

# Ce doped SiO<sub>2</sub> optical fibers for remote radiation sensing and measurement.

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## ABSTRACT

Scintillating materials, able to convert energy of ionizing radiation into light in the visible-UV interval, are presently used in a wide class of applications such as medical imaging, industrial inspection, security controls and high energy physics detectors.

In the last few years we studied and developed a new radiation sensor based on silica-glass fiber-optic technology. In its simplest configuration such device is composed by a short portion (about 10 mm) of scintillating fiber coupled to a photomultiplier through a suitably long passive silica fiber.

In this work, we present new results concerning the characterization of silica based Ce and Eu doped fibers glasses obtained by a modified sol-gel method and drawn by a conventional drawing tower for optical fibers. The radio-luminescence of Eu doped fibers is rather weak; moreover it displays a marked sensitivity increase during subsequent irradiations, preventing the use of such fibers in dosimetry. On the other hand Ce-doped fibers show very high radiation hardness, signal stability and reproducibility, and high sensitivity to radiations with energies from 10 keV to several tens of MeV. Numerous tests with photons (X and gamma rays), electrons, and protons have already been successfully performed.

At the early stage of its market introduction it is the smallest radiation sensor, also compared to MOSFET and diode technology and it appears to be the ideal choice for *in vivo* measurements in medical field or remote sensing.

## 1. INTRODUCTION

A useful strategy to dose reduction and technique effectiveness in modern diagnostics and therapy concerns the improvement of dose evaluation methodologies, particularly by *in vivo* real-time dosimetry. This aspect has been underlined in several situations, and a well known example is the case of mammography [1]. While dose evaluations by computer simulation and by using dedicated phantoms are commonly employed in most cases, *in vivo* dose monitoring is not routinely performed in diagnostic techniques. Till now, Mosfets, thermoluminescent dosimeters (TLD), diodes, and in some cases, electronic portal imaging devices (EPID) are commonly employed in therapy techniques [2-12]. Many drawbacks are known for these devices, particularly for radiotherapy applications: Mosfets suffer of low reproducibility and low radiation hardness; radiation hardness of diodes also appears to be poor; TLD have a low reproducibility and dose assessment is strongly delayed in time. Moreover, due to their macroscopic dimensions, such dosimeters generally do not allow the direct *in vivo* determination of the dose imparted to inner organs, although very recent studies seem to open the perspective of the use of Mosfets in brachytherapy and in intensity modulated radiotherapy (IMRT) of oropharynx and nasopharynx [8-10]. Finally, except for diodes, dosimeters nowadays do not allow a direct "real time" dose evaluation.

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A few systems for real-time dose monitoring based on composite fiber sensors are presently under development for medical applications. Dosimeters have been proposed based (i) on plastic scintillating fibers [13], (ii) silica glass fibers [14,15], and (iii) crystals joined to fibers [16-18]. In all cases, a small “active” portion of fiber or crystal is coupled to a passive fiber for the signal transport to a processing unit. Plastic fibers exploit the radio-luminescence (scintillation) phenomenon. Silica glass fibers are based on different optical phenomena like radiation induced darkening, luminescence from defects, activators or the population of trapping centers. Lastly, crystals joined to fibers exploit radio-luminescence (RL) and optically-stimulated luminescence (OSL) mechanisms.

Reproducibility and independence of the RL response upon accumulated dose are important requirements of RL-based composite fibers. Several properties of the material affects the RL response: among others, it can be strongly influenced by the presence of point defects, that can be present in un-irradiated materials and can be further activated or created by irradiation. After irradiation, their stability and concentration can also depend on temperature. As a consequence, changes of defects concentration during and after irradiation can lead to material RL sensitivity changes as evidenced in the case of glass doped with  $\text{Cu}^+$  ions and of  $\text{Al}_2\text{O}_3:\text{C}$  [15,17]. Such variations impose pre-irradiation treatments of the fiber [15] or the use of dose correction algorithms [17] lowering the overall reliability of the dosimetry system. Very recently, a real time OSL optical fiber dosimeter based on  $\text{KBr:Eu}$  was presented [18]: in this case too, OSL sensitivity changes depending on irradiation time were noticed.

