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ESTIMATING MAXIMUM TEMPERATURES ATTAINED DURING FIRES IN BUILDING STONEWORKS BY THERMOLUMINESCENCE: A CASE STUDY FROM UNCASTILLO, SARAGOSSA (SPAIN)

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ABSTRACT

Knowing the maximum temperatures attained by stonework during past fires in historical buildings is important to understand the damage caused to the materials and the subsequent weathering history after the fire. Thermoluminescence (TL) provides a tool to assess such temperature but different protocols exist. TL has been tested to assess the maximum temperature reached by a past fire on the surface of calcitic sandstone (frequently used in historical buildings in Saragossa, Central-Eastern Spain). We have prepared subsamples of this sandstone annealed from 200°C to 700°C. Quartz extracts from such samples were used for testing different TL protocols, from measuring the erosion of the whole TL glow curve of quartz extracted to test the Thermal Activation Characteristic (TAC) and other more recent approaches, such as comparing the sensitization of the integrated 110, 200 and 250-400°C peaks at different irradiation doses or a full predose protocol. The erosion of the TL glow curve of quartz seems to underestimate the attained temperature while the other tested protocols indicate that the temperature reached was 400°C.

KEYWORDS: historical fire, TL, quartz, firing temperature

1. INTRODUCTION

When looking back at the weathering history of a building, finding fires as a part of this history is not uncommon. Fire stands among the main catastrophic weathering agents for building stonework. Thus, dating of fires and knowing the maximum temperatures attained by stony materials during them will provide with valuable piece of knowledge towards understanding the level of damage caused.

Different methods have been tested to assess the maximum temperature reached by fires and the damage caused on building materials (see table 1).

Table 1. Summary on the methods proposed to assess the maximum fire temperature attained during accidental fires.

Method	Technique	Material	Paper
Color assessment	Image analysis	Mortar	Deng-Fong et al., 2004
Compressive strength	No data	Several stone types	Chakrabarti et al., 1996
Compressive strength, porosity		Concrete	Poon et al., 2001
Mineralogical-morphological measurements	DTA-TGA, XRD, SEM	Concrete	Handoo et al., 2002
	DTA-TGA	Cement pastes	Alarcón-Ruíz et al., 2005
Structure	Ultrasonic tests	Reinforced concrete	Cioni et al., 2001; Colombo and Felicetti, 2007
Color assessment	Image analysis	Concrete	Short et al., 2001
Petrographic examination, crack density	Microscopy	Concrete	Georgali and Tsakiridis, 2005
Mineralogy, colour changes	Microscopy, image analysis	Sandstone	Hajpal and Török, 2004
Petrographic examination	Microscopy	Concrete, limestone and lime mortar	Ingham, 2009
Physical properties	Schmidt hammer, microscopy, mechanical tests	Marble	Koca et al., 2006
Luminescence signal	Thermoluminescence	Concrete	Placido 1980; Chew et al., 1987 ; Pei et al., 1997

For stone, a number of physicochemical analyses are suitable for the point-by-point inspection of damage. Some of them provide information on the maximum temperature attained, and namely X-ray diffraction, Differential Thermal Analysis and Thermo-Gravimetric Analysis have been commonly used. Mineralogical analyses allow to roughly assess maximum temperature attained on the surface of stone during a fire rarely rise above 600°C (Gomez-Heras et al., 2008; 2009).

Below such temperature, the damage caused strongly depends on the maximum temperature, the heating time and the stone characteristics. In historical masonries, different marks of fire on blocks can be frequently observed (reddish and eroded blocks) and stones show variable states of preservation. The heating time can be more or less similar but strongly different temperatures can be reached on the different surfaces of blocks, as fire and heat transfer is difficult to predict (Sacadura, 2005; Viskanta, 2008). As the subsequent decay of the stone strongly depends on the maximum temperature, it would be very important to have a method to assess such temperature.

Thermoluminescence (TL) assessment of firing temperature has been tested on concrete by comparing the erosion of the TL glow curve to polymineral samples at different depths (Placido 1980; Chew et al., 1987; Pei et al., 1997). This approach has provided valuable information on the effect of fire at different depths and the maximum temperature reached with low precision, as the erosion is also strongly dependent on the firing duration.

