Investigation of UV optical fibers under synchrotron irradiation
Dan Sporea, Laura Mihai, Adelina Sporea, Alin Lixandru, Elke Braeuer-Krisch

To cite this version:
Dan Sporea, Laura Mihai, Adelina Sporea, Alin Lixandru, Elke Braeuer-Krisch. Investigation of UV optical fibers under synchrotron irradiation. Optics Express, Optical Society of America, 2014, 22 (25), pp.31473-31485. 10.1364/OE.22.031473 . hal-01572961

HAL Id: hal-01572961
https://hal.archives-ouvertes.fr/hal-01572961
Submitted on 8 Aug 2017

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L’archive ouverte pluridisciplinaire HAL, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d’enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.
Investigation of UV optical fibers under synchrotron irradiation

Dan Sporea,1* Laura Mihai,1 Adelina Sporea,1 Alin Lixandru,2 and Elke Bräuer-Krisch3

1National Institute for Laser, Plasma and Radiation Physics, 409 Atomistilor St., Magurele, RO-077125, Romania
2“Politehnica” University, 313 Splaiul Independenţei, Bucharest, RO-060042, Romania
3European Synchrotron Radiation Facility, BP 220 6, rue Jules Horowitz, 38043 Grenoble cedex, France
dan.sporea@inflpr.ro

Abstract: The focus of the present paper deals, for the first time, with commercial UV optical fibers, characterizing their behaviour as they are subjected to very high flux wiggler generated synchrotron radiation. Five distinct types of UV optical fibers, produced by three manufactures, were exposed to total doses between 5 Gy and 2000 Gy. The exposure to synchrotron radiation was performed in two campaigns. The tests were run off-line and considered the dependence of the radiation induced attenuation (RIA) as function of the total dose. The recovery of the radiation induced colour centres was studied at room temperature and after heating the samples up to 560 K. As a première, we also investigated through THz imaging and spectroscopy the irradiated optical fiber samples. Under these conditions, three of the optical fibers proved to be radiation resistant. The two optical fibers sensitive to synchrotron radiation exhibited a linear variation of the optical absorption at the wavelengths of $\lambda = 229$ nm, $\lambda = 248$ nm, and $\lambda = 265$ nm, for total doses between 60 Gy and 2000 Gy. These two samples showed also an increase of the optical absorption in the UV spectral range when heated to 560 K. The optical fibers sensitive to synchrotron radiation can potentially be used for on-line radiation dosimetry.

©2014 Optical Society of America

OCIS codes: (060.2270) Fiber characterization; (060.2310) Fiber optics; (300.6540) Spectroscopy, ultraviolet; (300.6495) Spectroscopy, terahertz; (340.6720) Synchrotron radiation.

References and links

1. Y. Morita and W. Kawakami, “Dose rate effect on radiation induced attenuation of pure silica core optical fibres,” IEEE Trans. Nucl. Sci. 36(1), 584–590 (1989), doi:10.1109/23.34505.
2. H. Henschel, O. Kohn, H. U. Schmidt, and J. Kirchhof, “Radiation-induced loss of rare earth doped silica fibres,” in Proc. Fourth European Conference on Radiation and its Effects on Components and Systems, RADECS 97, (Cannes, France, 1997) pp. 439–444, doi: 10.1109/RADCECS.1997.698961.
3. B. Brichard and A. Fernandez Fernandez, “Radiation effects in silica glass optical fibers,” in Short Course Notebook, New Challenges for Radiation Tolerance Assessment, (Cap d’Agde, France, 2005) pp. 95–138.
4. F. Berghmans, B. Brichard, A. Fernandez Fernandez, A. Gusarov, M. Van Uffelen, and S. Girard, “An introduction to radiation effects on optical components and fiber optic sensors,” in Optical waveguide sensing and imaging, W.J. Bock, I. Gannot, and S. Tanev, Eds., (Springer Series B: Physics and Biophysics, Dordrecht, The Netherlands, 2008) pp. 127–166.
5. S. Girard, J. Kuhnenn, A. Gusarov, B. Brichard, M. Van Uffelen, Y. Ouerdane, A. Boukenter, and C. Marcandella, “Radiation effects on silica-based optical fibers: recent advances and future challenges,” IEEE Trans. Nucl. Sci. 60(3), 2015–2036 (2013).
6. H. Henschel, O. Köhn, and H. U. Schmidt, “Optical fibres as radiation dosimeters,” Nucl. Instr. Met. 69(2-3), 307–314 (1992).
7. D. Sporea, A. Sporea, S. O’Keeffe, D. McCarthy, and E. Lewis, “Optical fibers and optical fiber sensors used in radiation monitoring,” in Selected Topics on Optical Fiber Technology, Y. Moh, S.W. Harun, H. Arof, Eds., (Intech, Vienna, 2012) pp. 608–652.

