



Optical bleaching of the 375 °C TL peak, $[\text{GeO}_4/\text{Li}^+]^0$ EPR center and OSL signal in irradiated natural quartz

G. Vaccaro^a, L. Panzeri^{a,b,*}, A.M. Monti^a, M. Martini^{a,b}, M. Fasoli^{a,b}

^a Dipartimento di Scienza dei Materiali, Università degli Studi di Milano-Bicocca, via Cozzi 55, IT-20125 Milan, Italy

^b INFN, Sezione di Milano-Bicocca, Piazza della Scienza 3, IT-20126 Milan, Italy

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ABSTRACT

The effect of exposure of quartz samples to blue (470 ± 30 nm) and UV (365 ± 10 nm) light has been studied. Thermoluminescence, TL, Electron Paramagnetic Resonance, EPR, and Optically Stimulated Luminescence, OSL, have been considered.

Looking at the OSL signal, while a reduction of the slow component is indeed induced by the UV exposure, a similar effect is observed, although with lower efficiency, under blue light exposure. Therefore, no conclusions can then be drawn as regards the effects induced on the OSL signal upon a change in the excitation wavelength.

The effect of the UV and blue light exposure on the so called Slowly Bleachable Peak, SBP [1], observed by TL at 340 °C, heating rate 5 °C/s, is presented. It is noticed a decrease of the SBP induced by UV exposure while no effect is caused by blue light exposure. A similar behaviour is detected when looking at the EPR signal of the $[\text{GeO}_4/\text{Li}^+]^0$ center, that is reduced by the UV light, while no effect is seen after exposure to blue light.

These results suggest that one of the electron sources originating the SBP can be identified as the $[\text{GeO}_4/\text{Li}^+]^0$ center.

1. Introduction

The identification of the defects responsible for trapping and luminescence emissions in quartz is not straightforward. In fact, the studies of TL and OSL give only a phenomenological picture, while the possibility of comparing the EPR features as a function of irradiation and thermal treatments with the luminescence properties can allow to determine which are the centers responsible for them. As regards the trapping centers, germanium, practically always present in quartz, is known to give origin to a number of centers that play a role in electron trapping: the diamagnetic $[\text{GeO}_4]0$ center, given by a Ge substituting for a Si, the paramagnetic $[\text{GeO}_4]^-$ center i.e. a $[\text{GeO}_4]^0$ center that has trapped an electron upon irradiation and the paramagnetic centers $[\text{GeO}_4/\text{M}^+]^0$, where M^+ is an alkaline ion, Li^+ or Na^+ [2–4].

McKeever et al. in 1985 [5] proposed $[\text{GeO}_4]^-$ as a trap responsible for the so called “110 °C” TL peak and recently this proposal found a confirmation in a series of measurements giving an excellent agreement between the decay of the $[\text{GeO}_4]^-$ EPR signal and the decay of the 110 °C TL peak at various temperatures [6]. In the present work we focus our attention to the $[\text{GeO}_4/\text{M}^+]0$ EPR signal, compared with the TL peaks at around 340 °C, using a 5 °C/s heating rate, and with the OSL features of quartz.

Looking at the TL glow curves of quartz it is difficult to determine a typical one, because they are strongly dependent not only on the concentration of impurities, but also on the previous irradiation and thermal treatments the quartz sample has been submitted to. So different quartz can present very different glow curves [7]. However, since the pioneering work by Aitken [8], quartz has been reported as presenting at least three main peaks, the already mentioned 110 °C and two peaks at 325 °C and 375 °C, always with a 15 °C/s heating rate. These latter are typically considered for dating purposes, because, due to their high temperature position, they are characterized by a sufficient stability for quaternary period dating.

A fundamental difference between the 325 °C and 375 °C peaks is their bleaching behaviour: under ambient illumination, the 325 °C TL peak bleaches very rapidly and, for this reason, it was appropriately called “rapidly bleaching peak” (RBP), whereas a slower bleaching rate was observed for the 375 °C TL peak, hence indicated as “slowly bleaching peak” (SBP) [1,9–11]. In particular, Spooner et al. [11] showed that for illumination between 400 and 700 nm, only the RBP is rapidly bleached, whereas the SBP is essentially unaffected, while with wavelength shorter than 400 nm, the RBP and SBP bleach effectively in a rapid and slow way, respectively. This different bleaching mechanism makes these two TL peaks utilizable for different purposes in dating.

