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On the luminescence signals of empty sample carriers

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Abstract

Luminescence dating is a leading technique for a large spectrum of Quaternary dating applications. Since the development of automated reader systems, handling great amounts of samples has become possible. A large quantity of data is produced in a short time and a detailed check of every single curve is often impractical. Therefore, it is important to be confident in excluding any kind of unwanted signal contributions, such as those from sample carriers. For commonly used types of steel and aluminium (Al) carriers from three laboratories, luminescence characteristics of spurious and radiation-induced signals are presented. TL and OSL emissions of discs show natural (Al) and regenerated thermally stable signals in the UV, UV-blue and red detection range. These signals have characteristic saturation doses of several hundred Gy. Furthermore, we demonstrate light insensitive signal components and phototransferred thermoluminescence (PTTL). Due to high scatter between discs, the proportion of unwanted disc signal contribution to the entire signal is difficult to predict, without direct measurement. The sources of these signals are possibly chemical compounds acting as luminophores or oxide layers (Al_2O_3 layers in case of Al discs).

Introduction

Since the beginning of thermoluminescence (TL) and later optically stimulated luminescence (OSL) dating much effort has been focused on avoiding unwanted signal contributions to those used for dating purposes (Aitken 1985, Aitken 1998). These contributions can originate from the sample itself or the measurement setup, e.g. the atmosphere during TL measurements or phosphorescence from the filters. The choice of the sample carrier is therefore of paramount importance for measuring a clean luminescence signal from the sample only.

The first TL measurements with relevance to recent dating applications were carried out with samples mounted on glass plates (Daniels et al. 1953). Several other materials were used as sample holders in the following decades, such as aluminium (Al), platinum, nickel, steel (e.g. Bøtter-Jensen 1997), or silver (e.g. Yawata and Hashimoto 2007). Only sparse information on the applicability of those substances is given in the literature (Berger 1982, Aitken 1998). The authors report on 'parasite' luminescence signals generated by Al holders. Today, most laboratories use stainless steel or Al carriers for sample placement in the commonly used Risø readers, usually termed as discs (flat plate) or cups (with depression). It should be mentioned, however, that one of the first materials used for TL dosimetric purposes was Al oxide (Al_2O_3) (Osvay and Biró 1980).

However, to our best knowledge, no systematic study has been published in the contemporary luminescence dating literature to prove the absence of parasite luminescence for commonly used sample holders. Standard measurement protocols do not check for unwanted signal contributions. Thus, the registered net luminescence (background subtracted) is routinely attributed in its entirety to the sample.

Since we found hints on spurious (i.e. non-radiation-induced) and dose dependent luminescence signals of a variety of different disc materials during experimental measurements, we conducted a study to investigate the features of these potentially problematic signals and their expected influence on dose determination. Therefore, we measured various sample carriers (Al and steel) from the luminescence laboratories in Oxford, Cologne and Bayreuth using different stimulation methods (TL, OSL, IRSL) and detection windows (UV: 340 Δ 80 nm, UV-blue: 420 Δ 30 nm, red: 625 Δ 25 nm and 630 Δ 30 nm). Furthermore, we examined the dose response characteristics and the bleachability of the signals as well as the phototransferred thermoluminescence (PTTL, e.g. Furetta 2010, Kalchgruber 2002) of empty discs. An exemplary equivalent dose determination (SAR protocol, after Murray and Wintle 2000) of a well-studied sediment sample acts as reference point for assessing signal interference. Finally, an alternative method for cleaning the discs is presented and its effectiveness is tested.

Materials and methods

Sample carriers

To investigate the luminescence behaviour of sample carriers, three kinds of empty discs or cups commonly employed in laboratories were tested:

1) Stainless steel discs used in Oxford and Cologne (Cr-Ni steel V4A, diameter 9.8 mm, thickness 0.5 mm)
2) Al discs used in Oxford, Cologne and Bayreuth (unknown Al composition, diameter 9.8 mm or 9.6 mm, thickness 0.45 mm)
3) Al cups, manufactured in Bayreuth (Goodfellow Al foil, purity 99.0 %, diameter 12.0 mm, thickness 0.1 mm)

From each kind of discs or cups, at least five exemplars were measured. We chose all discs randomly from the 'clean disc box' (for the applied cleaning procedure, see below) and measured them without further chemical and physical treatments. Regarding Al discs we tested both used and new discs. Al cups from Bayreuth were new, because cleaning without damaging is difficult due to their low thickness.

Disc cleaning procedures

To test the effects of disc cleaning procedures on spurious and dose-dependent signals two methods were applied on used discs in Cologne.

1) Steel and Al discs were cleaned in an ultra sonic bath in addition to mechanical rubbing (sponge) and washing with rinsing agent to remove remaining silicon oil. Afterwards, the discs were flushed with distilled water and purged in acetone.
2) A mechanically more severe procedure was applied to remove the oxidized layers from steel and Al discs. Several tens of discs were placed in a bottle of chalk suspension and kept on the shaking table for polishing for 24 h, or 72 h if they were scrubbed with scouring agent in advance. Washing with distilled water afterwards prevents measuring spurious signals from carbonates. The loss in mass is negligible.