#222748 - $15.00 USD  Received 11 Sep 2014; revised 23 Oct 2014; accepted 20 Nov 2014; published 12 Dec 2014
(C) 2014 OSA 15 Dec 2014 | Vol. 22, No. 25 | DOI:10.1364/OE.22.031473 | OPTICS EXPRESS 31473
8. E. V. Anoikin, V. M. Mashinsky, V. B. Neustreuev, and Y. S. Sidoren, “Effects of exposure to photons of various energies on transmission of germanosilicate optical fiber in the visible to near IR spectral range,” J. Non-Crys. Solids 179, 243–253 (1994).

9. S. Girard, B. Tortech, E. Regnier, M. Van Uffelen, A. Gusarov, Y. Ouerdane, J. Baggio, P. Paiillet, V. Ferlet-Cavoirs, A. Boukenter, J.-P. Meunier, F. Berghmans, J. R. Schwank, M. R. Shaneyfelt, J. A. Felix, E. W. Blackmore, and H. Thiencpont, “Proton- and gamma-induced effects on Erbium-doped optical fibers,” IEEE Trans. Nucl. Sci. 54(6), 2426–2434 (2007), doi:10.1109/TNS.2007.910859.

10. P. Lu, X. Bao, K. Brown, and N. Kulkarni, “Gamma-induced attenuation in single-mode and multimode, Ge-doped and P-doped optical fibers: A fiber optic dosimeter for low dose levels,” Can. J. Phys. 78(2), 89–97 (2000), doi:10.1139/p00-012.

11. S. Girard, J. Keurinck, Y. Ouerdane, J.-P. Meunier, and A. Boukenter, “γ-rays and pulsed X-ray radiation responses of germanosilicate single-mode optical fibers: influence of cladding dopants,” J. Lightwave Technol. 22(8), 1915–1922 (2004), doi:10.1109/JLT.2004.832435.

12. S. Girard, J. Keurinck, A. Boukenter, J.-P. Meunier, Y. Ouerdane, P. Charre, and M. Vié, “Gamma-rays and pulsed X-ray radiation responses of nitrogen-, germanium-doped and pure silica core optical fibers,” Nucl. Instrum. Meth. B 215(1–2), 187–195 (2004).

13. S. Ghosh, S. Das, M. C. Paul, K. Dasgupta, D. Bohra, H. S. Chaudhary, L. Panwar, P. K. Bhatnagar, and S. G. Vaijapurkar, “Evaluation of the performance of high phosphorous with germanium codoped multimode optical fibers for use as a radiation sensor at low dose rates,” Appl. Opt. 50(25), E80–E85 (2011), doi:10.1364/AO.50.000E80.

14. S. Girard, Y. Ouerdane, C. Marcandella, A. Boukenter, S. Quenard, and N. Authier, “Feasibility of radiation dosimetry with phosphorus-doped optical fibers in the ultraviolet and visible domain,” J. Non-Crys. Solids 357, 1871–1874 (2011), doi:10.1016/j.jnoncrysol.2010.11.113.

15. A. V. Faustov, A. Gusarov, M. Wuilpart, A. A. Fotiadi, L. B. Liokumovich, I. O. Zolotovskiy, A. L. Tomashuk, T. de Schoutheete, and P. Megret, “Comparison of gamma-radiation induced attenuation in Al-doped, P-doped and Ge-doped fibers for dosimetry,” IEEE Trans. Nucl. Sci. 60(4), 2511–2517 (2013), doi:10.1109/TNS.2013.2273273.

16. M. C. Paul, D. Bohra, A. Dhar, R. Sen, P. K. Bhatnagar, and K. Dasgupta, “Radiation response behavior of high phosphorous doped step-index multimode optical fibers under low dose gamma irradiation,” J. Non-Crys. Solids 355, 1496–1507 (2009), doi:10.1016/j.jnoncrysol.2009.05.017.

17. A. V. Faustov, A. Gusarov, M. Wuilpart, A. A. Fotiadi, L. B. Liokumovich, I. O. Zolotovskiy, A. L. Tomashuk, T. de Schoutheete, and P. Megret, “Remote distributed optical fibre dose measuring of high gamma-irradiation with highly sensitive Al- and P-doped fibres,” in Proc. SPIE vol. 8774, Optical Sensors 2013, F. Baldini, J. Homola, and R. A. Lieberman, Eds., March 3, 2013, pp. 877404.

