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Investigation on non-optically bleachable components of ESR aluminium signal in quartz

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ABSTRACT

Electron Spin Resonance (ESR) can be used as a method to estimate the age of sediment deposition using the paramagnetic centre related to aluminium impurities in quartz. This so-called Al-centre can be partially optically bleached and its signal intensity decreases in relation to time exposure to solar light, until it reaches a plateau value corresponding to a residual signal. This signal can be attributed to "Deep Aluminium Traps" (DAT) which cannot be reset by an exposure to sunlight. In this study, we have investigated the behaviour of the DAT signal in samples from different origins and ages. The intensity of the DAT signal has been isolated from the total aluminium signal by the exposure of different quartz samples to simulated solar light. We observed that the DAT intensities were sample dependant and therefore it should be determined for each sample. Moreover, DAT intensities of Pleistocene volcanic quartz increase with gamma laboratory irradiation, whereas DAT intensities of sedimentary quartz do not vary with added artificial doses. This suggests that DAT in quartz extracted from sediments must be inherited from the primary source of the quartz, and were saturated at the time of sedimentation. We thereby validate the ESR dating of quartz sediment protocol used so far.

1. Introduction

The Electron Spin Resonance (ESR) dating of quartz is a powerful method with a wide range of applications in geosciences and in archaeology. Quartz is a suitable, and often the only available, material for ESR dating of archaeological sites (Bahain et al., 2002; Despriée et al., 2011), fault movements (Ikeya et al., 1982; Lee and Schwarcz, 1994; Toyoda and Schwarcz, 1996), volcanic events (Imai et al., 1985; Miallier et al., 1994; Toyoda et al., 1995; Asagoe et al., 2011), or sedimentary deposits (Yokoyama et al., 1985; Falguères et al., 1988; Laurent et al., 1998; Voinchet et al., 2004, 2010). When dating quartz, in the majority of cases, the geological or archaeological event is dated and not the quartz growth. In order to date this geological or archaeological event, a zeroing of the quartz geochronometer is necessary, either by pressure, heating or optical bleaching.

Regarding marine, alluvial or aeolian sediments, an optical bleaching by sunlight of the light sensitive paramagnetic centres of quartz occurs during the transportation of the quartz grain by water or by wind, before deposition in the sedimentary sequence (Yokoyama et al., 1985). However, the bleaching mechanism of ESR centres of quartz by the sunlight is complex and is not fully understood. There are several paramagnetic centres in quartz and in this paper, we will focus on the most used, the aluminium-centre, for dating sediments. This Al-ESR centre cannot be totally zeroed when exposed to sunlight, or bleached, and we were interested in the non-optically bleachable component of this centre for quartz from different origins.

1.1. Literature review

Several optical bleaching experiments have been performed on aluminium paramagnetic centre of quartz in the past. "Bleaching curves" were constructed by plotting the ESR intensity of aliquots exposed to simulated solar light against the time of exposure (Walther and Zilles, 1994; Tanaka et al., 1997; Toyoda et al., 2000;

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Voinchet et al., 2003; Tissoux et al., 2007). All of the bleaching curves demonstrated a rapid decrease of the signal intensity during the first hour and then a slowdown of this decrease until the curve reached a plateau value after several hundreds of hours of artificial light exposure. The plateau value is considered as a maximum bleaching value, corresponding to electrons located in traps for which activation energy is higher than photon energy brought by light (Ikeya, 1993). The resulting ESR intensity corresponds to the "residual dose", which must be taken into account for dating (Falguères et al., 1988; Voinchet et al., 2003).

