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Ge-doped optical fibers for passive and active radiation detection modes

M. Benabdesselam, F. Mady, M. Cieslikiewicz-Bouet, W. Blanc, H. El Hamzaoui, A. Cassez, G. Bouwmans, M. Bouazaoui and B. Capoen

Abstract – Silica-based optical fibers made by Modified Chemical Vapor Deposition (MCVD) technique have been doped with different concentrations of germanium (Ge). After characterization of the thermoluminescence (TL) process in each of these fibers, their real-time responses were studied by radioluminescence (RL) under X-rays. First, the same counterintuitive trend was observed for the intensity of the two phenomena as a function of the Ge content, namely the decrease in TL and RL signals with increasing Ge content. This behavior was explained by the existence of thermally disconnected traps. Then, the RL response as a function of the dose-rate of the fiber doped with 1% Ge was judiciously chosen and investigated in detail. The results show that up to Total Ionizing Dose (TID) of 1.9 kGy(SiO₂), this response is not only insensitive to the accumulated dose effect but also exhibits an afterglow decay at the end of the radiation much faster than that of a Ce-doped fiber, a very useful feature where irradiations are repetitive as in the medical field of flash-therapy. In addition to the medical field where the Ge-doped fiber seems to be quite suitable, slightly larger core diameters would also allow this same fiber to meet the challenge of high sensitivity in the environmental radiation monitoring around nuclear power plants or radioactive waste storage areas.

Index Terms— silica, germanium, thermoluminescence, radioluminescence, dosimetry

During the last decades, optical fibers have opened the way for many different applications. Among these fibers, Germanium (Ge)-doped fiber, especially in its single-mode form (SMF), is widely used in the field of telecommunications for data transfer over long distances [1]. Another field where the fiber continues to prove very useful is undoubtedly that of the sensing of many environmental parameters such as temperature and pressure [2]. The other parameter we are concerned with in this work is that of dose and/or dose rate in the monitoring of radiations present in radiative environments, such as around nuclear power plants, radioactive waste storage sites and medical applications. In addition to their electromagnetic immunity [3] and their very small size, which allow them to reach difficult-to-access areas, appropriately doped fibers can be very sensitive to the radiation characteristics present in these non-accessible areas.

M. Benabdesselam, F. Mady and W. Blanc are with the Université Côte d'Azur, CNRS, Institut de Physique de Nice – INPHYNI UMR 7010, F-06108, Nice Cedex 2 France (e-mail: Mourad.Beabdesselam@univ-cotedazur.fr, Franck.Mady@univ-cotedazur.fr, Wilfried.blanc@univ-cotedazur.fr, M. Cieslikiewicz-Bouet, H. El Hamzaoui, A. Cassez, G. Bouwmans, M. Bouazaoui and B. Capoen are with Univ-Lille, CNRS, UMR 8523 – PhLAM – Physique des Lasers Atomes et Molécules, F-59000 Lille, France (e-mail: monika.bouet@univ-lille.fr, Hicham.el-hamzaoui@univ-lille.fr, andy.cassez@univ-lille.fr, geraud.bouwmans@univ-lille.fr, mohamed.bouazaoui@univ-lille.fr, bruno.capoen@univ-lille.fr)

To characterize this radiative environment, dosimetric solutions with optical fibers based on radiation induced attenuation (RIA) have been proposed. They include for example the method using long period gratings [4-5] or that based on the absorption band of P1 defect at 1570 nm present in phosphorus-doped silica-based fibers. This radiation-induced band shows a linear growth with the dose up to 500 Gy without any dependence on the dose-rate, temperature, or the nature of radiation [6-7].

Moreover, during the last twenty years, some studies on the potential of silica-based optical fibers for ionizing radiation dosimetry have been carried out using the thermoluminescence (TL) technique [8-11]. Using this technique, we have demonstrated that Ge-doped fiber is an excellent thermoluminescent dosimeter (TLD) with good detection efficiency for neutrons (0.8 and 14 MeV) and protons (63 MeV), also showing high sensitivity to X and γ photons (⁶⁰Co) [12]. However, although TL is a robust method that helps to understand the physical mechanisms resulting from the radiation/fiber interaction and their relaxation, the information on the total ionizing dose (TID) is only available retrospectively, i.e., once the irradiation has ceased. More recently, another dosimetric technique based on the study of optically stimulated luminescence (OSL) has been used to measure the absorbed dose with rare-earth (RE)-doped silica rods spliced to transport fibers [13-15]. But again, as in TL, the OSL measurements are post-mortem and the information on the absorbed dose of radiation is known only afterwards.

