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PALAEODOSE UNDERESTIMATION OF HEATED QUARTZ IN RED-TL DATING OF VOLCANIC CONTEXTS

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Abstract: Thermoluminescence (TL) dating is a valuable tool for chronometric dating of heated minerals and has been shown to agree very well with independent age control. Comparison with argon dating of samples from identical events, however, revealed age underestimations of volcanic eruptions dated by orange-red TL (R-TL) of quartz extracts from some xenolith samples, while good agreement was obtained for others. The underestimation is attributed to an apparent signal loss ("anomalous fading") which was experimentally observed for some, but not all samples investigated. The presence of significant amounts of feldspar or tridymite, which could be related to the observations, is excluded by IRSL (Infrared Stimulated Luminescence) and XRD analysis. While the data is not entirely conclusive, it leads to the current working hypothesis that exposure to high temperatures might be responsible for an effect similar to the anomalous fading phenomena observed for some feldspar luminescence. It therefore appears to be prudent not to sample xenoliths from high temperature context, like basalt dykes in volcanic context.

Keywords: thermoluminescence dating, red TL, volcanism, xenolith, quartz, fading.

1. INTRODUCTION

The dating of Upper and Middle Pleistocene volcanic events is problematic if volcanic K-feldspars (sanidines) for argon dating are absent. Therefore TL dating of heated detritical quartz extracted from crustal xenoliths became the main focus of a project on establishing ages for the Quaternary volcanism of the Eifel region in Germany. Based on previous research (e.g. Hashimoto *et al.*, 1987; Millaier *et al.*, 1991; Fattahi and Stokes 2000, 2003) and excellent results obtained by Zöller *et al.* (2014) after Richter and Krbetschek (2006) for other heated materials, the orange-red 620 nm emission (R-TL) of heated quartz was employed for dating. R-TL is especially useful for Middle Pleistocene application because of its high saturation dose and good agreement of dating results with independent age control was found (e.g. Fattahi and Stokes 2003). The luminescence of unheated quartz does, in general, not appear to be affected by anomalous fading (e.g. Lai and Fan, 2014). This was recently confirmed for the orange-red as well as the UV-blue TL signal from fine grain quartz, which was extracted from heated sediment (Zöller *et al.*, 2014). Quartz of volcanic origin, however, may display anomalous fading (e.g. Bonde *et al.*, 2001; Tsukamoto *et al.*, 2007), while no evidence for fading of quartz from xenoliths was reported (Miallier *et al.*, 1991). The quartz in this study is extracted from crustal xenoliths composed of Lower Devonian slates and quartzites from the Eifel area, Rhenish Slate Mountains, and therefore not of volcanic origin. Anomalous fading is thus not supposed to occur. Instead, meaningful eruption

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ages based on quartz luminescence are expected because the natural doses, as estimated from the dose-rates and the supposed ages of the eruptions, should be far below the saturation dose in all cases. For verification of the TL dating procedures the project also employs Ar/Ar dating on material from the identical eruption phases.

While age results will be published in detail elsewhere, we provide information on locations, rock type and context in **Table 1**. The comparison of apparent R-TL ages with Ar/Ar ages revealed good agreement for some samples, but significant TL age underestimates for others, in particular for xenoliths BT-1140 and BT-1143/44. Especially for sample BT-1140 the previous (van den Bogaard, 1990) and the new argon dating results agree and are in line with stratigraphic as well as palaeobotanical evidence. These argon ages therefore cannot be disputed. The R-TL underestimation thus indicates either an instability of the TL signals (thermal fading), the presence of non-quartz minerals in the samples or anomalous fading (athermal fading). The present contribution focusses especially on the observation of unexpected apparent signal loss ("anomalous fading") in some, but not all, quartz samples studied in this project.