In this paper, we propose a novel type of glass composite fiber dosimeter based on the RL emission of a small portion of glass fiber doped with Cerium. Previous investigations already demonstrated the interesting RL properties of these composite fibers [19-35]. Moreover, with the aim to better separate the Cherenkov emission from the scintillating signal [36,37] an alternative Eu doped fiber was produced, tested and compared with Ce-doped ones. Lastly, the response of Ce-doped fibers to gamma rays and electrons from radio-therapy equipments are reported.

## 2. MATERIALS AND METHODS

Rare earth-doped  $\text{SiO}_2$  powders were produced by a simple modification of the synthesis published elsewhere to obtain bulk doped-samples[23].

Specifically, the Sol solution is obtained with the following composition:

ethanol 99.9% 18 ml; TEOS 6 ml; water 3.6 ml and  $\text{Ce}(\text{Eu})(\text{NO}_3)_3 \cdot 12 \cdot \text{H}_2\text{O}$  solution in ethanol (10 mg/ml) in the amount needed to obtain the proper level doping in  $\text{SiO}_2$  (the most effective doping level for Ce and Eu ions is about 600 molar ppm).

Such composition is the basic recipe to obtain glasses with any rare earth doping in silica, and can be adopted to get both powdered glass or bulk cylinder depending from dehydration procedure and the following thermal treatment.

For glass powder preparation, the xerogel powder was obtained by rapidly drying the gel in a rotating evaporator; its grain size uniformity was improved by a successive milling with an agate mortar. Slow sintering up to 1100 °C in a quartz chamber in oxidizing atmosphere gave the final glass powder. This powder was subjected to a rapid thermal treatment (RTT) at 1600-1800 °C in order to improve the  $\text{Ce}^{3+}$  ( $\text{Eu}^{3+}$ ) scintillation efficiency and remove eventual OH content excess. After this treatment, the powder was inserted in a quartz tube at  $10^{-4}$ - $10^{-5}$  mbar and voids were eliminated by using an ultrasonic bath. The tube was finally sealed, connected to a support glass rod and inserted in a furnace at 2100 °C. A fiber with 220  $\mu\text{m}$  diameter was obtained by using the proper pulling speed.

With a slightly different procedure and composition, the Sol solution turned into Gel can alternatively be slowly dried in order to obtain a monolithic xerogel cylinder. After a proper thermal treatment it results in a completely vitrified glass monolith that can be fused and connected to a support rod in order to obtain a proper optical preform. Both kinds of preforms are shown in Fig. 1.

Powder in tube optical pre-forms lead to few meters long optical fibers; on the other hand bulk rod drawing permits to get about 50 m of defects free fiber from a single cylindrical rod of 10 mm diameter and 4 cm length.

The optical fiber dosimeters were composed by a portion of rare-earth-doped silica glass fiber (1 cm length, 220  $\mu\text{m}$  diameter), obtained by drawing such optical pre-form.

This portion was connected (Starlite S.r.l, Italy) to a commercial optical fiber by fusion splicing, which was optically coupled (Fraen Corporation S.r.l., Italy) to a photomultiplier tube (PMT) (Hamamatsu, R7400 P) operating in photon counting mode. The signal is processed by a circuitry and software properly designed (ELSE S.r.l., Italy). In order to maximize the transmitted light through the passive fiber the best results were obtained with a large core Hard Polymer Clad Multimode Fiber 0.48 NA high OH from Thorlabs (BFH48-200) or 3M (FT-200-URT).