The shapes of ESR Al-centre signals before and after optical bleaching are similar (Fig. 1). They are composed by the same hyperfine structure due to the nuclear spins and the quadrupole splitting related to ^{27}Al with $g1 = 2.0600208$; $g2 = 2.008325$; $g3 = 2.001948$ (Ikeya, 1993). Both Deep Aluminium Traps (DAT) and Optically Bleachable Aluminium Traps (OBAT) should correspond to a substitution of Si^{4+} atom by an Al^{3+} in the crystal lattice of quartz (SiO_4). The Al^{3+} ion being a lower charge cation than Si^{4+}, additional interstitial cations (generally H, Li or Na) tend to be located nearby the aluminium, yielding an over-all neutral charge. Under irradiation, one electron is usually ejected from the Al-centre, which then becomes paramagnetic (Weil, 1984).

However, even if the DAT or OBAT are related to the presence of aluminium in the lattice of quartz, their different behaviours towards bleaching (thermal and optical) suggest that the aluminium-centre should present several states. Indeed, thermal annealing experiments on quartz grains show several kinks in the annealing curve of the ESR aluminium signal before a total zeroing (Toyoda and Ikeya, 1991; Tissoux et al., 2008). It seems to attest to the presence of several rates of decay, related to several "depths" of aluminium traps. In the same way, Toyoda (1992) and Voinchet et al. (2003) have demonstrated that the bleaching curve of the aluminium-centre of quartz can be fitted using a double exponential function, indicating two kinetics of decay for the OBAT.

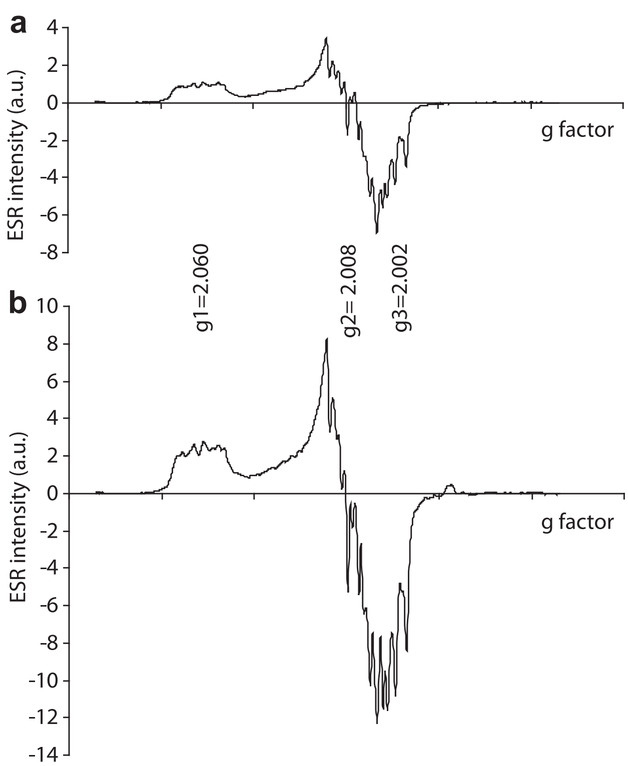

Fig. 1. Hyperfine structure in the bleached (a) and natural (b) quartz due to the nuclear spins and the quadrupole splitting related to ^{27}Al with $g1 = 2.0600208$; $g2 = 2.008325$; $g3 = 2.001948$ (Ikeya, 1993) observed by ESR.

1.2. DAT in sedimentary quartz and its implications in the dating protocol

The equivalent doses (D_E) accumulated by the Al-centres of quartz are commonly determined by the additive dose method using a multiple aliquots approach (Yokoyama et al., 1985). Several aliquots are irradiated at increasing artificial doses and the resultant ESR intensities of the natural and irradiated aliquots are plotted against the irradiation dose. In order to eliminate the intensity related to the DAT, an aliquot of the natural quartz is optically bleached and its residual intensity is subtracted from the ESR intensity of each of the aliquots before constructing the dose response curve. The equivalent dose (D_E) is then determined by extrapolation of the curve to zero (Fig. 2), by using a single saturating exponential function, a double-saturating exponential function or an exponential + linear function (Duval et al., 2009).

A better understanding of the residual dose and of the DAT behaviour towards irradiation is necessary to validate the ESR dating protocol of sedimentary quartz.