2. MATERIALS AND METHODS

Heated crustal xenoliths from several eruption centers in the Quaternary West Eifel (WEVF) and the East Eifel Volcanic Field (EEVF) were collected for luminescence analysis and samples for LA-Ar/Ar dating were obtained from the identical volcanostratigraphic layers. Samples analyzed here originate, amongst others, from the scoria cones "Wannen" and "Bausenberg" in the EEVF and the "Kirchweiler" and "Kalem" volcanoes in the WEVF. The homogeneity of the gamma radiation field was checked wherever possible with a "RadEye PRD" detector (small NaJ detector; Thermo Scientific) in the field. For external γ-dose-rate determination the volcanic rock material surrounding each xenolith was sampled for HpGe-γ-spectrometry in the laboratory. Internal dose-rates of the xenoliths were obtained from thick source alpha counting (U, Th) and ICP-MS (K).

Sample preparation for TL measurements was executed under subdued red laboratory light. The outer 2 mm of the xenoliths ware removed with a low speed water cooled saw in order to simplify dosimetry by removing the volume exposed to external α/β-radiation and the

Table 1. *Data and information on the samples mentioned in the text. (* polymineral MAAD data detected in blue wavelength; + alpha sensitivity or radionuclide content not measured because D_E already shows massive underestimation).*

Sample	Locality	Xenolith/rock type	Agreement with Ar-age within 2-σ uncertainty	Volcanic context, estimated temperature regime	Relative heating	max. R-TL signal loss observed (% prompt R-TL)	R-TL main peak temperature (°C)
BT-1137	Wannen	clay	yes (heating event dated by TL may be slightly younger)	late volcanic dyke fill	moderate	12	320
BT-1138	Wannen	slate	dito	in scoria	moderate to hot	7	320
BT-1140	Bausenberg	slate	no	in scoria	hot to very hot	18	350
BT-1141	Wartgesberg	slate	n.a. (but similar to ^{14}C data for post-/end-volcanism)	at edge of a volcanic bomb	moderate to hot	14	330
BT-1184*	Wartgesberg	slate	n.a. (but similar to ^{14}C data for post-/end-volcanism)	massive quartzitic slate; contact heated by lava flow	moderate to hot	10	320
BT-1143	Kalem	slate/siltstone	no	contact of basalt and scoria	very hot	n.a.	360
BT-1144	Kalem	slate/siltstone	no	in scoria with close proximity to basalt flow	very hot	12	340
BT-1148	Kirchweiler-Stolz	Slate/siltstone	no+	contact of basalt and scoria	very hot	4	350
BT-1149	Kirchweiler-Stolz	sandstone	no +	within basalt	extremely hot	15	350
BT-1151	Mendig	quartz	no +	within basalt	extremely hot	20	370
non-volcanic context	Manderscheid	quarzitic slate	n.a.	n.a.	n.a.	<0.2	330

remaining material was crushed carefully. The fine grained host rock of the xenoliths prevented the extraction of coarse grain quartz and therefore fine grain quartz separates (Fuchs *et al.*, 2005) had to be used instead.

TL measurements were executed at 5 K^{-1} up to 425/450°C under N_2. Some data was collected using a lexsyg luminescence reader (Richter *et al.*, 2012; 2013) equipped with a Hamamatsu H7421-40 PMT. This detection unit has an enhanced sensitivity in the red wavelength range compared to standard bi-alkaline PMTs and luminescence detection was restricted to ~590–650 nm by a combination of a Schott KG3 with an AHF ET620/60 filter. Some series of TL measurements involving long storage times were performed on a Daybreak 1150 TL-reader, equipped with a bi-alkaline PMT (EMI9635 B) and restricted detection wavelength to the orange-red by Chroma D620/40 and Corning HA03 filters. Excellent agreement in peak temperatures was observed between the two luminescence systems for identical samples at the identical heating rates. The lexsyg system has a much better signal to background ratio (**Fig. 1**) compared to the bi-alkaline PMT of the Daybreak or an IR-sensitive PM-tube. Reproducibility of the background (mainly heat radiation) was found to be excellent and not affecting results. This allows for a single background measurement for each aliquot at the end of the Single Aliquot Regeneration (SAR) sequence, which reduces the number of heating cycles and thus minimizes sensitivity changes throughout the SAR protocol. Glow curve peak temperatures were observed between 320 and 380°C for the relevant temperature range >300°C, with peaks occurring at temperatures comparable to other studies (Fattahi and Stokes, 2003).