18. A. V. Kir’yakov, S. Ghosh, M. C. Paul, Y. O. Barmenkov, V. Aboites, and N. S. Kozlova, “Ce-doped and Ce/Au-codoped alumino-phospho-silicate fibers: Spectral attenuation trends at high-energy electron irradiation and posterior low-power optical bleaching,” Opt. Mater. Express 4(3), 434–448 (2014), doi:10.1364/OME.4.000434.

19. D. Sporea, A. Sporea, and C. Oproiu, “Effects of hydrogen loading on optical attenuation of gamma-irradiated UV fibers,” J. Nucl. Mater. 423(1-3), 142–148 (2012).

20. http://www.esrf.eu/Accelerators/Operation/Modes.

21. S. Girard and C. Marcandella, “Transient and steady state radiation responses of solarization-resistant optical fibers,” IEEE Trans. Nucl. Sci. 57(4), 2049–2055 (2010), doi:10.1109/TNS.2010.2042615.

22. E. Bräuer-Krisch, R. Serduc, E. A. Siegbahn, G. Le Duc, Y. Prezado, A. Bravin, H. Blattmann, and J. A. Felix, “Effects of pulsed spatial fractionated microscopic beams on normal and tumoral brain tissue,” Special Issue of Mutation Research - Spatio-Temporal Radiation Biology MUTREV-7944 (to be published).

23. D. Sporea, A. Sporea, and C. Oproiu, “Comparative study on the degradation of UV optical fibers subjected to electron beam and gamma ray irradiation,” Opt. Fiber Technol. 19(6), 652–657 (2013).

24. D. Sporea and R. Sporea, “Setup for the in situ monitoring of the irradiation-induced effects in optical fibers in the ultraviolet-visible optical range,” Rev. Sci. Instrum. 76(11), 113110 (2005), doi:10.1063/1.2130932.

25. The Book on the technologies of Polymicro, www.polymicro.com.

26. M. J. Söderlund, J. J. Montiel Ponsoda, J. P. Koplow, and S. Honkanen, “Heat-induced darkening and spectral broadening in photodarkened ytterbium-doped fiber under thermal cycling,” Opt. Express 17(12), 9940–9946 (2009).

27. M. Leich, S. Jetschke, S. Unger, and J. Kirchhof, “Temperature influence on the photodarkening kinetics in Yb-doped silica fibers,” J. Opt. Soc. Am. B 28(1), 65–68 (2011).

28. J. Song, J. Guo, X. Wang, and J. Jin, “Temperature dependence of radiation-induced attenuation of optical fibers,” Chin. Opt. Lett. COL 10(11), 110604 (2012).

29. J. Jin, J. Liu, X. Wang, J. Guo, and N. Song, “Effect of color center absorption on temperature dependence of radiation-induced attenuation in optical fibers at near infrared wavelengths,” J. Lightwave Technol. 31(6), 839–845 (2013).

30. S. Girard, C. Marcandella, A. Morana, J. Perisse, D. Di Francesca, P. Paiillet, J.-R. Mace, A. Boukenter, M. Leon, M. Gaillardin, N. Richard, M. Raine, S. Agnello, M. Cannas, and Y. Ouerdane, “Combined high dose and temperature radiation effects on multimode silica-based optical fibers,” IEEE Trans. Nucl. Sci. 60(6), 4305–4313 (2013), doi:10.1109/TNS.2013.2281832.
1. Introduction

Optical fibers were extensively tested under irradiation in order to assess their radiation resistance. Most of the research targeted communication report on optical fibers for remote control of experiments or for data transfer in radiation environments. The irradiation effects were evaluated as function of the dose rate or total dose [1,2]. Spectral measurements were done on-line or off-line, mostly in the visible and near IR range, under gamma-ray, X-ray or neutron irradiation [3–5]. On the other side, optical fiber characteristics degradation was exploited in order to build radiation monitoring systems or dosimeters [6–8]. The investigated optical fibers were pure or doped silica with: Er [9], Ge [10–15], P [10,13,15–17], N [12], Al [15,17], Ce [18], Ce/Au [18]. In our previous work, we investigated the possible use of commercial available UV multimode optical fibers for radiation dosimetry, when they were irradiated with electron beam, gamma-ray, Bremsstrahlung or X-rays [19–21].

