INTERNATIONAL JOURNAL OF CONSERVATION SCIENCE

ISSN: 2067-533X Volume 11, Special Issue 1, 2020: 233-242 www.ijcs.uaic.ro

# TL AUTHENTICITY TESTS: COMPARISON BETWEEN MEASUREMENT METHODS FOR TEMPERATURE ESTIMATION DURING DRILLING

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***Abstract***

*Sampling represents an important step of the procedure to perform authenticity tests by ThermoLuminescence (TL). The result of the test could be compromised if this phase is not correctly carried out. During the drilling, in fact, a local temperature increasing could cause a loss of the luminescence signal. In this paper, the comparison between IR thermography imaging and electrical measurements is shown also in order to validate a specific conceived model. The research is aimed at implementing the procedure used to perform the TL tests with the measurement of the temperature reached during the drilling. The measurements were performed using a pottery sherd simulating the collection step. Results show the validity of the model and provide data useful to optimize the sampling in TL test methodology.*

***Keywords****: Thermoluminescence; IR thermography; Electrical measurements; true or false; Signal loss; Drill; DAQ board*

## Introduction

Thermoluminescence (TL), light emission during heating, is the basis of a technique for the realization of authenticity tests of ceramic and terracotta materials. The experimental methodology allows knowing if an artefact is "true or false". A true sample shows a light intensity signal proportional to the time elapsed since the manufacture in kiln or, in any case, the last heating at high temperature [1-2]. The signal emitted by a pottery sherd is, in fact, related to the time interval between the measurement of the TL signal in the laboratory and the last bleaching due to heating [3].

The TL signal is related to the luminescent emission of crystalline inclusions, essentially quartz and feldspars, contained in the sample that must be extracted by appropriate physical-chemical-preparation procedures.

From a theoretical point of view, the TL is a probabilistic phenomenon in that, as the temperature increases, the escape probability of electrons from the energy traps increases. This probability is proportional to the thermally stimulated luminescence signal [1]. Any heating before the measurement in the laboratory could cause a loss of the TL signal and then an underestimation of the presumed age [1].

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In spite of what happens for the application of the TL as a dating technique [4-10], authenticity tests require the collection of sample in terms of powder. Sampling has to be performed as non-invasive as possible and in correspondence of hidden areas. If, in fact, the test establishes the authenticity of the finding, the damage caused by the collection phase would not affect the worth of the artifact. The powder is collected by an adequate drill characterized by a low speed to avoid any increasing in local temperature. The heating, in fact, could compromise the TL test result causing loss of the luminescence signal [11-22]. This underestimation could implicate erroneous results in terms of false negatives. This means that the TL signal from an authentic sample could be confused with the noise and then considered as a fake.

In this field a multidisciplinary study started with the aim to estimate the temperature reached during the drilling. The knowledge of the temperature values reached by the sample during the collection allows evaluating any signal losses and/or if the measured data are underestimated and they need corrections. A preliminary study has been proposed by authors in [23]. The objective is the development of a method for the indirect temperature measurements simultaneously with the sampling in order to be included to actual protocol.

In this paper an improvement of this methodology has been addressed considering a more accurate modeling and a comparison through stardard solutions such as IR thermography. It is worth to mention that this latter technique implies high area occupied, high cost, limited memory and it is not able to perform measurements in depth. The proposed methodology, based on electrical measurements, addresses all these points. It represents a simple, low-cost and non-invasive solution, able to perform measurements run time and it is suitable to be implemented in embedded systems.

The temperature during drilling was extimated through output voltage measurements across a known reference resistor, in presence of a current [23]. At the same time IR images were acquired for temperature measurements [24-25]. The informations coming from the two methodologies have been compared and have been used also to validate the model. Thanks to the results further implementations of the model will allow the optimization of the protocol used for TL authenticity tests.

