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QUARTZ TL DATING ON SELECTED LAYERS FROM ARCHAEOMETALLURGICAL KILN FRAGMENTS: A PROPOSED PROCEDURE TO OVERCOME AGE DISPERSION

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Abstract: TL dating of archaeometallurgical kiln remains provides information about the chronological period of the corresponding activities. Due to the high temperature this material has usually been subjected to, changes in the TL sensitivity of the quartz grains and also indications of mineralogical alterations, can be present. The study provides absolute ages for kiln assemblages from two prehistoric sites on the island of Seriphos (Cyclades, Greece). Additionally, as the study highlights sources of potential errors, a methodological approach for luminescence dating of similar material is presented.

1. INTRODUCTION

Ancient slag heaps present a valuable source for the technological and archaeological study of early metal production processes. Crucial to the understanding of these sites is their dating, often complicated by the rarity of diagnostic pottery fragments necessitating the use of appropriate scientific techniques. TL has a great potential for absolute dating of archaeometallurgical remains, both slags and kiln fragments (Godfrey-Smith and Casey, 2003), due to the fine resetting of the 'luminescence clock' caused by the temperature of the smelting process.

Direct dating of slag by means of luminescence has proven problematic in the past (Elitzsch *et al*., 1983; Gautier, 2001), mainly due to the complex composition of the material and inaccuracies in microdosimetry calculations. However, Haustein *et al*., (2003) recently reported promising TL dating results based on successfully isolated mineral phases separated out of the slag body and the application of a detailed microdosimetry approach.

In the present study kiln wall fragments from two smelting sites on the island of Seriphos (Cyclades, Greece) were chronologically studied by applying a quartz TL dating protocol on selected layers separated from each sample fragment.

During the TL examination of the quartz extracts variations of the glow-curve characteristics were detected for the natural and laboratory-irradiated aliquots and 1st and 2nd TL glow read-outs, mainly for the low temperature range. The effects should be attributed to the distance between the walls and the fire inside the kiln and possibly to the different origin of the quartz inclusions.

The relation between the luminescence (TL/OSL) properties and thermal treatment has been the subject of many studies (Godfrey-Smith, 1994; Rendell *et al*., 1994; Bøtter-Jensen *et al*., 1995). One of the most pronounced results was the observation of a strong increase of the signal sensitivity, which was found to be related to the intensity of the thermal treatment applied, particularly for the red-TL emissions. The material of the study serves as an interesting case for the examination of the TL characteristics since both the temperature reached and duration of heating are in excess of that employed for pottery production.

Scanning Electron Microscopy was used to establish the degree of vitrification of the samples and Neutron Activation Analysis to provide the chemical profiles of the layers. The alkaline elements K and Rb were found to be significantly lower in the layers that were closer to the fire (referred to hereafter as inner layers, while outer layers correspond to those away from the fire, i.e. closer to the external surface of the kiln structure). These deviating values, which indicate a leaching process occurring sometime during burial (Schwedt *et al*., 2004), possibly introduce an additional error in luminescence dating (Zacharias *et al*., 2005) and the effect was used in order to discard layer-samples suspected to have undergone chemical alterations.

2. EXPERIMENTAL DETAILS

Material and sampling sites

The samples were collected from two sites of archaeometallurgical interest, Kephala (**Fig. 1**) and Phournoi, situated respectively at the north-western and northern part of the island of Seriphos. Both sites are relatively extensive deposits of slag, within which dispersed ceramic furnace lining fragments are found. The geology of the sites consists broadly of schist, frequently traversed by thin quartz veins. The soil layer above bedrock appears to be shallow in both cases ranging from a few up to 30 cm in depth.

The thickness of the furnace lining fragments varies from ca 1.5 to 4.0 cm and colour changes could be observed in cross section ranging from orange-red on their outer surface to a grayish-black vitrified layer on their inner surface, which often bears an attached layer of slag. Inclusions of refractory materials, such as quartz and schist, are seen in the ceramic matrix and these would have enhanced the heat-withstanding properties of the material (Freestone, 1989).