Two points in particular need to be checked:

i) Is the residual dose of each quartz distinctive? If the number of DAT is identical for all alpha-quartz measured by ESR, it should mean that the DAT concentration is inherent to the crystalline nature of the quartz. On the contrary, if different DAT concentrations are determined for each source of quartz, it should imply that these concentrations depend on the crystallisation conditions of quartz, notably temperature, composition of magma and pressure. In such a case, the residual dose has then to be determined for each quartz sampled for age calculation.
ii) What is the behaviour of DAT towards irradiation? Deep Aluminium Traps become paramagnetic when they are irradiated (Weil, 1984). Do both the Deep Aluminium Traps and

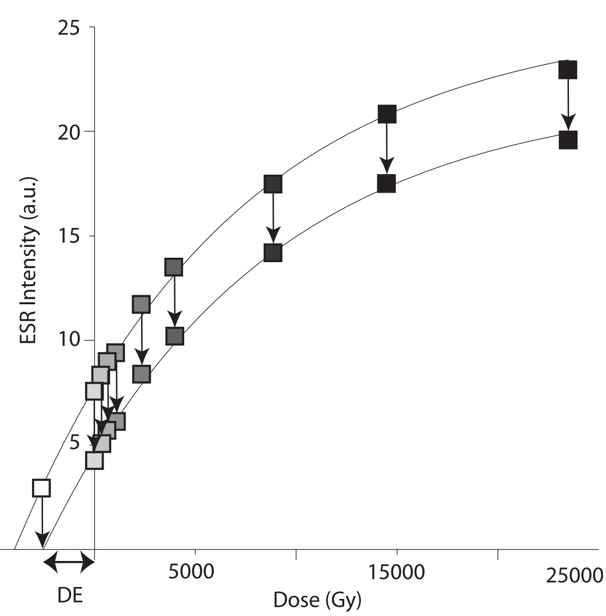

Fig. 2. Growth curves of aluminium-centre of quartz. The ESR intensity of the bleached aliquot (white square) is subtracted from the natural and irradiated aliquots (grey squares) and a new curve is built. The equivalent dose (D_E) of the OBAT is determined by extrapolation of this curve to zero.

Optically Bleachable Aluminium Traps concentrations increase with irradiation? In terms of dating, if the number of DAT increases similarly to the OBAT with irradiation, it must be taken into account when calculating ages.

In order to answer these questions, we have sampled quartz from various ages and environments. Sample dependence was studied on alluvial quartz from different fluvial systems in order to check the influence of the quartz origin on the DAT signal. Behaviour of DAT towards irradiation was then checked on quartz of different natures (sedimentary and volcanic) by a specific protocol including irradiation, ESR measurement, optical bleaching and new ESR measurement for all the aliquots of each sample.

2. Materials and methods

2.1. Sampling

Sample dependence experiments were performed on seven alluvial sedimentary quartz from the river Cher, France (4 samples), the river Meuse, France (1 sample) and the river Neckar, Germany (2 samples).

Dose response of DAT to irradiation was tested on six samples. Four are alluvial sedimentary quartz and two are volcanic tephra. Sampling was done in fossil alluvial deposits of the river Arlanzon (Spain), of the river Cher and river Sauldre (France) and from the river Neckar (Germany). The two volcanic samples are the Nishigo pyroclastic ignimbrite from Aizu Caldera and the Kanayama tephra from Numazawa Caldera (Japan), dated to 50 ka and 1.1 Ma respectively. These ESR experiments on volcanic quartz intend to provide information relating to the DAT and OBAT behaviour of recently crystallized minerals prior to any optical exposure or resetting.

A wide geographical distribution of sedimentary quartz was used, to avoid any local specificity and to guarantee the variability of the quartz sources (Fig. 3). Ages of all the deposits are known or estimated; they spread from about 50 ka to 1.3 Ma.