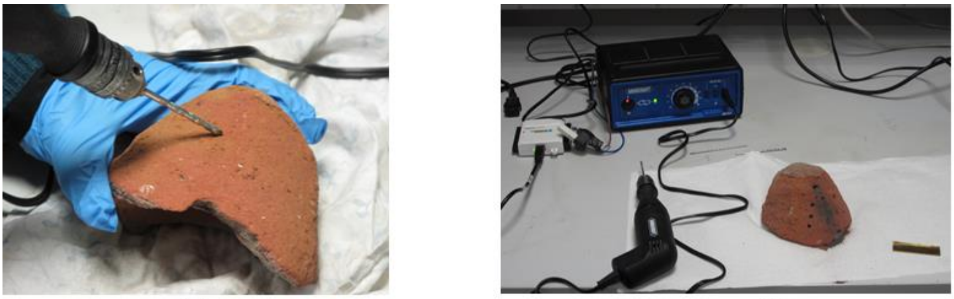
## Methods

In order to compare the temperature reached by sample during the drilling step, measured by infrared thermography and the temperature evaluation calculated using the model here proposed, we simulate the related phase of the TL authenticity tests performing this step on a historical terracotta.

A high precision Minicraft drill MB1012 is used. It ensures a perfect balance between power and accuracy. It is equipped with geared chuck for extra precision and twin bearing shaft for smother running. Different drill bits of different diameters are employed (2.0, 3.0 and 5.5 mm respectively). The drill is used with power adapter tranformer MB751 Electrotechnis-clers Werk, Berlin, Germany at variable speed to 1 up to 10. Figure 1 and Figure 2 show respectively the drill, the sample, and the main phases of the collection steps. We have done seven holes with different diameters ranging from 2 mm to 5.5 mm. Each hole corresponds to a measurements series that are listed in Table 1 with the relative drilling test information regarding the used diameter drill bit and the power transformer speed.

The experimental phase involved the simultaneous acquisition, during the drilling, of thermal images and electrical voltage values. It should be noted that both the solutions will be compared and the advantages of the novel principle based on electrical measurements will be shown. The experimental set-up is presented in Figure 3.

A thermal imaging camera FLIR® System B series with a spectral range of 7.5-13 μm and a measurable temperature range from -20 °C to +120 °C was used. The measurements were performed with controlled temperature (22 °C) and humidity (30%) at a fixed relative distance between camera and sample of 60 cm. The IR images are processed by the ThermaCAM Researcher basic 2.8 SR-1 software.



(a) (b)