* Corresponding author at: Dipartimento di Scienza dei Materiali, Università degli Studi di Milano-Bicocca, via Cozzi 55, IT-20125 Milan, Italy.
E-mail address: laura.panzeri@unimib.it (L. Panzeri).

The RBP is strictly related to the OSL signal in quartz and is extremely attractive for geological sediments dating, where solar illumination acts as a clock resetting for OSL signal.

In the last decades, the OSL technique has been largely used for dating and dosimetry. The fact that the signal resetting occurs because of exposure to sunlight rather than heating made OSL the preferred technique for geological sediment dating. A typical OSL shine down curve can be described by a decay with more than one component. Generally three main components, with largely different decay times, a fast, a medium and a slow component are reported [12,13]. In particular, the intensity of the fast component, obtained by integrating the first seconds of OSL signal, is directly used for dosimetry application. This component was related to an unknown electron source, characterized by a high activation energy and long lifetime, which is also responsible for the TL emission of RBP in natural quartz [14]. On the other hand, the role of the medium and the slow component should be clarified in order to properly estimate their contributions to the signal used for dosimetry.

The centers originated by Al substitution for Si are commonly considered to play a fundamental role in the mechanisms involving OSL and RBP. On the other hand, the situation regarding the SBP is even farther from clearness as compared with that of the RBP. Franklin [9] suggested that SBP is part of the “families” of centers responsible for 110 °C TL peak. Following this scheme and considering that we previously demonstrated the role of electron source of $[\text{GeO}_4]^-$ centers for the 110 °C TL peak [6], the “family” of Ge centers could be a possible candidate involved in the mechanism originating the SBP. Besides, McMorris [15] observed that a TL peak at 300 °C (heating rate 0.17 °C/s) decreases in the same fashion as the $[\text{GeO}_4/\text{M}^+]^0$ (where M^+ is an interstitial alkaline ion, Li^+ or Na^+) concentration as a function of thermal annealing up to 400 °C. It is worth highlighting that, even if McMorris did not explicitly identify this TL peak, by its position in the glow curve and by the bleaching properties under UV illumination discussed in that work, it is reasonable to expect that the peak found at 300 °C corresponds to the SBP. However, contrasting results were reported in the literature. By a TL, EPR, UV-vis and IR investigation, Guzzo et al. [16] proposed that $[\text{GeO}_4/\text{Li}^+]^0$ center is involved in the TL emission of the 220 °C peak and RBP, apparently excluding its contribution in the SBP emission. Besides, by TL, EPR and RL investigation Woda et al. [17] suggested that no direct correlation exist between the $[\text{GeO}_4/\text{M}^+]^0$ center and the SBP. Detailed information on formation and properties of $[\text{GeO}_4/\text{M}^+]^0$ center in quartz, only from EPR without TL and OSL studies, was widely reported in a lot of papers [18–21].

The aim of this work is an extensive experimental investigation on the behaviour under UV and blue illumination of the SBP, the $[\text{GeO}_4/\text{M}^+]^0$ centers and the OSL signal in order to find some possible correlations and to identify the centers involved in the TL and OSL mechanism. The main motivation for this study is related to the importance that these TL, EPR and OSL signals play in dating. Consequently, a better understanding of the physical mechanisms and of the involved centers is of great importance both from the theoretical point of view and for applications.

2. Materials and methods

A natural colourless (hyaline) quartz single crystal was crushed, grinded, and sieved to select grains 100–200 μm in diameter. Different aliquots were used for EPR, TL and OSL measurements. In order to increase the TL and OSL signal all sample were annealed at 800 °C for 10 min (in a pre-heated furnace controlled by a digital system and stabilized within 3 °C) before the irradiation.

