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Research paper

Insights into TT-OSL signal stability from single-grain analyses of known-age deposits at Atapuerca, Spain

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ABSTRACT

Single-grain thermally transferred optically stimulated luminescence (TT-OSL) dating has recently been applied to a number of the Atapuerca palaeoanthropological sites. As yet, however, there have not been any direct assessments of TT-OSL signal stabilities for specific grains used for dating. In this study, we undertake a series of TT-OSL suitability assessments on known-age samples from Gran Dolina and Sima del Elefante. Our results suggest that the Atapuerca samples contain populations or sub-populations of grains with suitably stable TT-OSL signals for dating over late-Early and Middle Pleistocene timescales. Equivalent dose (D_e) distribution analysis in combination with pulse-annealing assessments provides a useful means of identifying inter-grain and inter-sample differences in TT-OSL signal stabilities. We also show that obtaining D_e values using different preheat conditions may help to identify potentially problematic TT-OSL behaviours. Analyses of multi-grain aliquot TL curves for these samples reveal that 'bulk' TL signal loss experiments may provide limited insights into TT-OSL source trap lifetimes due to averaging effects, the dominance of grain populations that do not produce TT-OSL, and interference from slowly bleaching OSL components. Our results improve the robustness and precision of existing TT-OSL chronologies for units TD6-3 and TE16-TE17 at Gran Dolina and Sima del Elefante, and support the broader suitability of the single-grain TT-OSL approach at the Atapuerca sites.

1. Introduction

The Sierra de Atapuerca karst complex in northern Spain contains an extraordinary suite of palaeoanthropological sites (Fig. S1) and has collectively yielded some of the earliest human fossils in Western Europe, the largest accumulation of Middle Pleistocene hominin remains globally, and the oldest human mitochondrial DNA recovered thus far (e.g., Carbonell et al., 2008; Arsuaga et al., 2014). Establishing high resolution, numerical chronologies on the Atapuerca infill sequences is critical for reliable interpretation of the extensive fossil, lithic and palaeoenvironmental records – which continue to grow with every excavation season. The most significant fossil- and artefact-bearing sedimentary deposits in this endokarst system accumulated during the Early to Middle Pleistocene (e.g., Berger et al., 2008), and therefore lie beyond the datable range of conventional quartz optically stimulated luminescence (OSL). However, a number of recent studies have demonstrated the feasibility of obtaining alternative sediment chronologies at various Atapuerca sites using thermally transferred OSL (TT-OSL) dating of individual quartz grains (Arnold et al., 2013, 2014, in press; Arsuaga et al., 2014; Demuro et al., 2014).

For samples with inherently bright signal intensities, single-grain TT-OSL dating offers several advantages over more widely used multi-grain TT-OSL approaches, namely: a more reliable means of assessing bleaching adequacy and post-depositional history in complex sedimentary settings; the potential to isolate grain populations with more favourable TT-OSL signal/dose-response properties; and the ability to identify problematic sources of intrinsic equivalent dose (D_e) scatter, such as grain-to-grain variations in TT-OSL signal lifetimes. The latter is of particular interest because there has been some discussion about the long-term

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stability of multi-grain TT-OSL signals (e.g., Duller and Wintle, 2012). Laboratory measurements of electron-retention lifetimes for the TT-OSL source trap suggest that significant age underestimations might be expected for Early and Middle Pleistocene samples over typical burial temperature ranges (e.g., Li and Li, 2006; Adamiec et al., 2010; Shen et al., 2011). However, a recent synthesis of published known-age TT-OSL studies does not appear to provide widespread support for the predicted age shortfalls (Arnold et al., in press).

Reconciling potential differences between laboratory-derived lifetime predictions and field-based evidence for TT-OSL reliability remains an important issue. It is unclear, for instance, whether existing TT-OSL lifetime predictions are equally applicable to all samples or whether there are sample-specific differences in TT-OSL source trap lifetimes – as may be the case for the main OSL dating signal (the 'fast' component; cf., Wintle and Murray, 1998; Spooner and Questiaux, 2000). The number of published known-age TT-OSL comparisons >500 ka also remains limited (Arnold et al., in press), and this currently precludes more comprehensive reliability assessments over extended age ranges. Additionally, existing TT-OSL lifetime assessments have been made at the multi-grain scale using thermoluminescence (TL) loss or isothermal decay experiments performed with standard luminescence dating readers. Such experimental approaches may be hindered by a number of complications. In particular, the empirical uncertainties associated with fitted kinetic parameters (trap depth and frequency factor) are typically relatively large (e.g., Brown and Forman, 2012). Furthermore, it is possible that existing lifetime assessments may partly reflect multi-grain averaging effects arising from simultaneously measuring grain populations with different TT-OSL source trap properties. Single-grain TT-OSL dating offers the potential to evaluate, or even circumvent, such averaging effects. Indeed, the overall reliability of known-age single-grain TT-OSL comparisons performed at Atapuerca suggests that at least some of the grain populations in these samples exhibit sufficiently stable TT-OSL signals (Arnold et al., in press). As yet, however, there have not been any direct assessments of TT-OSL signal stability characteristics for specific grains used for single-grain D_e analysis.