High resolution dosimetry for absolute dose measurements is still a challenge in Microbeam Radiation Therapy (MRT) [22]. Presently, there is no commercial on-line dosimeter to accurately perform absolute dose measurements or to determine the peak and the valley dose, when large factors between ~10-100 are required, with an excellent dynamic range, in the case involving the combination of dose rates of about 16 000 Gy/sec, a relatively low energy spectrum around 100 keV and, an optimal resolution of a few microns. For this reason a rather novel approach was tested at the ID17 Biomedical beamline to investigate the possible use of OF-based dosimeters. In our preliminary study we show that OFs at very high doses are giving promising results, but with a size of 400 micron of the core of the OFs, high resolution dosimetry in MRT can’t be performed yet. The use of smaller diameter OFs will be investigated in the future.

The focus of the present paper is, for the first time, the behavior of commercial UV optical fibers as they are subjected to synchrotron radiation. Five different types of UV optical fibers produced by three manufactures were exposed to total doses between 5 Gy and 2000 Gy. The investigations targeted the evaluation of the optical absorption degradation in these optical fibers as function of the total dose received and after the samples were heated. The recovery of this characteristic at room temperature was also studied. The paper reports the radiation resistance of the tested optical fibers, as well as the possible use of UV optical fibers for synchrotron radiation dosimetry. In the case of two optical fibers we noticed an increase of the optical absorption after sample heating. This effect is quite an unusual one, and it has to be further evaluated. For the first time, according to our knowledge, irradiated optical fibers were studied by THz spectroscopy.

2. Materials and experiments

The results reported in this paper refer only to off-line measurements. The optical fibers subjected to synchrotron irradiation and temperature related tests are listed in Table 1 along with their major characteristics. All the samples have the length of 140 mm.
Table 1. Characteristics of the Investigated Optical Fibers and the Testing Conditions

| Product code | Type of optical fiber | Core/ cladding diameter (µm) | Jacket (µm) | Irradiated | Heated (560 K, “test3”) | Cooled (280 K, “test4”) |
|--------------|-----------------------|-------------------------------|-------------|------------|-------------------------|-------------------------|
| S1,2,        | high-OH silica/silica, modified core, hydrogen loaded | 400/ 440 | 480, Polyimide | X          | X                      | X                       |
| FVP400/UVMI (Polymicro Technologies) |                                |                |             |            |                         |                         |
| S2,          | high-OH silica/silica, modified core | 400/ 440 | 480, Polyimide | X          | X                      | X                       |
| FVP400/UVM (Polymicro Technologies) |                                |                |             |            |                         |                         |
| S3,          | pure fused silica core/ fluorine doped silica cladding; step index multimode; high-OH core | 400/ 440 | 480, Polyimide | X          | X                      | X                       |
| SFS400/440T (Fiberguide Industries Inc.) |                                |                |             |            |                         |                         |
| S4,          | silica /silica, step index multimode | 400/ 440 | 480, Polyimide | X          | X                      | X                       |
| UVS400/480 (Fiberguide Industries Inc.) |                                |                |             |            |                         |                         |
| S5,          | optical fiber for UV applications | 400 (core) | Acrylate | X          | X                      | X                       |
| HPSU400P (Oxford Electronics) |                                |                |             |            |                         |                         |

1 After the irradiation, for all the optical fibers the recovery was measured at room temperature (“test 2”).

2 Sample S1 was one more time heated to 560 K after it was cooled at 280 K (“test5”).

The five optical fibers were irradiated at the European Synchrotron Radiation Facility (ESRF), in Grenoble to check their possible use in a planned intercomparison project. A 3rd generation Synchrotron Radiation source is available at the ESRF, producing from the 6 GeV circulating electrons in the storage ring a high intensity highly collimated photon beam on ~30 experimental stations around the storage ring. The ID17 Biomedical beamline is equipped with a 21 pole wiggler insertion device to produce photons in the range of radiotherapeutic interest from about 50-350 keV. The actual filtered photon spectrum impinging on the Multi Slit Collimator (MSC) or on our samples for the broad beam irradiations is shown in Fig. 1.

The irradiation included two campaigns, and was performed in three steps, for eight total doses. During the first campaign samples of the five optical fiber types were irradiated up to 30 Gy, 200 Gy and 1000 Gy. The synchrotron was operated in the bunch mode, maximum machine current at 40 mA [23]. For the second campaign, two sets of pristine optical fibers were irradiated up to 5 Gy and 10 Gy, and the samples irradiated in the first round were further irradiated with a doubled total dose (i.e. 60 Gy, 400 Gy and 2000 Gy), while the synchrotron was operated in the 7/8 th filling mode, at maximum machine current of 200 mA [23]. The irradiated field was 70 mm x 27 mm. The irradiations were done at room temperature. With the different irradiation doses applied we expected at least to observe a linear behavior of the detector, which could be confirmed under certain conditions. Further exposures and measurements will be necessary to determine the lifetime of the detectors.