Specific TL protocols have also been used to assess the firing temperature of archaeological materials. Namely, the TL of quartz has been used to assess the temperature reached during intentional firing in the past on archaeological burned flints and pottery (Sunta and David, 1982; Aitken, 1985, Göksu et al., 1989). The TL glow curve of quartz shows four main peaks located at 110, 200, 325 and 375°C (Franklin et al., 1995). The 110°C peak has a half-life at room temperature of about two hours. It was early studied by Zimmerman (1971) who explained some characteristics of the pre-dose sensitization process.

Sunta and David (1982) used such characteristics to assess the firing temperature of quartz by using a pre-dose based technique, but Watson and Aitken (1985) and later on Koul et al. (1996) and Koul (2006) showed that it was not generally applicable.

Serious considerations have been made concerning the feasibility of the method as the preservation of this memory may be diluted with time, although the firing episode registers its effect in the quartz. The role of alkali ions was postulated to limit the capacity of the 110°C peak of quartz to remember the firing temperature using pre-dose sensitization technique (Koul, 2006).

However, the luminescence characteristics of the 110°C peak can be correlated to the thermal history of the quartz sample, although not only the firing temperature must be considered, but also the time or firing, firing and cooling rates and the heating environment (Aitken, 1985; Bøtter-Jensen, et al., 1995; Charitidis et al., 2000; Roque et al., 2004). The sensitization of the 110°C TL peak of quartz occurs within the temperature range between 200°C and 900-1000°C. This is the temperature interval at which most building fires occur. In a recent paper, Gartia (2009) studied paleothermometry of NaCl as evidenced from TL data and discussed possible implications in paleogeology and paleogeography.

Charitidis et al. (2000) investigated the sensitivity variation of all TL glow-peaks of quartz (they used synthetic quartz), which are highly dependent on the firing temperature (Chen et al., 1988). Charitidis et al. (2000) showed that the sensitivity of such glow-peaks is highly increased as the firing temperature increases: the rate of increase is low up to the firing of 500°C and very high for firing temperatures above 600°C. They observed that increase in the 110°C glow-peak dose response is highly superlinear. The glow-peaks at 200°C and the integral containing the high temperature glow-peaks (325°C and 375°C) dose responses are sublinear below 60 Gy, and became superlinear at higher doses. As an alternative method, Roque et al. (2004) used the entire TL glow curve to plot the ratio of the regenerated glow curve intensity to the natural one as a function of temperature to assess the annealing conditions, for fine quartz and feldspars grains of ancient pottery, with successful results.

Polymeris et al. (2007) investigated the possibility of using both TL and the optically stimulated luminescence (OSL) for monitoring the sensitization of quartz extracted from a fired ceramic, attributed to the annealing treatment only, the thermal activation to 500°C and the pre-dose effect. They found a relation between both TL and OSL sensitivities and the firing temperature and suggested that either luminescence technique would be able to assess the firing temperature.

The aim of this work is to assess by solely applying TL, the firing temperature at which the surface of a specific sandstone was heated. The rock used for test is the 'Uncastillo sandstone', commonly used continuously for construction of buildings from Roman times onwards in the region of Saragossa (Spain).

This is calcitic sandstone (Fig. 1) with a variety of quartz grain types (mono, polycrystalline and chert). This brown sandstone shows intense discoloration after fires and therefore facilitates to identify areas fired in the past. Studies on the maximum temperature attained by fires on similar sandstones have been reported by methods alternative to TL (Hajpal and Torok, 2004).

They reveal that elevated temperatures influence the texture and strength of the sandstone. In particular, cracks are generated in the calcite-rich cementing matrix related to shrinkage of clay minerals due to the loss of structural water at 450°C and more micro cracks at grain boundaries are observed when heating above 600°C.

We compare this information with our TL results. The TL was measured on subsamples of the sandstone taken from the historical quarry. A test sample from a straw loft fired accidentally in the 1990s (to an unknown temperature) was used to assess the temperature reached during a past fire. This sample was selected to avoid unnecessary sampling of heritage properties.

Different approaches have been considered to estimate the firing temperature, since unlike ancient fired ceramics the fire was accidental: it is expected to have reached a relatively low temperature (ca. 200-600°C), and to have heated the rock surface for a short timespan (minutes rather than hours) (Liritzis, 1982).