2.2. Sample preparation and analysis

For each sample, a 100–200 μm fraction of pure quartz was obtained using the chemical and physical protocol detailed in Yokoyama et al. (1985) and refined by Voinchet et al. (2003). Ten aliquots of each sample were irradiated with increasing doses from 260 to 25,000 Gy using a panoramic ^{60}Co gamma ray source providing a dose rate of 120 Gy h^{-1} (Dolo et al., 1996). One aliquot was kept as it is and called “natural”.

Natural and irradiated aliquots of each sample were then measured by ESR. Measurements were performed at 100 K with a Bruker EMX spectrometer using the experimental conditions proposed by Voinchet et al. (2004). Each aliquot was measured three times, and the tube was rotated 60° between measurements. The ESR intensity was determined using the height of the signal between the top of the first peak at $g = 2.018$ and the bottom of the 16th peak at $g = 2.002$ of the Al hyperfine structure (Toyoda and Falguères, 2003). Finally, an additive dose curve was built.

All the aliquots were then exposed to simulated sunlight of more than 1500 h using a Dr. Hönle SOL 2 solar simulator until the maximal optical bleaching of the OBAT was obtained (Voinchet et al., 2003).

The sample dependence of the quartz DAT was tested by measuring the residual dose of non-irradiated quartz after maximal optical bleaching. The absolute intensity of the residual dose for each sample was then determined through measurements in normalized conditions (identical weights and experimental conditions, calibrated tubes…).

The behaviour of the DAT towards irradiation was observed by comparison of the growth curve obtained on the natural and irradiated aliquots with the dose response curve constructed after maximal bleaching of the same aliquots.

3. Result and discussion

3.1. Sample dependence of alluvial quartz

The absolute intensity of the seven measured bleached samples shows a great variability, from 500 to 1200 in arbitrary normalized units (Fig. 4). It indicates that the residual dose in some samples can be up to two times higher than in other, for the same amount of quartz. Subsequently, the residual dose, and then the DAT concentration is sample dependant.

Conversely, three quartz deposited by the river Cher but extracted from two different fossil alluvial sheets from different areas and of different ages, give similar residual dose (Fig. 4). These

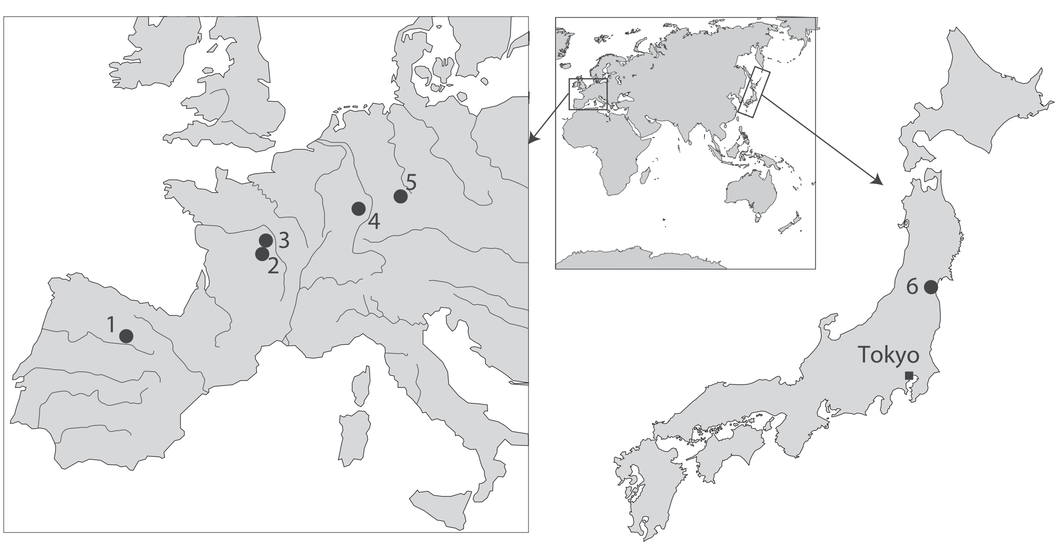

Fig. 3. Location map of the alluvial sediments (Europe) and volcanic tephras (Japan) sampled for this study. 1-river Arlanzon; 2-river Cher; 3-river Sauldre; 4-river Meuse; 5-river Neckar; 6-Sendai area.