TL and OSL measurements were performed with a Risø TL-DA-20 reader equipped with an EMI 9235QB photomultiplier mounting a 7.5 mm Hoya U-340 band pass UV filter (window 280–380 nm). For TL acquisitions, a heating rate of 5 °C/s was used. For OSL measurements,

an array of blue LEDs (470 \pm 30 nm) with a constant power of 54 mW/cm² was used as stimulation source. The acquisition of OSL signals was carried out at 125 °C for 180 s. The EPR spectra were acquired by a Varian spectrometer, operating in the X band with a power of 6.4 mW, modulation frequency of 100 kHz and modulation amplitude of 1 Gauss. A Bruker cavity (ER4102ST model and 3000 Q-factor) was used. We employed the Easyspin Matlab Toolbox to fit the EPR spectra and extract the g-values [22]. A 50 Gy dose was delivered to the samples by Risø ⁹⁰Sr-⁹⁰Y source (dose rate of 120 \pm 15 mGy/s) at room temperature.

Besides, a subsequent pre-heating at 200 °C for 10 s (this temperature was reached by heating the samples from room temperature with 5 °C/s heating rate) was carried out by Risø before the TL, EPR and OSL acquisitions. This additional treatment was necessary in order to erase, from the freshly irradiated samples, both the TL signal of the 110 °C peak and the EPR one of the $[\text{GeO}_4]^-$ center, signals that were not considered in this work. To investigate the bleaching effect under blue and UV illumination, the blue LEDs source of Risø at the power of 54 mW/cm² and a spectroline UV lamp (365 \pm 10 nm) at the power of 1 mW/cm² were used, respectively.

The details of irradiations, treatments, UV and blue illumination used for each TL, EPR and OSL investigation were reported in Table 1.

3. Results and discussion

The effect of UV and blue illumination on TL, EPR and OSL signals is reported in Figs. 1 and 2, respectively. In order to make the data in these figures easier to read, only some selected signals were reported. First, we discuss the properties of TL, EPR and OSL signals before illumination, subsequently we analysed the modifications induced by UV and blue illumination.

As shown in Fig. 1a, the TL glow curve of the sample before illumination (reported in blue) is characterized by two peaks at about 200 °C and 340 °C. As it is known, the position of peaks in TL glow curve depends on the heating rate and on the possible thermal lag depending on the sample holders (steel or aluminum discs or cups, also with different thickness, are typically used with Risø equipment). Consequently, an unambiguous and unanimous peak position assignment, from the sole glow curve, is often difficult to achieve. The temperature used to indicate some of the most known TL peaks (110 °C, 160 °C, 220 °C, 325 °C, 375 °C, etc.) refers typically to the one reported in the

Table 1

Step-by-step sequence of irradiations, treatments, UV and blue illumination used for each TL, EPR and OSL investigation.

| Step | TL Single aliquot | EPR Single aliquot | OSL Multiple aliquot |
|------|---|---|---|
| 1 | Irradiation at 50 Gy | Irradiation at 50 Gy | Irradiation at 50 Gy |
| 2 | Preheat at 200 °C for 10 s | Preheat at 200 °C for 10 s | Preheat at 200 °C for 10 s |
| 3 | TL acquisition up to 500 °C | EPR acquisition | OSL acquisition at 125 °C for 250 s |
| 4 | Irradiation at 50 Gy | UV or blue illumination for time, t_k^b | UV or blue illumination for time, t_k^a |
| 5 | Preheat at 200 °C for 10 s | EPR acquisition | OSL acquisition at 125 °C for 250 s |
| 6 | UV or blue illumination for time, t_k^a | Return to 4 | Return to 1 on a different aliquot |
| 7 | TL acquisition up to 500 °C | | |
| 8 | Return to 1 | | |

Note:.

^a $t_k = 0, 15, 40, 60, 120$ and 180 min.

^b $t_i = 0, 15, 25, 20, 22, 18$ and 30 min, hence the effective time under illumination after each step was: 0, 15, 40, 60, 82, 100 and 130 min.

