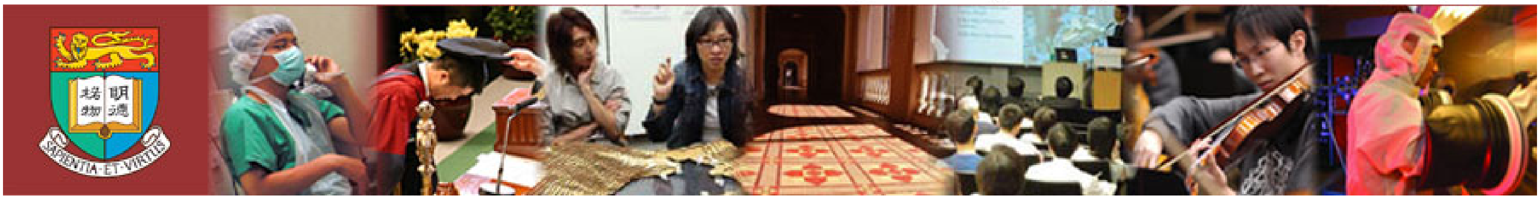

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Author(s)	**Liu, Z; Zhao, H; Wang, CM; Li, SH**
Citation	**Quaternary Geochronology, 2015, v. 30 n. pt. B, p. 363-368**
Issued Date	**2015**
URL	**http://hdl.handle.net/10722/216830**
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Estimation of Paleo-firing Temperatures Using Luminescence Signals for the Volcanic Lava Baked Layer in Datong, China

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Abstract: Eight paleo-fired samples from the baked layer in different depths under the lava and one unfired sample were collected from Datong, China. Fine quartz grains (4-11 μm) from samples were used for probing into relationship between luminescence signals and paleo-firing temperatures. Findings from the re-firing experiments indicated that using thermoluminescence (TL) and optically stimulated luminescence (OSL) sensitivity changes could estimate the paleo-firing temperatures of samples: (1) 110 ºC TL sensitivity change rate against the re-firing temperature can tell whether the sample has been fired to temperatures above 500 ºC or not; (2) 150 ºC TL sensitivity against the re-firing temperature can indicate whether the sample has been fired to temperatures above 900 ºC or not; (3) the more specific paleo-firing temperatures can be estimated by comparing the ratio of OSL and 150 ºC TL sensitivities against re-firing temperatures. Results showed that the paleo-firing temperatures of the eight lava-baked samples decreased exponentially with the distance from the lava. Based on the estimated temperature profile, the temperature of the lava was estimated to be about 1100 ºC.

Key words: Datong volcano; baked layer; temperature; luminescence; sensitivity; fine grain quartz

1. Introduction

Deducing the temperatures of the lava flows is of great significance for analyzing the flowing process of lava (Griffiths, 2000; Pinkerton et al., 2002), which helps to define the extents and degree affected by volcanoes. The paleo-firing temperature estimation of the baked layer beneath the lava can provide a possibility for assessing the temperature of the lava above. The estimation of paleo-firing temperatures also provides the information of the degree of zeroing for heated samples, which is essential for luminescence dating.

Several studies showed that luminescence sensitivity changes of quartz were related to the thermal history (Bøtter-Jensen et al., 1995; Wintle and Murray, 1999; Han et al., 2000; Poolton et al., 2000; Schilles et al., 2001; Li, 2002; Polymeris et al., 2006; Oniya et al, 2012). Therefore, many attempts using TL and OSL sensitizations of quartz were made to acquire paleo-firing temperatures. Sunta and David (1982) measured the sensitivity of the 110 ºC TL peak before and after the application of the pre-dose at different re-firing temperatures. They observed that for pre-fired samples the ratio of the latter to the former remained constant until the firing temperature was attained. The ratio began to increase significantly when the heating temperature was higher. Göksu et al. (1989) applied this method to determine the ancient heat treatment of flint. However, Watson and Aitken (1985) observed that the procedure developed by Sunta and David (1982) was not generally applicable. Their studies showed that the sensitivity of the 110 ºC and 230 ºC TL peak, respectively, may be associated with the maximum temperature the quartz had experienced. Therefore, to some extent, the quartz can preserve a memory of its thermal history. Polymeris et al. (2007) observed a relationship between TL and OSL sensitivities, and concluded that they could be used to assess paleo-firing temperatures. It was reported that samples that have been heated to different temperatures will display different in the plots of the sensitivity versus the re-firing temperature. However, most of the studies were carried out on archaeological samples with small palaeo-doses. The potential for assessing the paleo-firing temperatures based on luminescence sensitivity still needs further studies, particularly for geological samples.