The study we propose here is of interest in several respects. The first is the use of Ge-fiber manufactured by MCVD (Modified Chemical Vapor Deposition), a low cost and widely mastered process since it is this type of fiber that is used for long distance data transport. The second one is due to the high sensitivity of the Ge-fiber to different kinds of radiations and the third one is of practical relevance because from an applicative point of view, it is not required to splice any sensitive scintillator [16] or inorganic crystal [17] to the transport fiber since the Ge-doped fiber can play both the sensing and transport roles thus avoiding all the problems of splice brittleness, fragility and the losses that could be induced. Finally, a last consideration, unlike TL and OSL techniques, radioluminescence (RL) allows real-time evaluation of the radiation level [18-20]. It is important to note, however, that the present study differs from all the previous ones by the very wide range of dose rates, the use of Ge fiber both as a probe and for transport, and finally Ge contents (< 3 wt. %) that have never been tested and that prove to be the best indicated in terms of radiosensitivity.

I. EXPERIMENTAL DETAILS

A. Studied fibres

The studies mainly concern five germanosilicate optical fibers fabricated by the MCVD technique, with different Ge contents, at the FiberTech platform of the University of Lille [21,22]. The characteristics of these fibers are summarized in Table 1. In general, for optical fiber preforms fabricated using MCVD method, a pure silica tube (F300, Heraeus, Germany) with external/internal diameters of 30/25 mm was placed on the MCVD lathe and etched chemically to improve the quality of the internal wall. Then, several layers of pre-vitrified silica doped with germania were deposited at the temperature of 1900 °C and using appropriate contents of GeCl_4 , SiCl_4 and O_2 precursors. After pre-vitrifications, the diameter of the tube was reduced by viscous collapse to close completely this tube and to form the solid rod at 2200°C. This manufacturing process yielded homogeneous 40 cm-long rods with a diameter of 17 mm and the doped part of 4.2 mm. The content of GeO_2 was measured by a Cameca SX 100 microprobe and the profile of the dopant distribution was confirmed by refractive index profile (RIP) measurements (P106, Photon Kinetics) in the entire length of each preform. Subsequently, all preforms were drawn into optical fibers on the 12-meter drawing tower of FiberTech platform. Each fiber had a diameter of 125 ± 7 μm with a single coating for the Ge3, Ge6 and Ge9 fibers and with a double coating of a low refractive index polymer and protective coating for Ge01 and Ge1 fibers. The optical attenuation of these fibers was evaluated by the cut-back method. The transverse dimensions of the fabricated fibers were measured with a TM1000 tabletop scanning electron microscope (SEM). The diameter of the doped core for all the fibers was measured at 34 ± 2 μm .

Fibers	[Ge] (wt. %)	NA	Atten. (dB/km)	f_c (km/dB)
Ge01	0.1	0.4	32	1.25×10^{-2}
Ge1	1	0.4	42	0.95×10^{-2}
Ge3	3.5	0.093	12	0.77×10^{-2}
Ge6	6.6	0.127	16	0.79×10^{-2}
Ge9	9.5	0.152	16	0.95×10^{-2}

Table 1. Optical characteristics of the five studied Ge-doped fibers

Note that the last column of Table 1 corresponds to a correction factor f_c defined as the ratio between the numerical aperture (NA) and attenuation. It will be used in section II. B. For the RL studies, a length of 5 m of each fiber was used. The part of the fiber which was to be subjected to the irradiation, and which acts in fact as a dosimetric probe, was stripped over 1 cm with the aim of probing only the fiber core without interference with the possible cladding signal. The rest of the length was used to transport the signals to the detection device.

In the case of TL measurements, identical lengths of fibers (5m) were gradually stripped, cut in small pieces of a few cm, then cleaned with ethanol to remove any residues from the plastic cladding. These pieces were finally crushed to give a few mg of powder which is spread in an aluminum cupel.