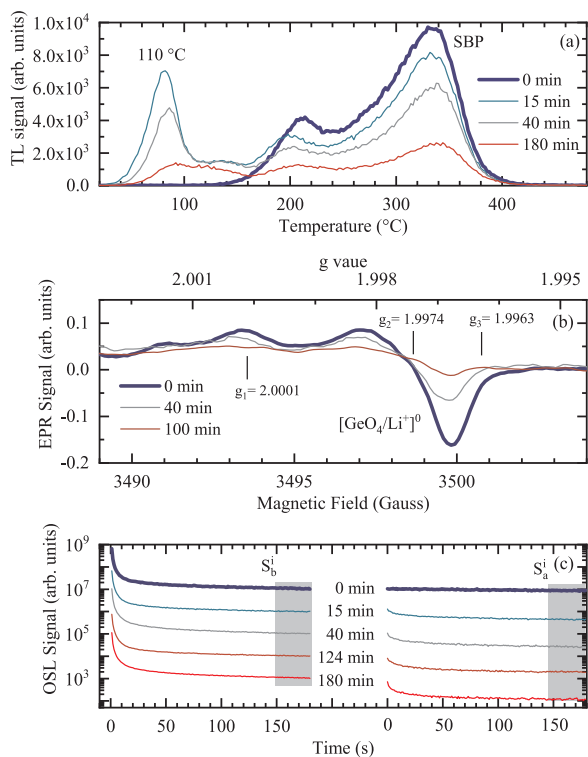


Fig. 1. a) TL glow curves, b) EPR spectra and c) OSL decays (vertically shifted to improve readability) of annealed sample following a UV-bleaching for different time. In TL glow curves the position of the SBP and 110 °C peak, we observe at about 340 °C and 80 °C are indicated in section a). As explained in the text, the two time ranges of OSL decays used to estimate effect before (S_b) and after (S_a) UV illumination on the slow component are highlighted by squares grey in section c). The time of UV illumination is indicated in each figure.

first literature works in which these peaks were observed and/or investigated, mostly using a 20 °C/s heating rate [23]. However some TL peaks of quartz are characterized by different physical properties in terms of bleaching, thermal stability, etc. Based on the optical bleaching properties considered in detail afterwards, we identified the peak that we observe at 340 °C with the SBP discussed in the introduction. Hereafter we focussed our attention only on the SBP.

In Fig. 1b, the EPR spectrum of the sample before UV illumination shows the well known composite signal of $[\text{GeO}_4/\text{M}^+]^0$ center [20]. It is important to note that, differently from the data shown in our previous work on the same quartz [6], the EPR signal due to $[\text{GeO}_4]^-$ center is missing in Fig. 1a as a consequence of the pre-heat at 200 °C for 10 s discussed in section Materials and Methods [6]. As reported in the literature, the signal of $[\text{GeO}_4/\text{M}^+]^0$ center is typically due to the superposition of four contributions: $[\text{GeO}_4/\text{Li}^+]_A^0$, $[\text{GeO}_4/\text{Li}^+]_C^0$, $[\text{GeO}_4/\text{Na}^+]_A^0$ and $[\text{GeO}_4/\text{Na}^+]_C^0$, where the subscript refers to a slightly different symmetry property of the center [4,19,20]. By the fit analysis of the line-shape [6], we estimated that, in our sample, and in agreement with our previous work, the predominant contribution to the $[\text{GeO}_4/\text{M}^+]^0$ EPR signal is due to $[\text{GeO}_4/\text{Li}^+]_C^0$ (about 70–75% of the whole signal). The other contributions affect only marginally the shape and intensity of the $[\text{GeO}_4/\text{M}^+]^0$ signal. For this reason, here we report only the parameters of $[\text{GeO}_4/\text{Li}^+]_C^0$ centers obtained by the fit: $g_1 = 2.0001$, $g_2 = 1.9974$ and $g_3 = 1.9963$. Despite, in the literature, a full agreement for the g values of $[\text{GeO}_4/\text{Li}^+]_C^0$ center is missing ($g_1 = 2.0014$, $g_2 = 1.9983$ and $g_3 = 1.9947$ [20] and $g_1 = 2.0000$, $g_2 = 1.9973$ and $g_3 = 1.9962$ [24]), our data are consistent with those reported by Weil [24]. Hereafter, the $[\text{GeO}_4/\text{Li}^+]_C^0$ center will be indicated, for simplicity, as $[\text{GeO}_4/\text{Li}^+]^0$ center.