The aims of the present study are threefold: (i) To undertake additional suitability assessments of recently published single-grain TT-OSL chronologies at Atapuerca via analysis of TT-OSL signal stability characteristics of dated grain populations. For this purpose, we focus on a sub-set of samples from Arnold et al. (in press) that display different types of D_e scatter and are potentially indicative of inter-sample variations in TT-OSL signal stabilities. (ii) To investigate a means of optimising D_e measurement conditions for the isolation of grain populations with suitable TT-OSL thermal stabilities at Atapuerca. (iii) To gain insight into the representativeness of TT-OSL source trap assessments made at the multi-grain scale using 'bulk' TL-loss experiments.

2. Sample details and experimental procedures

The five samples under consideration here were taken from independently dated late-Early and Middle Pleistocene horizons at the sites of Gran Dolina, Sima del Elefante and Galería (Fig. S1, Table S1). Full details of the sample positions, stratigraphy and associated age control are provided in Arnold et al. (in press). Samples F13 and F14 were taken from unit TD6-3 at Gran Dolina, which has been dated to 780–930 ka using palaeomagnetism (Parés and Pérez-González, 1995), TL dating of unheated sediments (Berger et al., 2008), and combined ESR/U-series dating of fossil teeth (Falguères et al., 1999). Samples ATE10-11 and ATE10-6 were taken from units TE17 and TE16 at Sima del Elefante, and bracket a major, continuous geomagnetic reversal that has been assigned as the Brunhes-Matuyama (B-M) boundary (Parés et al., 2006). Sample ATG10-3 (see Section 6) was collected from unit GIIIb at Galería, which has a combined chronology of $237\pm^{9}_{8}$ ka based on four ESR/U-series ages of herbivore teeth, two TL and infrared stimulated luminescence (IRSL) ages, and an ESR age on a stratigraphically correlated flowstone (Falguères et al., 2013).

Details of the TT-OSL dating procedures are provided in the Supplementary Information and in Arnold et al. (in press). Single-grain D_e estimates were measured using the single-aliquot regenerative dose (SAR) protocol shown in Table S2a, which makes use of a TT-OSL test dose correction. The feasibility of this single-grain approach is aided by the inherent brightness of the quartz TT-OSL signals for these samples (e.g., Fig. S2). Application of the SAR rejection criteria shown in Table S3 resulted in the acceptance of 6–9% of D_e measurements for further analysis. Multi-grain dose-recovery tests performed on paired sets of aliquots (non-dosed aliquots and aliquots given a known laboratory dose prior to measurement) support the general suitability of the SAR protocol used in this study (Table S4); net measured-to-given dose ratios of 1.04 ± 0.09 and 0.98 ± 0.06 were obtained for samples F13 and ATE10-11, respectively. Environmental dose rates were calculated using a combination of *in situ* field gamma-ray spectrometry (Arnold et al., 2012) and low-level beta counting (Bøtter-Jensen and Mejdahl, 1988) (Table S1). Additional high-resolution gamma-ray spectrometry measurements performed on a selection of samples confirm that the dated sediments are in present day secular equilibrium (Table S5).

3. Single-grain TT-OSL D_e characteristics

The single-grain TT-OSL D_e distributions for samples F13, F14, ATE10-11 and ATE10-6 have been presented in Arnold et al. (in press) and are summarised in Figs. 1 and S3. These datasets encompass the full range of D_e characteristics seen for Atapuerca infill deposits and provide insight into potential variations in TT-OSL signal stabilities between/within samples. Sample ATE10-11 displays low overdispersion (≤20% at 2σ) and the majority of D_e estimates are consistent with a single dose population centred on the weighted mean D_e value (Fig. 1a). The central age model (CAM) age for this sample is in agreement with the independent estimate of ≤780 ka (Table S1), suggesting that all of the accepted grains have suitably stable TT-OSL signals. This sample is representative of the vast majority of independently dated Middle Pleistocene samples analysed from Atapuerca and provides empirical support for the general reliability of the single-grain TT-OSL approach at these sites.

