, subjected to neutron irradiation: a - the UV-visible spectrum; b – detail of the attenuation spectrum in the 210 nm - 280 nm spectral range.

was also heated during the irradiation (Fig. 7b). The tested optical fibers were of hydrogen loaded type and exhibit a lower radiation hardening under heating conditions, as the temperature contributes to hydrogen diffusion.

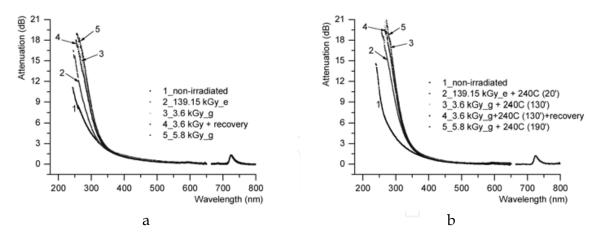


Fig. 7. The change of the optical attenuation of an optical fiber subjected to beta irradiation (total dose of 139.15 kGy_e) and to Bremsstrahlung (total dose of 5.8 kGy_g): a - at room temperature; b – heated at 240 °C during the irradiation.

Investigation of the effects of irradiation on these fibers has been also carried out by EPR measurements. As shown in Fig. 8, these measurements evidenced the presence of resonance signals in some non-irradiated samples. A detailed investigation carried out in Palermo has shown that these signals do not arise from intrinsic defects of silica.

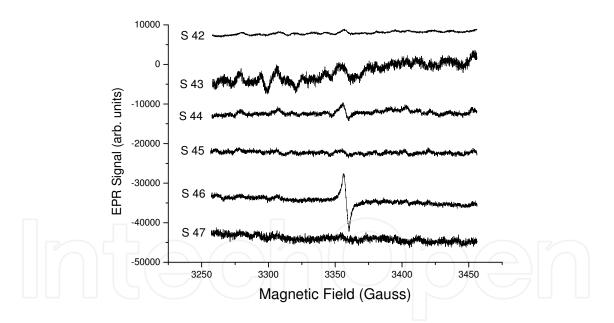


Fig. 8. The EPR signals for the non-irradiated samples corrected for sample mass (Power = 3.2 mW; modulation amplitude = 4 G; scanning time = 655 s; receiver gain = 9×10^2). Spectra are arbitrarily vertically shifted.

The EPR measurements carried out on the irradiated fibers are reported in Figs. 9 and 10. It is worth to note that in these figures the spectra recorded for the irradiated samples subtracted by spectra recorded for the respective pristine materials are reported, and show that the defects contents increase by irradiation. Furthermore, the higher radiation resistance of the samples S44, S45, S46 with respect to the other investigated fibers, already evidenced by optical measurements, is confirmed also by the EPR measurements.

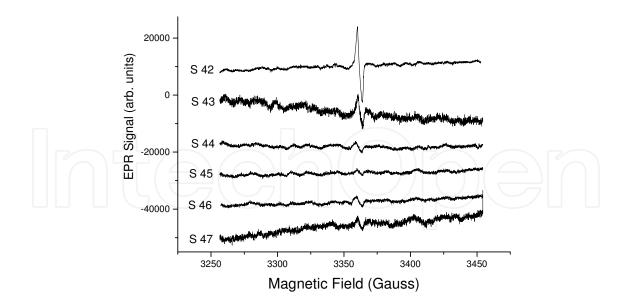


Fig. 9. The EPR spectra for 618 Gy irradiated samples corrected for sample mass, after nonirradiated signal subtraction (Power = 3.2 mW; modulation amplitude = 4 G; scanning time = 655 s; receiver gain: 1×10^2 for S 42 and 9×10^2 for the others). Spectra are arbitrarily vertically shifted.

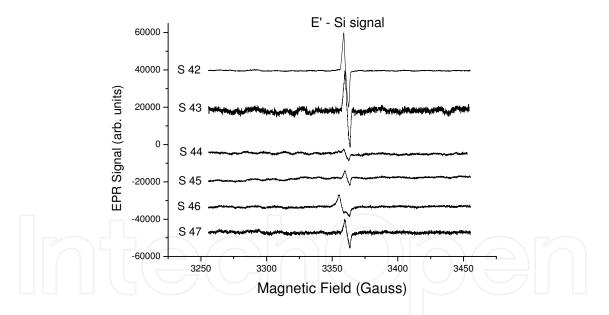


Fig. 10. The EPR spectra for 1800 Gy irradiated samples corrected for sample mass, after non-irradiated signal subtraction. (Power = 3.2 mW; modulation amplitude = 4 G; scanning time = 655 s; receiver gain: 1×10^2 for S 42 and 9×10^2 for the others). Spectra are arbitrarily vertically shifted.