For the beam dosimetry a PTW pin-point ion chamber (Fig. 2) was used to avoid ion recombination correction problems, either in a water tank or in solid water. The PTW pin-point chamber model TW31014-000981 has a chamber volume of 0.015 cm³, and was
calibrated with UNIDOS Webline T10022-000118. The dose rate was measured in water @ 2 cm depth for a field size of 2 cm x 2 cm. The reference dose was converted in dose rate including all standard corrections like temperature and pressure.

Fig. 1. MRT spectrum after filtering with 1.5 mm Al and 1.0 mm Cu.

Fig. 2. Set-up of the PTW water tank on the MRT goniometer using an PTW pin-point chamber being scanned vertically through the beam for absolute dose measurements in the broad beam configuration: 1 – pin-point chamber; 2 – PTW water tank; 3 – MRT goniometer.

For off-line, measurements removable SMA 906 connectors were mounted at each sample ends after they were carefully polished before each measurement, in order to reduce the coupling loss. Before the irradiation and after each irradiation step the optical absorption of the samples was measured between 225 nm and 900 nm. In each case, the spectral optical absorption was acquired three times, and the reported results corresponding to the mean values obtained, with a mean estimated measuring uncertainty of 0.014 OD.

An automatic setup was used to measure the spectral optical absorption. It is quite similar to that previously used in our investigations [20,24], but some additional functions were added. The newly designed setup makes possible simultaneous measurements of several optical fiber samples. It consists of the S2000 Ocean Optics optical fiber mini spectrometer; the Avantes FOM-UVIR400-2x8, optical fiber multiplexer; the Ocean Optics FVA-UV optical fiber manual attenuator; and the Analytical Instruments System UV/VIS deuterium-
tungsten light source, model DT1000CE (Fig. 3). The characteristics of the employed instrumentation are: optical fiber spectrometer (0.5 nm spectral resolution, integration time from 3 ms to 60 s, 12 bit data conversion), deuterium–tungsten light source (maximum output fluctuation better than 0.005% p–p, after a 30 s warm-up time), optical fiber manual attenuator (having a spectrally flat attenuation between 0% and 98% transmission, from 200 nm to 2000 nm), and optical fiber multiplexer (2 - 8 inputs configuration, switching time of < 60 ms for adjacent positions, optical throughput of about 60%, optical repeatability > 90%).

The optical signal at the output of the light source (1) is attenuated by the optical fiber attenuator (2) in order to avoid the saturation of the signal detected by the mini spectrometer (4), as the light source output is coupled through the spectrometer by the optical fiber multiplexer (3). The laptop (6) controls both the spectrometer and the multiplexer, to acquire (i) the spectra of the light source, (ii) the dark signal, and (iii) the optical signal passing through the sample optical fibers (5). These samples are coupled by connecting optical fibers between the corresponding input and the output of the multiplexer. One-by-one the samples are connected between the attenuator and the spectrometer, in such a way that their transmission spectrum is registered. The samples are scanned sequentially under the laptop control and the optical absorption spectra are computed by the LabVIEW VI provided by Ocean Optics, embedded into this application. Because an absorption spectrum is calculated after each measurement, the value of the optical absorption corresponding to each wavelength of interest, selected by the operator, is displayed. Up to five color centers of interest for which the dynamic of the change is evaluated can be selected by the operator. In our case, the wavelengths to be considered of interest, depending on the composition of the investigated optical fiber, are: \( \lambda = 215 \) nm; \( \lambda = 229 \) nm; \( \lambda = 248 \) nm; \( \lambda = 265 \) nm; \( \lambda = 330 \) nm. We selected these wavelengths as they are referred in the published literature [25]. No measurements were performed below 215 nm because of the limitations imposed by the spectral transmission of the connecting optical fibers and of the optical fiber multiplexer. In the meantime, an automatic correction of the absorption spectrum to compensate for the bias induced by the optical fiber multiplexer and the connecting optical fibers was included in the application software. During the data post processing, the absorption spectra are corrected as per the value corresponding to \( \lambda = 580 \) nm, wavelength where no radiation effects were observed. This correction was made by subtracting from the entire spectrum the absorbance corresponding to \( \lambda = 580 \) nm. In this way the displayed values are not affected by the imperfect connections. The measured values of the spectral absorption are represented in this paper as relative values. The absolute values have no significance because of: (i) the bias introduced by the connecting devices, and (ii) the various conditions in which the samples were irradiated and measured.