The D_e dataset for ATE10-6 (Fig. 1b) displays a higher overdispersion value (32 ± 4%), which was interpreted by Arnold et al. (in press) as being potentially indicative of heterogeneous bleaching prior to deposition. This interpretation is supported by the reliability of the 3-parameter minimum age model (MAM-3) result for this ≥780 ka horizon (Table S1). However, the possibility remains that this sample was exposed to very little (or even no) daylight when it was washed into the cave, and that the MAM-3 D_e estimate has inadvertently isolated grains with low TT-OSL lifetimes instead of genuinely well-bleached grain populations.

Samples F13 and F14 display D_e datasets with higher overdispersion values (~30%) and exhibit two discrete dose populations (Figs. 1c, S3). The finite mixture model (FMM) age for the main (upper) dose component is in good agreement with the independent estimate (780–930 ka) for both samples (Table S1). In contrast, the lower dose FMM components underestimate the expected minimum age of unit TD6 (>780 ka) by ~40–50%. Arnold et al. (in press) speculated that these unreliable low dose components may indicate the presence of grain populations with thermally unstable TT-OSL signals since all other obvious sources of

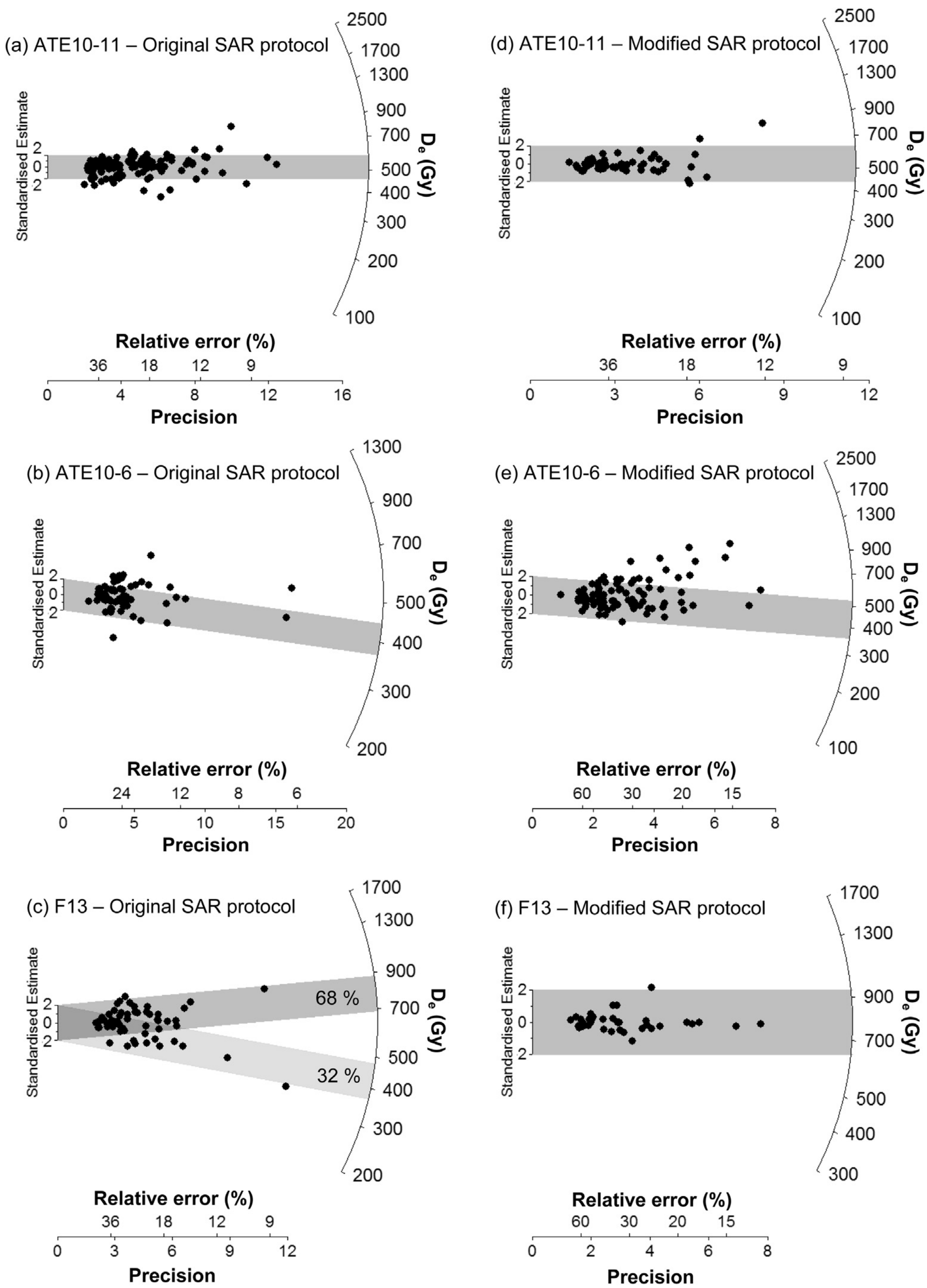

Fig. 1. Single-grain TT-OSL D_e distributions obtained for the Gran Dolina and Sima del Elefante samples using the original (Table S2a) and modified (Table S2b) SAR protocols. (a, d) Sample ATE10-11; (b, e) Sample ATE10-6; (c, f) Sample F13. The grey bands shown in Figures (a), (d) and (f) are centred on the CAM D_e values. The grey bands shown in Figures (b) and (e) are centred on the MAM-3 D_e values. In Figure (c), the weighted mean burial dose estimate of the main FMM component (i.e., that containing the highest proportion of individual D_e values) is shown as a dark grey shaded band. The additional dose component identified by the optimum FMM fit is shown as a light grey shaded band. The percentage of grains associated with each fitted FMM component is also shown.

intrinsic and extrinsic D_e scatter could be reasonably discounted.

4. Single-grain TT-OSL pulse-annealing experiments