The OSL shine down curve of the sample before the UV illumination,

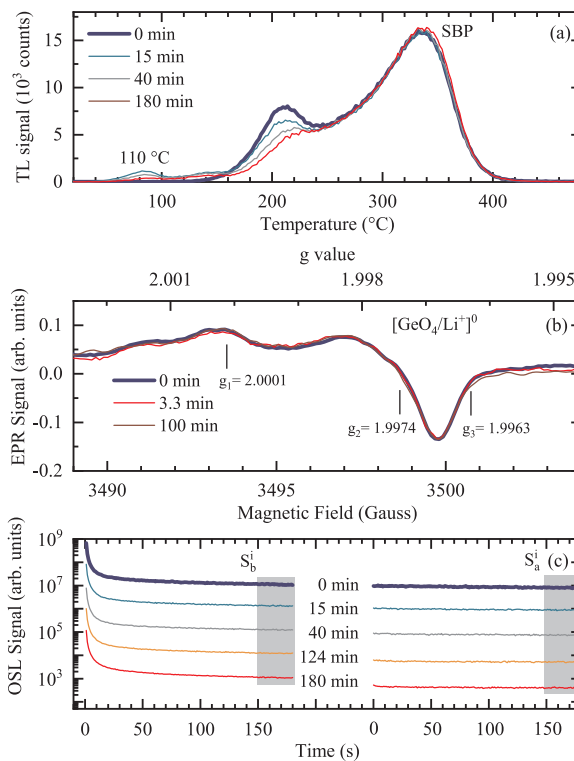


Fig. 2. a) TL glow curves, b) EPR spectra and c) OSL decays of annealed sample following a blue-bleaching for different time. In TL glow curves the position of the SBP and 110 °C peak, we observe at about 340 °C and 80 °C are indicated in section a). As explained in the text, the two time ranges of OSL decays used to estimate effect before and after blue illumination on the slow component are highlighted by squares grey in section c). The time of blue illumination is indicated in each figure.

shown in Fig. 1c and indicated as “0 min”, evidences the typical components of OSL decay: fast, medium and slow. However the intensity of the slow one is extremely high and a complete bleach is not reached during the OSL measurement. For this reason multiple aliquots were employed for OSL measurements whereas a single one for TL and EPR.

The detailed sequence of treatments, irradiations and UV/blue illuminations are reported in Table 1. The different sequence of steps used for each acquisition type (TL, EPR and OSL) is due to the intrinsic difference in each technique. The simplest sequence is that of EPR investigation in which the sample was irradiated only once and then exposed to UV/blue illumination for different times. On the contrary, for TL investigation, the same sample was irradiated and exposed to illumination after every acquisition (this because the heating of the sample during the read-out of TL measurement erases the signal itself). In this case, we previously verified that the SBP intensity was not affected by the repeated sequence of irradiation and heating due to the TL measurements. Finally, as mention before, multiple aliquots were used for OSL acquisition; however, the estimation of changes induced by different illumination times was evaluated by comparison of OSL signal before and after illumination for each aliquot.

As shown in Fig. 1a, UV illumination induces evident changes in the TL spectrum: the SBP gradually decrease on increasing the exposure time. Besides, the well-known 110 °C peak (observed at about 80 °C in our experiments) appears after the first UV exposure for 15 min and its intensity tends to be reduced increasing the exposure time. The findings related to the 110 °C peak are attributed to the phototransferred TL (PTTL) mechanism, in which electrons are transferred from one trap to another [25].

EPR spectra of Fig. 1b show that the signal of $[\text{GeO}_4/\text{Li}^+]^0$ center gradually decreases under UV illumination, without evident

modification in line shape, suggesting that all the center populations originating this EPR signal are bleached in the same way. For OSL measurements and referring to the Fig. 1c, we acquired for each aliquot the OSL decay; subsequently, the aliquot was cooled down to room temperature and exposed to UV illumination. Hence, the OSL decay was acquired again and the shine down curves acquired for different aliquots before and after different UV illumination time were reported in Fig. 1c. Before the illumination, each aliquot was characterized by a comparable OSL signal, whereas a strong dependence on the duration of illumination is evident. Indeed, one can note that the slow component contribution decreases on increasing the illumination time and, concurrently, the fast component becomes more evident.

