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Refractive index change dependence on Ge(1) defects in γ-irradiated Ge-doped silica

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We present an experimental study regarding the effects of the γ radiation on silica glass doped with Ge up to 10 000 ppm molar produced by the sol-gel technique. We have determined the irradiation-induced changes in the refractive index (Δn) as a function of the oxygen deficiency of the samples, evaluated from the ratio between the germanium lone pair centers (GLPC) and the Ge content. Δn at 1500 nm have been estimated using optical-absorption spectra in the range 1.5–6 eV. We have found that Δn is independent of Ge differences for GLPC/Ge values $<10^{-4}$, while it depends on Ge for larger oxygen deficiencies. In details, the oxygen deficiency can reduce the induced Δn of the investigated materials and our studies evidence that the photosensitivity of the GeO_2-SiO_2 glass is reduced until the GLPC concentration reaches values of $2\times10^{17}-5\times10^{17}$ defects/cm^3. We have also investigated the induced concentration of paramagnetic point defects [Ge(1), Ge(2), and E'Ge] using the electron-paramagnetic-resonance (EPR) technique. From the comparison of the optical and EPR data we have further found a relation between the induced optical-absorption coefficient at 5.8 eV and Ge(1) defects, a linear correlation between Ge(1) and Δn and the absence of a correlation between the other paramagnetic defects and Δn. These findings suggest that the Δn phenomenology is closely related to the Ge(1) generation mechanisms and this latter is affected by the oxygen deficiency.

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I. INTRODUCTION

Germanium-doped silica is widely used for the fabrication of optical devices such as fiber, sensors, and multiplexers,[1–3] or in general for applications that need the selectivity of wavelengths.[4] The technological applications of this material are related to the observed variation in the refractive index, Δn, (photosensitivity),[1,5,6] and to the second-harmonic generation (SHG).[7] These two effects have been related, on the one hand, to the presence of the twofold-coordinated Ge atom,[1,8–10] also named as germanium lone pair center (GLPC) $:=\mathrm{Ge}^{\bullet\bullet}$,[11,12] where each symbol - stays for a bond with an O atom and $\bullet$ represents an electron. On the other hand, to the generation of point defects such as Ge(1), Ge(2), and E'Ge centers.[1,7,9,13,14] All these latter defects are paramagnetic structures and, in particular, the Ge(1) is suggested to consist in an electron trapped on a fourfold-coordinated Ge atom $:=\mathrm{Ge}^{\bullet}=$.[8,15] On the structure of the Ge(2), two different models have been suggested: a single ionized GLPC $:=\mathrm{Ge}^{\bullet}$ [8,16] or a Ge(1) with a Ge atom as a second neighbor.[9,15] Finally, the E'Ge is constituted by a threefold-coordinated Ge atom with an unpaired electron: $\equiv\mathrm{Ge}^{\bullet}$.[17] Ge(1) and E'-Ge models have also been supported by theoretical calculations.[14,18]

The dependence of the refractive index, that is, a macroscopic propriety of the material, on some microscopic structures is explained by the fact that these structures are responsible for some optical-absorption (OA) bands[1] and by the fact that the optical absorption and the refractive index are linked by the Kramers-Kronig relations.[4] In particular, the GLPC has been related with the amplitude of an OA band peaked at $\sim$5.15 eV, called $B_{2\beta}$,[19] that at the same time is responsible for two photoluminescence (PL) bands peaked at $\sim$3.2 and $\sim$4.3 eV.[19,20] In addition, the presence of this defect causes an OA band at $\sim$3.8 eV that is 10^3 times less intense than the 5.15 eV component.[21] Moreover, an optical-absorption band at $\sim$4.5 eV has been related to the Ge(1),[15,21] and one at $\sim$6.3 eV is attributed to the E'Ge center.[21] More complex is the assignment of an irradiation-induced band at 5.8 eV that someone associate to the Ge(2),[15,21] while others attribute to the Ge(1).[8,22]

In a previous work, it was investigated the effect of the oxygen deficiency on Ge(1) defects generation by γ-ray irradiation.[16] It was found that oxygen deficiency, evaluated by the GLPC/Ge concentration ratio, could inhibit or favor the defect generation, in dependence of such ratio and the Ge doping level. In the present work, using a larger set of samples and the Kramers-Kronig relations, we examine the variation in the refractive index Δn at 1500 nm starting from the variation in the optical-absorption spectra in the range 1.5–6 eV induced by γ ray. The changes in the refractive index are investigated for samples with different GLPC/Ge ratios, where additional Ge-related point defects are induced by γ irradiation. The aim of this work is to determine how Δn is affected by the presence of point defects and, on the basis of the above considerations, by the oxygen deficiency of the samples.