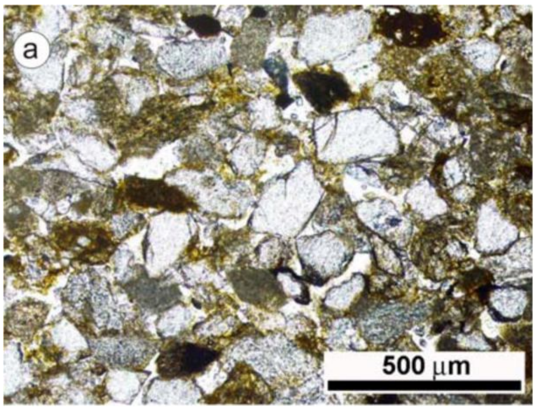

Figure 1. Cross-section slide of 'Uncastillo' calcitic sandstone.

2. METHODS

5 cm cubes of sandstone taken from the quarry were heated in an oven at different temperatures from 200°C to 700°C (in steps of 100°C) for 8 hours. These were used as subsamples. Moreover, a sandstone sample was collected in a building of the village of Uncastillo (Saragossa, Spain). Such sample was affected by a fire some years ago, with its surface layer (of 1 cm thick) showing a reddish color as a result of the fire damage. Quartz grains from all subsamples as well as from the main sample were extracted after cutting and gentle crushing by squeezing in a vice.

The powdered material was sieved to obtain the grains size fraction of 90-180 μm. The grains were treated with HCl to eliminate carbonates. The quartz fraction was separated by applying successive (1) etching with HF 20%, (2) gently mortar grinding, (3) HF 20%, (4) gently mortar grinding, (5) HF 20%, (6) and gently mortar grinding again, following the procedure proposed by Fernández Mosquera and Sanjurjo Sánchez (2008).

Quartz grains were mounted on stainless steel cups for TL. TL measurements were performed on a Risø TL\OSL DA-15 automatic reader, equipped with calibrated $^{90}Sr/^{90}Y$ beta source of 0.13±0.003 Gy s^{-1} dose rate. All TL signals were detected through a 7.5-mm thickness Hoya U-340 filter using 9635Q photomultiplier. The transmission characteristics of this filter drop rapidly for wavelengths above 360 nm, reaching 10% by 380 nm.

All aliquots were checked with infrared (IR) stimulation at ambient temperature to ensure the absence of any feldspar contamination. Four different protocols were applied to assess the firing temperature:

Protocol 1 (adopted from Placido, 1980)

The natural TL glow curve was measured on the quartz of all heated and unheated sandstone cubes. TL measurements were performed up to 500°C at a heating rate of 5°C s^{-1}. Subsequently, another TL measurement was performed to get a background curve (for background subtraction).

The curves were compared to assess the erosion at each annealing temperature. The quartz TL glow curve of the test sample was also measured and compared with the TL from subsamples to assess a probable temperature interval.

Protocol 2 (according to Charitidis **et al.**, *2000)*

The quartz from the subsamples and the main sample were irradiated with beta doses of 6, 12, 24, 48, 96, 140 and 192 Gy.

After irradiation, prompt TL measurements were performed up to 500°C at a heating rate of 5°C s^{-1}. The 110°C, 200°C glow-peaks and the 250-400°C area of the curve were integrated to build plots of TL vs. irradiation dose and TL vs. annealing temperature.

The TL vs. irradiation dose plot was used to compare the growth curve of the samples annealed at different temperatures and the main sample, while the TL vs. annealing temperature was used to compare the sensitization of the integrated peaks at different irradiation doses.

Protocol 3 (Aitken, 1985)

The thermal Activation Characteristics (TAC) of the quartz extracted from the test sample were deducted. The following steps were applied:

1. Administration of beta test dose (0.4 Gy)
2. TL up to 160°C (1°C s^{-1} heating rate)
3. Administration of 25 Gy beta dose
4. TL up to 450°C (1°C s^{-1} heating rate)
5. Repeat of steps 1-2

Protocol 4 (Polymeris* et al.*, 2007)

This test consists on a full pre-dose protocol. 5 discs with 5 mg of quartz grains of the test sample were mounted on steel cups. The normalized TL sensitivity versus annealing temperatures for the test sample quartz, will deal with pure thermal sensitization, TAC sensitization and pre-dose sensitization of both the 110°C TL peak and the high temperature peak glow curve part.