**Fig.1.** The historical terracotta (a) and the drill (b) used for the sampling.

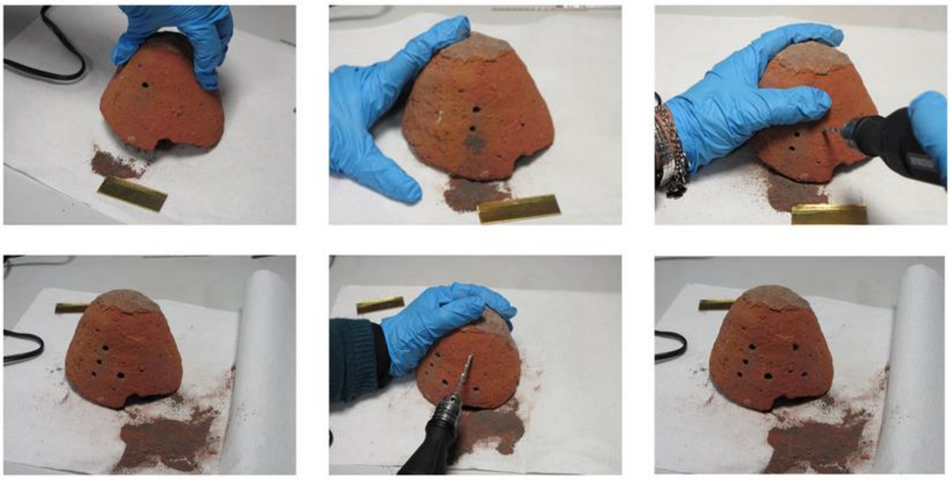


**Fig.2.** The main phases of the drilling on the terracotta sample.

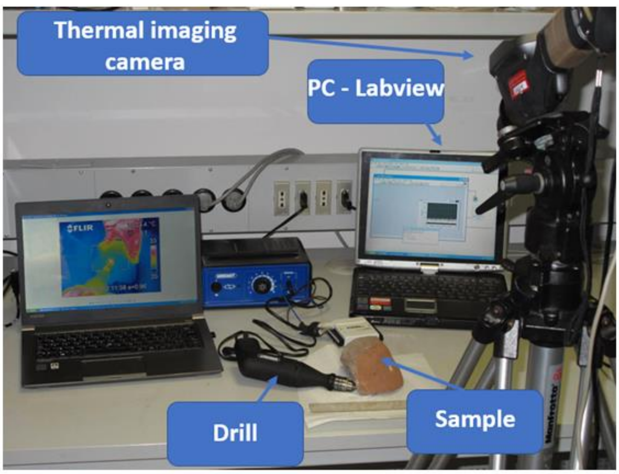



**Fig.3.** The experimental set up.

**Table 1.** Measurement drilling series and the information regarding the diameter bit.

| Measurement series | Diameter bit |
|---|---|
| I | |
| II | |
| III | 5.5mm |
| IV | |
| V | |
| VI | 2.0mm |
| VII | 3.0mm |

The emissivity of the surface active and passive blades was measured prior to thermal imaging measurements when blade reaches the standard temperature values of 60 °C and 30 °C respectively. Emissivity (ε), the relative power of a surface to emit heat by radiation, was calculated. This last is a dimensionless quantity depending to the temperature of the sample and the wavelength of used energy; its value range from $0 < \varepsilon < 1$ [24-25].

Concerning the electrical measurements were performed with a DAQ board NI-6009, a resistor (R) of 0.68 **Ω**-20 W and a laptop using a LabView routine in order to implement an automatic measurement system. In particular, the DAQ board has been used to measure the voltage ($V_m$) across the resistor R that is connected in series with the power supply of the drill. A sample rate of 1 kHz has been selected for the experiments and, in order to decrease the noise level, improving the resolution of the entire architecture of measurement, a differential acquisition solution has been considered. The LabVIEW routine used to perform the measurements is composed of a DAQ assistant block used as interface for the $V_m$. A loop execution has been selected in order to acquire continuously. A virtual oscilloscope has been used to show the $V_m$ signal. A block write to measurement file has been used to save the data in a text file in order to be processed in MATLAB®. Figure 4 shows a picture of the architecture used during the experiments.

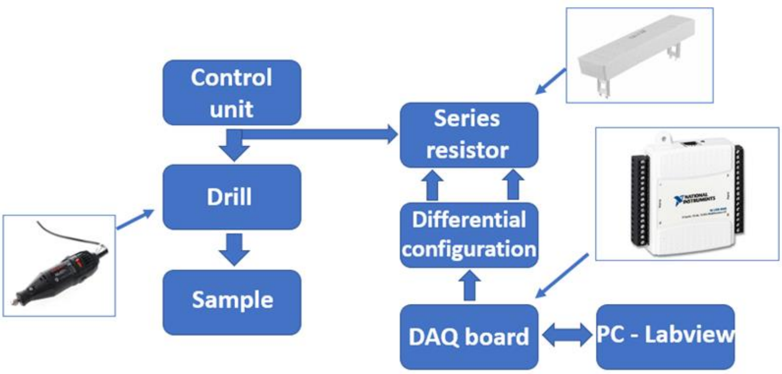



**Fig.4.** Schematization of the setup used to perform the electrical measurements.

## Measurement strategy

The measurement strategy here pursued is based on the estimation of electrical parameters and a non-invasive approach in order to estimate the variation of temperature dissipation during the drilling of an archaeological pottery sherds object of a TL authenticity test. The schematic diagram of the solution is shown in Figure 5. It is composed of five steps.