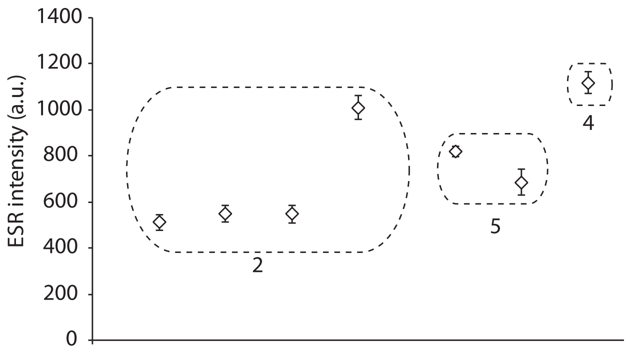

Fig. 4. Sample dependence of the DAT ESR intensity. 2-river Cher; 4-river Meuse; 5-river Neckar.

three quartz originate from a single source in the Massif Central Mountains and this could suggest that the DAT concentration in sedimentary quartz relies upon the specific geological source of the quartz. This assumption should be corroborated by measurement of several quartz samples extracted from the same magmatic or volcanic rocks and optically bleached. In the same river valley, a fourth quartz sample gave a very different residual dose from the three others. It is obvious that rivers cross various types of rocks on their pathway and then can carry different kinds of alluvia, inducing some mixing of quartz grains from various origins in the sedimentary deposits. Moreover, the origins of the quartz present in the alluvia deposited in terraces of different ages by the same river can be miscellaneous as the river can cut different stratigraphic layers of bedrock with time. Consequently, the sample dependence demonstrated that it is essential to determine the residual dose for each sedimentary quartz that has to be dated.

