Ancient TL

www.ancienttl.org · ISSN: 2693-0935

Vandenberghe, D., Jain, M. and Murray, A., 2008. *A note on spurious luminescence from silicone oil.* Ancient TL 26(2): 29-32. https://doi.org/10.26034/la.atl.2008.420

This article is published under a *Creative Commons Attribution 4.0 International* (CC BY): https://creativecommons.org/licenses/by/4.0

© The Author(s), 2008

A note on spurious luminescence from silicone oil

D.A.G. Vandenberghe[1,2,3], M. Jain[2] and A.S. Murray[1]

1. Nordic Laboratory for Luminescence Dating, Department of Earth Sciences, University of Aarhus, Risø DTU, DK-4000 Roskilde, Denmark
2. Radiation Research Department, Risø DTU, Technical University of Denmark, DK-4000 Roskilde, Denmark
3. Laboratory of Mineralogy and Petrology (Luminescence Research Group), Department of Geology and Soil Science, Ghent University, B-9000 Gent, Belgium

(Received 28 August 2008; in final form 17 December 2008)

Introduction

Previous studies have shown that the slow component of quartz OSL exhibits considerable thermal stability and dose response, which suggests that this component could be used to date beyond the age range covered by conventional luminescence techniques (Singarayer et al., 2000; Singarayer and Bailey, 2003; Jain et al., 2003; Rhodes et al., 2006). The first step in all slow component studies is to distinguish the slowly decaying dosimetric signal from a background level, obtained (for instance) from a blank disc that is put through the same measurement sequence as the sample. In view of the thermal stability and bleaching characteristics of the slow component, background evaluation using quartz grains which have been heated or bleached is not thought to be appropriate. Measurements of the background have previously been reported – for instance Li (2007) observed that background count rate varied with optical stimulation power, while Singarayer (2002, her Fig. 2.4) found that, at a constant stimulation power, the background from both blank and "dead" discs increases with measurement temperature.

In the course of our investigations into the potential of the slow component for dating, we observed anomalously high background count rates from blank silicone-sprayed stainless steel substrates (cups or discs). This high background appears to originate with a spurious signal from the silicone oil. The signal can be observed upon stimulation of the silicone using blue diodes at an elevated temperature, such as during the high temperature clean-out (at 280°C) that is usually inserted between each cycle of the SAR protocol to minimize recuperation (Murray and Wintle, 2003). Investigations of the slow component involve more stringent heat treatments and possibly stimulation at elevated temperatures; as such these measurements are particularly sensitive to interference from such spurious luminescence.

Analytical facilities

The measurements reported here were performed using an automated Risø TL/OSL DA-20 reader equipped with blue LED's emitting at 470nm (FWHM 20nm). Luminescence was detected through a 7.5 mm thick Hoya U-340 detection filter placed in front of a bialkali EMI 9235QA photomultiplier tube. Details on the measurement apparatus can be found in Thomsen et al. (2008). The background signals were obtained from stainless steel substrates (cups or discs). The silicone oil used throughout this work is "Rüsch Silkospray", which is manufactured by Willy Rüsch GMBH, D-71394 Kernen-Rommelshausen, Germany.

Experiments and results

The existence of a spurious OSL signal is demonstrated in Fig. 1. A blank silicone-sprayed stainless steel cup was first heated to 500°C, and subsequently stimulated for 100 s using blue diodes at 50°C. This was followed by a further measurement at the same temperature but without switching on the blue diodes. This measurement cycle, with and without stimulation, was then repeated at progressively higher stimulation temperatures up to 275°C. Finally, the experiment was repeated for stimulation temperatures of 50°C, 125°C and 275°C. The aliquot was not exposed to ionising radiation in the course of the experiment. Fig. 1a shows some of the observed signals; the spurious (non-radiation-induced) signals do not decay significantly during the 100 s of stimulation. The average count rate is plotted as a function of stimulation temperature in Fig. 1b. The OSL signal (squares) progressively increases with stimulation temperature from 125°C onwards. The signals recorded without optical stimulation (circles) are indistinguishable from the PM dark count at room temperature (dashed line). At least in this experiment, the spurious signals appear to be reproducible (open symbols in Fig. 1b).