II. EXPERIMENTAL

The employed materials have been produced by the sol-gel technique starting from a weighted mixture of tetraetil-orthosilicate and tetraetil-orthogermanate. In Table I, the Ge and GLPC contents of all investigated samples are summarized. The nominal Ge content was tested for some samples by inductively coupled plasma mass spectrometry or by instrumental neutron activation analysis measurements while

TABLE I. Sample nickname, Ge content, GLPC content.

Sample	Ge content (atoms/cm^3)	GLPC (defects/cm^3)
A0	2.2×10^{20}	Not detected
A1	2.2×10^{20}	2.2×10^{16}
A2	2.2×10^{20}	1.5×10^{17}
A3	1.4×10^{20} [a]	4.9×10^{17}
A4	1.4×10^{20} [a]	2.5×10^{18}
A5	2.2×10^{20}	5.0×10^{18}
B1	2.2×10^{19}	1.0×10^{15}
B3	2.7×10^{19} [a]	1.6×10^{16}
B4	2.7×10^{19} [a]	5.6×10^{17}
B5	1.8×10^{19} [b]	4.5×10^{17}
C1	2.2×10^{18}	Not detected
C3	5.5×10^{18} [a]	Not detected
C4	5.5×10^{18} [a]	1.3×10^{16}
C5	1.7×10^{18} [b]	6.8×10^{16}

[a] Determined by inductively coupled plasma mass spectrometry, error of ~10%.

[b] Determined by instrumental neutron activation analysis measurement (Reference 23), the error is ~2%. In all the other cases the content is the nominal one. In the sample name the letter refers to the Ge content and the number to the preparation route described in the experimental section.

the GLPC contents were estimated as described in the following by optical measurements.

The densification of the aerogels was performed using opportune routes of temperature and atmosphere to obtain different ratios between the doping level and the concentration of the GLPC. The sample A0 was heated up to 1200 °C at a rate of 0.3 °C/min, then it was kept at this temperature for ~45 min before being cooled down to room temperature. Up to 800 °C, the sample was under a constant flux of O_2 and N_2 while during the other parts of the treatment it was in ambient atmosphere. The sample A2 was obtained using the procedure of the A0 sample with the difference that starting from 800 °C it was kept in He atmosphere and then it was cooled in N_2 atmosphere.

The samples A1, B1, and C1 were heated at a rate of 0.2 °C/min from room temperature up to 400 °C, and they were kept at this temperature for 4 h before reaching 800 °C at a rate of 0.6 °C/min. During this treatment the samples were kept in a O_2 and N_2 atmosphere. After these routes, they were heated up to 1200 °C in a He ambient at a rate of 1.2 °C/min, and maintained for half an hour in these conditions before to return to room temperature in N_2 atmosphere.

The samples named A3, B3, and C3, and that named A4, B4, and C4 have been obtained by the same gel. They were heated at a rate of 0.3 °C/min from 25 °C to 1150 °C, then they were kept at this temperature for 24 h before returning to room temperature. Until 700 °C, the samples were under a O_2 flux while during the other part of the process a low-pressure atmosphere was used. The difference in the preparation of the two sample sets is the fact that the A4, B4, and C4 materials were kept under a He flux for 30 min at 700 °C, before going to the low pressure. The value of the latter, $\sim5\times10^{-8}$ atm, is one order of magnitude higher than that used for the A3, B3, and C3 samples. Finally, the samples A5, B5, and C5 were obtained using the same route of samples A3, B3, and C3 but with different vacuum value. The employed sol-gel samples are disks with diameter ~4 mm and thickness values inside the range ~0.5–1 mm, with the major surfaces optically polished.