B. Experimental setup (irradiations, TL, RL)

The irradiations were carried out in air at room temperature (RT) using an X-ray generator (XRG3D, Fisher Scientific) equipped with a Cu anticathode operating at 30 kV. The dose and dose-rate characteristics of the radiation beam were controlled by means of an electrometer (UnidosE) connected to an ionization chamber (PTW 23342) always placed just below the center of the beam. Considering the air/silica conversion factor, all the indicated dose rates are in silica. During RL measurements, each fiber was irradiated on its stripped end over 1 cm, the rest of the fiber still present in the radiation chamber was protected by a lead shield whatever the size of the beam.

Before TL measurements, the crushed fiber was irradiated with a total ionizing dose (TID) of 500 Gy (SiO_2). It was then kept in darkness during 30 s before reading. TL measurements have been performed between RT and 450°C with a linear heating rate of 1°C/s. During heating, the TL signal was detected by means of a UV-Visible PM tube.

The RL measurement system essentially consisted of a photon counting PMT (H7421-40) with an integration time of 1s, connected to an optical block with fibered outputs [12]. To avoid unwanted light interference, all measurements were performed in total darkness.

Finally, for a better signal-to-noise ratio, whether in TL or RL mode an interference filter centered on the main emission of Ge defects at 400 nm (3.1 eV) was always placed in front of the PMTs.

II. RESULTS AND DISCUSSION

A. Thermally stimulated luminescence (TL)

Figure 1 shows the TL curves obtained on the 5 fiber preforms with different Ge contents. Depending on the dopant content,

each of these curves is characterized by a main peak located between 200 and 250°C, corresponding to an energy depth of the traps between 1.2 and 1.4 eV in the silica band gap [23]. Also depending on the doping, the phosphorescence which designates the fraction of the TL signal around RT is more or less important. Except for the lowest Ge concentration, the general trend is the decrease of the TL signal with the increasing Ge amount in the fiber. The variation in position of the main peak means that, depending on the Ge concentration, different rearrangements of the trap defects associated with this dopant take place in the silica matrix. In other words, depending on the amount of Ge, the trap defects associated with these TL peaks are located at deeper or shallower levels within the energy band diagram of silica. It is important to note that the fibers drawn from these preforms show the same TL behavior. This confirms the fact that drawing does not induce the formation of any new thermoluminescent defect.

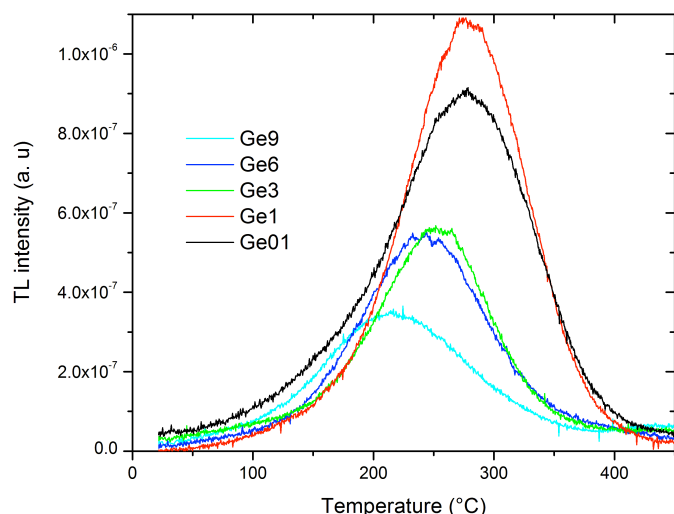


Fig. 1. TL curves obtained observed on the 5 fiber preforms under the same conditions of irradiation and readout

Figure 2 represents a superimposition of two spectra. One is the RL spectrum obtained at RT one of the fibers (here Ge1), the other is the TL spectrum obtained on the powder from the same fiber at the temperature of peak maximum. Both spectra are characterized by a broad emission composed of a main band centered at about 3.1 eV as well as a small one around 2.7 eV, already observed by TL on Ge doped fibers [9,11]. These two TL emitting centers are oxygen deficient centers (ODC) that are vacancies associated with Ge and Si atoms respectively [24]. These centers having trapped electrons during irradiation are reformed during the relaxation TSL process through radiative recombinations on these centers between the electrons and the thermally released holes.