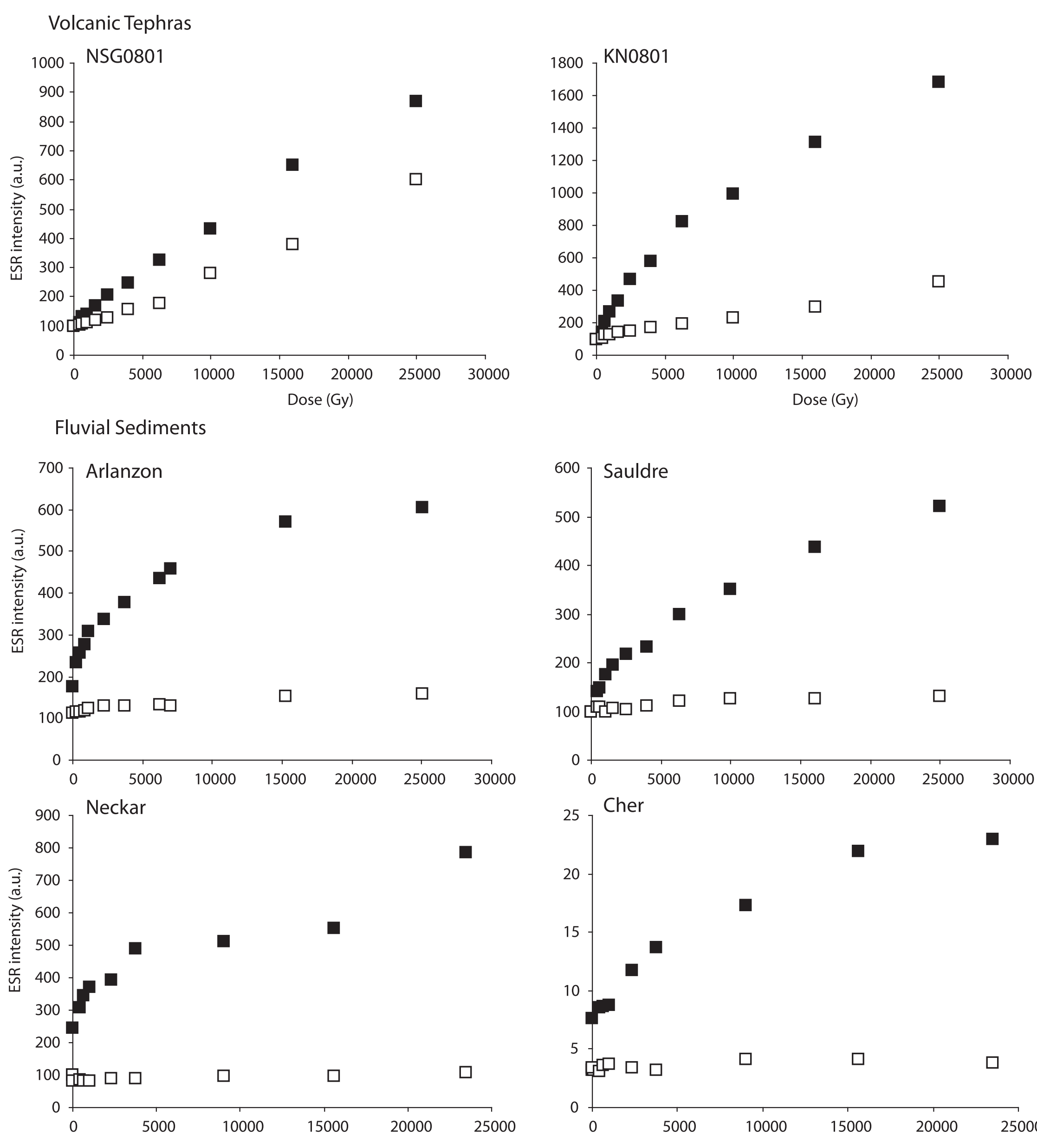

Fig. 5. Growth curves of aluminium-centre in quartz extracted from volcanic tephras and from sedimentary alluvia (full squares) and ESR intensity of the residual dose, related to the DAT of quartz, after more than 1500 h of optical bleaching of each natural and irradiated aliquots (open squares) against the irradiation dose.

3.2. Behaviour of DAT towards irradiation

Dose response curves for fluvial and volcanic samples before and after optical bleaching are presented in Fig. 5. The growth curve of non-bleached aliquots shows the dose response of Al-centres (OBAT + DAT) towards irradiation, whereas the bleached aliquots curve shows the behaviour towards irradiation of DAT only. This experiment allows to distinguish clearly two groups of samples according to their geological natures: one corresponds to volcanic tephra and the other to alluvia.

The volcanic quartz samples show an increase of the (OBAT + DAT) ESR intensities with irradiation that can be fitted by an exponential function + linear term for NSG0801 and single saturating exponential function for KN0801. For the two tephras, the dose response curves constructed after a maximal optical bleaching show an increase. These DAT dose response curves can be fitted with a similar function to the (DAT + OBAT) ones. It suggests that the Deep Aluminium Traps and the Optically Bleachable Aluminium Traps ESR intensities increase similarly with irradiation in the volcanic tephra. In such volcanic quartz, the significant increase of the DAT dose with irradiation indicates that these radiosensitive traps are not yet saturated.