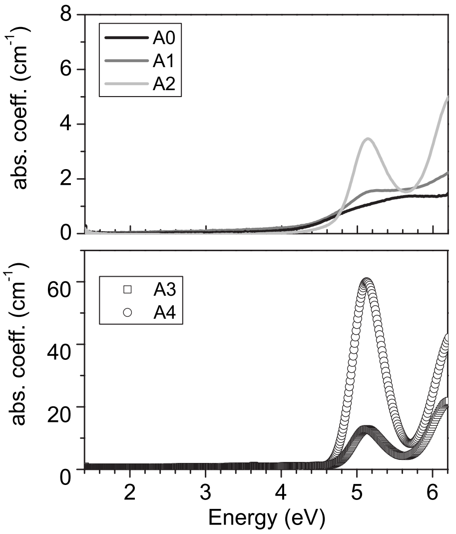

FIG. 1. Optical-absorption spectra acquired for the samples A0 (black line), A1 (medium gray line), A2 (light gray line), A3 (□), and A4 (○) before irradiation.

All the experiments and measurements were carried out at room temperature. The γ irradiation was performed using a ^{60}Co source with a dose rate of ~2 kGy/h and arriving at the doses of ~2 and ~10 kGy. The OA spectra were acquired in the spectral range 1.5–6 eV using a spectrophotometer Jasco V-560. The PL spectra have been recorded with a Jasco 6500 using a 150 Watt Xenon lamp. The electron-paramagnetic-resonance (EPR) measurements were performed using a Bruker EMX spectrometer working at 9.8 GHz with a modulation of the magnetic field with frequency of 100 kHz and all other experimental parameters regulated so that the EPR signal was neither distorted nor saturated by the microwave power. To estimate the paramagnetic centers concentration the double integral of the EPR signals were determined and compared to that of a reference sample having the same dimensions and containing a known concentration of E'-Si centers.[24]

III. RESULTS

A. Optical-absorption effects

In Fig. 1, we report typical OA spectra acquired for the as-grown A0, A1, A2, A3, and A4 samples. Excluding the sample A0, in all these spectra we can clearly see the presence of the $B_{2\beta}$ band at 5.15 eV with intensity dependent on the sample. In particular, the band is more intense in the A4 sample and less intense in the A1. As regards the A0 sample, we note that no PL activity associated to the GLPC was

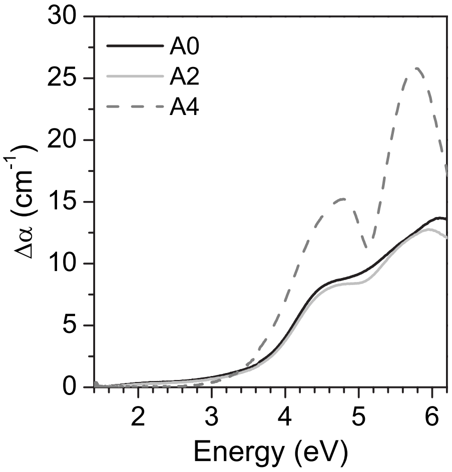

FIG. 2. Induced optical-absorption coefficient ($\Delta\alpha$) in samples A0 (black line), A2 (light gray line), and A4 (medium gray dashed line) at the irradiation dose of ∼10 kGy.

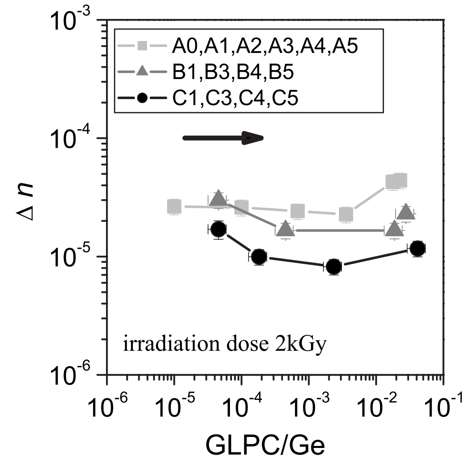

FIG. 3. Induced variation in the refractive index (Δn) at the dose of ∼2 kGy as a function of GLPC/Ge ratio in samples A (—■—), B (—▲—), and C (—●—); the arrow indicates the direction of increase in the sample number reported in the label.

detected even if in the OA spectra the absorption coefficient is different from zero at 5.15 eV, so concluding that the absorption in this sample is due to other centers.