In this paper, we implemented experiments to acquire the relationship between the luminescence sensitivity and firing temperatures. Methods determining the paleo-firing temperatures were established and applied to the samples from a lava-baked layer in Datong, China.

2. Samples and equipment

Datong volcanic group is located in the Datong Basin in the northern part of China. More than 30 volcanoes erupted in the area of about 900 km^2. The volcanic eruptions in this region lie between Quaternary loess layers or lacustrine sediments. Samples used in this study were collected from Yujiazhai profile. The lava in this area has an age of about 300 ka (Li and Sun, 1984). The lacustrine sediments that have been baked by the lava flow had a distinct red color compared with the unfired layer. Eight paleo-fired samples from the lava-baked layer and one unfired sample beneath the baked layer (No: YJZC-7) were collected (Fig. 1). The unfired sample was used to heat to different known temperatures for the investigation of the link between the luminescence sensitization and firing temperatures in the laboratory. A portion of the unfired sample was used as a reference for comparisons.

Sample preparation was carried out under red light in the laboratory. All the samples were treated with 30% H_2O_2 and 30% HCL to remove the organic materials and the carbonates. They were treated with 30% fluorosilicic acid for 5 days for the removal of feldspar contamination. After that, fine grains in the range 4-11 μm were selected according to Stoke's Law using acetone and deposited on stainless steel discs. The purity of the quartz was checked with infrared stimulation (Li et al., 2002).

Luminescence measurements were performed using a Daybreak 2200 TL/OSL automatic system. A ^{90}Sr/^{90}Y beta source, delivering ~0.05 Gy s^{-1}, to quartz in stainless steel discs, was used for irradiation. The luminescence signals were detected through two 3 mm U-340 filters in all observations. A SX2-10-13 muffle oven with the maximum temperature of 1350 ºC was used for heating the samples to different temperatures in the laboratory.

Throughout the paper, laboratory heated samples are referred to as fired samples, the temperatures as the firing temperatures. The samples from the lava-baked layer are called paleo-fired samples, heated to paleo-firing temperatures. Heat treatment to the fired or paleo-fired samples in the laboratory is referred to as re-firing.

3. Experimental procedures

A portion of the unfired sample was heated to different temperatures. The unfired fine-grain quartz sample was divided into 12 subsamples. Eleven were heated to various temperatures ranging from 100 ºC to 1100 ºC in steps of 100 ºC for 15 minutes in the muffle oven, and then cooled naturally. The sample without any thermal treatment was used as a reference for comparison.

The 12 fired samples and the 8 paleo-fired samples were used for re-firing experiments. Each sample was divided into 8 subsamples for re-firing at temperatures ranging from 300 ºC to 1000 ºC in steps of 100 ºC for 15 minutes in the muffle oven. Then, thermoluminescence (TL) and optically stimulated luminescence (OSL) signals induced by a dose of 10 Gy were measured. A heating rate of 5 ºC/s was used for the TL measurements to 260 ºC. The OSL signals were measured at 125 ºC for 100 seconds. The sensitivity of 110 ºC TL, 150 ºC TL, and OSL signals were obtained.

4. Results

4.1 110 ºC TL sensitivity against re-firing temperature

The 110 ºC TL sensitivity against re-firing temperature of fired samples are illustrated in Fig. 2a. For samples that have been heated to 500 ºC and lower, the 110 ºC TL sensitivity increases sharply when the re-firing temperature was around 500 ºC. It decreases slightly for the temperature above 500 ºC. For fired samples that have been heated to more than 500 ºC, it remains unchanged. Therefore, if there is an obvious peak around 500 ºC in the 110 ºC TL sensitivity curve, it indicates that the sample has not been heated to temperatures higher than 500 ºC. On contrary, if there is no obvious peak at about 500 ºC in the 110 ºC TL sensitivity curve, the sample might have been heated to temperatures higher than 500 ºC.