Measurement setup

The luminescence measurements were carried out on different Risø DA-12, DA-15 and DA-20 readers in Cologne, Oxford and Bayreuth, equipped with a standard bialkali photomultiplier tube (EMI 9235QB). In addition, for the red detection a cooled trialkaline photomultiplier tube (EMI 9658B) described in Fattahi and Stokes (2005) was used in Oxford. The luminescence was measured placing the following filters consecutively between the disc and the photomultiplier:

1) Hoya U340 (7.5 mm, 340 Δ 80 nm),
2) Combination of Schott GG400 (3.0 mm), Corning 7-59 (2.0 mm), Schott BG39 (1.0 mm) and HA3 (4.0 mm): UV-blue detection centred at 420 nm,
3) Chroma D410/30x interference filter for UV-blue detection (410 Δ 30 nm) for IRSL,
4) Chroma D630/60 M (630 Δ 30 nm, Cologne) or Omega D625/DF50 (625 Δ 25 nm, Oxford) interference filters for red detection, respectively.

The heating rates were set to values of 2 or 5 K s^{-1} and the measurement chamber was flushed with N_2 for two minutes before each measurement exceeding 160°C, except where indicated otherwise. Maximum temperatures for UV TL (UVTL) and UV-blue TL (BTL) measurements were 500°C and for red TL (RTL) 450°C. The background for each measurement was recorded immediately afterwards. For OSL measurements of the UV emissions, blue LEDs (470 Δ 30 nm) and for IRSL measurements, infrared LEDs (870 Δ 40 nm) were used.

The discs received radiation doses from $^{90}Sr/^{90}Y$ β-sources delivering around 5 Gy min^{-1} (DA-12 and DA-15) or 7 Gy min^{-1} (DA-20). These dose rates are usually calculated for mineral grains mounted on the discs and not for the discs themselves. Therefore, the dose rates are approximations.

Dose response measurements

For dose response measurements, blank discs were irradiated with incremental doses up to ca. 1 or 2 kGy and subsequently TL and OSL were measured. A standardized luminescence efficiency value, with units cts $(K\ Gy)^{-1}$ for TL and cts $(s\ Gy)^{-1}$ for OSL, respectively, allows comparing measurements with differing parameters, e.g. heating rate or measurement channels (suggested by M. Krbetschek, pers. comm.). The TL signal was integrated over the thermally stable range of 300-400°C for UVTL and BTL. To avoid the influence of increasingly noisy net signals above 350°C, the integration interval for RTL was lowered to 250-350°C.

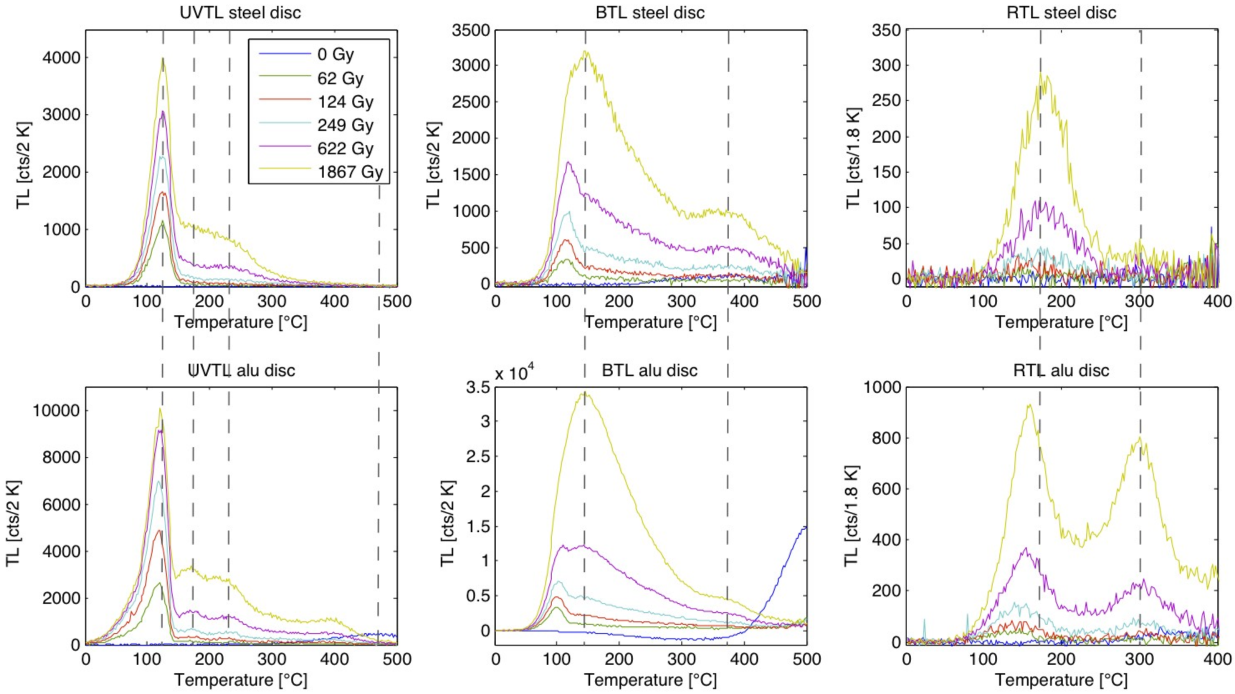

Figure 1: *Natural and regenerated TL signals from single steel and Al discs for different detection wavelengths. All measurements were performed in Cologne and carried out at 5 K s^{-1}. For further details, see text.*

The OSL/IRSL decay curves were measured at 125°C read temperature after a preheat of 260°C (OSL) for 10 s and read temperatures of 50°C after a preheat of 250°C for 60 s or 270°C for 10 s (IRSL).