The ESR Al-signals of sedimentary quartz also show a systematic increase of the (OBAT + DAT) intensities with irradiation. The mathematical function allowing the fitting of the experimental data sets changes depending on the sample, reflecting the variability of quartz in our panel. In the case of the Neckar River sample, the fitting was impossible, probably due to the presence of unbleached grains from the bedrock in the samples. On the contrary, the DAT number doesn't increase significantly with irradiation in any sample of sedimentary quartz whatever the bedrock, the age of deposition, the river location, the mixture of grain etc. The most likely hypothesis is that all the DAT in sedimentary quartz are presently full in the natural samples, implying a saturation of the ESR signal. Indeed, DAT cannot be reset by exposure to solar light, only by a heating at sufficient temperature. Consequently, the DAT accumulate in the crystal lattice of the sedimentary quartz grains either from the crystallisation of the quartz or from the quartz's last exposure to heat. As our quartz samples shouldn't have been exposed to heat (such as emitted from a volcanic event) during their history, we conclude that the DAT accumulate in the crystal lattice from the quartz formation, and the river systems from which our samples come from carry alluvia eroded from old European geological blocks: Proterozoic to Hercynien bedrock of the Iberian cordillera for the river Arlanzon, Triassic sandstones for the river Neckar, Triassic bedrock of the northern part of Massif Central for the river Cher, and alteration of Cretaceous deposits for the river Sauldre. The DAT ESR signal of all these quartz was probably already saturated before the erosion phase, and so at the time of deposition in the sedimentary sequence (Fig. 6).

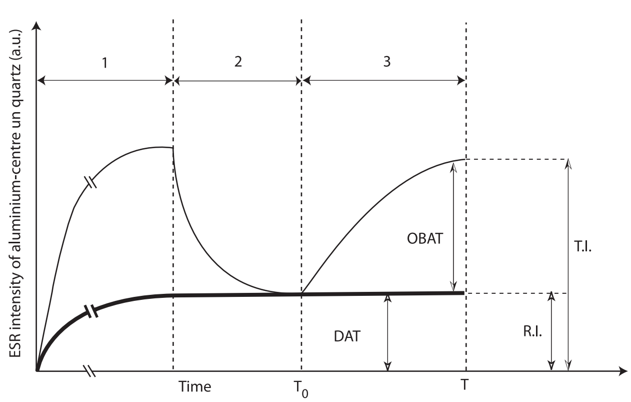

Fig. 6. Bleaching history of the Optically Bleachable Aluminium Traps (OBAT) and of the Deep Aluminium Traps (DAT) of quartz grains. 1: Geological period; 2: Zeroing, Optical bleaching; 3: Archaeological period. RI: residual intensity; TI: total intensity.

4. Conclusion

The residual dose related to aluminium paramagnetic centres observed in quartz by ESR after a maximal optical bleaching is generated by the presence of Deep Aluminium Traps in the crystal, which cannot be reset by solar light. This residual dose, which depends on the concentration of DAT, seems to be specific to the bedrock from which quartz originates. It must be determined for all the sedimentary quartz when ESR geochronology studies are performed.

The paramagnetic DAT are created by irradiation of aluminium diamagnetic defects in the quartz lattice. Their number increases with irradiation, until all the aluminium defects have become paramagnetic. In concrete terms, the DAT intensities increase with irradiation in young quartz from volcanic tephra but not in quartz extracted from fluvial sediment. We came to the conclusion that the DAT in sedimentary quartz must be inherited from the primary source of very old quartz and were saturated at the time of source rock erosion.

In the current dating protocol, the residual dose of aluminium-centre in quartz is determined by bleaching the natural aliquot and then this residual dose is subtracted from the ESR intensity of each irradiated aliquot. In this study, we demonstrated that the DAT does not increase with irradiation when the dated sediments were produced by the erosion of very old parent-rocks, as found in Central France. Consequently, the residual dose of the irradiated aliquots being equal to the residual dose of the natural aliquot thus validates this dating protocol. Nevertheless, our results on recently formed quartz lead us to recommend a thorough investigation of the origin of the sediment. The question should be asked systematically whether the DAT in sedimentary quartz is saturated or not and, when in doubt, an irradiated aliquot should be checked. In case of non-saturated DAT, the DAT intensity for each aliquot should be determined and subtracted from the intensity used for the D_E determination.

Concerning volcanic quartz, the formation of quartz is dated. The bleaching step does not exist and considering a similar behaviour of DAT and OBAT towards irradiation, we can assume that saturation or not of DAT at present does not change the D_E calculation.

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