The OA spectra have been measured also in all the other samples here reported and by a Gaussian decomposition the $B_{2\beta}$ band has been isolated to determine the GLPC content using Smakula's formula[1]

$$Nf \approx \alpha_{\mathrm{MAX}}\Gamma\Lambda \times 9.111 \times 10^{15}, \qquad (1)$$

where N is the concentration of defects, f is the oscillator strength $f \sim 0.1$,[8,25–27] α_{MAX} is the absorption coefficient at the maximum position, Γ is a line-shape parameter that for a Gaussian profile is 1.0645,[1] and Λ is the full width at the half maximum (∼0.46 eV).[20,21,26] The error on the so estimated GLPC concentrations is ∼20% and it depends essentially on the different values attributed to their oscillator strength. This formula has been used for A1, A2, A3, A4, B4, B5, and C5 materials. At variance, in the A5 sample the $B_{2\beta}$ band amplitude overcomes the maximum limit of detection of our system (∼4 optical density) so the estimation of the GLPC concentration was carried out using the amplitude of the band at 3.8 eV and the known ratio of 10^{-3} to the $B_{2\beta}$.[21]

For some samples (B1, B3, and C4) the initial OA was too low to determine the $B_{2\beta}$ and it was necessary to estimate the GLPC content from the PL at 3.2 eV excited at 5.15 eV. The defect content was then determined by comparing the PL amplitude with that of samples for which this PL is linearly correlated with the $B_{2\beta}$ amplitude and the latter can be estimated. In the samples C1 and C3 the GLPC activity was undetectable even using PL measurements so for these samples the GLPC/Ge ratio is assumed to be the same of the samples produced in the same way with higher Ge content and a detectable GLPC activity.

In Fig. 2, the differences between the OA spectra acquired for the samples A0, A2, and A4 at the dose of 10 kGy and the spectra recorded for the as-grown samples are reported. From the comparison of these spectra we observe that the amplitude of the induced optical absorption at 4.5 and 5.8 eV is similar for the A0 and A2 samples while the amplitude at these energies is higher in the A4 sample. In particular, the absorption coefficient at 4.5 and 5.8 eV is 8 and 12 cm^{-1} in A0, 7.8 and 12 cm^{-1} in A2 while it is 14 and 26 cm^{-1} in A4. In the spectra of Fig. 2, the dip at ∼5.2 eV in the sample A4 can be considered the consequence of the partial bleaching of the $B_{2\beta}$ activity. Instead, the different profile at ∼6.3 eV could be influenced by the different concentrations of the induced E'Ge. In general, the shape of the OA spectra of the other samples agrees with that of A samples, showing main contribution at 4.5 and 5.8 eV.

In Figs. 3 and 4, we report the variations in the refractive index calculated from the induced optical absorption in the different samples and measured at the irradiation doses of ∼2 kGy (Fig. 3) and 10 kGy (Fig. 4).

In particular, the Δn values have been calculated using a decomposition of the induced OA by fitting with sets of Gaussian bands and the equation reported in Ref. 4

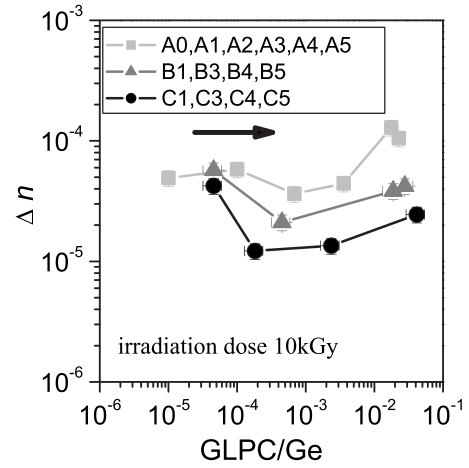

FIG. 4. Induced variation in the refractive index (Δn) at the dose of ∼10 kGy as a function of GLPC/Ge ratio in samples A (—■—), B (—▲—), and C (—●—); the arrow indicates the direction of increase in the sample number reported in the label.

$$\Delta n(w) = \frac{c}{\sqrt{\pi}} \sum_i \frac{\Delta\alpha_i B_i}{w_i^2 - w^2}, \tag{2}$$

in which c is the speed of light in vacuum, $\Delta\alpha_i$ is the variation in the absorption coefficient at the maximum of the i component peaked at the frequency w_i, B_i is $\sqrt{2}\sigma$ (σ is the standard deviation) of the i Gaussian component, and w is the frequency corresponding to 1500 nm. The induced absorption in the sample A5, in the energy range of the $B_{2\beta}$, has been determined by fitting the tails of the Gaussian that describes the $B_{2\beta}$ since, as already noticed, this band always overcomes the maximum detection limit.