For the purposes of sampling, the sites had been divided into smaller sub-units based on the observed distribution of the material in each case (Georgakopoulou, 2005). The samples were collected from the surface of the heaps, as excavation has not been carried out so far. This study includes three samples from the Phournoi 1 unit and five samples from Kephala (**Fig. 2**) collected from the sub-units Kephala 1 and Kephala 2. It should be noted that the Kephala deposit is significantly more extensive than Phournoi and therefore a larger number of samples was deemed necessary. In the selection of kiln fragments for TL examination preference was given to samples of higher thickness that carried little or no attached slag, to avoid possible contamination.

Chemical analysis

Since the 1970's, mineralogical studies on archaeological pottery report significant variations in alkali metal concentrations due to environmental alterations during burial. Picon (1976) established the relationship between the calcareous nature of the pottery (i.e. Ca >4%) and its high-fired/over-fired state. Within the following years, the alteration of the extensive glassy phase, which can be rich in potassium, together with the crystallization of zeolites, was proposed as an explanation (Buxeda *et al*., 2001 with references therein). Since potassium significantly contributes to the dose rate estimates, the potential implications of potassium leaching for luminescence dating was the subject of a recent study (Zacharias *et al*., 2005) regarding the TL dating of Bronze Age pottery.

After removing the outer 0.5 cm part of the fragments, the samples were sliced almost parallel to the surface based on the visually observed colour variations, thus providing two to three layers from the same specimen. Due to the intensive thermal treatment the material had undergone during the smelting process, where the temperature stayed for several hours at about 1200°C, a degree of

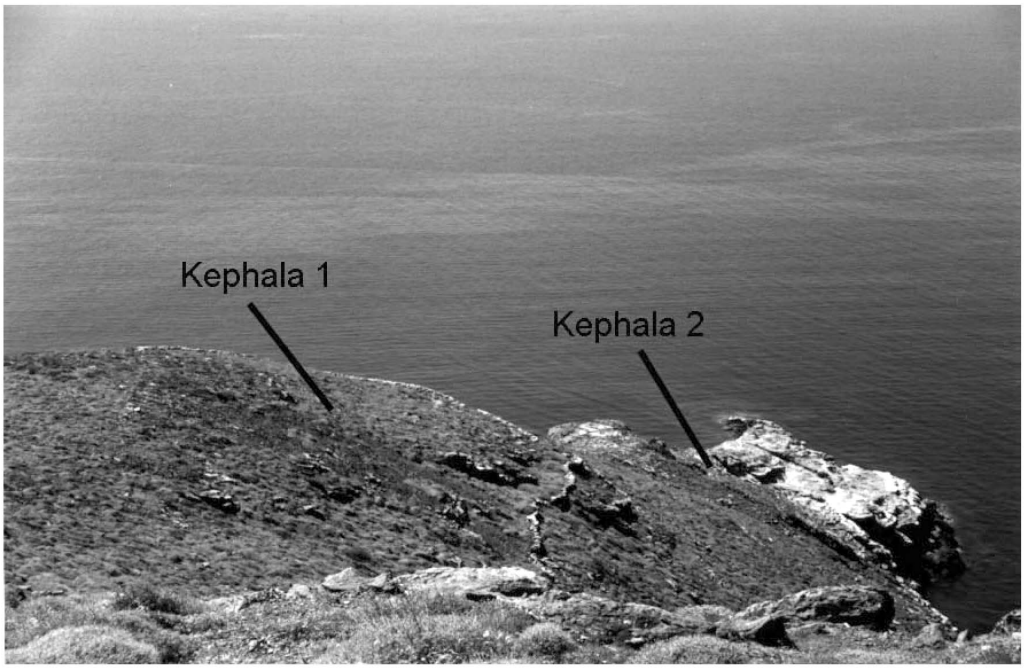

Fig. 1. *The slag heap of Kephala on Seriphos, showing the two main sampling sub-units Kephala 1 and 2.*

vitrification should be expected for the fragments. In order to get an estimation of the temperatures to which the furnace walls were exposed, fresh breaks from almost all layers were removed for examination under the scanning electron microscope. A characterisation of the degree of vitrification of the samples is given in **Table 1**. An almost complete vitrification was observed for the inner furnace wall layers, indicating temperatures in excess of 1050°C (Tite and Maniatis, 1975).