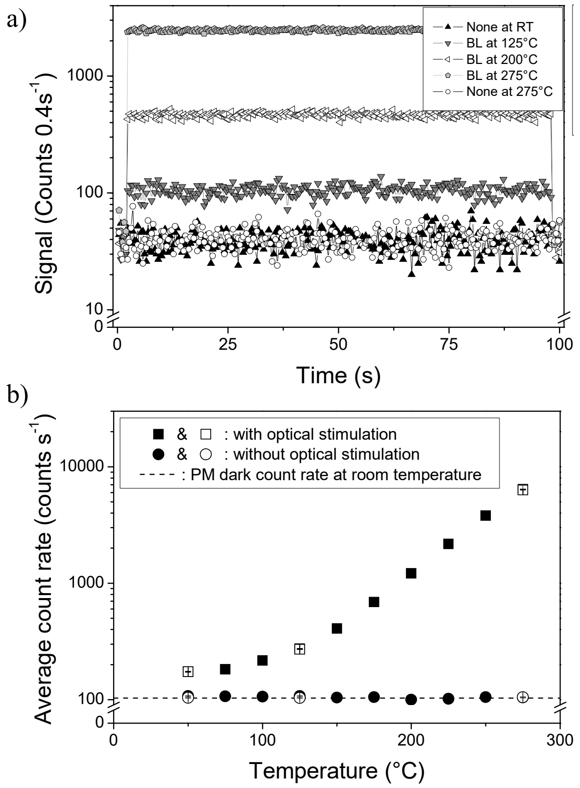

Figure 1: *(a) Signals observed from an annealed blank silicone-coated stainless steel cup during stimulation with blue diodes at various temperatures. RT refers to room temperature (~20°C) (b) Average count rate plotted as a function of stimulation temperature. The squares and circles represent the count rates observed with and without optical stimulation, respectively; the open symbols indicate repeated measurements. The dashed line marks the PM dark count rate at room temperature. Note that the aliquot was not beta irradiated during the course of this experiment.*

Spurious OSL was also monitored over longer stimulation times. Fig. 2 shows the OSL signal (normalised to the light-level observed in the first second of stimulation) from a blank silicone-sprayed disc that was stimulated for 1500 s at 250°C; the disc had previously been heated up to temperatures of as much as 600°C. Although the signal appears to remain relatively constant over the first 100 s (inset Fig. 2), on the longer timescale it can be seen to decay slowly over the entire 1500 s of stimulation to ~90% of the initial value.

We investigated whether the spurious signals originate with the silicone oil, and whether they exhibit a preheat and/or dose dependence. This experiment used 4 stainless steel cups; 2 had been used previously ("old cups") and 2 had never been used before ("new cups"). The two old cups were carefully cleaned with propanol, and all four cups were examined under a microscope to ensure that there were no quartz grains adhering to them. One old and one new cup were sprayed with silicone oil, while the other two were not. All cups were then put through a measurement sequence consisting of a ~16 Gy beta dose, preheat of 10 s at 300°C, stimulation with blue diodes for 100 s at 125°C, ~16 Gy beta dose, preheat of 10 s at 300°C and stimulation with blue diodes for 100 s at 250°C. The experiment was then repeated for preheat temperatures of 500°C and 600°C. After this, the experiment was further repeated for the preheat at 300°C twice, once without dosing and once with dosing. Finally, the aliquots were once more put through the measurement cycles employing the 300 and 600°C preheats, but this time the signal was recorded without switching on the blue diodes. The observed average count rates are summarised in Table 1.

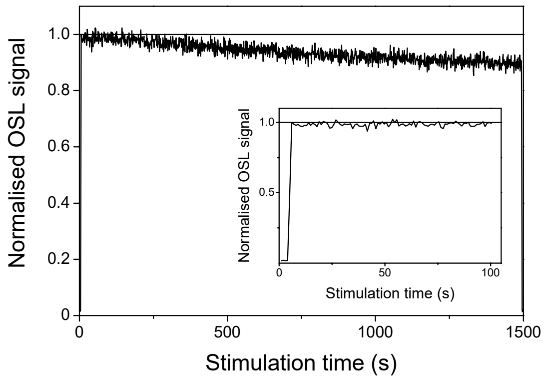

Figure 2: *Signal observed from an annealed blank silicone-sprayed stainless steel cup during stimulation with blue diodes for 1500 s at 250°C; the signal has been normalised to that observed in the first second of stimulation. The inset shows the signal observed during the first 100 s of stimulation.*

The signals measured from the new cup without silicone oil stand out in that they are independent of the thermal pre-treatment and measurement temperature, and have an intensity that is comparable to that of the signals measured without optical stimulation (Table 1, last two columns). For the three other cups, the OSL signals measured at 250°C are significantly higher than both the background level and the corresponding light levels at 125°C. For the silicone-sprayed cups, stimulating at 125°C generally increases the light-level above that measured for the cups without silicone oil; however, there is no detectable dependence of the signal on pre-treatment (in contrast to results of measurements performed at