From the data of Figs. 3 and 4, we note that when the GLPC/Ge ratio is $\leq 10^{-4}$, Δn is quite independent from the Ge doping level and from the oxygen deficiency. At variance, when the GLPC/Ge ratio increases, Δn depends on the Ge doping level. In particular, for both dose values we observe that the induced Δn in the samples C3 and C4 is lower than that induced in the sample C1 and, moreover, for C3 and C4 samples Δn is almost the same. In sample C5 we observe that at the dose of ~2 kGy the induced change is similar to that of C3 and C4, instead at the higher dose (Fig. 4) the induced Δn is larger.

As regards the samples B, Δn is quite independent of GLPC/Ge ratio at the dose of ~2 kGy, while for the dose of ~10 kGy a decrease followed by a recovery is observed as a function of the GLPC/Ge ratio. We note that the samples B4 and B5, with a GLPC content of $\sim 5 \times 10^{17}$ defects/cm^3, tend to show similar variation in the refractive index of the B1 sample having $\sim 10^{15}$ defects/cm^3.

Finally, the A samples, having the highest Ge doping level, show a photosensitivity that is equivalent or superior to that of A0, both at ~2 and ~10 kGy.

B. Paramagnetic defects

No EPR signal was present in the spectra acquired before the irradiation in each sample. After the first irradiation dose of 2 kGy, it is possible to identify the presence of the signals related to different types of Ge point defects at g ~ 2 in the EPR spectra.

In Fig. 5, the EPR spectra acquired at the dose of ~10 kGy for the samples A0, A2, and A4 (from top to bottom) are reported. A decomposition of the EPR spectra using the reference line shapes of Ge(1), Ge(2), and E'Ge reported in Ref. 16 has been carried out. In the spectrum of the sample A0 it is possible to identify the presence of Ge(1) while the presence of the E'Ge signal is evidenced by the decomposition and the Ge(2) signal is absent. Also in the spectra of A2 and A4 samples the Ge(1) and E'Ge signals are present. Moreover, in these cases the Ge(2) signal is observed, as tested by the presence of a negative signal at g ≈ 1.9867 (see zoom of Fig. 5) that is associated to this type of paramagnetic point defect.[28] The concentrations of every species were calculated using the EPR spectra decomposition. It is important to note that Ge(1) concentrations of these materials are 10^{17} defects/cm^3 (A0 and A2), 2.3×10^{17} defects/cm^3 (A4) while the Ge(2) are induced only in samples A2 and A4 in the concentration of 0.2 and 4.3×10^{17} defects/cm^3, respectively. At this dose the induced E'Ge concentration are $\sim 1.5 \times 10^{16}$, 2.5×10^{16}, and 1.3×10^{17} for A0, A2, and A4 samples, respectively.

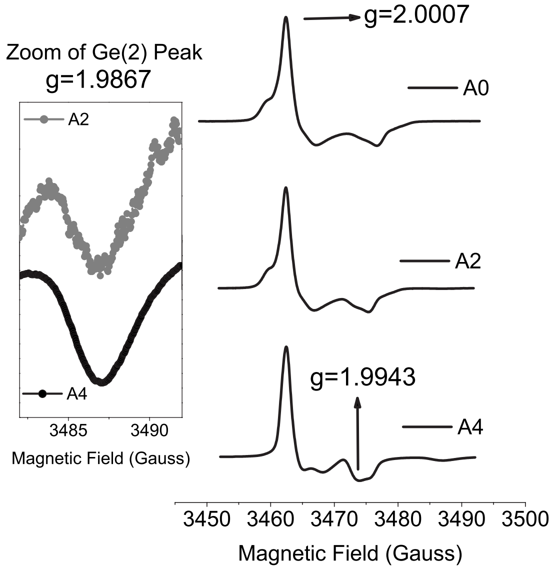

FIG. 5. EPR spectra acquired after the irradiation at ~10 kGy in A0, A2, and A4 samples, from top to bottom. In the inset, the zooms regarding the peak at about 3487 G, attributed to the Ge(2) EPR signal, are reported.