Given the large doses required to regenerate luminescence signals, the full SAR protocol (Murray and Wintle, 2000) was compared to a 'short' SAR protocol (Richter and Krbetschek, 2006) which employs fewer dose points. Likewise other studies (e.g. Ganzawa *et al.*, 2005), the observed sensitivity changes of R-TL were always smaller than the generally accepted 10% threshold and R-TL growth curves do not show large deviations from linear growth for most samples (data to be published elsewhere). D_E-values obtained for standard and 'short' SAR protocols are identical (at 1σ) with similar precision and dose recovery tests were also equally satisfying (<10% deviation from unity). Subsequently the 'short' SAR protocol was employed for age determination because of constraints in machine time available.

3. POTENTIAL CAUSES OF DOSE UNDERESTIMATION IN QUARTZ LUMINESCENCE DATING

The obtained dose underestimations indicate either towards the instability of the TL signal, the presence of non-quartz minerals in the samples or anomalous fading.

Because only TL signals above 300°C are considered here, instability of the TL signal does not appear to be a reasonable explanation for the Middle to Late Pleistocene time range under consideration. Temperatures reached in volcanic eruptions are high, certainly beyond the α- to β-quartz phase transition at 573°C, which is quick, reversible and consists of minor displacements of atoms (Kleber *et al.*, 1998). But higher temperatures can be reached where the second phase transition to the quartz polymorph phase of tridymite occurs (in the range beyond

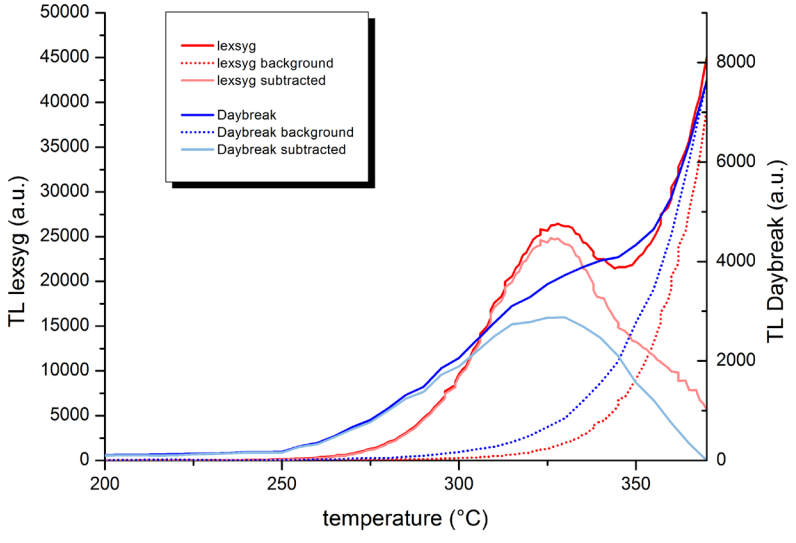

Fig. 1. *Natural TL glow curves for 620 nm detection window (see text) for lexsyg (red) and Daybreak (blue) luminescence reader of sample BT-1137. The raw NTL data is given as solid lines, the background as short dotted line and the subtracted data as faded solid lines. The data was scaled to the approximate same maximum background level. Note that the detection windows are not fully identical. (Readers are referred to the online publication for color coding).*

900°C), which is a slow but substantial process, involving the entire restructuration of the crystal lattice. As tridymite is known to occur in some Eifel volcanoes we unavailingly tested five HF etched and unetched xenolith samples (**Fig. 2**) showing significant TL age underestimation for the occurrence of tridymite by x-ray diffraction (XRD) with a traceability between 1 and 2%. XRD spectra show exclusively quartz components and we failed to detect tridymite, which should be present in xenoliths at large quantities if conditions would have been right for its formation and considering the significant TL-age underestimation.