Neutron Activation Analysis (NAA) at the NCSR Demokritos requires about 130 mg of the powdered and dried sample. The samples are irradiated for 45 min in the Demokritos' reactor, at a thermal neutron flux of ca $3 \cdot 10^{13}$ n $cm^{-2}s^{-1}$, including two SOIL-7 standard samples. Each sample is measured twice, 7 and ca 20 d after the irradiation. A Ge γ-detector covering the energy range 80-1600 keV is used for the measurements. NAA provided the concentrations of the entire set of 26 elements (Hein

Fig. 2. *Examples of furnace lining fragments from Kephala.*

Table 1. *Chemical compositions of the studied fragments, according to NAA and α-counting measurements. A description of the colour and degree of vitrification of the samples is also given. The second letter of the sample code indicates the position of the layer from A (outermost), to C (innermost) ones.*

				Sampling sites / Samples				
Samples	**U (μg g^{-1})**	**Th (μg g^{-1})**	**K (wt %)**	**Rb (μg g^{-1})**	**Na (wt %)**	**Ca (wt %)**	**Colour**	**Vitrification**
Site KEPHALA 1								
KEPH1AA	2.96±0.09	11.40±0.23	2.13±0.06	73.80±1.50	1.71±0.08	1.54±0.15	reddish-orange	medium
KEPH1AB	3.07±0.10	12.50±0.25	1.62±0.05	41.30±1.05	1.74±0.08	1.66±0.15	grayish-black	high
KEPH1BA	3.15±0.10	11.20±0.23	2.09±0.05	75.60±1.70	2.03±0.09	1.44±0.15	reddish-orange	medium
KEPH1BB	3.07±0.10	10.90±0.20	1.30±0.04	49.00±1.10	1.90±0.10	1.65±0.20	grayish-black	high
KEPH1C	3.41±0.10	11.50±0.22	2.12±0.05	81.90±1.66	1.11±0.08	0.89±0.12	reddish-orange	medium
Site KEPHALA 2								
KEPH2BA	2.58±0.08	10.80±0.19	2.18±0.05	92.40±2.00	1.09±0.10	0.87±0.15	reddish-orange	medium - high
KEPH2BB	2.29±0.08	10.00±0.22	2.13±0.04	109.00±3.05	1.04±0.08	0.84±0.12	grayish-brown	medium - high
KEPH2C	2.27±0.08	10.20±0.20	2.46±0.05	94.40±1.90	1.15±0.10	1.57±0.15	reddish-orange	medium
Site PHOURNOI								
PH1AA	2.79±0.10	14.20±0.25	3.38±0.08	158.50±3.12	1.21±0.08	0.82±0.11	reddish-orange	medium
PH1BB	2.93±0.09	14.70±0.23	2.39±0.05	94.80±2.95	1.39±0.09	1.81±0.15	grayish-black	high
PH1DA	3.48±0.11	15.70±0.25	2.79±0.08	118.00±3.50	1.66±0.08	1.36±0.12	reddish-orange	medium
PH1DB	3.25±0.10	16.60±0.25	1.80±0.05	77.60±2.55	1.50±0.10	1.70±0.15	reddish-orange	medium
PH1DC	3.35±0.10	14.80±0.22	2.86±0.06	117.00±3.25	1.68±0.11	1.71±0.15	grayish-black	high

et al., 2002). It was observed that the layers from the same fragment exhibit concentration profiles very similar and within the experimental errors, except for the alkaline elements of K and Rb for which a significant reduction is observed in the layers closer to the fire (**Table 1**). The only exception is the inner most layer-sample PH1DC, which has higher K and Rb concentrations similar to those of the outer layer PH1DA. In **Table 1**, the concentrations of the elements relevant for the dose rate estimation, namely U and Th, show only insignificant variations, possibly due to heterogeneities inherent to the material.

Finally, the concentrations of Na and Ca are given in order to emphasize that the observed K and Rb deviations are not associated with Na enriched values despite the non-calcareous nature of the material (Buxeda *et al*., 2001).