Cycle	Treatment	Count rates (cts/s) observed for: Old cup with silicone spray		Old cup without silicone spray		New cup with silicone spray		New cup without silicone spray	
		at 125°C	at 250°C	at 125°C	at 250°C	at 125°C	at 250°C	at 125°C	at 250°C
1	Dose + 10 s at 300°C + optical stimulation	308 ± 2	341 ± 2	310 ± 2	707 ± 3	205 ± 1	354 ± 2	275 ± 2	212 ± 1
2	Dose + 10 s at 500°C + optical stimulation	289 ± 2	1785 ± 4	268 ± 2	1189 ± 4	271 ± 2	2855 ± 5	193 ± 1	145 ± 1
3	Dose + 10 s at 600°C + optical stimulation	218 ± 2	4120 ± 7	170 ± 1	1095 ± 3	239 ± 2	5306 ± 7	143 ± 1	146 ± 1
4	No dose+ 10 s at 300°C + optical stimulation	237 ± 2	3906 ± 6	155 ± 1	1066 ± 3	271 ± 2	5015 ± 7	129 ± 1	131 ± 1
5	Dose + 10 s at 300°C + optical stimulation	240 ± 2	3681 ± 6	157 ± 1	1025 ± 3	268 ± 2	4554 ± 7	133 ± 1	128 ± 1
6	Dose + 10 s at 300°C + no optical stimulation	86 ± 1	85 ± 1	86 ± 1	84 ± 1	85 ± 1	84 ± 1	89 ± 1	81 ± 1
7	Dose + 10 s at 600°C + no optical stimulation	84 ± 1	83 ± 1	81 ± 1	83 ± 1	81 ± 1	84 ± 1	79 ± 1	85 ± 1

Table 1: *Average count rates (± 1 standard deviation) observed from various blank stainless steel cups after various preheat treatments and at two different measurement temperatures (125°C and 250°C). Except where indicated otherwise (no dose; no optical stimulation), the aliquots received a beta dose of ~16 Gy prior to each measurement and were stimulated using blue diodes.*

250°C). Finally, it is also interesting to note that spurious signals can be observed from a cup that was cleaned after previous use with silicone-oil.

From the observation of spurious OSL signals at 125°C following high temperature treatments, it is reasonable to ask whether a spurious signal is also emitted during measurement in conventional (i.e. fast component) SAR routines. To examine this in greater detail, a stainless steel cup was sprayed with silicone oil and put through a measurement sequence that contained the main heat treatments of a conventional SAR protocol. The sequence consisted of preheating for 10 s at 260°C, measuring the signal at 125°C both with and without stimulation using the blue diodes, applying a cutheat to 220°C, and measuring the signal at 280°C, again both with and without blue diode stimulation. This sequence was repeated 8 times in total. The average count rates are plotted as a function of measurement cycle in Fig. 3. It can be seen that the signals with and without blue light stimulation at 125°C are not identical (solid and open circles, respectively). The optically stimulated luminescence signals do not change throughout the period of measurement (38 s) and the average count rates remain relatively constant over the 8 measurement cycles. The overall average count rate (± 1 standard error) is 169 ± 4 cts.s^{-1}. This value falls within the range of observations for a new cup without silicone oil (Table 1). Thus, it seems unlikely that the signal stimulated at 125°C originates with spurious OSL from silicone oil. On the other hand, spurious OSL signals can be clearly observed at a stimulation temperature of 280°C; this signal progressively increases with measurement cycle, after an initial decrease (solid squares in Fig. 3). It is concluded that conventional SAR measurements of the quartz fast component at 125°C do not usually suffer interference from spurious OSL from silicone oil. It should be noted that, even if there had been a spurious OSL signal at 125°C, the SAR protocol would be self-correcting so long as this signal was a constant underlying the fast component signal; it would, however, reduce our ability to detect light levels of a comparable intensity.

Discussion and conclusion

Thermally stimulated spurious luminescence signals from silicone oil have been observed in combination with aluminium but not with stainless steel (Murray, 1981). We are unaware of any previous observations of spurious OSL in this context.