Anomalous fading is well known to occur in feldspar minerals (e.g. Wintle, 1973; Spooner, 1994; Huntley and Lamothe, 2001). Therefore the quartz separates were checked by IRSL measurements for the presence of larger amounts of feldspar (Mauz and Lang, 2004). Quantities of non-quartz minerals below the detection limits of IRSL and XRD appear to be too low in order to dominate the R-TL results. We therefore also preclude other contamination as a likely reason for the age underestimates.

Consequently, the possibility of "anomalous fading" of quartz in some of our samples has to be considered, even though previous R-TL dating of heated quartz in several studies yielded ages consistent with independent methods. For example the R-TL age obtained for heated quartz extracted from baked loess in a Palaeolithic hearth agrees well with magnetic dating and calibrated radiocarbon ages (Zöller *et al.* 2014). Results of our experiments in the apparent signal loss are presented and discussed.

4. EXPERIMENTAL DESIGN AND RESULTS OF SIGNAL LOSS STUDIES

Because of the large doses required for fading tests and restrictions in machine time a multiple aliquot fading test was employed. The natural TL (NTL) of the quartz fine grain extracts was read out and the discs were subsequently irradiated by an external β-source with a dose roughly equivalent to the obtained D_E. After storage/transfer times between 5 minutes and 180 days the regenerated TL and background was read out. For each