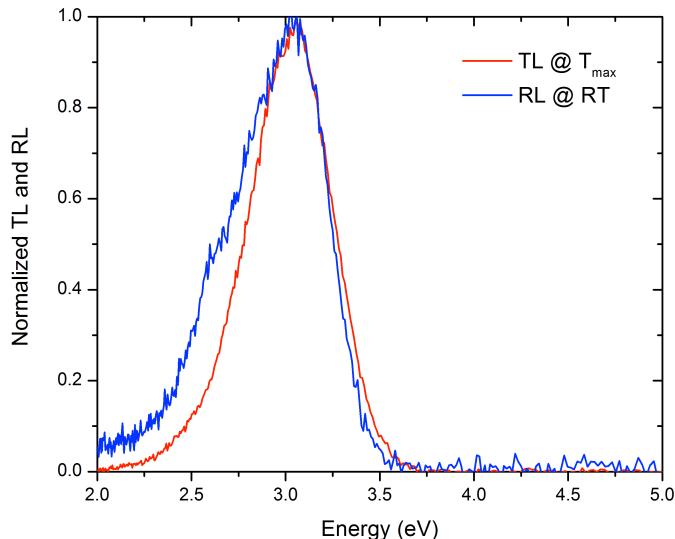


Fig. 2. Normalized spectra of RL obtained on the fiber Ge1 at RT (a) and of TL on the powder of the same fiber at T_{max} of the peak (b).

B. Radiation Induced Luminescence (RL)

Figure 3 shows the evolution over time of the RL signal levels from the 5 fibers at different dose rates. These signal levels are highly reproducible with a standard deviation of no more than 2% at all tested rates, which constitutes some reliability in the real-time dose rate estimation. Whether during irradiation or in its absence, the levels of the plateaus are relatively stable with no sensitivity variation with the accumulated dose.

As in TL, the spectrally resolved RL shows broad emission resulting from radiative recombinations on the same centers as those observed in TL, namely the Si- and Ge-ODC centers located at 2.7 and 3.1 eV respectively (Figure 2). The latter being by far the most significant radio-luminescent center.

Not surprisingly, on all fibers, the measurement of the different plateau levels indicates a linear evolution of the RL signal as a function of the dose rate. At the highest irradiation rates, we notice that for low Ge contents, there appears a form of signal overshoot before stabilizing. This feature, present only in the low Ge content fibers, is even more pronounced on the least doped one (Ge01). This signal overshoot has already been observed by other authors in Ge-doped fibers under more energetic photons, but its origin is still unknown [19]. Such an overshoot might be due to Ge impurities. However, it has already been observed in the RL response of some RE-doped silica-based fibers too.

About these evolutions over time of the RL signals in Figure 3, it should be noted both the promptness and stability of the RL sensitivity at all tested dose rates without any accumulated dose effect. Note that at the end of each RL reading cycle, the TID reaches the level of 1.675 kGy(SiO₂).

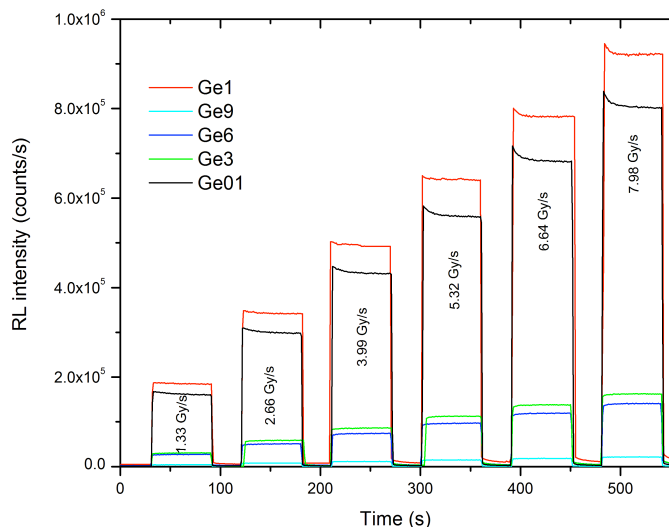


Fig. 3. Variations of the RL signals for the 5 fibers as a function of the dose rate (in Gy(SiO₂)/s)

As shown in Figure 4, this very stable dose-rate information, independent of the dose where the TID reaches at the end of the cycle, the level of 1.7 kGy(SiO₂), is undoubtedly a remarkable advantage over other radio-luminescent fibered rods used where the RL plateaus are in permanent change both during irradiation and after its stop [13]. It should be stressed that the independence on the TID is valid with a 1 cm-long sample. The result may be different with longer probe fibers, as the radiation induced attenuation (RIA) should have a stronger influence on the collected signal. The prompt presence or not of the plateau in the RL response may also be due to a competition during the irradiation between the filling of the traps on one side and that of the recombination centers on the other.