TL properties of the quartz aliquots

The layer-samples were gently crushed and the coarse product was dry sieved to obtain the 105-125 and 125-160 μm grains. After chemical treatment with HCl (10%) and H_2O_2 (5%) solutions, the remaining was separated using the non-toxic sodium heteropolytungstate in order to receive the fraction which is rich in quartz. Finally, HF (40%) and HCl (10%) treatment completed the purification of the quartz.

TL glows were recorded using a Littlemore TL 711 set, equipped with an EMI 9635QA photomultiplier and a Corning 7-59 blue filter. Measurements ran at a heating rate of 1.5°C s^{-1} under pure nitrogen gas flow and the irradiations were administered with a ^{60}Co γ-source providing 1.60 Gy min^{-1}.

In **Fig. 3**, the natural TL and the additionally irradiated glow curves are shown for both the inner and outer layers of the fragment KEPH1A. An intense TL signal can be observed for the less fired aliquots (KEPH1AA), in the low temperature range, centred at around 150°C in contrast to that of the high-fired aliquots (KEPH1AB).

For the examination of the suitability of the material for dating, a stable section of the glow curve has to be determined by the plateau test (**Fig. 4**). All samples were found to have a plateau in the range of ca 240-360°C. At higher temperatures, the thermal background becomes dominant and background correction seems increasingly uncertain.

Another observation regarding the TL properties of the material concerns the layer-sample KEPH1C. This fraction provided only one layer due to its small thickness (ca 1.5 cm). In **Fig. 5**, the 1st TL recordings are shown (upper plot) in which the intensities in the low tempera-

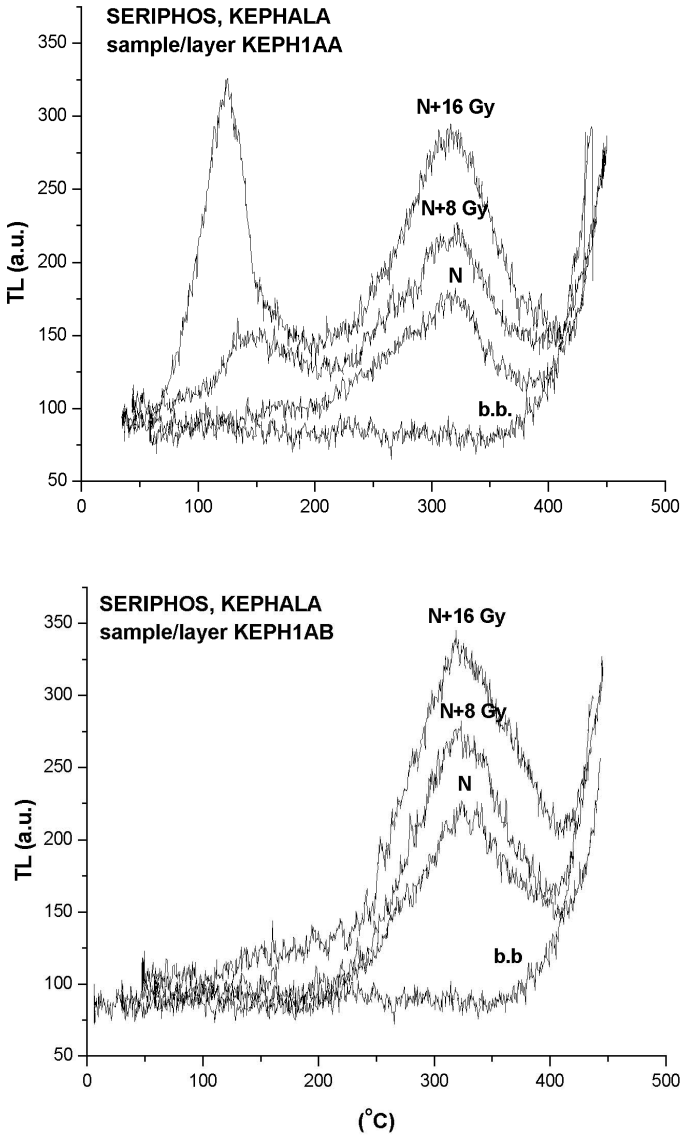

Fig. 3. *Glow curves for quartz aliquots from the layers KEPH1AA (upper) and KEPH1AB (lower plot). The natural (N) and additional irradiated glow curves are shown. b.b. stands for the black body signal.*