The following steps were applied:

1. Annealing to a temperature between 200°C to 600°C in 100°C steps.
2. Administration of beta test dose (2 Gy)
3. TL up to 500°C (1°C s^{-1} heating rate)
4. Administration of beta test dose (2 Gy)
5. TL up to 500°C (1°C s^{-1} heating rate)
6. Administration of 50 Gy beta dose
7. TL up to 500°C (1°C s^{-1} heating rate)
8. Administration of beta test dose (2 Gy)
9. TL up to 500°C (1°C s^{-1} heating rate)

3. RESULTS AND DISCUSSION

Besides the TL results, mineralogical changes have been observed in the annealed subsamples during the quartz extraction. The steps considered to extract quartz, showed that clean quartz was obtained after the step 2 in the subsamples annealed to 500°C upwards, while samples annealed up to 400°C or lower temperatures were clean after step 6 of the procedure proposed by Fernández Mosquera and Sanjurjo Sánchez (2008). This is probably due to the effect of heating. The obtained clean quartz was also higher (approximately double relative quartz content) in the samples annealed to 500°C upwards (Fig. 2), while the calcite content remains constant, as this mineral disintegrates above 750°C. This is probably related to cracks caused by the shrinkage of clay minerals in the calcite-rich cementing matrix due to the loss of structural water at 450°C.

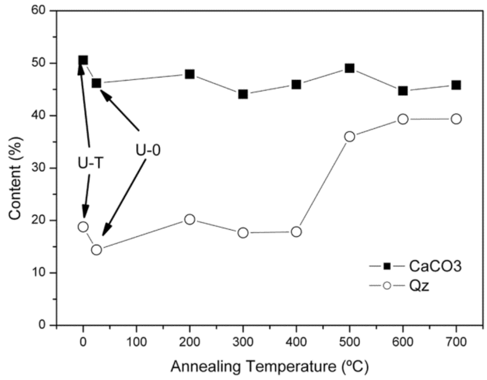

Figure 2. Plot of approximate content in calcite (squares) and quartz (circles) of the subsamples vs. the annealing temperature (U-T: test sample; U-0: unheated subsample).

The comparison of natural TL curves of the quartz from the fired and unheated subsamples (protocol 1) with the test sample (erosion of the TL glow curves) provides a possible maximum temperature of the fire on surface near 300°C (Fig. 3).

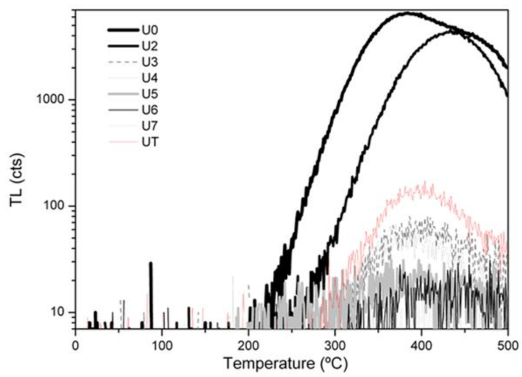

Figure 3. Erosion of the TL glow curve after annealing at different temperatures.

Comparing TL glow curves generated after different beta doses and annealing temperatures (protocol 2), plots of TL response vs. different annealing temperatures were constructed (Fig. 4). Such plots indicate that the sensitivity of the 110°C peak is higher at low doses at low annealing temperatures (200 to 400°C) while differences are not observed at high annealing temperatures (above 500°C). The 200-210°C glow peak and the 250-400°C interval showed important sensitivity changes at higher doses and higher annealing temperatures (from 400 to 700°C) and specially, at a dose of 48 Gy.