In Fig. 6, we report the induced concentration of Ge(1) defects at the dose of 2 kGy as a function of the GLPC/Ge ratio. We observe that the induced Ge(1) concentration is independent both of Ge and GLPC content and GLPC/Ge ratio when the latter is lower than 10^{-4}. At variance, for higher values of this ratio we find a variable dependence. In particular, for samples named C the induced concentration decrease for GLPC/Ge values $>10^{-4}$, and at the same time it appears independent both of Ge and GLPC content. In the samples of B type a decrease in the induced Ge(1) concentration followed by a recovery is observed. Finally in the samples A the Ge(1) induced by the radiation have concen-

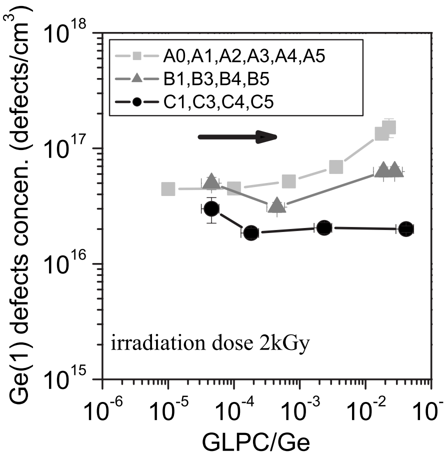

FIG. 6. Induced concentration of Ge(1) point defects at the dose of ~2 kGy as a function of GLPC/Ge ratio in samples A (—■—), B (—▲—), and C (—●—); the arrow indicates the direction of increase in the sample number reported in the label.

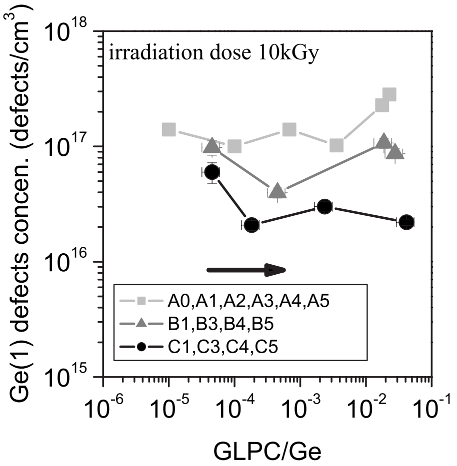

FIG. 7. Induced concentration of Ge(1) point defects at the dose of ~10 kGy as a function of GLPC/Ge ratio in samples A (—■—), B (—▲—), and C (—●—); the arrow indicates the direction of increase in the sample number reported in the label.

tration values similar or higher with respect to that of sample A0.

The concentrations of Ge(1) induced at the dose of 10 kGy are reported in Fig. 7. Even for this dose we observe that the Ge(1) are induced independently of Ge and GLPC content when the GLPC/Ge ratio is inferior to 10^{-4}. For higher values in samples named C the induced concentration is always lower than that induced in C1. As observed at the dose of 2 kGy, also at 10 kGy for the samples B a decrease followed by a recovery of the induced Ge(1) concentration is observed. Finally, for the samples A the induced Ge(1) concentration appears to depend from GLPC/Ge only for samples A4 and A5 where the GLPC concentration is higher than 2×10^{18}.

IV. DISCUSSION

The presence of the 5.8 eV band in every employed material leads to some considerations regarding the attribution of this band to the Ge(2) defect. In fact, at 10 kGy the samples A0 and A2 have similar Ge(1) concentration ($\sim10^{17}$ defects/cm^3) and show similar values of the induced absorption coefficient ($\sim$12 cm^{-1}) at 5.8 eV; furthermore, the A4 sample, with double the Ge(1) concentration (2.3×10^{17} defects/cm^3), has double induced absorption at 5.8 eV ($\sim$26 cm^{-1}). These latter findings together with the absence of Ge(2) in A0 and A2, and the high Ge(2) content of A4 ($\sim4.3\times10^{17}$ defects/cm^3), suggest that the latter defect does not significantly affect the optical absorption at 5.8 eV, that at variance should be related to the Ge(1), as proposed in.[8,22,29]

From Figs. 3 and 4, regarding the refractive index changes, and Figs. 6 and 7, that regards the induced Ge(1) concentration, we observe many similarities in the dependence on GLPC/Ge ratio of these two radiation effects, the only differences appear for the C5 sample. Considering these similar dependencies, in Fig. 8(a) we report the variation in the refractive index as a function of the induced concentration of the Ge(1).