The main idea is based on the assumption that the sampling area is interested by a temperature increment correlated with the rotation of the drill. Therefore, the current and the power consumption of the drill will change. This current, named $I_a$, in presence of a resistor R, creates a differential of potential $V_m$ (see step 1 of Fig. 5). This voltage is the physical quantity that will be measured and will be correlated with the variation of temperature during the drilling through the adoption of the analytical model.

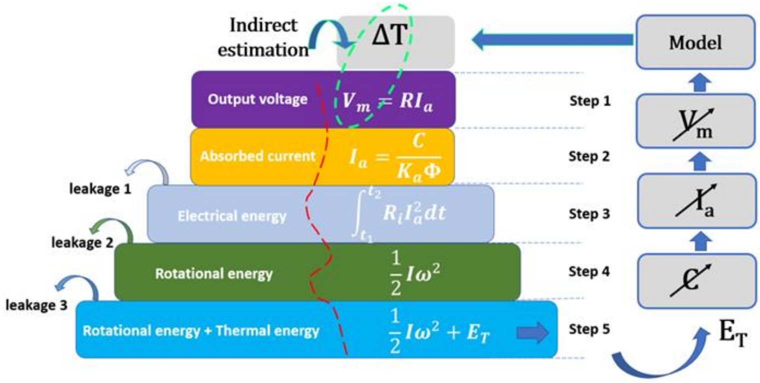



**Fig.5.** Block diagram of the non-invasive measurement method for the estimation of the temperature during drilling.

The current $I_a$, as shown in the step 2, is the absorbed current of the drill which starts to flow when it starts the rotation. It is correlated with the torque C, the excitation flux Φ and the constant $K_a$ that depends on the construction parameters of the drill that is an electrical machine. This latter parameter is mainly correlated with the geometry, the number of poles, type of windings, magnets [26]. Now the assumption is the absence of mechanical load. It is worth noting that $I_a$, will flow through the excitation circuit of the drill which presents a resistance $R_i$ (see step 3). As consequence, an electrical power will be dissipated ($R_i$ $I_a^2$) and, considering a time interval $t_2$-$t_1$, it is possible to define the electrical energy absorbed during the activity of the drill in the considered time interval.

During this step, leakages (leakage 1) as consequence of Joule effects of the excitation coil can occur. This energy, neglecting the leakages 2 (mainly given by electro-mechanical dissipative effects), will imply a rotational effect and an energy which can be expressed as the inertial moment I multiplied by the square of the angular velocity ω (see step 4).

In presence of a mechanical load, such as the effect of the drilling procedure of same samples, a thermal energy $E_T$ will be dissipated (see step 5) in accordance with the equation (1). As consequence, an influence of the term C occurs and, considering the equations of the step 2 and step 1 respectively, the current $I_a$ and the voltage $V_m$ will increase. In order to correlate its amplitude variation, with the variation of temperature, the equation (2) will be considered. In this context, it should be observed that the energy $E_T$, can be modelled as:

$$\mathbf{E_T} = \Psi\boldsymbol{\lambda}\frac{\mathbf{S\Delta T}}{\mathbf{L}}\boldsymbol{\Delta}\mathbf{t} \quad (1)$$

where, λ is the thermal conductivity of the material under analysis, S is the contact surface, L is the length of the contact, Δt is the time interval during the thermal effect and ΔT is the measurand. It is worth noting that equation (1) improves the approach pursued by authors in [23], through the adoption a term Ψ, which is a coefficient, experimentally estimated, able to take into the account the leakages presented in Figure 5. The paramenter has been estimated through a comparison with a set of data coming from the IR camera. The indirect estimation of ΔT can be done through the inversion of equation (1) and imposing that the thermal power corresponds with the electrical power ($E_T$/Δt=$V^2_m$/R):

$$\Delta T \approx \frac{V_m^2 L}{\Psi \lambda S R} \quad (2)$$

## Results and discussion

The experimental phase regarded the simultaneous measurements of the voltage $V_m$ and of the temperature by Infrared Thermography for all the sets of sampling (see Table 1 and Methods section).