![Fig. 3. Measuring setup: 1 – light source; 2 – optical fiber attenuator; 3 – optical fiber multiplexer; 4 - optical fiber mini spectrometer; 5 – optical fiber sample; 6 – laptop.](image-url)
For on-line measurements during the irradiation, the multiplexer channels are scanned periodically and subsequent values of the optical absorption at the specified wavelengths are saved and displayed as function of time, or if the dose rate is provided by the operator, as function of the total dose. The operation is performed for each selected multiplexer channel.

The spectral optical absorption of the samples was measured at the National Institute for Laser, Plasma and Radiation Physics, after each irradiation session. As some manufacturers’ data indicate a recovery of the optical absorption even at room temperature, after 10 days from the irradiation, some samples were measured one more time in order to observe the recovery.

Our investigation included also the study of the recovery of irradiation induced color centers as the samples from the second campaign were exposed to a prolonged heating process, at 560 K. The heating was done with a heating rate of 3.45 K/min. (Fig. 4). After the 560 K temperature was reached, the samples were kept at this temperature for 20 min.

![Fig. 4. Temporal profile of the heating process.](image)

Another assessment of the irradiation effects on UV optical fibers was done through THz imaging and spectroscopy. We compared, for the first time according to our knowledge, the THz spectral reflectance of two optical fiber samples type S1, one subjected to 5 Gy and the other exposed to 2000 Gy.

3. Results and discussions

Figures 5-7 illustrate the development of colour centres in optical fiber samples listed in Table 1. The plots of the spectral optical absorption can be used to assess the radiation hardness of the investigated optical fibers. The data are presented separately for different irradiation campaigns as the irradiation conditions are different (i.e. the irradiation geometry) and the time interval between the irradiation and the measurements in the laboratory is also different (some recovery effects can be present). Only results for the samples affected under specific circumstances by the processes to which they were subjected are mentioned in this paper, while for the other samples only comments are provided, to limit the paper length.

A comparative representation of the five optical fibers in the UV spectral range before the irradiation and after they were exposed to synchrotron radiation at 5 Gy and 10 Gy is given in Fig. 5. At these total doses, the most insensitive optical fiber is S3. All the other optical fibers undergo slight changes of the optical absorption, excepting the S5 for which the optical absorption exhibits a peak at 250 nm, for a dose of 10 Gy. For higher total doses (30 Gy, 60 Gy, 200 Gy, 400 Gy, 1000 Gy, and 2000 Gy) the S3, S4, S5 proved to be quite radiation resistant as the UV transmission does not change too much. Exceptions are S1 and S2 (Figs. 6-7). The effect of the irradiation at 30 Gy and 200 Gy is quite similar while some differences are present under 1000 Gy exposure. The data indicate a higher sensitivity of the S1 sample...
(hydrogen loaded) as compared to the S2 sample, both for the 1000 Gy and 2000 Gy dose. In order to highlight the contribution of the colour centres formation and recovery at different wavelengths some Figures include an insert.

For some optical fibers we noticed that the data collected at 30 Gy, 200 Gy, 1000 Gy, have higher values than those corresponding to the second round of irradiation (60 Gy, 400 Gy and 2000 Gy). Because the time intervals elapsed between the irradiation at ESRF and the laboratory measurements are different in the two cases we suspected that some recovery phenomenon is active even at room temperature. For this reason, in the next step of our investigation we measured the irradiated optical fibers after they were kept at room temperature for 10 days. All the samples irradiated at 5 Gy, 10 Gy, and 2000 Gy shown a drop of the optical absorption after they were kept for 10 days at room temperature (tests denoted as “test2”, as compared to initial measurements – “test1”). The only exception is S5 irradiated at 10 Gy, when no recovery was observed (Fig. 8). In this case, a slight decrease can be noticed at 265 nm. The recovery of the optical absorption at room temperature, for the dose of 2000 Gy, is illustrated in Fig. 9.

Fig. 5. UV spectral absorbance for the pristine optical fiber samples (a), irradiated optical fiber samples at the total dose of 5 Gy (b) and 10 Gy (c).

We were also interested on the recovery process as the samples are subjected to temperature stress. For the total dose of 2000 Gy we heated the samples up to 560 K and kept them to this temperature for 20 min. The results for the five samples are given in Fig. 9, designated as “test3”. For samples S4 and S5 heating of the optical fiber to 560 K contributes a little bit to the decrease of the optical absorption as compared to the effect of the storage at room temperature.