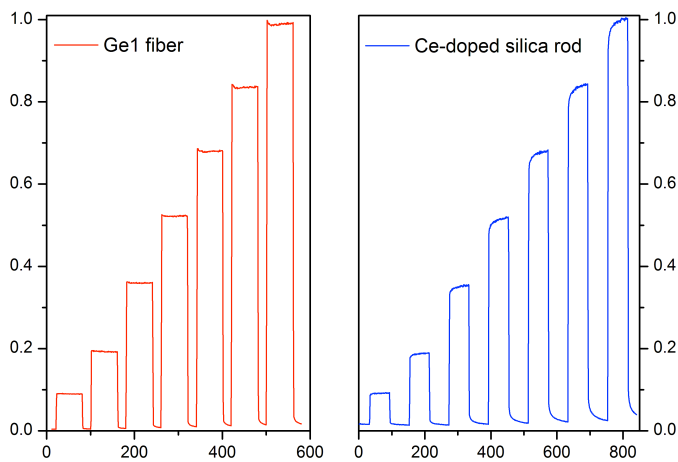


Fig. 4. Comparison of the normalized RL temporal evolutions at different dose rates of Ge1 (a) and a Ce-doped silica rod (b).

Similarly, after the end of the irradiation, we note that the afterglow decay of the RL signals takes place much faster with the Ge-doped fiber than with a Ce-doping [13]. This is not surprising since this decay is associated with the phosphorescence of shallow traps whose concentration is much higher in Ce-doped fibers than in Ge-doped ones. Indeed, as shown in Figure 1, at the irradiation temperature (which is RT), the phosphorescence of Ge-doped fibers is very

weak, and the concentration of shallow traps is very much lower than that of the dosimetric trap around 250°C. This faster return to zero of the RL signals also constitutes another advantage in the case of pulsed irradiations.

Figure 5 represents the evolution of the normalized intensities of the TL and RL signals as a function of the Ge content. For the RL, two curves are shown, corresponding to raw and corrected data. The corrected values are obtained by dividing the measured intensities by the f_c factor (Table 1), which considers the opto-geometrical fibers properties. It is important to note that this correction does not change fundamentally the curve behavior of the RL intensity versus Ge content. As a reminder, the RL process is observed following the prompt recombination of the radiation-induced electron-hole pairs during irradiation while the TL mechanism is due to recombination of the thermally stimulated release of the remaining trapped charges at different depths long after the end of the irradiation.

Although these two processes are different, we observe quite similar evolutions as a function of the Ge content, whatever the tested dose-rate: after a slight increase of the response between 0.1 and 1% of Ge, it then decreases while the Ge content increases, so that there is almost no RL response for the most heavily doped fiber (Ge9).

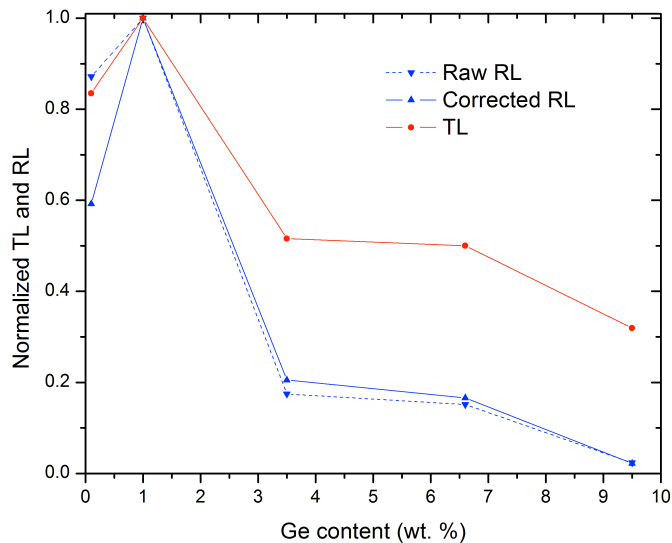


Fig. 5. Evolution of normalized TL and RL signal intensities as a function of Ge content in the five fibers. The corrected RL values consider the correction factor f_c in Table 1.