Experimental details and results

Thermoluminescence signal

- Steel discs

Steel discs from the Cologne and Oxford laboratories were heated to 500°C (UVTL and BTL) or 450°C (RTL) and TL was measured first without irradiation, then after incremental β-doses. Between the dose steps, a test dose was given to record potential sensitivity changes.

No signal above background could be detected for non-irradiated steel discs except for a small 380°C peak in the blue range (Fig. 1).

Significant TL signals resulted from exposure to ionizing radiation. UVTL and BTL showed a strong 110°C peak, whereas for BTL this peak slightly shifts towards higher temperatures or is superposed by another peak on its high temperature shoulder for high doses (> 1 kGy). At higher temperatures, we observed at least two other peaks which form a broad continuously decreasing shoulder between 150 and 300°C in the UV range. Little UVTL signal is detected above 300°C. A similar shoulder occurs in the blue window, followed by a distinct peak centred at 380°C in the high temperature region. In the red detection window, steel discs are far less sensitive to irradiation. We only observed signals above background noise for doses > 250 Gy (peak at 180°C).

-Al carriers

We applied the same measurement routine to Al discs from the Oxford and Cologne laboratories. In contrast to steel discs, Al discs yielded a notable TL signal without artificial irradiation. The peak positions for this spurious signal are 480°C (UV) and ≥ 500°C (UV-blue) with count rates extending from a few hundred cts K^{-1} in the UV and red (no peak observable) up to several thousand cts K^{-1} in the blue detection window. For BTL, however, difficulties with the background subtraction led to a negative net signal and the 'natural' peak may also suffer from this problem. The natural UVTL and BTL signal is not depleted completely by measurements up to 500°C. Hence, remaining trapped charge appears to cause a spurious signal also in subsequent measurements (data not shown).

Following β-irradiation, the 110°C UVTL peak of Al discs appears at slightly lower temperatures in

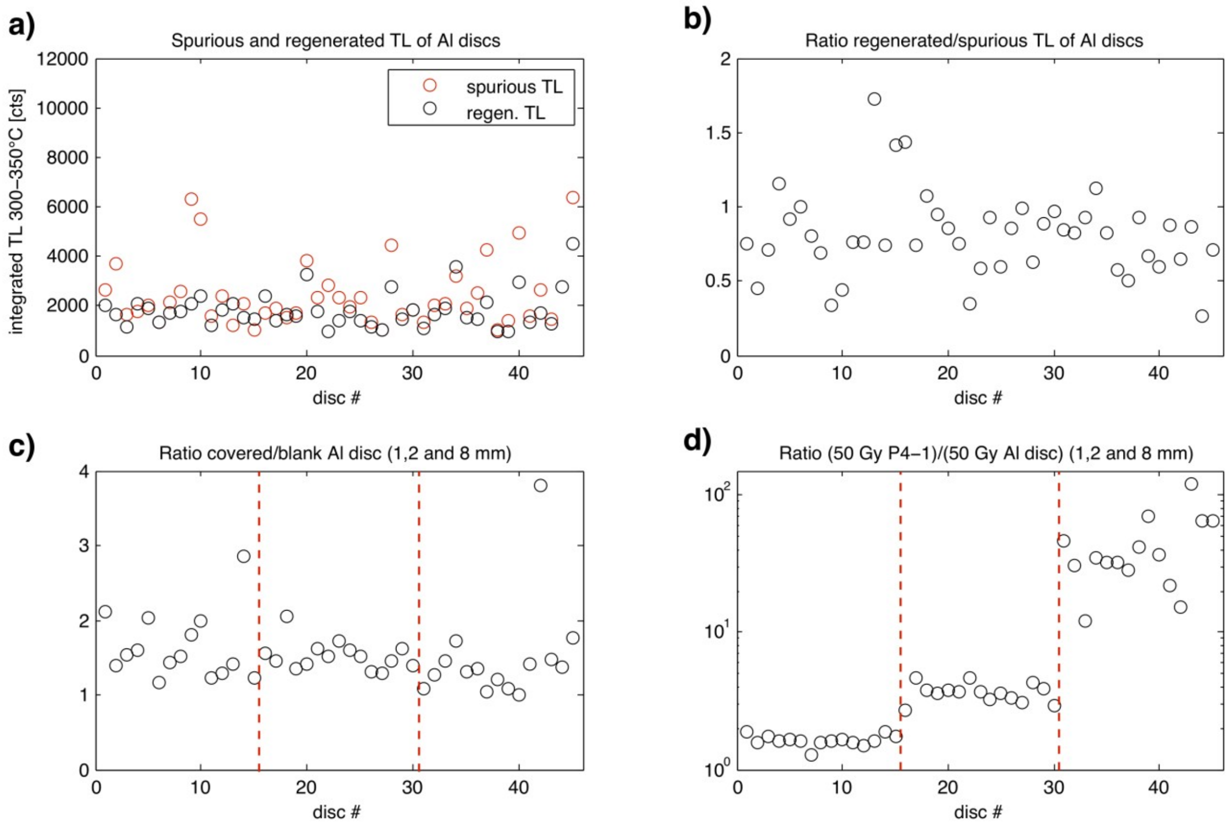

Figure 2: *Results of the experiment to measure the influence of grain coverage on unwanted signal contributions of used Al discs (UV detection range). Experimental conditions and detailed descriptions are given in the text.*

comparison to steel due to the higher thermal conductivity of Al and thus reduced thermal lag between heater plate and upper disc surface. We observed three other UVTL peaks at 180, 220 and 400°C for β-doses > ca. 250 Gy (Fig. 1). Similar to the BTL emission of steel, Al discs yield a 110°C peak that is surpassed in growth by a peak at 140°C for doses > 600 Gy. A further small BTL peak of Al is centred at 380°C. The RTL emission of Al discs is dominated by two maxima at 150 and 300°C. The low temperature peak seems to correspond to the RTL peak observed for steel discs, whereas steel discs only show a very weak 300°C peak.