In the case of S3 the sample heating produces an increase of the optical absorption in the 250 nm - 450 nm spectral band. A quite unusual behaviour is present in the case of S1 and S2,
when the temperature stress to 560 K induces a significant increase of the optical absorption over the entire UV spectrum.

The increase of the optical absorption under heating is not an expected effect, as usually the temperature increase produces a recovery effect [3–5]. However, recent investigations support the idea of the increase of optical attenuation under thermal excitation. Heat-induced darkening was studied for Yb-doped optical fibers [26,27], Ge/P co-doped optical fibers [28], and more recently for Ge or P doped pure silica or Fluorine-doped optical fibers [29,30]. It was suggested that thermal activation of additional colour centres, the increase of the absorption cross section, and the temperature dependence of the colour centre absorption are responsible for this effect. The measurements were done at 600 nm, 633 nm, 670 nm, 1310 nm, and 1550 nm. According to our knowledge no data were reported on this phenomenon in the UV spectral range, as some of our present results suggest. For this reason, a separate, more thorough investigation has to be carried out on this issue.

![Graph showing optical absorption](image)

Fig. 6. The irradiation induced optical absorption for S1 (a) and S2 (b), at the total doses of 30 Gy, 200 Gy and 1000 Gy.

![Graph showing optical absorption](image)

Fig. 7. The irradiation induced optical absorption for S1 (a) and S2 (b), at the total doses of 60 Gy, 400 Gy and 2000 Gy.

In the next step of our investigation we cooled the optical fiber samples at the temperature of 280 K for four days and we repeated the optical spectral absorption measurements. The newly acquired data are designated in Fig. 9 as “test4”. The sample S1 was one more time heated up to 560 K and its spectral optical absorption was evaluated, in “test5” (Fig. 10).

The samples cooling do not induce significant changes in the case of samples S1, S3, and S5 as compared to the first heating process results. In the case of samples S2 and S4 the cooling of the irradiated and heated optical fibers changes the spectral optical absorption to values closed to those measured after the irradiation. The second heating of sample S1 does
not produce any modification of the absorption spectrum. This complex dynamics of the irradiation induced colour centres has to be further examined through a separate investigation.

Fig. 8. The irradiation induced optical absorption for S5, at the total dose of 10 Gy, for two tests run at 10 days apart.

Fig. 9. The irradiation induced optical absorption for S2 (a), S3 (b), S4 (c) and S5 (d) at the total dose of 2000 Gy, for four tests.
In Fig. 11 is given, for comparison, the variations of the optical absorption, in the case of the most radiation sensitive optical fibers (S1 and S2), as it was measured for the wavelengths cited in the literature as being of interest [5,24]: \(\lambda = 215\) nm; \(\lambda = 229\) nm; \(\lambda = 248\) nm; \(\lambda = 265\) nm; \(\lambda = 330\) nm. The sample S1 exhibits a linear variation of the optical absorption over the dose range from 60 Gy to 2000 Gy, at the wavelengths of \(\lambda = 215\) nm, \(\lambda = 229\) nm, \(\lambda = 248\) nm, and \(\lambda = 265\) nm. In the case of sample S2 the optical absorption at \(\lambda = 215\) nm presents a non-linear variation with a trend of saturation for doses higher than 400 Gy. At wavelengths \(\lambda = 229\) nm, \(\lambda = 248\) nm, and \(\lambda = 265\) nm the change of the optical absorption with dose is quite linear. For both samples (S1 and S2) the optical absorption remains almost unchanged at \(\lambda = 330\) nm, with the dose increase.

Some previous publications focused on the measurement of glass refractive index in the THz spectral range [31–33]. We completed the test run on the irradiated optical fibers by evaluating the change of their spectral reflectance in the THz domain. The measurements included in this paper are for sample S1. The results were obtained with the TeraView TPS Spectra 3000 model spectrometer with the RIM module, operated in the frequency domain mode, with a scanning resolution of 0.05 mm. Some of the results are provided in Fig. 12. The change of the samples reflectivity in the THz range, for two irradiation doses (5 Gy and
2000 Gy) is illustrated in Figs. 12(a) and 12(c). These images present the sample scanned along the optical fiber axis. In Figs. 12(b) and 12(d) are given the THz spectra in the spectral range 0-1.6 THz, along the longitudinal line passing through the dot marked on the optical fiber core. The THz spectra of sample S1 jacket, cladding-core interface and core, for the total irradiation dose of 5 Gy and 2000 Gy are provided in Figs. 12(e), and respectively 12f.