To explain the probable reason behind this decrease in TL and RL signals with Ge content, we suggest examining the mechanisms involved in these two phenomena. Figure 6 shows the energy band diagram of Ge-doped silica. In the band gap are shown some levels as well as the various mechanisms that occur during the two luminescence processes following a creation of electron-hole pairs by irradiation (1).

In this diagram, MT refers to the main trap whose corresponding peak is the one observed in TL (Figure 1) and ODC- designate trapped state centers that constitute recombining centers for both TL and RL radiative emissions. Except for arrow 5 which will be discussed below, the arrows numbered 2, 3 and 4 correspond respectively to the trapping of holes by MT, to the capture of electrons by the ODC centers

thus forming the trapped states centers ODC- and finally the recombination of holes on the ODC-.

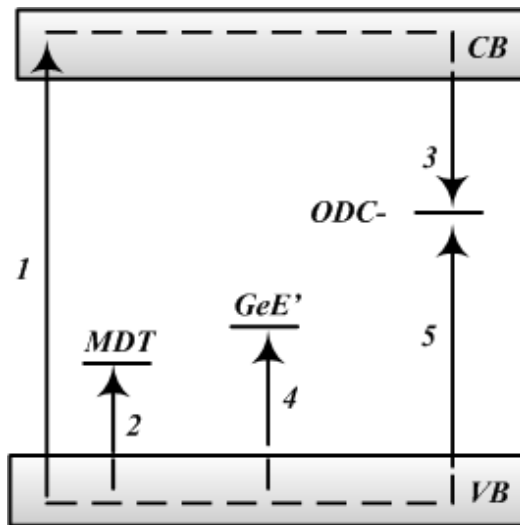


Fig. 6. Energy band diagram of Ge-doped silica matrix. Creation of electron-hole pairs (1), Trapping of holes by the main trap MT (2), Electron capture by ODC centers (3), Electron-hole recombination on ODC- centers (4), Hole trapping by deep hole traps TDT (5)

Contrary to one might have thought in view of the design of the most radiation sensitive fiber, the increase in the Ge content in the fibers does not imply that of the population of these TL/RL emitting centers, mainly Ge-ODCs. The general phenomenon of luminescence decrease as a function of dopant concentration has been observed in many materials doped with different ions (Tb, Eu, Mn, ...) [24,25]. It has already been reported in the literature and has been termed as quenching concentration [26]. However, in our case, the phenomenon at the origin of the decrease of the two luminescence signals is probably other than the aggregation of luminescent dopant ions. Indeed, based on a model built by F. Mady [27] to explain the specific features of TL processes in Ge-containing fibers by simulating the Density of Trapped States (DOTS), that are in perfect agreement with those extracted from experimental thermograms, we think rather to a hole trapping mechanism on a deep and very stable level that must be competing with the ODC- trapped state centers. These competing traps must be deep enough to avoid relaxing during the TL readout since they do not result in any TL peak on the thermogram (Figure 1). They can be referred to as thermally disconnected traps (TDT). They would constitute precursors and that when holes get trapped there (arrow 5), should form Ge-related trapped hole centers, known as GeE'. The latter have been shown to form under irradiation in Ge-doped silica [28]. Isochronal annealing experiments showed that these centers, which absorb in the UV, anneal only above 500°C and certainly at higher temperatures when the heating is linear (1°C/s) as it is the case during TL experiments and where the maximum temperature reaches only 450°C. Therefore, increasing Ge concentration tends rather to the formation of precursors that lead to the production of GeE' deep hole trap

centers at the detriment of Ge-ODC recombination centers at the origin of radiative emissions of TL and RL.