The positions of the TL peaks in the various detection windows for steel and Al discs are roughly the same for all measured discs. However, the intensities of the luminescence emissions and the relation of peak heights vary considerably between individual discs.

We also detected low spurious TL signals in the UV range for Al cups as a slowly growing shoulder from 200 up to 450°C. Following β-irradiation up to ca. 1 kGy we found UVTL peaks at 110°C and at around 400°C for doses > 500 Gy (see supplementary data at www.aber.ac.uk/ancient-tl). BTL glow curves of several Al cups exhibit a very weak peak at 110°C after the highest regeneration dose (> 1 kGy). In general, used and cleaned Al and steel discs produce much higher spurious and regenerated signal intensities in the UV and blue detection range than new Al cups. RTL emissions of Al cups were not investigated.

Fig. 2 presents the results of an experiment to measure the influence of grain coverage on unwanted signal contributions of used Al discs. All signals were recorded in the UV detection window. Fig. 2a shows the integrated TL (300-350°C) of the spurious signal and the regenerated signal after 50 Gy β-irradiation for 45 discs. The ratio of these signals for each disc is plotted in Fig. 2b. The annealed discs were then irradiated with 50 Gy and immediately covered with annealed quartz grains (BT781, loessic sample from Nussloch, Germany, unit P4-1, 100-200 µm, heated at 500°C for 1 h) using masks of different diameter (discs 1-15: 1 mm; discs 16-30: 2 mm; discs 31-45: 8 mm). Fig. 2c shows the ratio of the regenerated TL signal of grain-covered discs and uncovered discs

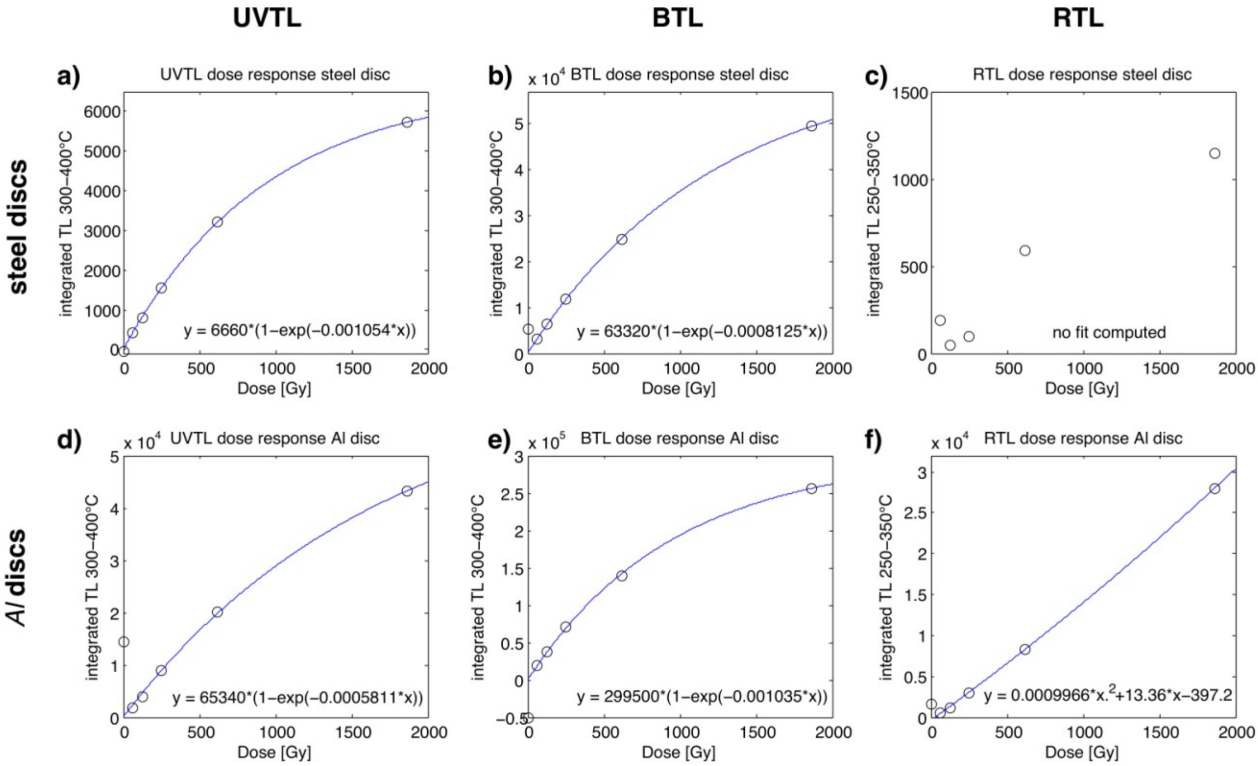

Figure 3: *TL dose response curves of steel and Al discs for different emissions. The investigated discs are the same as shown in Fig. 1. Integration limits are as indicated on the ordinate. For further details, see text.*

(measured after cleaning the discs individually afterwards). These measurements indicate that the effect of light shielding through grain coverage is negligible. The fact that the ratio is higher than 1 is probably a result of the cleaning procedure. Fig. 2d shows the ratio of the 50 Gy irradiated quartz sample (mounted on Al discs using the same order and mask diameters as in Fig. 2c) and the regenerated signal after the same dose of the same discs without quartz layer.