We applied the observations above to the paleo-fired samples. The 110 ºC TL peak sensitivity against re-firing temperature of the paleo-fired samples are shown in Fig. 2b. All the curves had a peak at about 500 ºC. However, from field observations and studies, some of the samples were certainly heated to temperatures above 500 ºC. Studies have shown that the TL sensitivity of quartz increases proportionally to the dose received before the thermal activation around 500 ºC, which is called pre-dose effect (Aitken, 1985). After being paleo-fired, the natural samples were naturally given large pre-doses during the process of sedimentation, before the re-firing in the laboratory. This pre-dose effect causes the TL sensitivity to increase significantly at the re-firing temperature of 500 ºC for the paleo-fired samples, even for the samples have been paleo-fired at temperatures higher than 500 ºC. The pre-dose effect affects the judgment on whether the paleo-firing temperature was above 500 ºC or not. Therefore, we conclude that using the 110 ºC TL sensitivity alone is not sufficient for indicating the paleo-firing temperatures, because it is not only affected by the thermal history, but also the pre-dose effect (Li, 2002).

4.2 110 ºC TL sensitivity change rate against re-firing temperature

Several studies have investigated the pre-dose effect in quartz (Zimmerman, 1971; McKeever, et al., 1985; Yang and McKeever, 1990; Rendell, et al., 1994; Li, 2002). The pre-dose effect is particularly important for the paleo-fired samples because they are about 300 ka of age, of equivalent to a dose of 1300 Gy. The pre-dose effect gives rise to the different responses for the fired and paleo-fired samples as shown in results in section 4.1. We define the sensitivity change rate as the ratio of sensitivity at a temperature and the sensitivity of the temperature 100 ºC lower of the same sample. For example, the sensitivity change rate at 600 ºC is the ratio of the 110 ºC TL sensitivity at 600 ºC against the corresponding value at 500 ºC.

The 110 ºC TL sensitivity change rates against re-firing temperature for the fired samples are shown in Fig. 3a. For samples that have not been heated to more than 500 ºC, an obvious peak at about 500 ºC appears in the curve of

110 ºC TL sensitivity change rate versus re-firing temperature, but does not appear for samples that have been heated to more than 500 ºC.

The relationship between 110 ºC TL sensitivity change rate and re-firing temperature for the paleo-fired samples are shown in Fig. 3b. The curves have a peak at about 500 ºC for the samples with a distance of more than about 100 cm from the lava, but the peak does not appear for the samples of less than about 100 cm from the lava, except for a sample of 10 cm away from the lava. We deduce that the samples of less than about 100 cm from the lava had been heated to more than 500 ºC, while the samples with a distance of more than about 100 cm from the lava had not. The exception of the sample 10 cm from the lava will be discussed later.

4.3 150 ºC TL peak sensitivity against re-firing temperature

The 150 ºC TL peak sensitivity changes against re-firing temperature of fired samples are shown in Fig. 4a. A large difference between the samples of the firing temperatures lower than 900 ºC or above was observed. It has indicated that curves have a distinct peak at about 900 ºC for samples that have been heated to temperatures lower than 900 ºC. No peak was observed for samples that were heated to 900 ºC or above. This difference offered a means for distinguishing samples of the firing temperature below and above 900 ºC.

The 150 ºC TL peak sensitivity changes against re-firing temperature of the paleo-fired samples are plotted in Fig. 4b. The curves of the three samples near the lava, with the distance of 10, 40, 55 cm from the lava, do not have a peak at about 900 ºC, which suggests that the samples have been heated to 900 ºC or above. However, other samples (except one 128 cm away from the lava) have a peak at about 900 ºC, which implies that the samples have not been heated to 900 ºC.

4.4 OSL/150 ºC TL sensitivity against re-firing temperature

The ratio of OSL sensitivity and 150 ºC TL sensitivity (OSL/150 ºC TL sensitivity) against re-firing temperature of fired samples are displayed in Fig. 5. It shows that each curve of fired samples is separated from the curve of unfired sample (YJZC-7) before the re-firing temperature reaches the temperature the sample has been heated to. The two curves are overlapping with each other for the re-firing temperature above. The method is best suitable for samples fired 500 ºC and above.

OSL/150 ºC TL sensitivity changes against re-firing temperature of the lava-baked samples are illustrated in Fig. 6. Each curve of the paleo-fired samples is compared with the curve of the unfired sample. Some of them do not have overlaps, however, there is an intersection, after which, the two lines have the similar trend of changing with temperature. The temperature of the intersection is regarded as the paleo-firing temperature. Using this method, the temperatures of the samples were acquired as shown in Table 1. We consider that there are differences between natural and laboratory simulation. Complicated influencing factors were involved for natural samples compared to the fired samples.