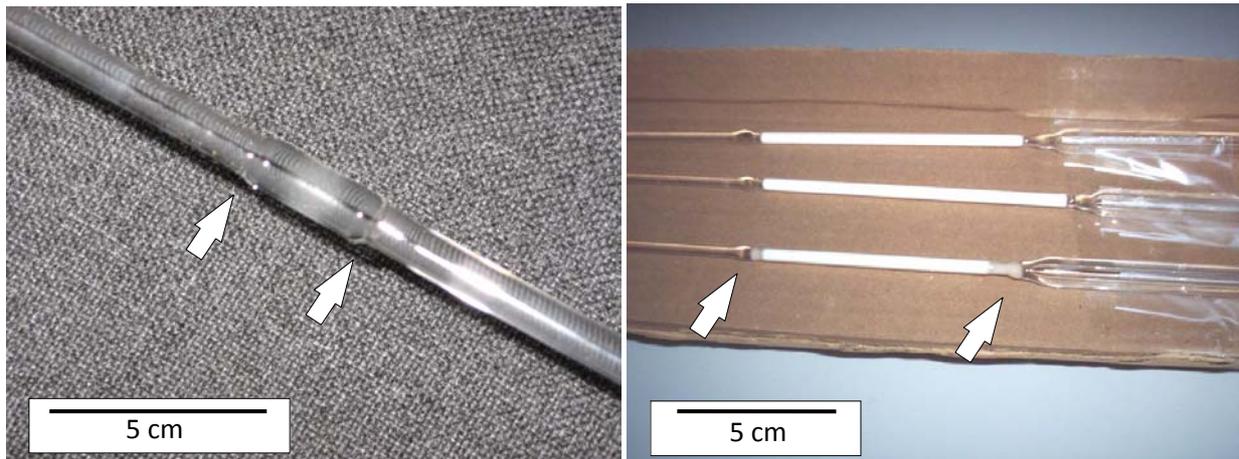


Fig. 1: Left: detail of the active bulk rod welded in the optical preform. Right: optical preform obtained by the “powder in tube” technique. Arrows are pointed on the welding point.

In order to subtract the Cerenkov contribution to the signal response a double optical fiber geometry has been adopted. In this experimental setup the first photomultiplier is connected to the active scintillating fiber, and the second one is connected to a passive silica fiber identical to the first but without the cerium activated portion. A schematic drawing of the system is shown in Fig. 2 together with the photograph of the data acquisition unit and of a fiber.

Radio-luminescence (RL) emission spectra were collected after X-ray irradiation at room temperature (RT) using a Philips 2274 X-ray tube operated at 20 kV; the detection system was a CCD (Jobin-Yvon Spectrum One 3000) coupled to a monochromator operating in the 210–780nm range.

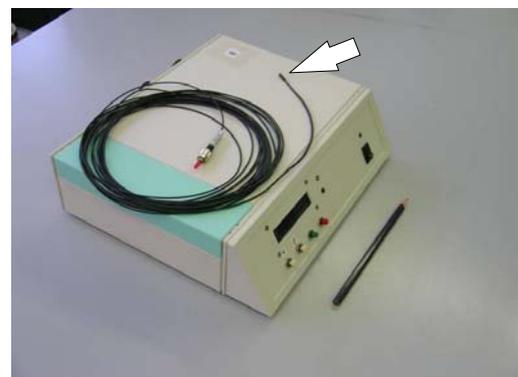
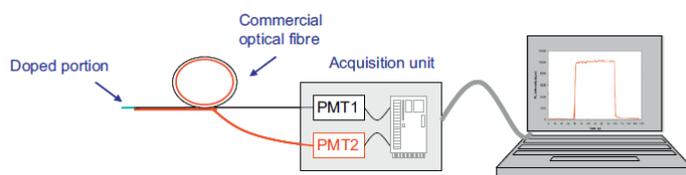


Fig.2 : Schematic drawing of the dosimetric systems in double optical fiber geometry (left) and picture of the acquisition unit and the fiber sensor (right) with the active end pointed by the arrow.

Different source of ionizing radiation have been employed in this work: irradiations with soft X rays were carried out by a Machlett OEG 50 X-ray tube at the University of Milano-Bicocca (Milano, Italy); hard X rays and 6 MeV electrons irradiations were performed by a Clinac 2100 CD (Varian, USA) medical linear accelerator at the Azienda Ospedaliera Maggiore della Carità in Novara, where also irradiations with a  $^{60}\text{Co}$  radiotherapy unit (Alcyon) were performed. Finally, tests with 38 MeV protons were carried out by an accelerator source at the Ispra European Joint Research Center (Ispra, Italy).