-Dose response

Growth curves of UVTL and BTL emissions of steel discs can be fitted to single saturating exponentials with characteristic saturation doses (D_0) of ca. 950 Gy and 1.23 kGy, respectively (Fig. 3). For low β-doses (< 200 Gy), the dose response can be approximated with a linear fit. In this range, the dose response amounts to about 3 cts (K Gy)$^{-1}$ for UVTL and 24 cts (K Gy)$^{-1}$ for BTL. For higher doses, those values decrease due to saturation. The RTL signals in the thermally stable region are too low to estimate their dose response. The UVTL and BTL growth curves of Al discs show similar behaviour with approximately linear growth up to doses of 500 Gy (UVTL) and 300 Gy (BTL) and characteristic saturation doses of 1.72 kGy and 970 Gy, respectively. In contrast, the RTL Al emission reveals supralinear increase for doses < 2 kGy. Up to onset of saturation, the dose response of Al discs can be expressed as ca. 20 cts (K Gy)$^{-1}$ for UVTL, ca. 180 cts (K Gy)$^{-1}$ for BTL and ca. 4-6 cts (K Gy)$^{-1}$ for RTL emissions.

Test dose monitoring shows little sensitization for steel discs and Al discs and cups. The effect is most distinct for the 110°C TL peak in the UV and blue detection window and the BTL high temperature peaks of Al discs, but not quantifiable for other high temperature peaks.

-Growth rate of the spurious signal

As spurious and dose-dependent signals of Al discs may derive from oxide layers, the signal response was investigated for different durations of oxygen exposure. Fresh chalk-polished Al discs (Cologne, procedure see above) were stored in an acetone bath in a closed bottle to prevent contact with oxygen. Batches of 5 discs were successively removed at defined times. The remaining time to the UVTL measurements is then the oxygen exposure time. Those were set to 10 minutes (approximated 0 days), 2, 5, 20 and 41 days. The TL signal was integrated over the range 250-450°C.

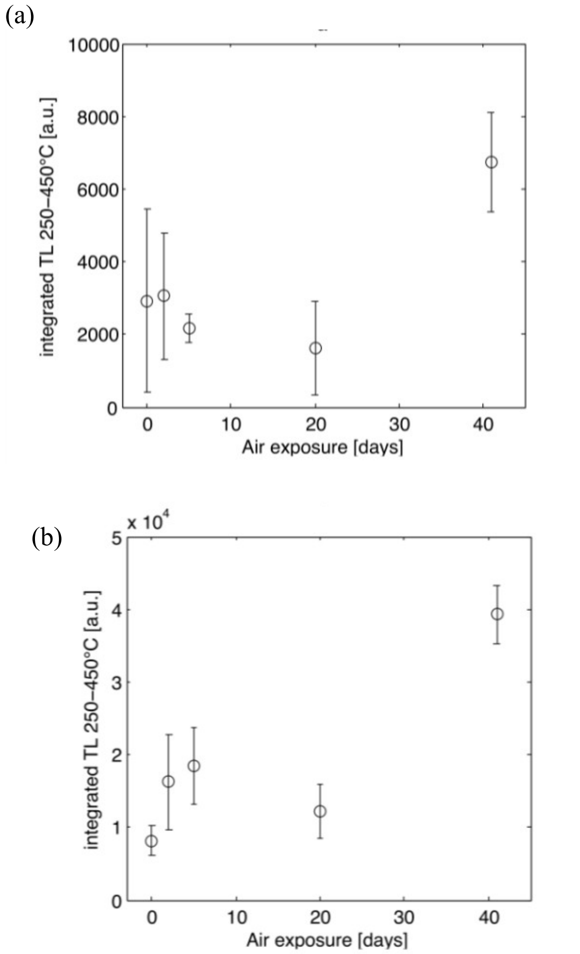

Figure 4: *Growth of (a) spurious and (b) 250 Gy regenerated UVTL signals of Al discs with air (oxygen) exposure duration. The error bars represent the standard deviation of 5 discs each.*

We observed no growth, within measurement uncertainty, up to oxygen exposure durations of 20 days due to high scatter of the spurious TL signal between discs (Fig. 4a). For longer contact with air, the discs showed a substantially increased signal. In order to check whether there is a correlation between spurious and regenerated signal, all discs received a β-dose of 250 Gy after initial spurious signal readout (Fig. 4b). We calculated the ratio of both signals in the same temperature interval. The ratio values (regenerated signal/spurious signal) denote high disc-to-disc scatter and suggest that both signals are not closely correlated (data not shown). However, Fig. 4 indicates that longer oxygen exposure durations generate both stronger spurious and radiation-induced TL signals.