The main purpose of the present note is to point out the existence of spurious signals in the specific context of studies related to the quartz slow OSL component. No comprehensive investigations of the behaviour of silicone oil as a function of measurement conditions were carried out. However, we can add that the spurious OSL signal associated with silicone oil can behave in an unpredictable manner, increasing first as it receives cumulative heating, and then dropping down in intensity (Fig. 3). Furthermore, although the signal shows no significant

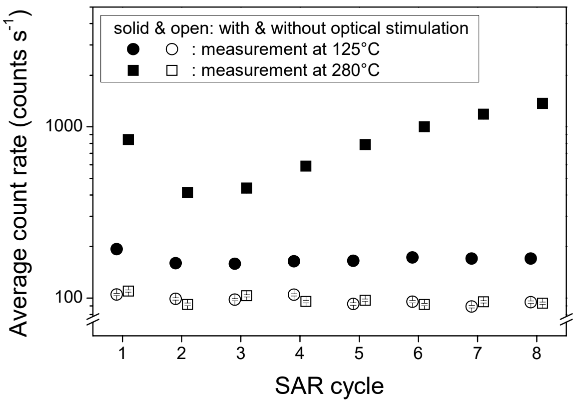

Figure 3: *Effect of the repeated thermal treatments typical of a SAR protocol on the signals (expressed as average count rates) observed from a blank and silicone-coated stainless steel cup. Stimulation was performed using blue diodes at 125°C and 280°C (circles and squares, respectively); the open symbols indicate the signals observed without optical stimulation. The aliquot was not beta irradiated during the course of this experiment.*

decrease over short stimulation times (40-100 s; see Figs. 1 and 2), it can be seen to decay slowly but steadily over longer stimulation times (of the order of 1-2 ks; Fig. 2). Finally, it is worth pointing out that the intensity of the spurious signal may be dependent on the amount of silicone oil sprayed upon a disc (see Table 1).

Measuring the slow component usually implies detecting low signal levels. From our observations above it is concluded that silicone oil can significantly interfere with such measurements, and that the spurious "slow component" is not easily corrected for. The original reason for using silicone oil was to ensure a mono-layer of grains during beta irradiation. This was because the beta dose rate is strongly dependent on both the build-up material in front of the grains (air for a mono-layer, otherwise other grains), and backscatter from the substrate (stainless-steel in the case of a mono-layer, otherwise other grains). Thus it would be unwise to avoid the use of silicone oil. However, we recommend that one should always test for the presence of spurious signals by measuring blank and silicone-oil coated substrates. Based on our Table 1, the same recommendation holds for cleaned substrates that have a history of use with silicone-oil. Indeed, there are probably better silicone degreasing agents available than the propanol we have used.

Acknowledgements
Financial support of the Fund for Scientific Research – Flanders (FWO-Vlaanderen) is gratefully acknowledged (DV: Postdoctoral Fellow). We thank Henrik Christiansen and Lars Bøtter-Jensen for discussions, and J.-H. Choi and G.A.T. Duller for their constructive comments on the manuscript.

References

Jain M., Murray A.S., Bøtter-Jensen L. (2003). Characterisation of blue-light stimulated luminescence components in different quartz samples: implications for dose measurement. *Radiation Measurements* **37**, 441-449.

Li B. (2007). A note on estimating the error when subtracting background counts from weak OSL signals. *Ancient TL* **25**, 9-14.

Murray A.S. (1981). *Environmental radioactivity studies relevant to thermoluminescence dating*. DPhil thesis, University of Oxford, 404p.

Murray A.S., Wintle A.G. (2003). The single aliquot regenerative dose protocol: potential for improvements in reliability. *Radiation Measurements* **37**, 377-381.

Rhodes E.J., Singarayer J.S., Raynal J.-P., Westaway K.E., Sbibi-Alaoui F.Z. (2006). New age estimates for the Palaeolithic assemblages and Pleistocene succession of Casablanca, Morocco. *Quaternary Science Reviews* **25**, 2569-2585.

Singarayer J. (2002). *Linearly modulated optically stimulated luminescence of sedimentary quartz: physical mechanisms and implications for dating*. DPhil thesis, University of Oxford, 345p.

Singarayer J.S., Bailey R.M. (2003). Further investigations of the quartz optically stimulated luminescence components using linear modulation. *Radiation Measurements* **37**, 451-458.

Singarayer J.S., Bailey R.M., Rhodes E.J. (2000). Potential of the slow component of quartz OSL for age determination of sedimentary samples. *Radiation Measurements* **32**, 873-880.

Thomsen K., Bøtter-Jensen L., Jain M., Denby P.M., Murray A.S. (2008). Recent instrumental developments for trapped electron dosimetry. *Radiation Measurements* **43**, 414-421.

Reviewer
J.H. Choi

Editors' comment: An alternative solvent for removal of silicone oil is ethyl methyl ketone, also known as butanone.