In addition to the optical fiber specified in Table I, several irradiated samples (S7.7; S7.9; S1-440B and S3-440B) were investigated through EPR for comparative studies both in Bucharest and Palermo. The results of EPR measurements performed in Bucharest are given in Figs. 11-13 (spectra are arbitrarily vertically shifted and are not normalized for the samples weight).

These measurements evidence the presence of E'-Si centres (Pacchioni et al., 2000; Karlitschek, 1995) and of the H(I) defects in some of the irradiated samples (Skuja, 1998). The presence of H(I) defects, identified by the doublet of lines split by about 74G, is evidenced by the magnified spectrum.

The same measurements have been done in Palermo showing qualitatively the same results, and supporting the calibration procedure to individuate specific point defects induced by irradiation. Furthermore, the quantitative analysis carried out in Palermo, enabled to estimate the concentrations of E'-Si and H(I) point defects as reported in Table II.

Optical fiber sample nickname	Sample mass (g)	E'-Si defects (centers/cm ³)	H(I) defects (centers/cm ³)
S7.7	0.124	8.2 x 10 ¹⁶	1.2 x 10 ¹⁵
S7.9	0.132	5.7 x 10 ¹⁶	$1.0 \ge 10^{15}$
S1_440B	0.023	$3.3 \ge 10^{16}$	Not detected
S3_440B	0.020	$3.1 \ge 10^{16}$	Not detected
SSU1.1	0.052	Not detected	Not detected

Table II. The concentration of E'-Si and H(I) point defects.

For some non-irradiated and irradiated samples luminescence measurements were performed (Fig. 14). These measurements have shown that in the non-irradiated samples no PL associated to intrinsic point defects of silica could be detected.

As shown in Fig. 14, in the irradiated fibers S7.9, S1_440B and S3_440B, the luminescence centred at about 4.4 eV excited at 5.0 eV associated to the oxygen deficient ODC(II) defects is detected (Skuja, 1998).

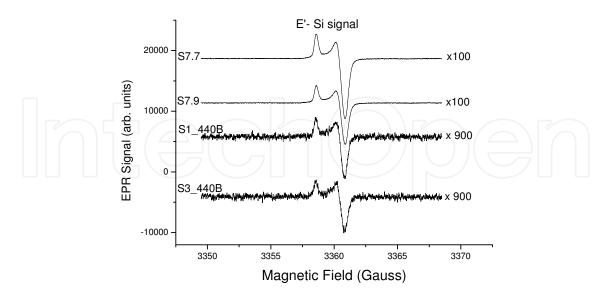


Fig. 11. The EPR signals for some irradiated samples at 113 kGy (samples S7.7 and S7.9) and at 40 kGy (samples S1-440B and S3-440B). Power = 0.1 mW; modulation amplitude = 0.1 G; scanning time = 655s; receiver gain = 1×10^2 for S7.7 and S7.9 and 9×10^2 for S1_440B and S3_440B.

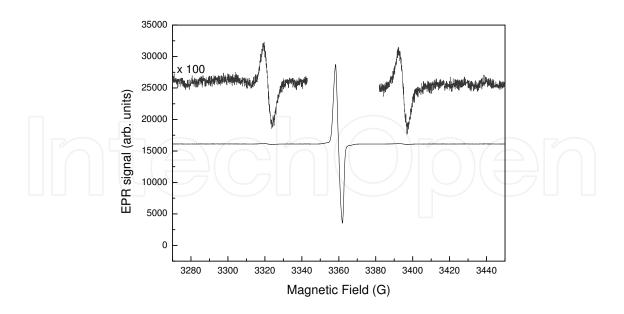


Fig. 12. The EPR signals recorded in Bucharest for the S7.7 sample irradiated at the dose of 113 kGy.