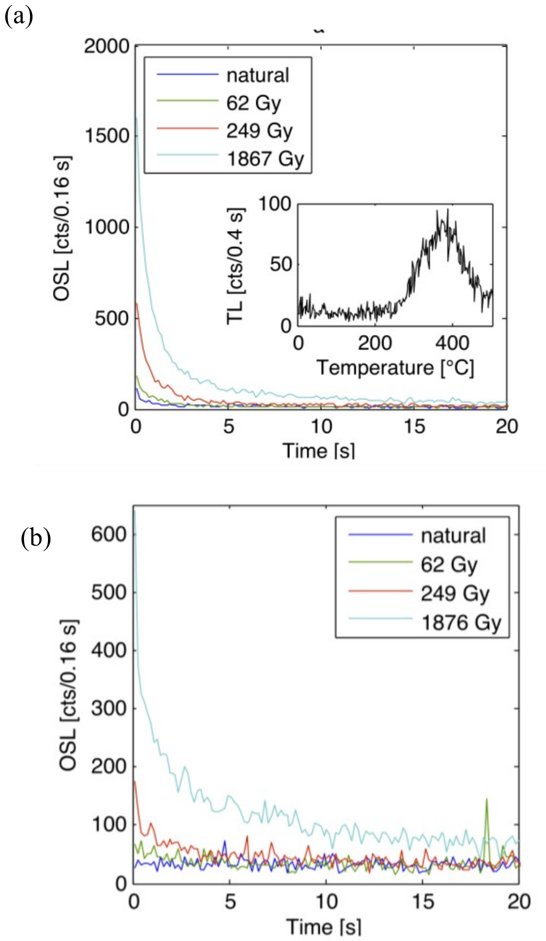

Figure 5: *Natural (0 Gy) and regenerated OSL signals of single (a) steel and (b) Al discs after preheat (260 °C for 10 s). The inset in (a) shows the residual UVTL measured after β-irradiation of 1867 Gy and 40 s blue optical stimulation (470 Δ 30 nm). For further details, see text.*

Optically and infrared stimulated signals

-OSL

All steel discs were preheated to 260°C for 10 s at a rate of 5 K s^{-1} to simulate conditions equal to routine OSL measurements. We observed negligible OSL signals without artificial irradiation, but count rates up to 20 cts $(s\ Gy)^{-1}$ for β-doses < 100 Gy (Fig. 5). Pronounced scatter of signal intensity and growth for single discs is typical. Al discs show OSL signals of about 2 cts $(s\ Gy)^{-1}$ after a 260°C preheat for 10 s up to the highest regeneration dose. Al cups produce OSL signal above background in the range of 10 cts $(s\ Gy)^{-1}$ in the first one or two measurement channels after irradiation, indicating a rapidly decaying signal component (supplementary data). As for steel discs, initial OSL signal intensities of Al cups differ considerably from cup to cup. Due to low signal-to-

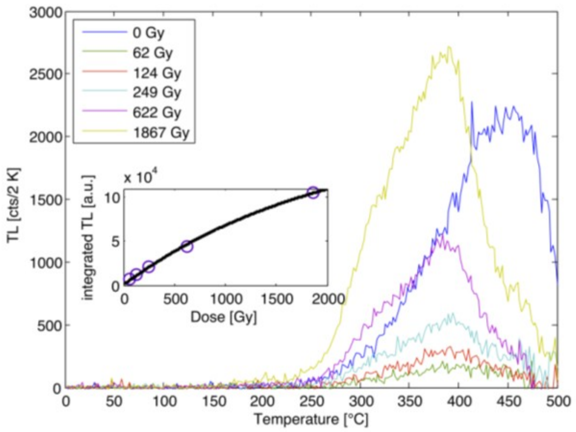

Figure 6: *Dose response of residual UVTL after blue optical stimulation (470 Δ 30 nm) for 40 s for one Al disc. The inset displays the signal growth for integrated TL (340-440°C) as a function of the regeneration dose, fitted to a single saturating exponential (y = a*(1-exp(-b*x)), where a = 1.74E+05, b = 4.95E-04 and x is dose in Gy).*

noise ratios of the test dose signals we cannot provide any information on potential sensitivity changes for OSL of steel and Al sample carriers.

-IRSL

We also checked the IRSL signals from discs during common IRSL (feldspar) measurement conditions. After a 270°C preheat for 10 s or 250°C for 60 s IRSL was measured at 50°C. Steel discs as well as Al cups completely lack IRSL signals (natural and after irradiation). Al discs reveal a dose dependent, slowly decaying signal with count rates in the range of 2-5 cts $(s\ Gy)^{-1}$, but no natural IRSL signal (supplementary data).

-Residual TL

After OSL signal depletion and subsequent TL measurement we detected a non-bleachable or slowly bleachable signal component at high temperatures for both steel and Al discs. An example of the residual UVTL of a steel disc after 1867 Gy β-irradiation is shown in the inset of Fig. 5a. In general, the residual UVTL signal of Al discs shows higher count rates than that for steel discs (Fig. 6). Furthermore, the peak positions of natural and regenerated residual glow curves of Al discs differ significantly. As the empty discs are handled under room light conditions (and therefore light sensitive components should be removed), it is probable that the 0 Gy peak in Fig. 1 (lower left) is identical to the 0 Gy peak shown in Fig. 6. The dose response of Al residual UVTL is approximately linear up to β-doses of 500 Gy, but shows exponential saturation for higher doses ($D_0 \approx 2$ kGy, Fig. 6).

-Photo-transferred thermoluminescence (PTTL)

For the measurement of the PTTL the discs were first irradiated with 250 Gy, twice annealed to 500°C, and subsequently two cycles of OSL and UVTL were measured without further irradiation. The OSL signals (max. about 1500 cts s^{-1}) of the Al discs show an initial increase before decaying exponentially as expected from a typical OSL curve. This is contrary to the OSL signals observed after lower preheat temperatures. Optical stimulation induced a weak PTTL signal (ca. 60 cts K^{-1}) with a peak at 190°C (Fig. 7c). The dose response of the PTTL signal was not investigated. Our results indicate that steel discs are free from PTTL signals.