As regards the effects of blue illumination on TL, EPR and OSL signals reported in Fig. 2, they are markedly different from those due to the UV illumination just discussed above. Indeed, by inspection of TL glow curves of Fig. 2a one can note that the SBP is not affected by the exposure to blue light. On the contrary, data trends of the 110 °C peak is similar to those observed under UV illumination: the 110 °C peak is induced after the first illumination and it decreases gradually on increasing the illumination time. The investigation of the $[\text{GeO}_4/\text{Li}^+]^0$ EPR center reveals that no alterations in terms of intensity and line shape of this signal are detectable (see Fig. 2b). Finally, the OSL measurements suggest that blue illumination only partially modifies the signal. Indeed, the decays in Fig. 2c show that after blue illumination only the slow component is appreciable and its contribution decreases on increasing the illumination time.

In order to verify that the changes were indeed due to the illumination (UV or blue) and not to the elapsed time between the measurements, we repeated the same sequence of TL, EPR and OSL experiments but without illumination. In this case, no significant modifications were observed.

The quantitative analysis and comparison of UV and blue bleaching effects by TL, EPR and OSL measurements shown in Figs. 1 and 2 is summarized in Fig. 3. The contribution of the SBP was estimated by integrating the glow curves over an interval of 20 °C around the peak maximum temperature, whereas the double integral of the EPR signal

was considered for the $[\text{GeO}_4/\text{Li}^+]^0$ center. These values, normalized to those corresponding to the signal detected before UV/blue illumination, are shown in Fig. 3a. As qualitatively anticipated, blue illumination does not modify the signal of the SBP and of the $[\text{GeO}_4/\text{Li}^+]^0$ center up to the exposure time explored (see the dashed-line in Fig. 3a). On the contrary, both TL and EPR signals decrease in the same way as a function of UV illumination time. In particular, to a first approximation, the signal of both SBP and $[\text{GeO}_4/\text{Li}^+]^0$ center can apparently be described as a mono exponential decay (see the continuous-line in Fig. 3a). The same trend observed for the SBP and the $[\text{GeO}_4/\text{Li}^+]^0$ center under UV and blue illumination strongly points out the close correlation between these TL and EPR signals. In particular, our findings suggest that the electron source originating the SBP can be identified as the $[\text{GeO}_4/\text{Li}^+]^0$ center. The different behaviour under UV and blue illumination is even more remarkable if one considers that in our experiments the blue illumination had a power higher by a factor 50 than the UV one (70 when the number of photons is considered).

In order to estimate the effect of UV and blue illumination on OSL signal and to find a possible correlation with the effects discussed above on TL and EPR experiments, we decided to focus our attention only on the slow component of OSL decay. In fact the signal of the SBP and of the $[\text{GeO}_4/\text{Li}^+]^0$ center is not affected by blue illumination; on the contrary, it is well known that the OSL fast component rapidly reduces under the same illumination. This suggests that the fast component is not related to the mechanisms responsible for the changes induced by illumination to the SBP and to the $[\text{GeO}_4/\text{Li}^+]^0$ center. To evaluate quantitatively the effect of UV and blue exposure on the slow OSL component, we calculated the quantity S_b^i/S_a^i by integrating the OSL signal over an interval from 150 to 180 s, before (S_b^i) and after (S_a^i) the exposure (see Figs. 1c and 2c). The superscript “i” indicates the illumination time. As one can observe from Fig. 3b, the exposure to both UV and blue illumination induces a gradual decrease of the slow component of OSL signal, more evident for the UV illumination. However, no evident correlation was found between the slow component and the SBP or $[\text{GeO}_4/\text{Li}^+]^0$ center (see the continuous line in Fig. 3b).