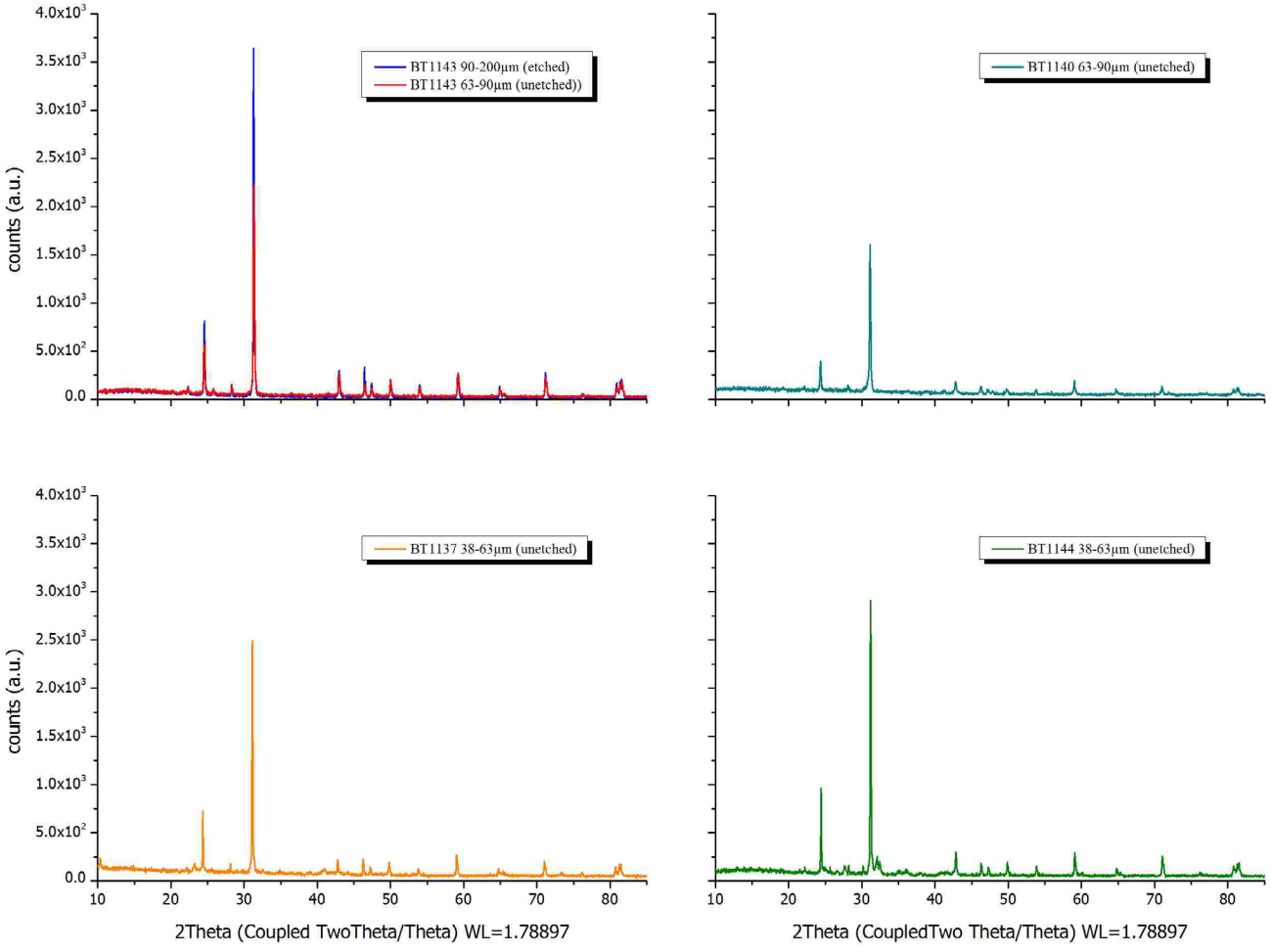

Fig. 2. *Results of XRD analysis of four different unetched fine to medium grain size extracts, together with etched material for one of these samples (BT-1143).*

storage time 4–6 aliquots were measured and mean TL intensities of a 40°C temperature integral around the peak temperature were used for R-TL-signal calculation. All aliquots were normalized against their natural TL and signal loss is expressed as the ratio to the prompt (5 minutes transfer time kept constant) R-TL (**Fig. 3**). An additional experiment investigated the effect of storage temperature, but no differences for one week storage between room temperature and 70°C was observed for two samples.

Most samples investigated (**Table 1**) exhibit some R-TL-signal loss (**Fig. 3**). However, maximum signal loss is 20% after 13 days and most samples exhibit no further or only minor signal reduction after the initial first day. In fact, only four out of eight samples show a clear negative slope of the linear fit, indicating clear fading/signal loss with time (we refrain from providing g-values due to the large doses beyond the linear dose range involved), while almost the entire signal loss occurs within the first day for the others. The losses in signal do not appear to be identical between samples despite the apparent geological similarity of the material. The luminescence behavior of the samples from such a similar geological origin is assumed to be very similar, *a priori* at least for the slates, and any differences observed may be due to changes caused by volcanic activities, especially by different temperature histories. To test this, material from the unheated Lower Devonian sandy siltstone bedrock was treated identical to the samples for dating and heated under air in the laboratory for temperatures below and beyond the quartz phase transition temperatures (2 hours at 500, 700 and 950°C followed by slow overnight cooling). The 130 Gy regenerated TL show peaks at about 130, 155, 210, 290, 330 and 370°C. For peak temperatures of relevance for dating an obvious sensitization of the 330°C peak when heating to 700°C or more can be observed (**Fig. 4**). Comparison of this prompt regenerated TL with the signal measured after 2 days of storage at room temperature revealed no signal loss at all for a second set of samples, and no short term fading appears to be present.