To examine whether grain-to-grain variations in TT-OSL source trap lifetimes can explain the additional dose dispersion apparent in some of these samples, we performed single-grain pulse-annealing experiments on samples F13 and ATE10-6 using the same grains that had been measured for D_e analysis (e.g., Fan et al., 2011). In these experiments, the measured grains were left in their original grain-hole positions, bleached with blue light stimulation at 360 °C to remove any remnant TT-OSL signals, and then subjected to repeated cycles of the pulse-annealing protocol shown in Table S2c. The preheat used to induce the TT-OSL signal (PH_2; step 4) was increased in increments of 20 °C (from 180 °C for 10 s to 360 °C for 10 s) between subsequent pulse-annealing cycles to examine possible inter-grain/inter-sample variations in the thermal stability of the TT-OSL signal.

The two grain populations in the F13 D_e dataset exhibit noticeably different TT-OSL pulse-annealing curves (Figs. 2a, S4). Grains from the reliable, high dose FMM component display peak TT-OSL signal production after a PH_2 of 280–300 °C for 10 s (Fig. 2a), consistent with multi-grain aliquot pulse-annealing results presented by Tsukamoto et al. (2008) and Jacobs et al. (2011). In contrast, grains from the unreliable, low dose FMM component display more significant TT-OSL signals following relatively low PH_2 temperatures of 180–250 °C for 10 s (Fig. S4). On average, peak TT-OSL production occurs after a lower PH_2 of 260 °C for 10 s for the unreliable grain population (Fig. 2a). Together, these results suggest that inter-grain differences in TT-OSL signal stability likely account for the discrete dose populations seen in the F13 D_e dataset. Grains yielding unreliably low D_e values appear to exhibit non-trivial TT-OSL contributions from relatively low temperature source traps.

Sample ATE10-6 exhibits only minor grain-to-grain variations in TT-OSL signal stability (Figs. 2b, S5). Grains from the low and high dose tails of the D_e distribution display indistinguishable TT-OSL pulse-annealing curves (Fig. S5). On average, peak TT-OSL production occurs after a PH_2 of 280 °C for 10 s for both groups of grains (Fig. 2b), consistent with trends seen for the reliable, high dose FMM population of sample F13. These results indicate that the additional dose dispersion in the known-age D_e dataset of ATE10-6 likely arises from extrinsic sources (heterogeneous bleaching) and that the MAM-3 has not inadvertently isolated grains with below-average TT-OSL lifetimes.