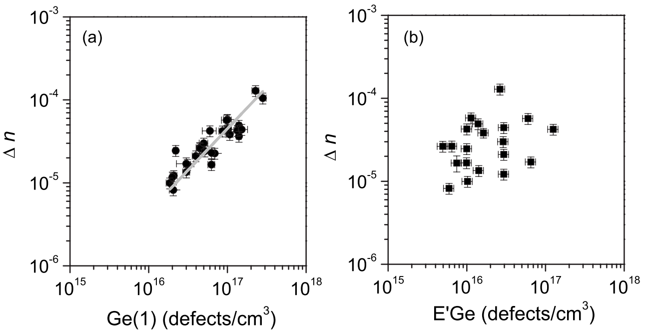

FIG. 8. (a) Induced variation in the refractive index (Δn) as a function of the induced concentration of Ge(1) defects in all the investigated samples; the line represents the linear best fit $\Delta n = 4.5\times10^{-22}\times\text{Ge}(1)$. (b) Dependence of Δn on the E'Ge concentration.

The data of Fig. 8(a), show a linear correlation between Ge(1) concentration and Δn. In particular, we find that

$$\Delta n = \kappa \text{Ge}(1)_{\text{conc}}, \tag{3}$$

where $\kappa = (4.5 \pm 1.5)10^{-22}$ cm^3 and Ge(1)$_{\text{conc}}$ is the Ge(1) concentration. It is important to note that the presence of E'Ge with the consequent presence of an OA band peaked at 6.3 eV,[21] could affect the observed correlation between Δn and Ge(1)$_{\text{conc}}$. To verify the relevance of the E'Ge on the investigated refractive index change in Fig. 8(b) we report Δn as a function of E'Ge concentration. It is found that the variations in Δn are clearly uncorrelated with the E'Ge concentration in the dose range explored. These findings suggest that the main contribution to the irradiation induced variation in the refractive index arises from Ge(1) generation.

The data on Δn reported in Figs. 3 and 4 indicate that the oxygen deficiency reduces the photosensitivity for low Ge content at both the reported doses. This effect can be compensated by the increase in the GLPC. These findings, basing on the relation between Δn and Ge(1) defects, are justified by the competition between two oxygen-deficiency effects. In fact, on the one hand, the oxygen deficiency reduces the efficiency of the Ge(1) generation, as reported in Ref. 16 and as confirmed by the behavior of the samples C reported in Figs. 6 and 7. On the other hand, the presence of GLPC, that are potential electron donors,[8,12] increases the electron trapping by tetra-coordinated Ge atoms. This effect, for example, determines the behavior of B samples.

Moreover, the increase in the set of samples here investigated permits us to affirm with more confidence that the reduction effects in the photosensitivity are activated when the ratio GLPC/Ge is $\sim10^{-4}$ and that they are compensated by the increase in the GLPC defects that produce the recovery of the photosensitivity. This aspect is evidenced looking to the data of samples with same Ge doping level and variable GLPC content both for Δn (Figs. 3 and 4) and for Ge(1) (Figs. 6 and 7). The total recovery takes place when the GLPC concentration is inside the range $2\times10^{17}-5\times10^{17}$ defects/cm^3, as it is possible to deduce thanks to the data regarding the samples A2, A3, B4, and B5. The effect of the presence of GLPC could be relevant also because the bleaching of their absorption band due to irradiation of the sample should induce a negative change in the refractive index.[1,21] We note that in the investigated samples even if present the effect of bleaching is overwhelmed by the positive change in the refractive index induced by Ge(1) generation, showing that the latter effect is predominant.

V. CONCLUSION

The reported experimental results evidence a relation between the induced changes in the refractive index (macroscopic γ irradiation effect) and the Ge(1) generation (microscopic effect of γ irradiation). Moreover, support to the assignment of the 5.8 eV induced optical-absorption band to the Ge(1) point defect is found together with suggestion that Ge(2) point defect has not significant absorption at this energy. On the basis of the found relation it is possible to explain the dependence of Δn on the oxygen deficiency. In fact, the latter reduces the photosensitivity of Ge-doped silica for low doping level because it inhibits the efficiency of Ge(1) generation mechanisms. At variance, the photosensitivity is recovered when the GLPC content is so high to compensate the first effect with an higher content of possible electron donors. In particular, our data suggest that the reduction effect is activated at 10^{-4} GLPC/Ge while the GLPC concentration necessary to compensate this effect is $>2\times10^{17}$ defects/cm^3.

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