### 3.RESULTS AND DISCUSSION

A first set of measurements was performed in the past on differently doped glasses in order to define the best candidates for further experiments. Variables in this first phase consisted in doping with different rare earth ions (cerium, europium, gadolinium, terbium, ytterbium) and in variation of sintering conditions for Ce doped sol-gel matrices (either oxidizing or reducing atmosphere ) [22-27]. Following these previous tests, further investigation was restricted to Cerium and Europium doping. Therefore scintillating glass fibers were produced and tested by means of X-ray irradiation. In Fig. 3 we report the RL emission spectra of the doped powders. The Ce-doped one displays the 5d-4f allowed transition band, while Eu-doped powder displays the  $^5D_0 - ^7F_j$  4f-4f transition lines of  $\text{Eu}^{3+}$ . The emission spectrum of Eu doped powder is at higher wavelengths with respect to that of Ce-doped samples; in principle this should favor the discrimination, by filtering, between scintillation light (produced in the doped fiber portion) and Cerenkov light (produced also along the passive part) which is more intense in the UV region.

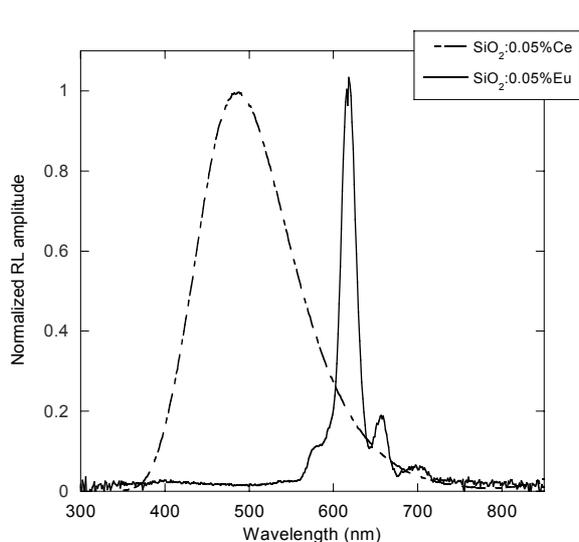


Fig. 3: RL emission spectra of Eu and Ce doped silica powders used for fibre pre-forms measured at RT under X-ray irradiation (20 kV).

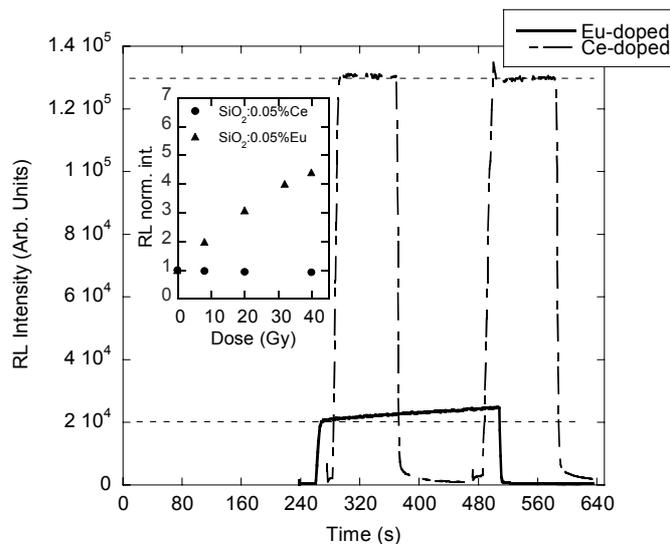


Fig. 4 : Typical RL signal versus irradiation time performed under x-ray irradiation. (32 kV). The dashed line represents the initial RL intensity of the Eu-doped fibre. Inset: RL normalized intensity of Ce- and Eu- doped fibres versus X-ray