5. D_e determination using higher preheat temperatures

In light of the complex TT-OSL thermal stabilities characteristics of sample F13, we investigated whether it was possible to isolate (or maximise) TT-OSL contributions from higher temperature source traps by modifying the SAR D_e measurement conditions. For this purpose, a series of single-grain D_e measurements were made on new batches of grains using the modified SAR protocol shown in Table S2b. This protocol uses a PH_2 of 290 °C for 10 s, which is designed to favour TT-OSL production from higher temperature source traps. For consistency, all four preheat treatments (PH_1 to PH_4) were kept the same to mirror the original SAR D_e measurement protocol. Multi-grain dose-recovery tests confirm the suitability of the modified SAR protocol for D_e estimation (Table S4), yielding net measured-to-given dose ratios of 1.02 ± 0.07 for F13 and 1.03 ± 0.10 for ATE10-11. The single-grain rejection criteria statistics remain largely unaltered for each sample when using the original or modified SAR protocol (Table S3), suggesting that similar grain populations are being isolated for D_e analysis in both cases.

The F13 D_e dataset obtained using the modified SAR protocol is characterised by a single FMM component rather than two discrete dose populations (Fig. 1f) and displays little or no overdispersion. The resultant CAM age of 858 ± 74 ka is statistically indistinguishable from that obtained for the main FMM dose component of F13 and F14 using the original SAR protocol, and is in agreement with the independent age estimate of 780–930 ka for layer TD6-3 (Table S1). These results confirm that the scatter in the original D_e dataset arises from intrinsic sources and that higher preheat temperatures are seemingly effective at isolating reliable TT-OSL signals for all accepted grains in this sample.

The use of a modified SAR protocol does not significantly alter the D_e characteristics (Fig. 1d–e) or resultant chronologies (Table S1) of the two samples from Sima del Elefante. The overdispersion values remain unaltered at ±2σ in both cases, supporting the D_e interpretations and age model selections made on the original datasets. The ATE10-6 distribution contains a larger number of D_e values in comparison with the original dataset, enabling the heterogeneously bleached nature of this sample to be more clearly identified (Fig. 1e). Importantly, the replicate datasets for ATE10-11 and ATE10-6 demonstrate that higher SAR preheat temperatures do not adversely affect single-grain TT-OSL dating reliability for Atapuerca samples that otherwise do not contain grains with thermally unstable TT-OSL signals.

6. TL insights into TT-OSL source traps

To determine whether it is possible to identify differences in the source(s) of the TT-OSL signals at the multi-grain scale, we measured bulk TL responses for a selection of samples in both the UV (270–380 nm) and blue (340–480 nm) emission bands (Fig. 3a–b). For sample ATG10-3, we carefully separated (using electrical wire) previously measured grains that produced TT-OSL signals from those that did not produce TT-OSL signals and mounted the two groups of grains on different multi-grain aliquots (see Fig. S6 for purity of grain separates). Interestingly, the 3 mm aliquots of F13 and ATE10-6 share the same set of TL peaks in their UV and blue emission bands; the inter-sample differences in TT-OSL