Signal loss after irradiation can be attributed to a number of phenomena (e.g. Aitken, 1985; Zöller, 1995) and relating the empirical observation to a particular phenomenon is not always straightforward. In the case of anomalous fading in feldspar it is explained as a result of tunneling recombination between electron-hole pairs (Aitken, 1985; Visocekas *et al.*, 1994). Low temperature measurements to gain detailed insight into phosphorescence processes of our samples were not possible. Nevertheless, phosphorescence was observed for samples BT-1140 and BT-1144, which show minor sensitivity changes with regeneration cycle during SAR procedures and thus allow direct comparison of two identical regenerations without having to normalize the data. After reading the natural TL, the samples were irradiated, TL and background immediately measured, before the same dose was applied again and the phosphorescence observed over a period of 5 hours before the TL was read out again. For sample BT-1140 two different preheats (230°C and 260°C for 30 s) were employed after irradiation, while none was required for BT-1144. The measured phosphorescence signal was close to machine background after 5 hours and negligible phosphorescence of an identically irradiated empty sample carrier was found. For sample BT-1140 the relative signal loss of the R-TL-peak between prompt measurement and after 5 hours delay was identical for non- as well as preheated material to PH 230°C/30 s, while somewhat larger for PH 260°C/30 s. For both samples the proportional R-TL signal reduction of the 330°C TL-peak between the two regenerations after this time delay of 5 hours is similar to the signal loss

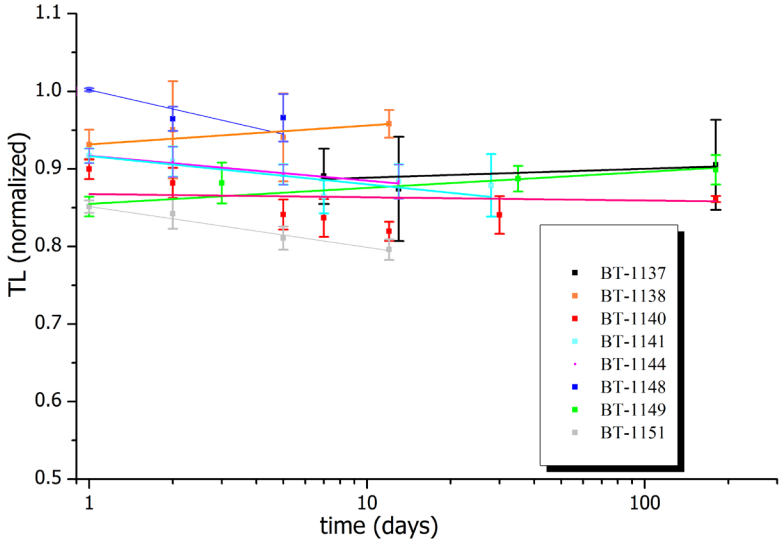

Fig. 3. *Results of TL signal loss (fading) experiment for different samples and storage times. Data is normalized to the natural TL and a linear fit was applied.*

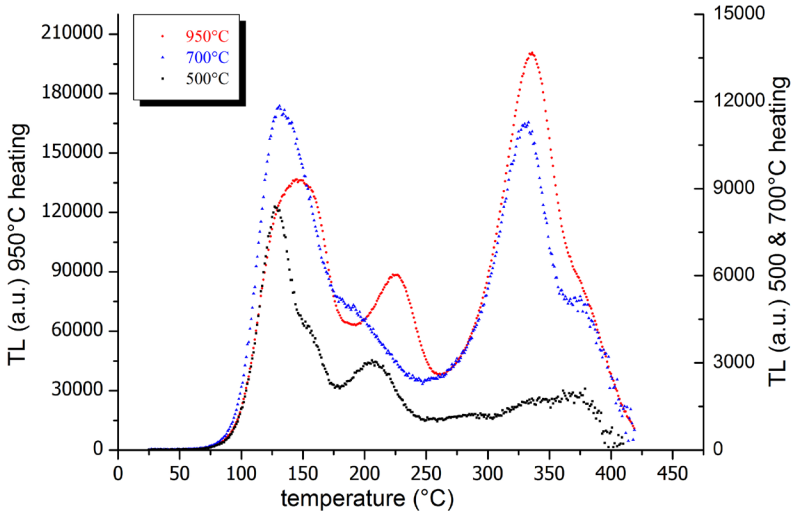

Fig. 4. *Regenerated (130 Gy) R-TL glow curves (background subtracted) from fine grain quartz of crushed and chemically treated Lower Devonian material (geological source from non-volcanic context) for heating in air at 500, 700 and 950°C for 2 hours (no peak-alignment). Note the different scale for the sample heated to 950°C.*

observed in the fading experiment after one day. This indicates that some of the signal loss of our samples after regenerating the signal, which is assumed to be the cause of the SAR age underestimations, occurs rather quickly after irradiation. To test this we repeated a SAR measurement (3 aliquots) using identical parameters except a delay of 3 days after each regeneration irradiation for samples BT-1140 and BT-1144 which are both showing age underestimations of >50%, thus presumably being affected by fading. In both cases the measured D_E increased, but only by 13 and 4%, respectively, which is unsufficient to solely explain the underestimation of the doses.