However, as shown in fig.4 the Eu doped fiber is apparently not suitable for dose assessment. In fact the RL signal is weaker than in Cerium doped glasses; moreover its dependence on the radiation dose prevents a reliable dose evaluation. Due to such drawbacks only cerium doped fibers were considered in further tests as dosimetry devices. It was suggested that the sensitivity increase are due to the presence of deep traps [32,33]. As such traps are progressively filled by prolonged irradiation, the fraction of carriers being captured per unit time compared to the one reaching the luminescence centers is progressively reduced resulting in an increased scintillation efficiency. Once deep traps are almost completely filled the prompt scintillation emission reaches its maximum value. Such effect seriously compromises the suitability of a material as a radio-luminescent dosimeter. The presence of traps could also be the cause of the slight phosphorescence signal observed in Ce-doped fibers after the end of the irradiation pulse (Fig. 4). Studies are in progress to better explain the physical origin of the trapping defects and to eliminate, or at least minimize, their contribution. The consequence of this phenomenon on the practical use of the dosimeter is nevertheless of slight importance, since it implies only a slight increase in the background contribution that must be subtracted from the total counts, when repeated measurements are performed in a relatively short time. Tests at higher energy have been performed by means of a radiotherapy particle accelerator showing the response linearity versus dose as reported in fig. 5.

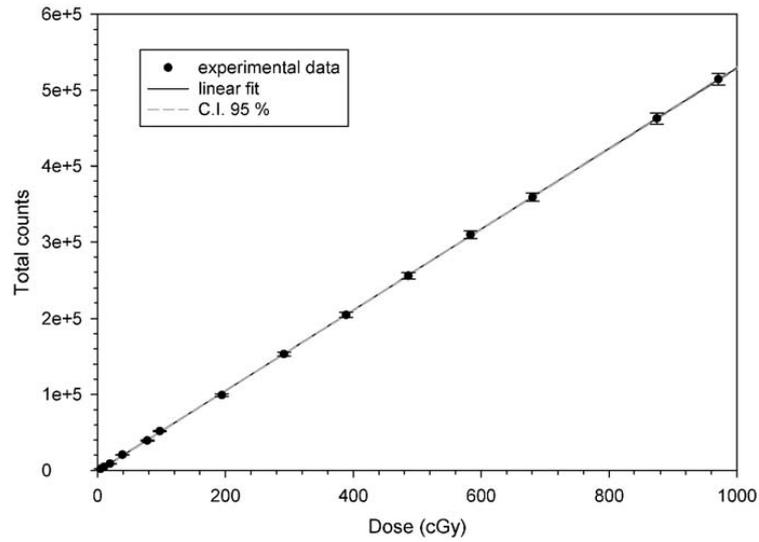


Fig. 5: Dose response of the Ce-doped dosimeter measured using a 6 MV photon beam. The data points represent the integrals of the RL versus time curves, after the background subtraction. Error bars correspond to 1SD.

The reproducibility of the dosimeter, evaluated as standard error of the mean of 10 consecutive measurements, was estimated equal to approximately 0.5%, coherently with typical values of ionization chambers. The dosimeter prototype exhibited a linear dose response over the whole dose range investigated, as shown in Fig. 5 The data points represent the integrals of the RL versus time curves, after background subtraction; the error bars correspond to 1SD. A straight line fitted to the experimental data ( $R^2 = 0.999$ ) is also drawn, together with the corresponding 95% confidence interval (CI).

The typical RL signal versus dose and time, measured by exposing the doped fiber to  $^{60}\text{Co}$  photons (0.36 Gy /min dose rate ) is shown in Fig. 6.

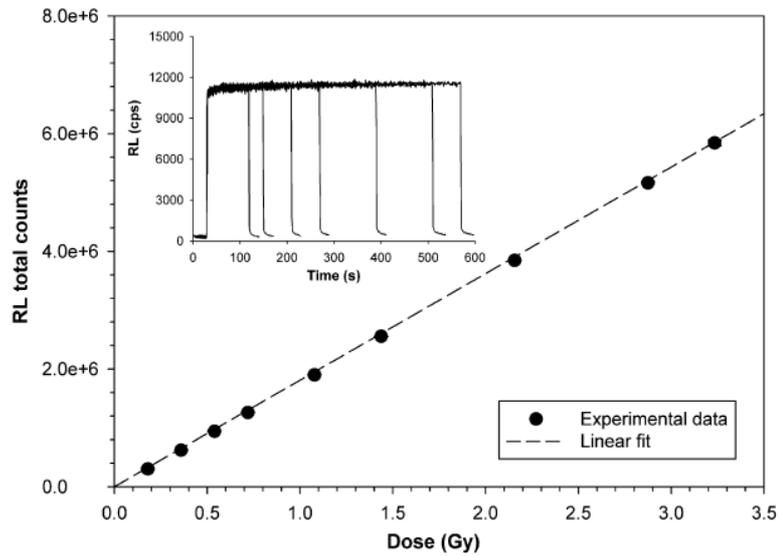


Fig. 6: Dose-response curve of the Ce-doped fiber dosimeter under  $^{60}\text{Co}$  photon irradiation. The inset shows the stability of the RL signal with increasing the irradiation time.