Considering the effect of blue (470 nm) and UV (365 nm) illumination on the $[\text{GeO}_4/\text{Li}^+]^0$ center, it is reasonable to expect that this defect is involved in the so called post-blue violet stimulated luminescence (VSL). This technique was proposed by Jain, 2009 [26] to extend the datable range using traps not accessible by blue light but depleted by 405 nm one. Hernandez and Mercier [27] showed that large part of the post-blue VSL signal can be associated with the temperature region between 370 and 390 °C and suggested that it mainly originates from traps associated with the TL peak at 375 °C in quartz. We can, therefore, suggest that $[\text{GeO}_4/\text{Li}^+]^0$ is one of the electron source for the VSL signal. Such defect, however, is not necessarily the only one involved in VSL. First of all, it is interesting to note that, to our knowledge, $[\text{GeO}_4/\text{Li}^+]^0$ EPR signal has never been detected in natural quartz unless freshly irradiated. The fact that, in our sample, we could easily observe the signal after imparting just 50 Gy, suggests that, either in our quartz the concentration of such defect is unusually high, or that its thermal stability is much less than the one the temperature position of its TL peak would suggest. If this signal would manifest unusual fading, it should be taken into account because it would make the dose response curve in VSL dating unreliable leading to age underestimation. In order to clarify these open questions, further investigation on different quartzes and on the thermal stability of the $[\text{GeO}_4/\text{Li}^+]^0$ EPR signal would be required.

Finally, the fact that the same temperature region of a glow curve is considered for TL dating of fired materials (pottery, ceramic artifacts, etc.) indicate that different defects contribute to the TL signal around 375 °C. Hernandez and Mercier, 2015 [27], in fact, showed that most of the charges contributing to the TL signal in this temperature region are not sensitive to the violet stimulation. However, in cases where the violet bleachable fraction of the glow curve is significant, and no fading

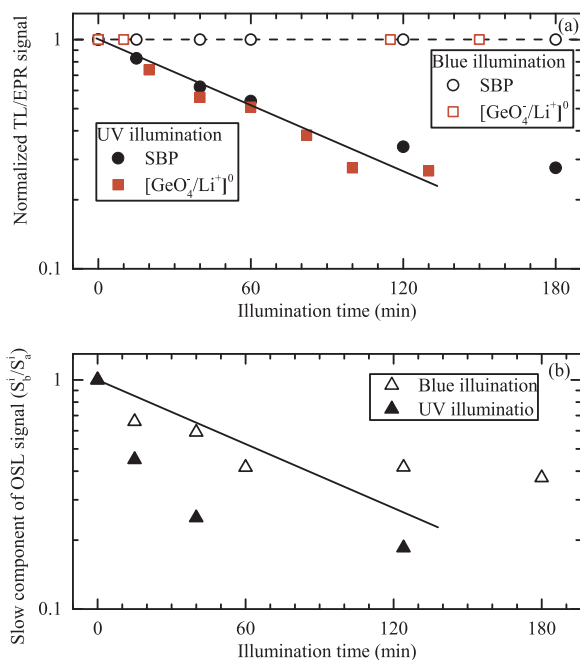


Fig. 3. a) The SBP TL and $[\text{GeO}_4/\text{Li}^+]^0$ EPR center signals as a function of UV and blue illumination time. The continuous-line and dashed-line are reported as guide for the eye. b) Slow component contribution of OSL signal as a function of UV and blue illumination time. In this figure, the same continuous-line shown in Fig. 3a is also reported for comparison.

of the $[\text{GeO}_4/\text{Li}^+]^0$ EPR signal occurs, this effect should be considered because a partial bleaching of the 375 °C peak could affect the reliability also of TL dating where, usually, exposing quartz to light is not considered an issue.

4. Conclusion

To summarize, a clear correlation between the SBP and $[\text{GeO}_4/\text{Li}^+]^0$ center was found by TL and EPR investigation. In particular, we found that the intensity of the SBP and $[\text{GeO}_4/\text{Li}^+]^0$ center decreases in the same way under UV illumination, whereas no modification was induced by blue illumination. These findings strongly suggest that $[\text{GeO}_4/\text{Li}^+]^0$ center plays the role of electron source for the TL signal of SBP.

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