At this point, by introducing for the first time THz spectroscopy as a tool to investigate radiation effects in optical fibers, we consider its potential for the estimation of radiation induced changes as the THz equipment employed makes possible some sort of tomography helping to explore different “layers” of the optical fiber in conjunction with the THz spectral evaluation of radiation induced changes separately for the optical fiber constituents (core, cladding, coating/ jacket). Additionally, it is possible to assess by THz spectroscopy the modification induced by the ionizing radiation on the refractive index or the dielectric constant of different layers forming the optical fiber. The radiation induced damage/ change of the material forming the jacket or optical fiber external reinforcing structure can be also assessed by the proposed method. Figures 12(e) and 12(f) clearly identify the modifications in the THz spectrum of the optical fiber constituents at low (5 Gy) and high (2000 Gy) synchrotron radiation doses.

![Fig. 12. The results of THz investigations on sample S1 in frequency domain mode: a – the reflectance imaging along the optical fiber (XY plane), for the dose of 5 Gy; b – the THz reflectance imaging along the depth of a line passing in the optical fiber core (XZ plane), for the dose of 5 Gy; c – the reflectance imaging along the optical fiber, for the dose of 2000 Gy; d – the THz reflectance imaging along the depth of a line passing in the optical fiber core, for the dose of 2000 Gy; e – the THz spectra of the optical fiber core, core-cladding interface, and jacket, for the dose of 5 Gy; f – the THz spectra of the optical fiber core, core-cladding interface, and jacket, for the dose of 2000 Gy.](image-url)
4. Conclusions and future work

We investigated for the first time the degradation of five commercially available UV optical fibers under synchrotron irradiation. The irradiations were done at the ESRF synchrotron accelerator, in Grenoble. The dose rates were 70.69 Gy/sec/mA and 65.49 Gy/sec/mA for the two campaigns. The total doses were varied between 5 Gy and 2000 Gy. Under these conditions, the S3, S4, and S5 optical fibers proved to be radiation hardened, while the most sensitive optical fiber exposure to synchrotron radiation were S1 and S2. As an exception, the S5 exhibits a peak of the optical absorption at 250 nm, for the total dose of 10 Gy. All the optical fibers showed a recovery of the optical absorption after storage for 10 days at room temperature.

The post irradiation heating of the samples at 560 K induced a slight recover from the irradiation induced colour centres in S4, and S5 optical fibers. As exceptions we notice the behaviour of samples S1 and S2, cases when the optical fiber heating induced, post irradiation, an increase of the optical absorption. Samples S1, S3, and S5 cooling do not produce significant modification of the spectral optical absorption. After the cooling to 280 K, samples S2 and S4 showed an optical absorption spectrum quasi identical to those obtained following the irradiation to 2000 Gy. This phenomenon of unexpected change of the colour centres behaviour with temperature is quite unusual and has to be further investigated.

The samples S1 and S2 shown a linear dependence of the optical absorption at $\lambda = 229$ nm, $\lambda = 248$ nm, and $\lambda = 265$ nm, for total doses between 60 Gy and 2000 Gy, after the second exposure to synchrotron radiation. At $\lambda = 215$ nm sample S2 presents a saturation of the optical absorption, while sample S1 optical absorption has a linear variation. For both samples (S1 and S2), the optical absorption remains almost unchanged with the dose increase at $\lambda = 330$ nm. This linear dependence on the dose can be used for on line synchrotron radiation dosimetry, if the increase of the optical absorption is monitored at the specified wavelengths. By selecting in an appropriate manner the type of the optical fiber to be subjected to radiation and the dose rate, optical fiber based dosimeters can be developed for on-line dosimetry.

The THz spectral measurements highlighted very clearly the irradiation induced changes in the reflectivity of sample S1.

Acknowledgments

The Romanian authors acknowledge that this work was supported by the Romanian Executive Agency for Higher Education, Research, Development and Innovation Funding (UEFISCDI), project “Sensor Systems for Secure Operation of Critical Installations” grant 8/2012. The procurement of the THz spectrometer was financially supported by UEFISCDI, grant 8PM/2010. The cooperation took part in the frame of the COST Action TD1205 “Innovative Methods in Radiotherapy and Radiosurgery using Synchrotron Radiation (SYRA3)”. The authors would like to thank Dr. H. Requardt for technical assistance during the irradiation of the samples, and to Mattia Donzelli for providing Fig. 1.