C. Radioluminescence dosimetry (RLD)

The excellent dosimetry TL properties of the Ge-doped silica fiber have already been demonstrated [9] so that we focus only on the detection properties in active mode by RL. In figure 7 is represented the linear RL response of the 5 fibers as a function of the dose rates. As it was the case in TL, the sensitivity of the RL response globally tends to decrease with increasing the dopant content and that the most radio-luminescent fiber is the one doped with 1% of Ge. Hence, whether the targeted application is, active detection by RL or passive dosimetry by TL, the 1% Ge-doped fiber (Ge1) seems to be the most suitable system. Indeed, for the six dose-rates reported on the graph, the RL sensitivity of the Ge01 and Ge1 fibers is about one decade larger than that of the Ge3 and Ge6 fibers and even almost two decades larger than that of the Ge9. This trend of decreasing RL signal with the Ge content is in very good agreement with the results reported by other authors [19] about Ge-doped fibers with concentrations of 3.59, 4.74 and 7.03 wt.%.

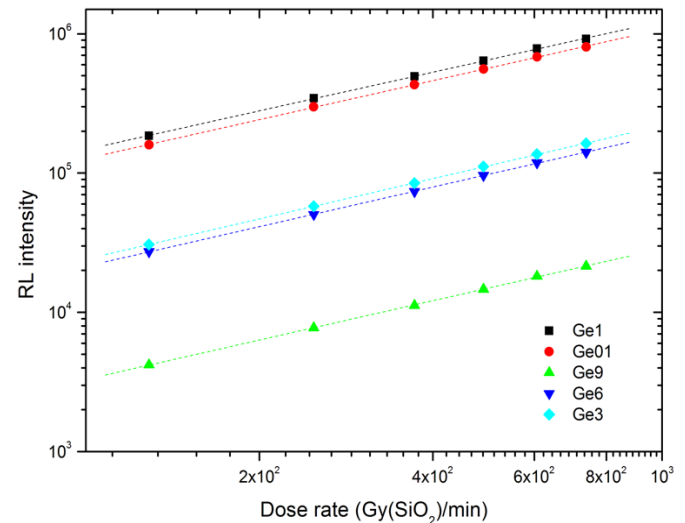


Fig. 7. RL response to different dose rates of the five Ge-doped fibers

The reason for this decrease is probably as explained for the TL process: the increase in Ge content tends more to increase the population of thermally disconnected traps that constitute the precursors of GeE' centers, more than the number of the recombinant Ge-ODC centers, which are at the origin of the radioluminescence of these fibers.

Based on these results and considering that all these fibers would show the same trend, we investigated the RL response of the most sensitive of them, namely Ge1, and tested its detection performance over a wide range of dose rates. This study was conducted on two fiber samples: the first one denoted sample 1 consists of a 4m-long Ge1 fiber with one end stripped over 1cm to act as a probe, the other end being directly connected to the detection device. This means that the same Ge1 fiber is used both for the detection of the irradiation dose-rate and the transport of the resulting RL signal to the detector. Sample 2 is a length of 1cm of the same Ge1 fiber spliced to a 50 μm multimode (MMF) transport fiber. This

second configuration is generally the most often used in the literature, especially when the probe cannot be drawn into an optical fiber over long distances, as is the case of probes based on quartz [29], or alumina single crystal [30] or RE-doped silica rods [13-15,31,32]. It is worth noting that in both configurations, only 1 cm of Ge1 fiber is used as a probe and is exposed to the radiation beam. All the rest of the two configurations is protected by a lead screen in the irradiation chamber and measurements are done in the absence of any ambient light.

Figure 8 shows the variations of the RL response as a function of the dose-rate obtained using the two configurations. In general, we observe that the responses of both configurations increase linearly with the dose-rate with a sensitivity is 3.5 times higher for the all-Ge fiber set (sample 1). The difference between the two responses must be due to the splicing losses in sample 2 since the sensitive part is the same in the two cases and that the transport of the RL signal is ensured by larger core fiber (diameter 50 μm) in the case of sample 2. This difference is not huge but could be significant when detecting very low dose rate levels. Indeed, as shown by the colored arrows in Figure 8, one can easily detect 2 mGy/s with sample 1 (the all-Ge fiber set) whereas with sample 2, the detection limit is almost one decade above (≈ 20 mGy/s).