A satisfactory stability of the signal with increasing the irradiation time can be observed. Consequently the amplitude of the curve enables a direct measurement of the dose rate, and the total dose can be simply obtained by integrating the signal over the time.

Further tests were performed by means of electrons at the energy of 6 MeV showing very good results in agreement with X and gamma rays.

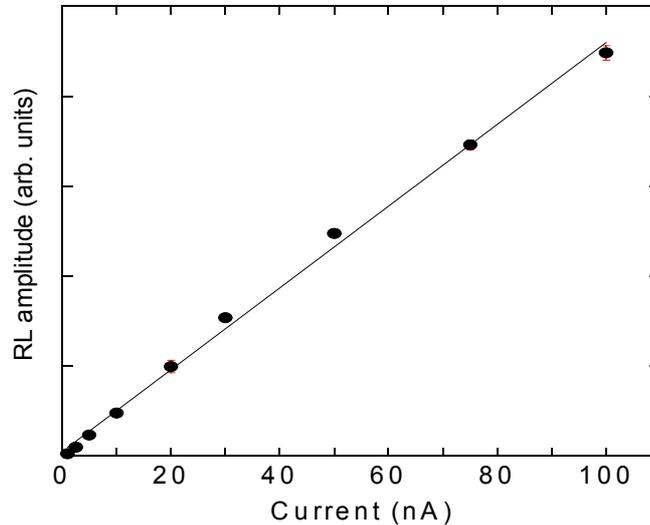


Fig. 7: Dose-response curve of the Ce-doped fiber dosimeter under 38 MeV proton irradiation.

More recently, due to the increasing importance of the hadron therapy new measurements were performed under protons radiation as the curve reported in fig. 7 shows.

Preliminary tests seem very encouraging, since the linearity and the reproducibility shown is good and in agreement with previous data.

#### 4. CONCLUSIONS

Up to now, tests with promising results have been performed with fibers doped with cerium, for which the relevant parameters for dosimetry aims have been found to be the following:

- small volume (fiber diameter 100-200  $\mu\text{m}$ ), negligible radiation field perturbation (leading to high spatial resolution, and allowing to perform very accurate "point dose evaluations")
- possibility of simultaneous multiple point measurements (using several fibers connected to the same measuring unit)
- possibility of remote measurement (distant from the radiation field)
- real time and "in vivo" use (dose rate - maximum time resolution 10 ms - and total dose progressively accumulated can be simultaneously evaluated during irradiation)
- high sensitivity and reproducibility comparable to that of ionization chambers
- independence of sensitivity upon accumulated dose
- independence of the response from the environmental conditions (temperature up to at least 50  $^{\circ}\text{C}$ , pressure, humidity of the air)
- linearity of the response over a wide range of doses/dose rates
- negligible ageing of the detector, without change of its characteristics (high radiation hardness)
- non toxicity

So, the optical-fiber based dosimeter will be employed:

- for in vivo measurements on patients, allowed also by the not necessity of any electrical supply, and therefore for the development of methodologies for measuring and reducing patient exposures, while maintaining and/or improving diagnostic/clinical information from existing and emerging imaging techniques; the dosimeter can be employed also for developing broadly applicable methodologies for assessing and reducing dose to peripheral tissues for radiotherapy in general and, in particular, for more advanced and innovative procedures having potential for wide use;
- for the development of methodologies for better assessing and reducing exposure to medical staff for procedures resulting in potentially large doses or complex radiation fields (e.g. interventional radiology).

Apart from medical applications, the characteristics of these doped fibers could allow their employment in other fields of radiation measurement, like for example the radiation control in nuclear reactors. For such purpose, sensitivity to neutrons could be improved by suitable co-doping or coatings with elements showing high neutron cross section .

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