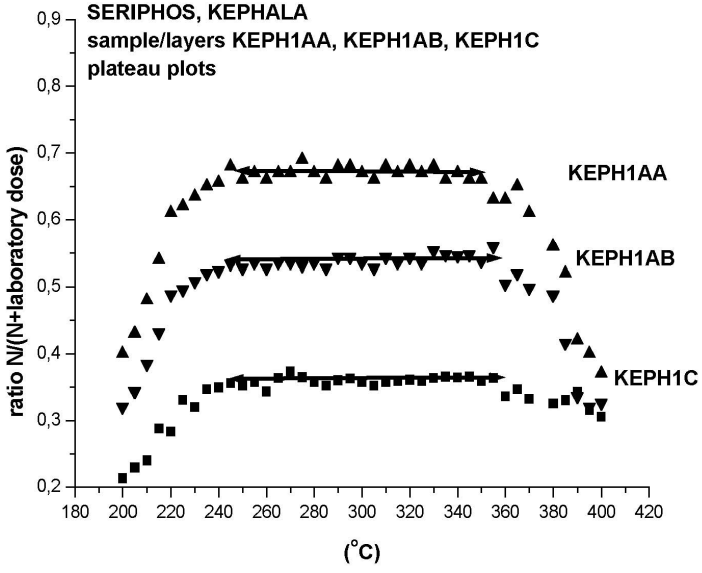

Fig. 4. *The plateau test for the sample-layers KEPH1AA, KEPH1AB and KEPH1C. The ratios N/(N+laboratory dose) for the samples KEPH1AB and KEPH1C are divided by a factor of 2 and 3 respectively, for better clarity.*

ture range show an average sensitivity similar to that of the samples KEPH1AA and KEPH1AB. In the case of sample KEPH1C however, we emphasise the signal intensity of the 2nd TL glows (lower plot) in the low temperature range, which exhibits a variation that seems to be related to the dose applied before the 1st TL reading (pre-dose effect). Nevertheless, in all TL spectra the prominent peak centred at ca 320°C was present.

TL dating procedure

For the equivalent dose estimation D_e, the additive procedure of the *foil technique* (Michael *et al*., 1997) was applied. Using the *foil technique*, a normalised growth curve is produced as follows: each point of the curve (r_L) is given by the ratio of the natural TL intensity, or the natural TL plus a laboratory dose (D_L), to the TL intensity of the 2nd glow of the same aliquot after irradiation with a test-dose (ρ) for inter-aliquot normalisation.