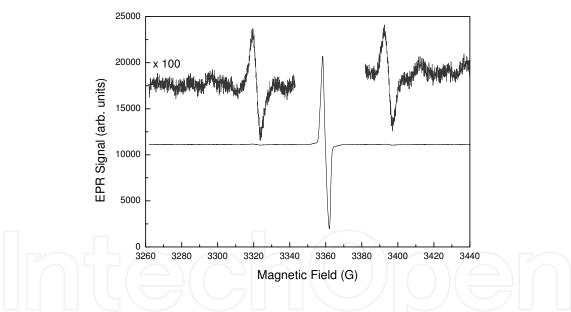


Fig. 13. The EPR signals recorded in Bucharest for the S7.9 sample irradiated at 113 kGy.

Sample S.7.7 was a common, 1000 μ m core diameter optical fiber, gamma irradiated up to 113 kGy, in 4 steps, with sample heating at 100 °C for 4 h between each irradiation step. The optical fiber S.7.9 was a solarization resistant optical fiber of 1000 μ m core diameter, gamma irradiated to 113 kGy. Samples S1-400B and S3-440B were of the same type (enhanced UV transmission), irradiated by gamma-rays up to a total dose of 40 kGy, the first one was also subjected to temperature stress (140 °C for 4 h, between the irradiation steps). In Fig. 14, the SSU1.1 fiber, not showing emission, is reported for comparison. Photoluminescence excitation spectra confirmed these attributions by evidencing an excitation band centred at 5.0 eV, as reported in literature for ODC(II) (Skuja, 1998). The presence in the sample S7.9 of

the ODC(II) emission band agrees with the observation of the H(I) defects that, as reported in the paragraph 2, can be considered a product of the reaction between ODC(II) and H species dissolved in the sample (Skuja, 1998). As regards the samples S3_440B and S1_440B, the absence of H(I) centers signal is also compatible with the much lower amplitude of the emission band of ODC(II), proving a lower content of these latter defects.

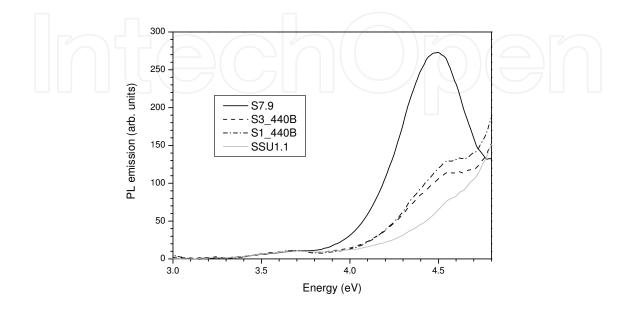


Fig. 14. Photoluminescence spectra excited at 5.0 eV of fibers irradiated at 113 kGy (sample S7.9), at 40 kGy (samples S1-440B and S3-440B) and at 500 kGy (sample SSU1.1).

5. Future work

Further investigations on the generation/recovery of colour centres in the UV-visible spectral range are of interest as new radiation hardened optical fibers are developed and more accurate measuring means are available. We shall focus in the near future on the evaluation of the irradiation effects on commercially optical fibers from different vendors in order to assess the role played in such cases by the dopants, core/cladding ratio, temperature stress. On-line measurements have to be extended to have a better understanding of the colour centre dynamics. Tests at higher irradiation dose and dose rates will be run for gamma and beta rays irradiation. To complement the inter-laboratory comparison on EPR measurements a supplementary inter-comparison dedicated to photoluminescence investigations will be organized between laboratories in Romanian and Italy.

6. Conclusions

Eleven types of commercially available optical fibers operating in the UV spectral range were investigated as they were subjected to gamma, beta, and neutron irradiation. The evaluation of the irradiation induced colour centres was done by off-line optical absorption measurements, EPR, and luminescence. E'-Si and H(I) defects were revealed by EPR, the

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ODC(II) diamagnetic centres were identified by photoluminescence. From the optical absorption measurements it is observed that the induced absorption increases for all the investigated doses suggesting a growth of the concentration of induced colour centres, with a prominent role of the E' centers and of the NBOHC. A dependence of irradiation effects on the materials is found.

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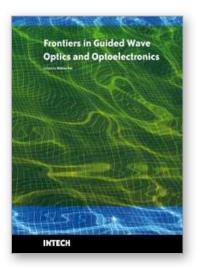
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