5. DISCUSSION AND CONCLUSIONS

The interpretation of the results from the experiments presented here are not straightforward because too many parameters might be involved, especially as the geological source of the samples does not appear to be uniform and the duration as well as temperature history appear to be different.

The observed palaeodose and thus age underestimations cannot be related exclusively to the phenomena of phosphorescence and indicate towards other problems with these samples, including long term fading which is difficult or impossible to detect under laboratory conditions.

While the dosimetry for such samples is not straightforward, the dependencies on the external dose are reduced due to the presence of an internal dose (between 50 and 90% of the total dose rate). We therefore exclude problems in dosimetry as the reason for the observed severe age underestimations, because external dose-rates would have had to be underestimated by a factor of four in several instances in order to obtain agreement with the Ar-age estimates.

The observed phosphorescence might explain some of the age underestimation for the samples measured, especially for samples with low or no preheating, and point towards the potential problem of extrapolation of short term fading tests to geological times. But phosphorescence certainly cannot account entirely for the severe underestimations observed. Nevertheless, it appears necessary to conduct further experiments with pulse irradiation (Bailey *et al.*, 2005) or employ elevated temperature irradiation, which is easily achieved with lexsyg system.

The heating experiment of a Devonian rock from non-volcanic context shows that not all samples necessarily loose signal and that the source of xenoliths is neither identical, nor uniform for the Eifel Volcanic Fields, as it was assumed originally. This is also indicated by the observed differences in grain sizes of the samples and by the differences in agreement of R-TL-ages with argon dating. However, the experiment reveals a very strong sensitization of the 330°C peak at heating to 700°C or higher and that the occurrence of higher temperature peaks is not necessarily a function of heating temperature. Samples from volcanic contexts with high temperatures (i.e. basalt), however, tend to have the main R-TL-peak at higher temperatures (340–370°C at 5 K s^{-1}) and it is these samples showing age underestimation, while samples with main TL peaks at 320/330°C do not and are originating from volcanic context involving lower temperatures (**Table 1**). No clear correlation between R-TL-peak temperature, volcanic context and fading rate can be established at this stage. However, there appears to be tendencies of increased signal loss with higher peak temperature and a relation might be present with the duration of high temperature exposure, which is in contrast to our laboratory experiment.

The original sampling strategy in this project was focused on obtaining xenoliths which have likely experienced the highest temperature during the volcanic eruption to be dated in order to exploit the enhancement of R-TL in quartz heated beyond the second-phase transition (Schilles *et al.*, 2001) and ensure complete zeroing of the geological signal during the eruption. Agreement of R-TL ages with argon dating is, however, preferentially obtained for large samples from volcanic context expected to be related to lower temperatures, while R-TL ages especially for small xenolith samples from 'hot' context, like basalts, disagree with argon dating. A brick red discoloration of slate xenoliths may be sufficient to indicate zeroing of the TL signal (see Zöller *et al.* (2014) and unpublished heating plateau tests from a 18 cm thick brick red slate xenolith). But such discoloration depends on the availability of iron for oxidization during heating (e.g. Bessey, 1950) whereas other xenoliths may not be discolored in the same way at identical temperature. Despite the data presented not being entirely conclusive, it appears to be more prudent to sample xenoliths from context which are associated with lower temperatures and face the increased possibility of obtaining samples not having been sufficiently heated for TL-dating due to an incomplete zeroing process - "Keep away from too hot!".

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