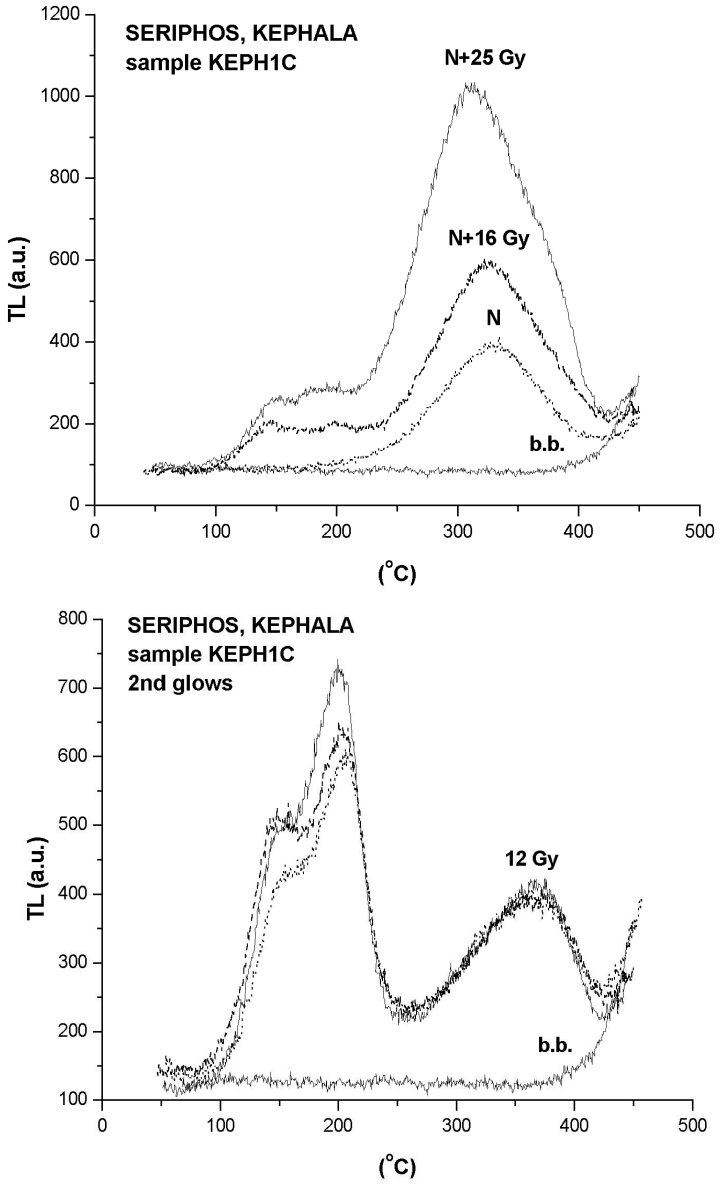

Fig. 5. *Natural and additional irradiated glow curves for quartz aliquots of sample KEPH1C (upper plot). The 2nd glows of the same aliquots are given in the lower plot.*

Equation 6 in Michael *et al*., 1997, also considers for any supralinearity effects may be present in the measurements. But in cases when selecting a test-dose (ρ) which provides a ratio r_0 (natural/test TL intensity) approximately equal to one, the need for correcting for supralinearity is not a necessity, resulting in the simplified form of equation (**Eq. 2.1):**

$$r_L = \frac{D_e + D_L}{\rho[1 + k(D_e + D_L)]} \qquad (2.1)$$

By introducing **Eq. 2.1** in a software program, the paired values r_L, D_L and also the value of ρ are inserted, thus providing the D_e and k values, were k is a correcting factor that accounts for sensitivity changes due to the pre-dose effect.

In **Fig. 6**, the normalised growth curves for the layers KEPH1AA and KEPH1AB and KEPH1C are shown. The condition that allows not introducing supralinearity effects was also tested in our measurements by considering a correction of 10% resulting at slightly deviating D_e values from those estimated in **Fig. 6**. This is an advantage of the normalization procedure followed in the *foil technique* since it reduces the measurement time and can be generally applied for additive dose TL measurements.

Dose rate estimation.

A PIPS thick-source α-counting technique (Michael and Zacharias, 2000) was used to check the samples for possible secular disequilibrium effects. The technique produced values very similar to those measured with NAA and listed in **Table 1**. The β-dose rate (D_β) was estimated using the K, U and Th values of the samples given in **Table 1**. To estimate the γ-dose rate, the radiation field generated by rocks (marble and granite bodies), soil and kiln wall fragments, has to be determined. This was done by measuring the environmental (γ- plus cosmic-) dose rate of the soil and kiln fragments *in situ* using a portable NaI scintillator. In addition to that, a numerical evaluation was used (Zacharias *et al*., 2002) based on the following equation (**Eq. 2.2**):

$$D_\gamma = pD_\gamma^{soil} + qD_\gamma^{kiln} + (1\text{-}p\text{-}q)\, D_\gamma^{rocks} \qquad (2.2)$$

where p and q are factors representing the percentage of the average γ-dose rate resulting from the soil and the kiln material respectively, located in an area of a half sphere and within a 50 cm radius, centred at the sampling points.

3. RESULTS AND DISCUSSION

In general, only moderate enhancement of the TL intensity for the less fired layers could be observed for the temperature range of interest to the D_e estimations. These layers also show strong TL signals in the low temperature range while for the high fired ones the signal intensity is reduced within the same temperature range. Although these observations seem to contradict what is known in the literature about enhancement following higher thermal treatments, one should keep in mind that the mate-

rial of the study had experienced temperatures in the range of 900 - 1200°C for several hours.