Influence of cleaning procedures

Since the BTL emissions of both steel and Al discs showed the strongest luminescence, we used them as an indicator of signal reduction attributed to the chalk cleaning procedure. The polishing of steel discs with chalk reduced the spurious and dose-dependent BTL signals by about 40-50 % but did not suppress them completely (data not shown). Though natural signals are lacking, we still observed a prominent 110°C peak of about 10 cts $(K\ Gy)^{-1}$.

Al discs showed a natural BTL signal with a peak at 450°C (ca. 100-200 cts K^{-1}) and a regenerated emission of 1-2 cts $(K\ Gy)^{-1}$ after air storage of several days. Accordingly, the disturbing signal can be effectively reduced but not fully eliminated.

Influence of the disc signals on D_e determination

In order to test the effect of unwanted signal contributions of sample discs during an equivalent dose determination, a standard SAR protocol was carried out (Murray and Wintle 2000) using a coarse grain quartz sample (BT781) whose reliable luminescence characteristics are known (Zöller et al. 1988, Tissoux et al. 2010). Half of 10 steel and half of 10 Al discs were annealed (500°C for 30 s), the other half remained untreated prior to grain deposition (aliquots of 2 mm diameter). A preheat temperature of 240°C, a cutheat of 220°C and a read temperature of 125°C were chosen and a hot bleach (OSL at 280°C for 40 s) was applied at the end of each SAR cycle. Excluding one outlier (annealed steel disc no. 4, supplementary data Fig. 3a), the mean D_e values determined with natural discs are ca. 20 % (steel, n = 4) and 35 % (Al, n = 5) higher than those measured with annealed discs (n = 5 each). This indicates that the natural disc signal contributes verifiably to the initially measured geological signal of the sample. In addition, the measurement uncertainty of each single D_e value increases substantially if the discs are not annealed before the measurement. This effect is more distinct for steel

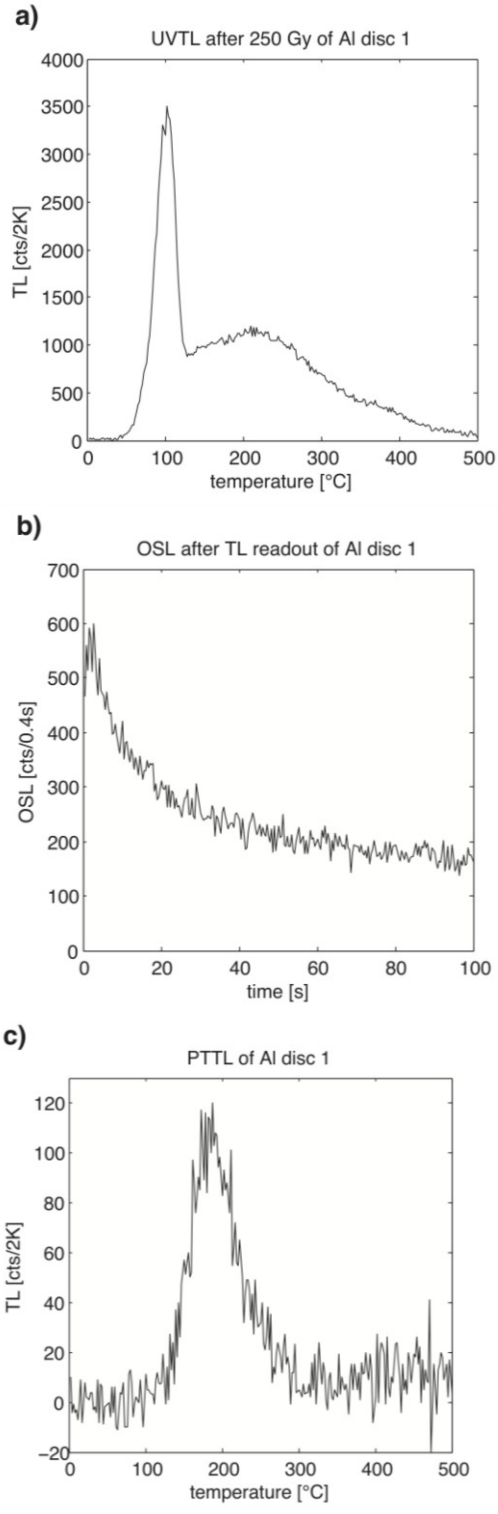

Figure 7: *PTTL measurement in the UV of an Al disc after a β-dose of 250 Gy was given: a) background subtracted TL measurement up to 500 °C after irradiation, b) subsequently recorded blue stimulated OSL signal, c) photo-transferred TL signal after optical signal depletion.*

than for Al discs, i.e., the mean uncertainty increases by about 90 % and 72 %, respectively, if the discs remain untreated prior to the measurement. Differences in the recycling ratio for annealed and non-annealed discs are not significant. However, statistical validity is limited by the small number of measured aliquots and further measurements are needed (supplementary data).

Discussion

The striking difference of TL peak temperatures for spurious and radiation-induced signals from Al discs (Fig. 1) suggests that different kinds of traps are involved in charge storage, but that potentially the same suit of recombination pathways are used owing to the common emission band (although detailed spectral measurements would be needed in order to confirm this). We observed several UVTL and BTL peaks between 100-400°C that cannot be distinguished without curve fitting. The RTL glow curve on the other hand consists of only two visually distinguishable peaks, suggesting involvement of fewer types of charge traps. In contrast to Cr-Ni steel, the charge occupation sites connected to the spurious and the dose-dependent signal of Al both include non-bleachable (insensitive to visible light) residual TL, suggesting that full signal erasure may only be possible by heat treatment.