From the data illustrated in Table 1, the paleo-firing temperatures of the eight lava-baked samples decrease exponentially with the distance from the lava (Fig. 7). The results show that the sample of 10 centimeters away from the lava might have been fired to more than 1000 ºC. The sample of 158 centimeters beneath the lava might have been heated to about 300 ºC. Based on the estimated temperature profile, the temperature of the lava above was about 1100 ºC (Fig. 7). We would like to point out that all the re-firing treatments were carried out in the muffle oven for 15min in the laboratory, which was shorter than the baked time by lava in nature. This may lead to overestimation of the firing temperature (Han et al., 2000).

5. Discussions

Our observations showed that the luminescence sensitization had a close relationship with the firing temperature. The findings from the re-firing experiments indicated that TL and OSL sensitivity changes against the re-firing temperature can be used to estimate the paleo-firing temperatures of samples. The results obtained by using the three methods, which utilizing 110 ºC TL sensitivity change rate, 150 ºC TL sensitivity and the ratio of OSL/150 ºC TL sensitivity, can be well compared with each other. The 110 ºC TL sensitivity is able to distinguish whether the sample has been fired to temperatures above 500 ºC or not for the fired samples. However, the 110 ºC TL signal is pre-dose dependent (Li, 2002), therefore, the sensitivity change of each re-firing temperature may give a spurious result in the temperature estimation for the paleo-fired samples. The pre-dose effects can be minimized by using the 110 ºC TL sensitivity change rate, because the same pre-dose was applied for all re-firing temperatures of a sample. The 150 ºC TL sensitivity can be used to identify samples that have been heated to ≥ 900 ºC.

By using the ratio of OSL sensitivity to 150 ºC TL sensitivity, we can estimate the paleo-firing temperature to which the sample has been heated. It can be obtained by comparing the OSL/150 ºC TL sensitivity versus re-firing temperature curve of the paleo-fired samples with that of the unfired sample. Both OSL and 150 ºC TL sensitivity have also been affected by pre-dose effect (Li and Chen, 2001). Both signals have different responses to thermal treatment. Ratio matching provides a way of demonstrating the thermal effect. We interpret that the OSL/150 ºC sensitivity ratio has combined both effects of OSL sensitivity and 150 ºC TL sensitivity to re-firing temperatures. The combination can give a better resolution to the paleo-temperature of sample being heated. We deduce that the ratio of OSL/150 ºC TL sensitivity is dominated by the maximum temperature the sample had experienced. When the re-firing temperature is lower than the fired temperature, the maximum temperatures of the unfired and fired sample are different, so that the two lines are separating. When the re-firing temperature is higher than the fired temperature, the max temperatures of the unfired and fired sample are the same, so that the two lines are overlapping. Hence, the temperature can be constrained by comparing the curve of fired/peleo-fired samples and the unfired one.

The peaks at curve of 110 ºC TL and 150 ºC TL sensitivity against re-firing temperature indicate that the phase changes shift from α-quartz to β-quartz at 573 ºC, and β-quartz to β-tridymite at 870 ºC occurred. The 110 ºC TL sensitivity rate increases significantly to a peak around 500 ºC for samples that have heated at less than 500 ºC, i.e. α-quartz. For samples heated to 500-900 ºC, i.e. β –quartz, the 110 ºC TL sensitivity change rates do not have a peak at about 500 ºC. For the sample that has been heated to above 900 ºC, i.e. β-tridymite, the 110 ºC TL sensitivity change rates have a peak around 500 ºC. This may explain the exception of the sample 10 cm away from the lava, which has similar behavior as samples heated below 500 ºC in the 110 ºC TL sensitivity change rate curves (Fig. 3b). The sample was heated to the highest temperature among the lava-baked samples, because it is the closest to the lava. The result from the OSL/150 ºC TL sensitivity against re-firing temperatures suggests that this sample was heated to temperature above 1000 ºC. Similarly, the phase changes of quartz have been demonstrated in the 150 ºC TL sensitivity. It is noted that quartz turns back to α-quartz when it cooled to temperature of 573 ºC or lower. However, the sensitivity changes of quartz luminescence signals are irreversible. It was explained as a result of transferring holes from non-luminescence traps to luminescence traps (Zimmerman, 1971).