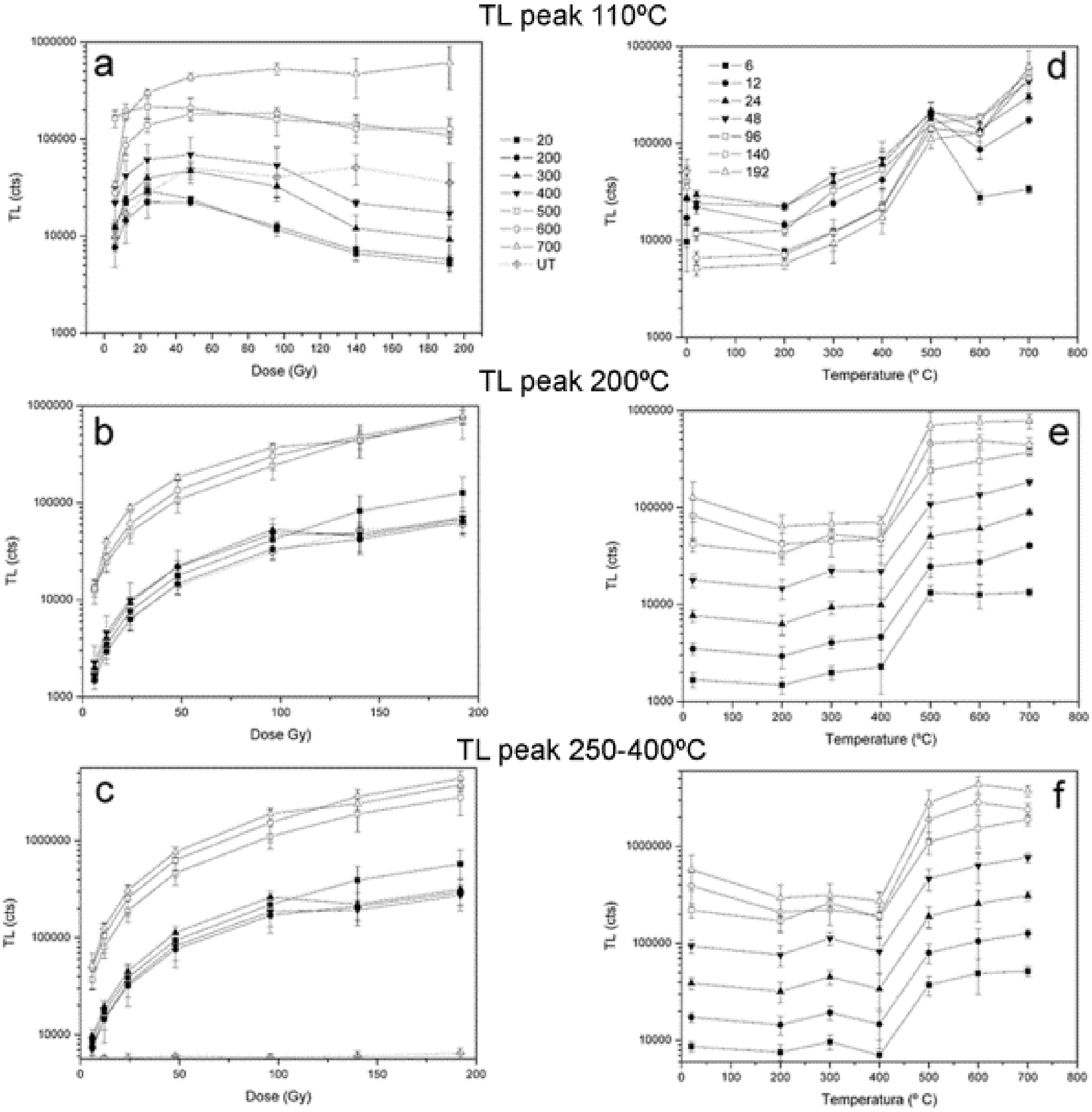

Figure 4. Plots of TL growth curves for the integrated peaks of subsamples and the test sample after irradiation with growing beta dose (a, b and c), and sensitization at different temperaturas (d, e and f). U-T: test sample.

The 110°C TL peak shows a sublinear saturating dose response. At low annealing temperatures (below 500°C) the peak dose response decreases with increasing doses above 48 Gy. The high temperature peaks (200°C and 250-400°C) show a superlinear behaviour after annealing at 500°C and above this temperature, but also superlinear dose response at high doses whether they have been annealed or not.

From these plots we can conclude that doses between 24 and 96 Gy are the most suitable to calculate firing temperatures by building up reference dose growth curves. The behaviour of the peak 110°C allows us to measure firing temperatures by building up a curve (and calculate the slope) between 200°C and 400°C annealing temperatures. The behaviour of the peaks 200-210 and 350°C allows constructing curves between 400°C and 700°C, at the same doses, to build up a high temperature calibration curve. Interpolation of the TL measured at these peaks after the suggested doses could provide the firing temperature of sandstone samples exposed to past fires.

As Fig. 4 reveals, the subsamples were also subjected to heating at around 400°C in the past.

In case of the lower re-firing temperatures, in the range of 200 to 400°C, the glow curves exhibit no significant changes, neither in shape nor in the intensity.