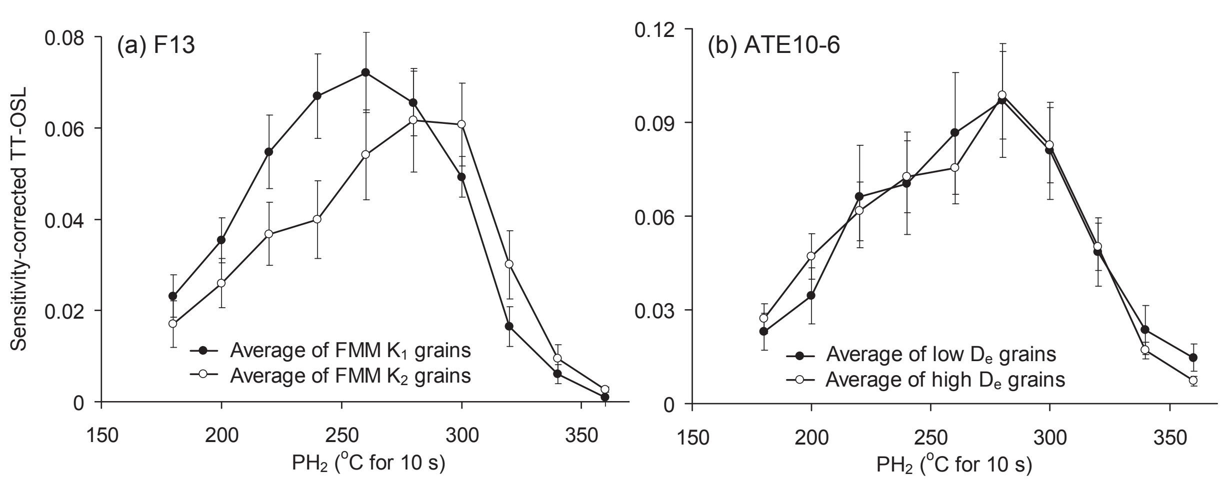

Fig. 2. Average pulse-annealing TT-OSL curves for grain populations with low and high D_e values from samples (a) F13 and (b) ATE10-6. Accepted grains with net T_n signals >50 counts/0.17 s were included in this analysis. The average curves for F13 have been derived using grains from the low dose (k_1) and high dose (k_2) FMM components. The average curves for ATE10-6 have been calculated from grains with low D_e values of <400 Gy and high D_e values of >600 Gy. The D_e cut-off values of <400 and >600 Gy equate to deviations of ±100 Gy from the CAM D_e for this sample.

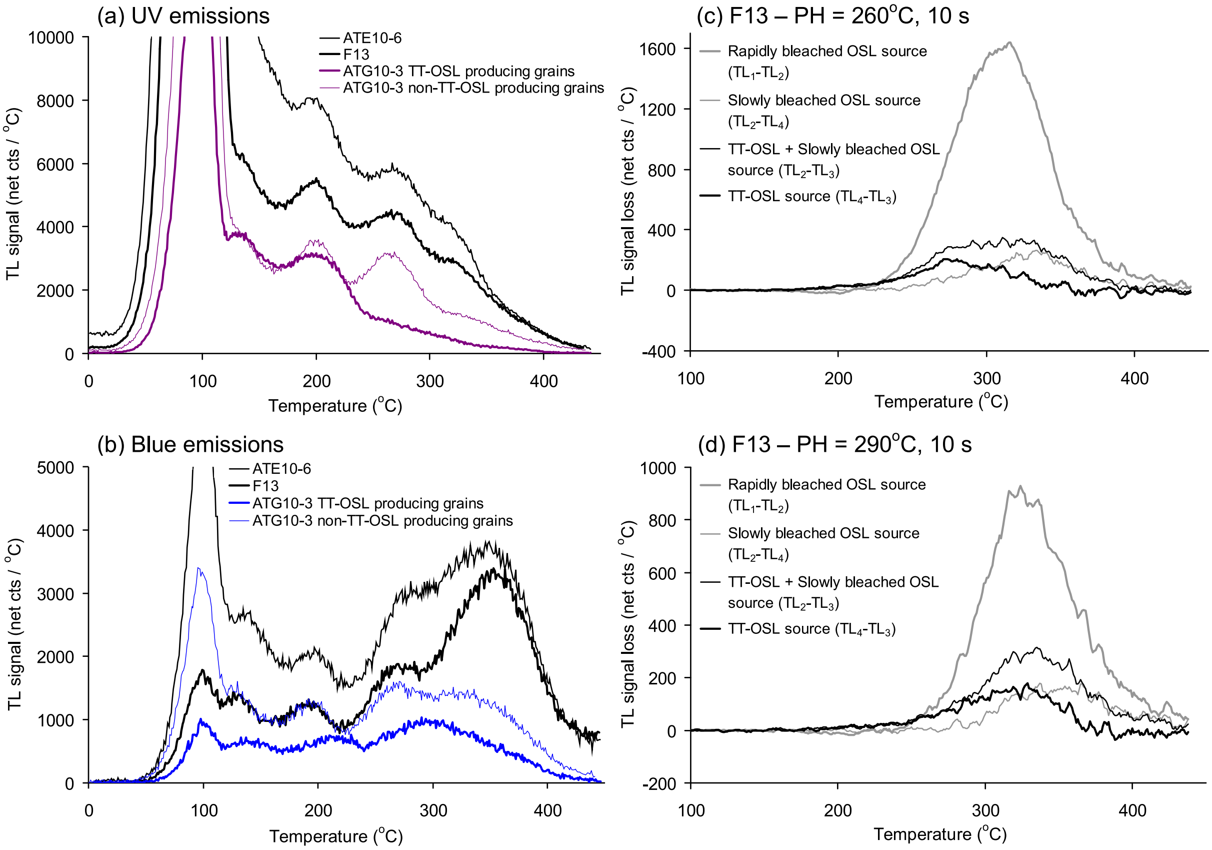

Fig. 3. (a)–(b) Multi-grain aliquot, background-subtracted TL glow curves for a 450 Gy dose measured in (a) the UV band using Hoya U-340 filters, and (b) the blue band using a combination of Schott BG-39, Corning 7-59 and LEE 0.6ND filters. (c)–(d) 450 Gy UV TL subtraction curves for sample F13 calculated using the TL measurements shown in Table S6 and preheats of (a) 260 °C for 10 s, and (b) 290 °C for 10 s. TL measurements were performed at a heating rate of 5 °C/s. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