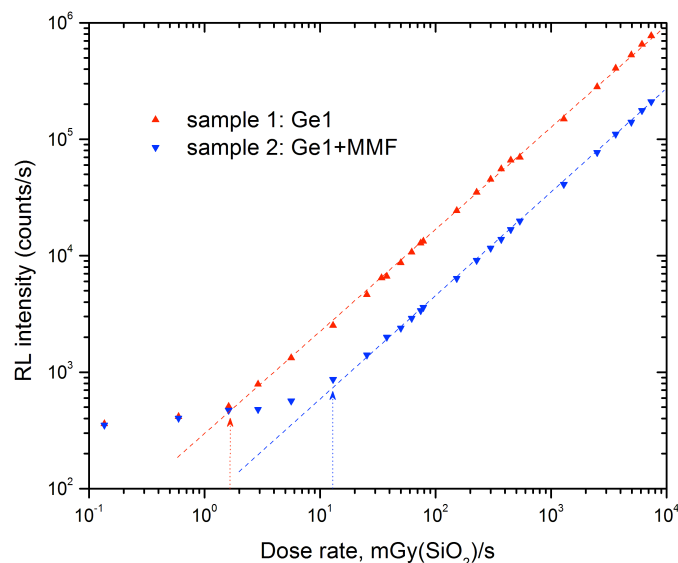


Fig. 8. Dose-rate dependencies of the RL from the two configurations

In comparison, the fibred RE-doped rods studied by ourselves [11] or by other authors [31], exhibited detection limits much lower, namely between 10 and 100 $\mu\text{Gy}(\text{SiO}_2)/\text{s}$, but in terms of robustness, these assemblies present a degree of fragility due to the splicing between the rod (probe) and the transport fiber. In addition, one must keep in mind that in terms of dimensions, the sensitive volume of the rods (length 10 mm, diameter 1.6 mm) is about 1000 times larger than that of the Ge1 fiber samples (length 10 mm, diameter 34 μm). This suggests that with Ge1 fibers of slightly larger diameters, we could expect to reach much lower detection thresholds and at least equivalent to those obtained with rods.

III. CONCLUSION

In summary, the TL study of five optical fibers doped with different Ge contents was rich in information about luminescence mechanisms during and after irradiation. It is well known that the TL technique allows to reveal the electrical nature (electron or hole) and sometimes even the origin (GeE' , Ge-ODC) of the trapping and recombination centers of the charge carriers. In our case, the weak TL signal at RT reflects the low concentration of shallow traps, which have a strong impact on the evolution kinetics of the RL signal both during and especially after the end of the irradiation.

The similar intensity evolution of both TL and RL signals with increasing Ge content is explained, for the first time, by the competition between the different possibilities for released holes during both processes. It seems more in favor of hole trapping on deep levels, allowing the formation of GeE' centers rather than recombination on trapped electron states leading to the reformation of Ge-ODC centers which gives rise to the TL and RL signals.

After TL, the RL responses of the fibers were then investigated. The 1% doped fiber, which was found to be the most sensitive among the five fibers, has been studied at several dose rates. In addition to the relatively low cost of MCVD synthesis of the Ge-doped optical fiber, the dosimetric properties of its RL response are interesting in several aspects: *i*) the first is its linearity over a wide range of dose rates covering in particular those encountered in radiotherapy (10-100 mGy/s); *ii*) the second is its stability during irradiation, without any effect of the accumulated dose, contrary to the many probes already proposed for such an application and *iii*) the third lies in the fast afterglow decay, which is undoubtedly a very useful feature in the case of repetitive pulsed irradiations as it is the case of the flash-therapy for example.

In addition to the medical field where the Ge-doped optical fiber seems to be quite suitable, slightly larger core diameters would also allow this same fiber to meet the challenge of high sensitivity in the environmental radiation monitoring around nuclear power plants or radioactive waste storage areas. Nevertheless, we must keep in mind that our measurements were performed with X-ray photons of 30 keV, well below the kinetic energy levels (a few hundred keV) of the photons leading to the appearance in the optical fibers during irradiation of a spurious light due to the Cerenkov effect in the optical fibers during irradiation. For an accurate estimation of the RL response, this effect must necessarily be considered in the case of high energy photons or electrons delivered by accelerators or γ photons from nuclear environments.

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