It has been demonstrated that the paleo-firing temperature estimation using the 110 ºC TL signal is precluded by the pre-dose effect. However, the pre-dose effect can be minimized when using the rate of the 110 ºC TL sensitivity change, because the same pre-dose is applied to the sample. Only the thermal effect will dominate the 110 ºC TL sensitivity change rate. Similarly, the ratio of OSL sensitivity and 150 ºC TL would have small impacts of pre-dose, because the pre-dose effects affect both of the signals (Chen et al., 2000). Another advantage of using the ratio is that no normalization is required for the aliquots.

6. Conclusions

Luminescence signals of 110 ºC TL, 150 ºC TL and OSL of quartz have close relationship with the paleo-firing temperatures. The sensitivity of the signals can be affected by their thermal and ionizing radiation histories. The pre-dose effects on the signals can be minimized when rate or ratios are used for thermal history study. Significant changes in luminescence sensitivity happened at temperatures that are coincident with phase change temperatures of the quartz.

Acknowledgements

We thank Prof. Yan-Chou Lu, Xu-Long Wang and Jin-Feng Liu for very helpful comments. We thank the anonymous referee for the constructive comments. This work was supported by NSFC grant (40972208) and China Geological Survey grant (1212011120147). This study was financially supported by the grants to Sheng-Hua Li from the Research Grant Council of the Hong Kong Special Administrative Region, China (Project no. 7028/08P, 7033/12P and 17303014).

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Table 1

Paleo-firing temperatures of the paleo-fired samples from the baked layer at Datong

Distance from the lava(cm)	Results from 110 ºC TL peak	Results from 150 ºC TL peak	Results from OSL/150 ºC TL peak
10	<500 ºC	≥900 ºC	>1000 ºC
40	≥500 ºC	≥900 ºC	800 ºC
55	≥500 ºC	≥900 ºC	700 ºC
81	≥500 ºC	<900 ºC	550 ºC
97	≥500 ºC	<900 ºC	500 ºC
107	<500 ºC	<900 ºC	400 ºC
128	<500 ºC	>900 ºC	400 ºC
158	<500 ºC	<900 ºC	300 ºC

Figure captions:

Fig. 1. Location of samples in the profile and distances of samples from the lava above.

Fig. 2. 110 ºC TL sensitivity changes with re-firing temperatures for (a) the fired samples with different temperatures and (b) the paleo-fired samples from baked layer.

Fig. 3. 110 ºC TL sensitivity change rate with re-firing temperatures for (a) the fired samples with different temperatures and (b) the paleo-fired samples from the baked layer.

Fig. 4. 150 ºC TL sensitivity changes with re-firing temperatures for (a) the fired samples with different temperatures and (b) the paleo-fired samples from the baked layer.

Fig. 5. OSL /150 ºC TL sensitivity changes with re-firing temperatures of the fired samples with different temperatures.

Fig. 6. OSL/150 ºC TL sensitivity changes with re-firing temperatures of the paleo-fired samples from the baked layer.

Fig. 7. Relation between paleo-firing temperatures and distance from lava.

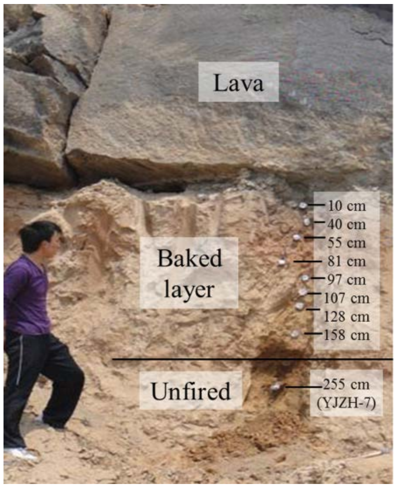
Lava
Baked layer
10 cm
40 cm
55 cm
81 cm
97 cm
107 cm
128 cm
158 cm
Unfired
255 cm (YJZH-7)

Fig. 1.