One possible explanation could be that the 370 nm emission band, which is associated with the $[H_3O_4]^0$ hole center (Krbetschek, *et al*., 1997) and is responsible for increments in the luminescence centers, is not present or eliminated in over-fired quartz. Additionally, induced sensitivity changes also depended on the origin of the quartz (Lima *et al*., 2002) and on the filter used during the measurements.

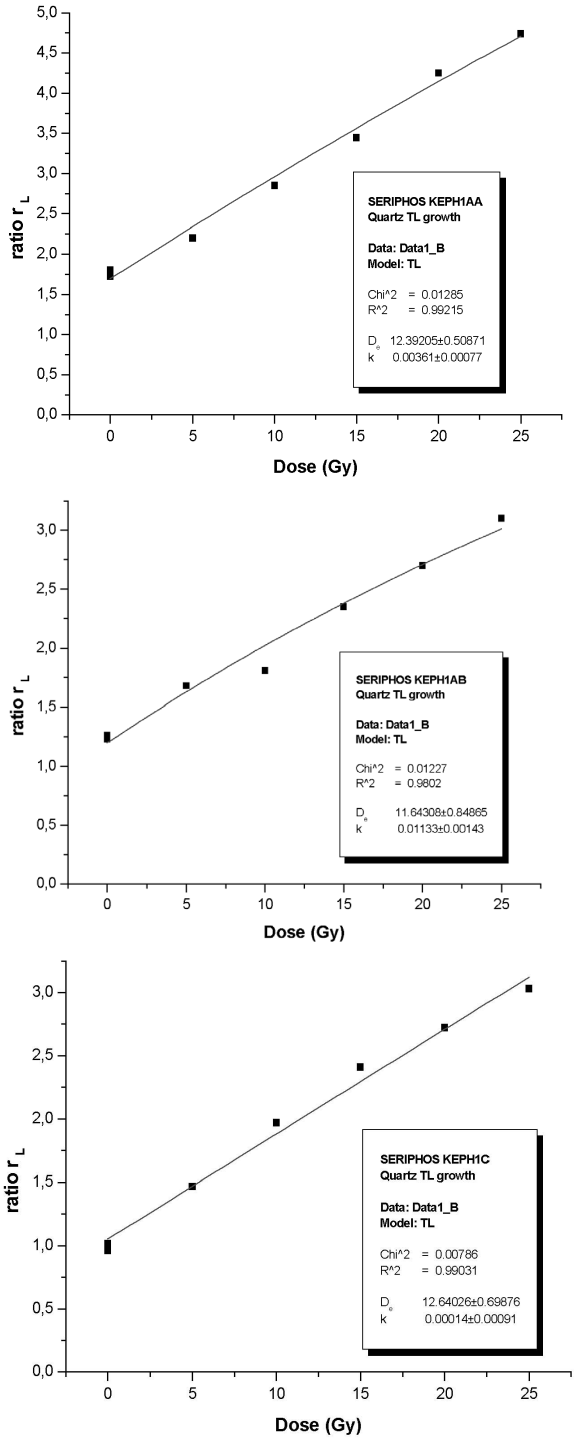

Fig. 6. *The growth curves for samples KEPH1AA (test-dose, ρ =7 Gy) KEPH1AB (ρ=8.6 Gy) and KEPH1C (ρ =12 Gy) for D_e estimation.*

The appearance of the pre-dose effect (k-factor, **Fig. 6**) is also material dependent and associated with the development of saturation, which was present in some of the high-fired samples of the study. In **Fig. 6**, the growth curves from each of the layer-samples KEPH1AA, KEPH1AB are given where, despite the normalisation procedure applied, the r_L ratio points produce D_e values with a deviation of ca 6%.

The chronological results presented in **Table 2** show systematic higher ages for those layers that are suspected to have undergone mineralogical alterations. Thus, the mean TL ages estimated for every site are based on the results from the less fired sample-layers pointing to the need for discarding highly vitrified layers when routine dating is attempted.