sources evident in the single-grain analyses (Fig. 2) cannot be readily discerned from multi-grain TL measurements alone. There are, however, clear differences in the TL properties of the "TT-OSL producing" and "non-TT-OSL producing" grains from ATG10-3; the latter exhibit the same TL peaks as the bulk (unsorted) aliquots of F13 and ATE10-6, while the former are devoid of a 280 °C peak in the UV band and display a different set of high temperature TL peaks in the blue band.

A series of additional TL measurements were made on these aliquots after administering various preheat, OSL and TT-OSL treatment steps (Table S6), and the resultant data were used to calculate TL subtraction curves (Figs. 3c–d, S7). These TL signal loss experiments are similar to those employed elsewhere for TT-OSL trap lifetime assessments (e.g., Adamiec et al., 2010; Shen et al., 2011). Three important findings emerge from these experiments: (i) TL signal losses recorded after TT-OSL stimulation (TL_2 minus TL_3 in Table S6) are additionally influenced by the depletion of slowly bleaching OSL components (Fig. 3c–d). Separate measurements are needed to isolate TL signal losses caused by slow OSL stimulation (TL_2-TL_4) and to ascertain the net effects of TT-OSL production on the resultant TL subtraction curves (TL_4-TL_3). (ii) The use of higher preheat temperatures (290 °C, 10 s) can isolate TT-OSL signals from a similar TL glow curve region to that associated with the fast component OSL signal (~325 °C TL trap; Fig. 3d). (iii) Aliquots of TT-OSL producing grains and non-TT-OSL producing grains from ATG10-3 both show TL losses following TT-OSL stimulation (TL_4-TL_3) (Fig. S7). As the latter grains do not actually display TT-OSL signal decays (Fig. S6), it follows that the transferred charge is being preferentially re-trapped at sites that are not related to the fast component OSL signal (e.g., the 280 °C TL trap, which is absent in the UV TL curve of the TT-OSL producing grains).

7. Discussion and conclusions

The results of this study provide several useful insights into TT-OSL dating suitability at the single- and multi-grain scales. Our known-age Atapuerca case studies highlight that TT-OSL thermal stability characteristics may differ between sites, and that some samples may exhibit pronounced inter-grain variations in TT-OSL trap lifetimes. It is worth emphasising, however, that all of the published known-age samples from Atapuerca contain at least some sub-populations of grains with suitably stable TT-OSL signals. Moreover, it seems possible to isolate sufficiently stable TT-OSL signals in potentially problematic Atapuerca samples by varying the SAR preheat conditions.

The TL experiments reveal that it may not be appropriate to generalise about TT-OSL signal stability characteristics from multi-grain studies alone. Bulk signal TL loss experiments may provide limited insights into true TT-OSL source trap characteristics due to averaging effects, the dominance of non-TT-OSL producing grain populations, and interference from slowly bleaching OSL components. To avoid such complicating factors, it would be preferable to undertake TT-OSL lifetime assessments on individual grains or very small aliquots. However, lower signal intensities and larger parameter uncertainties are likely to limit the widespread feasibility of this approach. Known-age comparison studies therefore