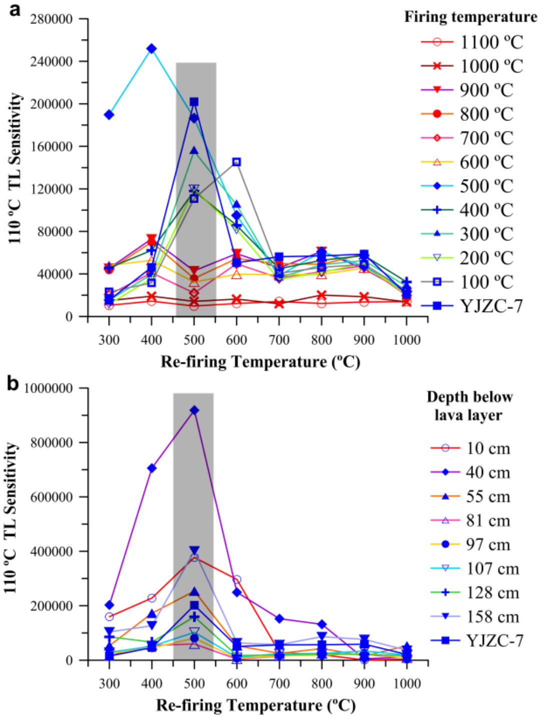
a
110 ºC TL Sensitivity
0
40000
80000
120000
160000
200000
240000
280000
Re-firing Temperature (ºC)
300
400
500
600
700
800
900
1000
Firing temperature
1100 ºC
1000 ºC
900 ºC
800 ºC
700 ºC
600 ºC
500 ºC
400 ºC
300 ºC
200 ºC
100 ºC
YJZC-7
b
110 ºC TL Sensitivity
0
200000
400000
600000
800000
1000000
Re-firing Temperature (ºC)
300
400
500
600
700
800
900
1000
Depth below lava layer
10 cm
40 cm
55 cm
81 cm
97 cm
107 cm
128 cm
158 cm
YJZC-7

Fig. 2.

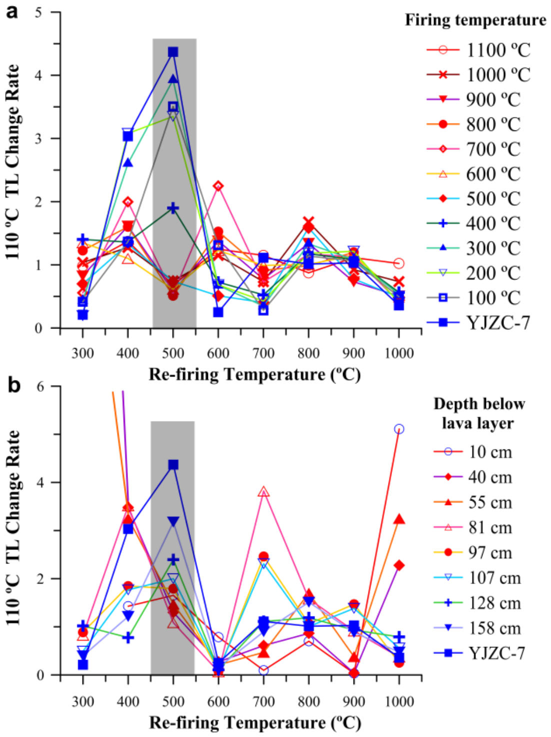
a
110 ºC TL Change Rate
0
1
2
3
4
5
300
400
500
600
700
800
900
1000
Re-firing Temperature (ºC)
Firing temperature
1100 ºC
1000 ºC
900 ºC
800 ºC
700 ºC
600 ºC
500 ºC
400 ºC
300 ºC
200 ºC
100 ºC
YJZC-7
b
110 ºC TL Change Rate
0
2
4
6
300
400
500
600
700
800
900
1000
Re-firing Temperature (ºC)
Depth below lava layer
10 cm
40 cm
55 cm
81 cm
97 cm
107 cm
128 cm
158 cm
YJZC-7

Fig. 3.