However, the intensity of glow curves measured after re-firing at higher temperatures, i.e. 500-700°C, was seen to be increasing with the increase in the re-firing temperature and thereby indicating the enhancement in the sensitization. This change has been observed for all glow peaks, including the 110°C (plots d, e and f). Furthermore, plots a, b and c indicate that this is the case for the intensity of each dose applied in the framework of the dose response study.

The TAC test (protocol 3) provided a probable maximum temperature near 400°C (Fig. 5).

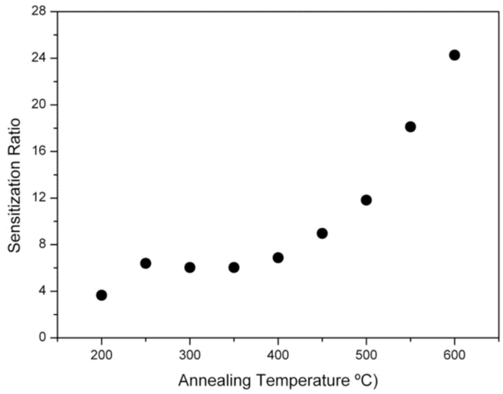

Figure 5. Results of the TAC performed on the test sample

This result is very similar to the results provided by the test of Polymeris et al. (2007). In such latter test, 110°C TL integrated signal was plotted versus the re-firing temperature for the sandstone material of the subsamples (Fig. 6).

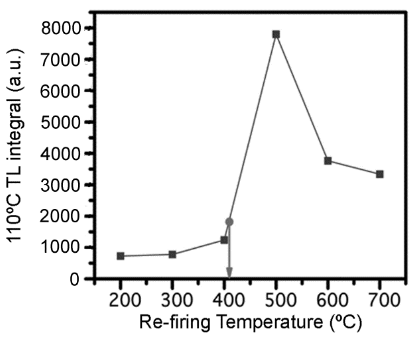

Figure 6. 110°C TL integrated signal versus re-firing temperature for the sandstone material of the subsamples (squares). The subsamples were also fired in the past at 400°C. By interpolating the intensity measured using the test sample into the calibration curve (circle) the firing temperature of the latter is also estimated at 400°C.

However, results of the protocol 4, similarly to the corresponding results of the protocol 2, suggest that the subsamples were also fired in the past at 400°C. By interpolating the intensity measured using the test sample into the calibration curve the firing temperature of the latter is also estimated at 400°C. This question poses a problem to assess our results that still must be taken with care. Further tests are needed with completely unfired material. At this point, we do not know what has occurred with the quarry samples to explain this result, but it is possible that a wildfire occurred in the past in the quarry front, as wildfires were relatively frequent in the area in the last decades. Some considerations can be extracted from this experience for next tests:

(a) Completely unfired samples must be used and more subsamples should be prepared for annealing test in shorter steps (e.g. 50°C).

(b) It is advisable to use thinner layers from the stone surface to depth (e.g 0.2 mm thick) and to assess the attenuation of the firing temperature with depth.

4. CONCLUSIONS

Accidental fire is the cause of irreversible damage on building stonework. A number of physicochemical analyses are suitable for assessing both the maximum temperature attained and the damage caused by such fires, but TL can be a very precise method. In the present work, the application of different TL protocols was studied towards assessing the temperature attained during a fire, in quartz extracted from calcitic sandstone.

The erosion of the TL glow curve of quartz provides information on the effect of fire at different depths in the rock, but seems to underestimate the attained temperature, as the erosion is also strongly dependent on the firing time. The Thermal Activation Characteristics tests seems to be more suitable and provides the maximum temperature attained (near 400°C in our test sample). However, the use of all the TL glow-peaks (approach of Charitidis et al., 2000) and the use of the entire TL glow curve (Polymeris et al., 2007) indicates that both the test sample and the subsamples were heated at around 400°C in the past. The results of the present study reveal the usefulness of the sensitization of the entire TL glow curve.

Nevertheless, further work is required with unfired sandstone to assess the reliability and precision of each protocol. Such work should additionally consider possible alterations in the shape of the glow curve, the use of more sub-samples, annealed in shorter steps (e.g. 50°C) and the use of thinner surface-depth layers (e.g 0.2 mm thick) to assess the attenuation of the firing temperature with depth.

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