While luminescence dating on thick and high fired (temperatures in excess of 900°C) ceramics is attempted, the examination of the following parameters should be incorporated in such studies: a) chemical and mineralogical investigation to examine for concentration variations in the body, b) isolation of sample-layers to check whether a thermal gradient is present, c) application of a dating technique which makes use of a normalisation procedure.

The present study indicated that the metallurgical activities on both sites took place broadly in the first half of the third millennium BC, which corresponds to the Aegean Early Bronze Age (EBA) I-II periods. Slightly older age estimation was obtained for Phournoi, but this is still within the 1-sigma error of the Kephala average ages, so an earlier date for this site cannot be concluded with certainty. The results are of particular importance as this is a period of prominence for Cycladic metallurgy and the sites presented here will significantly enhance understanding of this early production industry.

4. SUMMARY

Even non-calcareous but high-fired metallurgical fragments are potentially susceptible to mineralogical alterations and mainly, potassium leaching effects.

Thick ceramic fragments from metallurgical activities exhibit variable luminescence signals, necessitating the selection of homogeneous, less-fired layers following mineralogical and analytical criteria. It is expected that minerals within the same layer experienced similar thermal-cycles and inter-aliquot normalisation procedures can reduce scatter on the TL measurements.

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Table 2. *TL dating results. The water content is given by the ratio (saturation wet weight – dry weight)/(dry weight) and an error of 0.04 was assumed for dose rate calculations due to annual moisture fluctuations. A mean TL age is also given for every site, calculated on the basis of the TL ages of the less fired layers (in bold), namely: KEPH1AA, KEPH1BA, KEPH1C for Kephala 1, KEPH2BA, KEPH2C for Kephala 2 and PH1AA, PH1DA for Phournoi. (σ; estimated following error propagation)*

TL DATING RESULTS								
Site KEPHALA 1								
Sample	**Grain size (μm)**	**Water content**	**D_β (Gy ka^{-1})**	**D_{cenv} (Gy ka^{-1})**	**total D (Gy ka^{-1})**	**D_e (Gy)**	**Age (ka, ±1 σ)**	**Av. age (ka, ±σ)**
KEPH1AA	105-125	0.12	1.85±0.18	0.81±0.04	2.66±0.19	12.39±0.51	**4.66±0.38**	
KEPH1AB	105-125	0.12	1.58±0.17	0.81±0.04	2.39±0.17	11.64±0.85	4.87±0.50	
KEPH1BA	105-125	0.11	1.82±0.18	0.82±0.04	2.64±0.19	12.09±0.55	**4.58±0.39**	
KEPH1BB	105-125	0.10	1.55±0.16	0.82±0.04	2.37±0.18	11.66±0.59	4.92±0.45	
KEPH1C	105-125	0.11	1.88±0.17	0.81±0.04	2.69±0.20	12.64±0.70	**4.70±0.44**	
								4.64±0.23
Site KEPHALA 2								
KEPH2BA	105-125	0.13	1.75±0.15	0.87±0.04	2.62±0.17	12.40±0.61	**4.73±0.38**	
KEPH2BB	105-125	0.13	1.09±0.09	0.87±0.04	1.96±0.15	9.60±0.67	4.90±0.51	
KEPH2C	105-125	0.12	1.95±0.13	0.87±0.04	2.82±0.18	13.20±0.75	**4.68±0.40**	
								4.71±0.28
Site PHOURNOI								
PH1AA	105-125	0.10	2.64±0.20	0.78±0.04	3.42±0.21	16.76±0.85	**4.90±0.39**	
PH1BB	105-125	0.10	2.08±0.20	0.78±0.04	2.86±0.20	14.73±0.74	5.15±0.44	
PH1DA	125-160	0.09	2.40±0.18	0.78±0.04	3.18±0.19	15.74±0.78	**4.95±0.38**	
PHIDB	125-160	0.09	1.78±0.15	0.78±0.04	2.56±0.15	13.95±0.73	5.45±0.43	
PHIDC	125-160	0.09	2.41±0.18	0.78±0.04	3.19±0.20	16.43±1.15	5.15±0.48	
								4.92±0.27

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