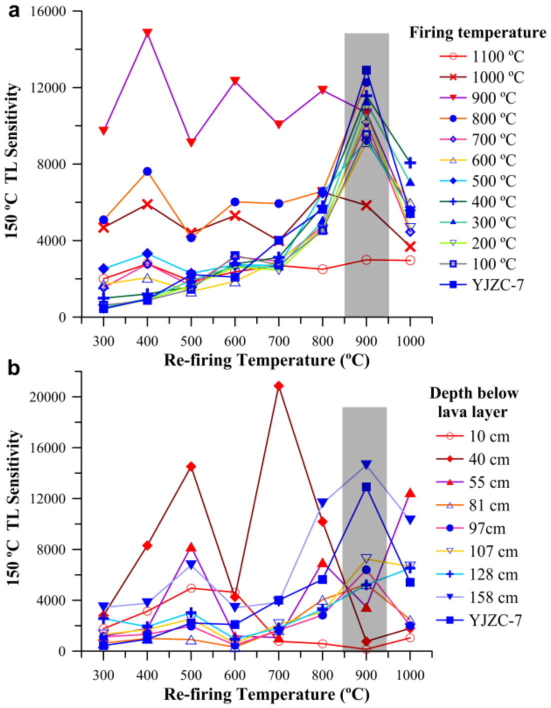
a
b
150 °C TL Sensitivity
Re-firing Temperature (ºC)
Firing temperature
1100 ºC
1000 ºC
900 ºC
800 ºC
700 ºC
600 ºC
500 ºC
400 ºC
300 ºC
200 ºC
100 ºC
YJZC-7
Depth below lava layer
10 cm
40 cm
55 cm
81 cm
97cm
107 cm
128 cm
158 cm
YJZC-7
0
4000
8000
12000
16000
20000
300
400
500
600
700
800
900
1000

Fig. 4.

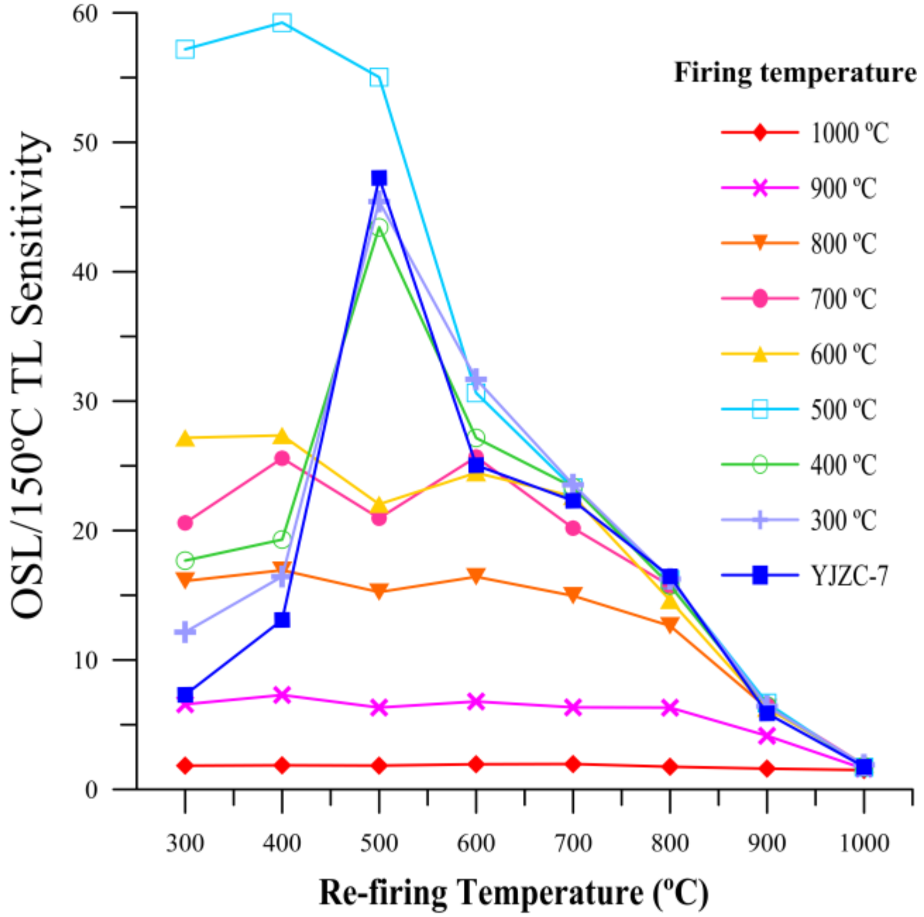
60
50
40
30
20
10
0
OSL/150ºC TL Sensitivity
300
400
500
600
700
800
900
1000
Re-firing Temperature (ºC)
Firing temperature
1000 ºC
900 ºC
800 ºC
700 ºC
600 ºC
500 ºC
400 ºC
300 ºC
YJZC-7

Fig. 5.