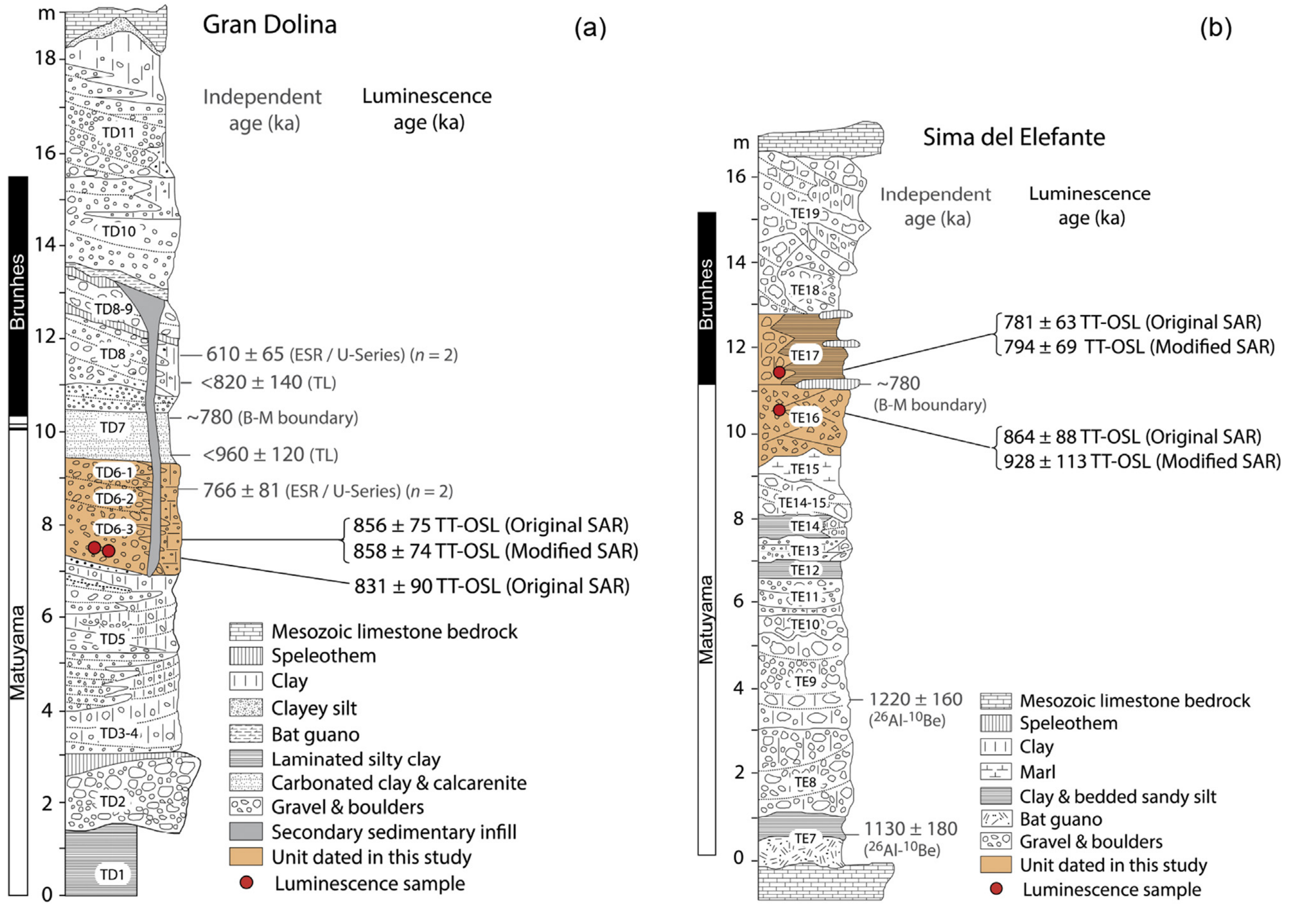

Fig. 4. Generalised Stratigraphic sequences for (a) Gran Dolina and (b) Sima del Elefante showing the single-grain TT-OSL ages and independent age control for sampled (and adjacent) horizons. See main text and Arnold et al. (in press) for further details of published age control.

remain the most informative and practical means of assessing TT-OSL signal stabilities over Early and Middle Pleistocene timescales. The presence of unreliable low dose components in known-age single-grain D_e datasets may be a useful indicator of unstable grain populations. Likewise, D_e plateau tests performed using various SAR preheats may be a useful means of identifying potentially problematic TT-OSL behaviours.

The suitability assessments undertaken here reveal that single-grain TT-OSL analysis is necessary at Atapuerca to avoid systematic dating uncertainties arising from extrinsic (partial bleaching) and intrinsic (signal stability) sources of D_e scatter. Our results confirm the suitability of the TT-OSL chronologies published previously for Gran Dolina and Sima del Elefante, and enable us to refine the existing age estimates. A weighted average TT-OSL age of 851 ± 46 ka is obtained for *Homo antecessor* palaeoanthropological horizon TD6-3 at Gran Dolina using the original and modified SAR protocols ($n = 3$) (Fig. 4a). The corresponding weighted mean TT-OSL ages for units TE16 and TE17 at Sima del Elefante are 789 ± 47 ka and 890 ± 70 ka, respectively ($n = 2$ in both cases) (Fig. 4b).

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.quageo.2015.02.005.

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