Due to the high affinity of Al to O, aluminium oxide layers build up within a few minutes when the (hypothetically) clean disc is exposed to oxygen. These layers reach thicknesses of up to 30 nm on pure Al at high temperatures (> 300°C) (Ostermann 2007). Diffusion of O is then not possible anymore and the layer protects against continuing corrosion (Bargel and Schulze 2008). Possibly, intrinsic defects of Al_2O_3 are responsible for part of the luminescence signal of the Al discs.

Similar to silicon dioxide (SiO_2), Al_2O_3 can also facilitate vacancies and interstitials (due to impurities) in its molecular structure, such as oxygen vacancies which in quartz act as hole traps (Kelly and Laubitz 1967, Preusser et al. 2009). Oxygen vacancies are described in Al_2O_3 as well, but acting as traps for electrons instead of holes (Hakanen et al. 1997). The remarkable luminescence sensitivity of Al_2O_3 is commonly used for dosimetric applications, where the dosimeters are additionally doped with e.g. C, Fe or recently with Tb and Tm (Osvay and Biró 1980, Aypar 1986, Akselrod et al. 1993, Barros et al. 2010). Luminescence properties are then, however, strongly influenced by the elements used for doping.

Stainless steel, as used for the discs, contains small amounts of carbon and silicon which might together form luminophores (carbides) (pers. comm. M. Krbetschek). It cannot be excluded that those contribute to the luminescence signal of the investigated steel discs. Furthermore, we cannot rule out that a small layer of Cr and Fe oxides (a few atom layers thick, arising from the admixture of Cr to the alloy) on Cr-Ni steel, causes part of the dose-dependent luminescence signal (Blasek and Weihert 1979, Bargel and Schulze 2008).

Although we observed minor dissimilarities between glow curves of steel and Al discs, the main peaks seem to correspond for both materials and all measured emissions (Fig. 1). This fact suggests that there is one common source for all disc materials.

Previous studies dealing with spurious luminescence from silicone oil revealed ambiguous findings. In combination with Al discs, considerable spurious TL signals were found, but not with steel discs (Murray 1981). Vandenberghe et al. (2008) reported that silicone oil (Willy Rüsch GmbH) potentially contributes to disturbing OSL signals. In both investigations, sample carrier materials are not described in detail. In contrast, Fuchs (2001) found silicone oil (from the same manufacturer) on Al cups to be free from spurious and radiation-induced OSL signals. The fact that Vandenberghe et al. (2008) observed disturbing signals of used and cleaned steel cups indicates that adherent silicone residues are a potential source. This would imply that conventional cleaning techniques (as described above) are not capable of removing silicone oil completely. As silicone is made up of silicon and oxygen atoms (among others), there might be chemical reactions during heat treatment or storage resulting in some kind of luminophore. Spurious signal levels of new sample holders possibly depend on the cleaning procedure prior to first use (if cleaned at all) because in some workshops lubricant oils (containing forms of silicone) might be used for disc or cup manufacturing. Beside the technique mentioned above, there are also alternative methods to remove silicone relics and to clean the discs, such as using propanol or butanone (Vandenberghe et al. 2008) or short acid treatments (e.g. 1-2 % aqua regia or diluted phosphoric acid for a few minutes).

In summary, there are two main sources potentially causing the observed disc signals: luminescent chemical compounds of the carrier material itself, or some kind of contamination resulting from grain deposition (silicone oil) that resists the cleaning procedures. Similar glow curves for both investigated materials favour contaminations as main source. However, further investigation is needed for conclusive determination of the origin of the observed signals.

For TL measurements it is clearly advisable to use Al cups of the type described above or steel discs, despite lower thermal conductivity and higher costs for steel discs. Al discs show lower unwanted signals with regard to OSL measurements. During our investigations we applied relatively high β-doses. The majority of luminescence samples require less irradiation for D_e determination, so that the effect of the disc signal is less pronounced. However, when measuring very dim samples or for basic studies, one should always be aware of the 'disc problem' and its influence on weak luminescence signals.

The contribution of the disc signal to the entire recorded signal depends only weakly on light shielding by the grain-covered area of the disc for coarse grains (Fig. 2), but shielding is likely to be more effective for fine grain layers. Correction for disc influence requires careful investigation of each individual disc, especially in the case of Al discs. Consequently, alternative and chemically more inert disc materials (nickel, silver, rhodium, gold etc.) should be investigated in detail.

Conclusions

Spurious and dose-dependent TL and OSL emissions from commonly used sample carriers were investigated in this study. We observed:

1) Al discs showed significant TL and OSL signals in the UV, UV-blue and red detection range.
2) Steel discs showed no significant spurious but dose-induced signals in the UV and UV-blue windows.
3) For Al cups (from Bayreuth) we found no spurious signals for UVTL and generally low sensitivity to irradiation.
4) The provided cleaning procedures for discs (Al and steel) can reduce spurious and dose-dependent signals but cannot eliminate them completely.
5) For most bright samples, the influence of disc signals is expected to be negligible but further investigation for the case of single grain discs is needed.

These phenomena seem to be widespread among laboratories as they were observed in at least three luminescence dating facilities and with various kinds of sample holders. However, this paper is not a comprehensive luminescence study of specific disc materials. The influence of spurious and dose-dependent signals on particular measurements is different for each disc and sample and difficult to assess because of highly differing characteristics between discs. In short, the résumé of this paper is to alert the reader to the problem of unwanted luminescence contributions from the sample holder and to advise careful measurement of these signals when measuring relatively young and/or dim samples (including single grains).

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