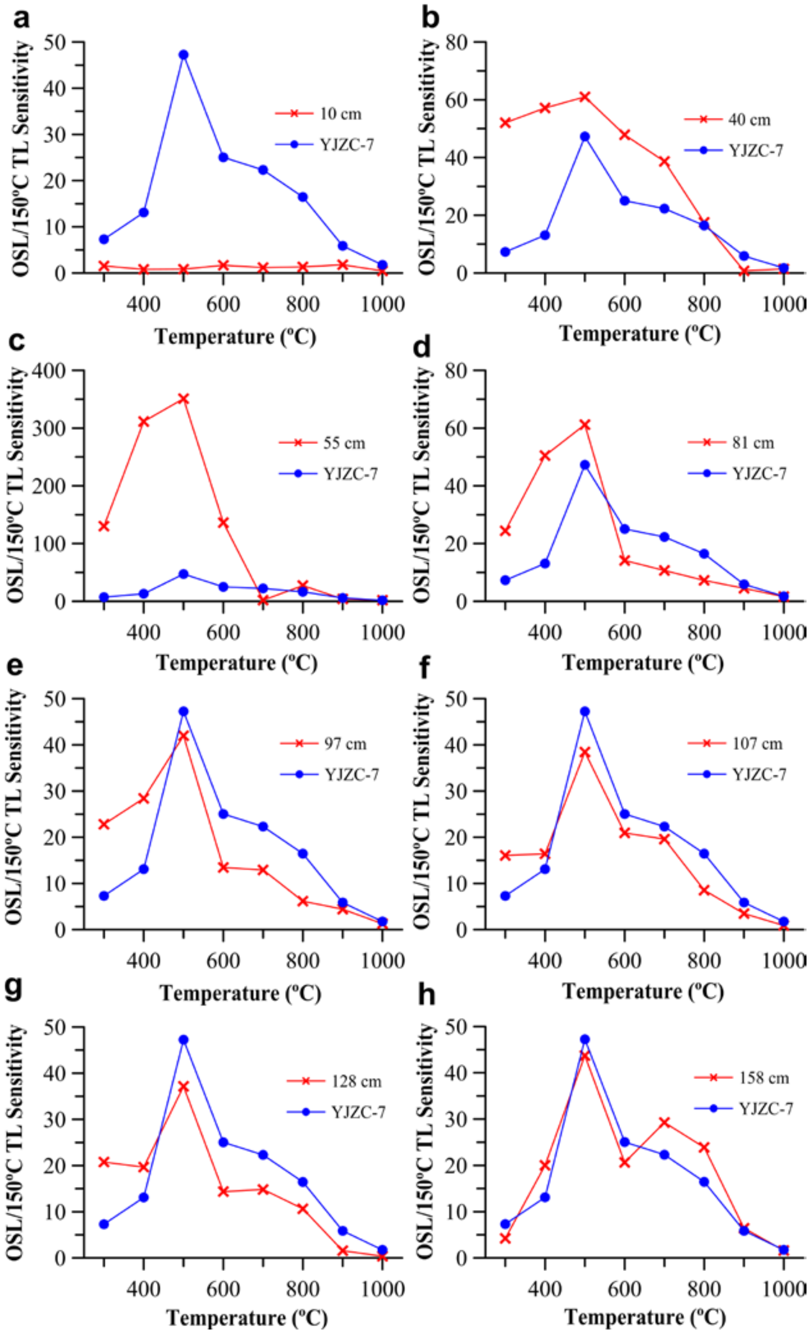
a
b
c
d
e
f
g
h
OSL/150ºC TL Sensitivity
Temperature (ºC)
10 cm
40 cm
55 cm
81 cm
97 cm
107 cm
128 cm
158 cm
YJZC-7
0
10
20
30
40
50
60
80
100
200
300
400
600
800
1000

Fig. 6.

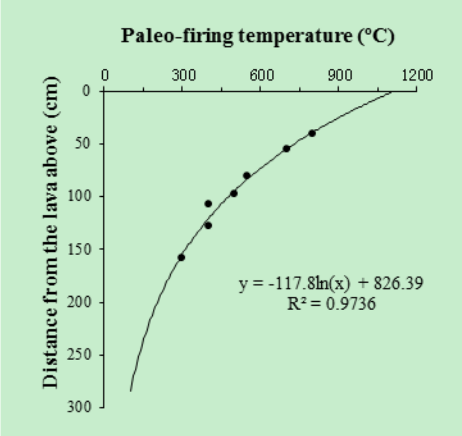
Paleo-firing temperature (°C)
Distance from the lava above (cm)
0
300
600
900
1200
0
50
100
150
200
250
300
y = -117.8ln(x) + 826.39
R² = 0.9736

Fig. 7.