Different series of IR images were acquired from the start of drilling with a time interval of 5 seconds and the related temperature data were obtained considering the maximum value in a ROI (Region Of Interest) placed in the center of the hole (Figure 6).

The behaviour of the temperature values vs. time (Figure 7) shows that the infrared radiation emitted by the object reaches the maximum intensity in the first seconds and then it becomes stable at an average value of 50 °C. The data of all the sets of measurements show a similar behavior. In Figure 8, the voltage trend as a function of drilling time is plotted for the II series.

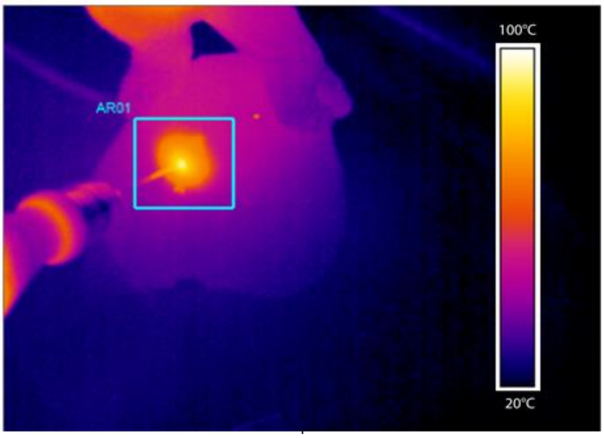



**Fig.6**. IR image processed by ThermaCAM software and ROI including each hole.

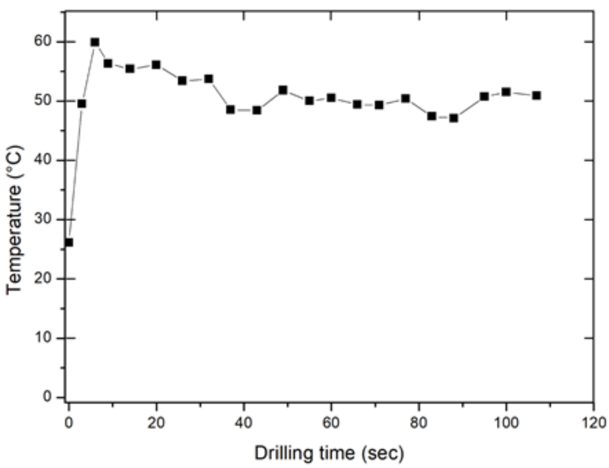



**Fig.7.** Behavior of the temperature values measured by Infrared Thermography vs. drilling time.

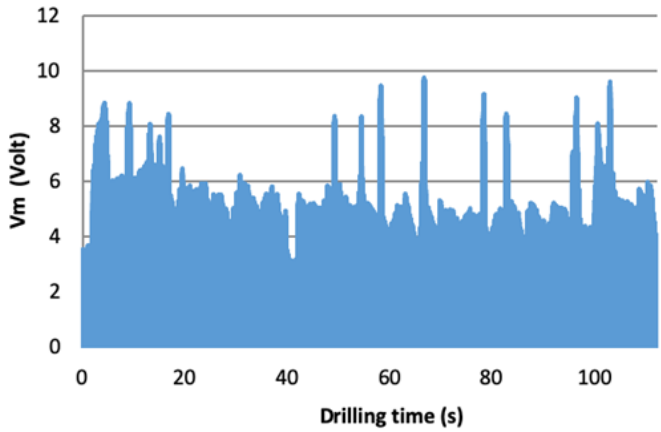



**Fig. 8**. Trend of the voltage vs. drilling time for the II measurement series.

***Data comparison***

The approach presented in this paper is based on the comparison between the data acquired by thermal imaging and the electrical measurements. Figure 9 shows as example the comparison between the data acquired by electrical measurements and the values of the temperature obtained by IR acquisitions.

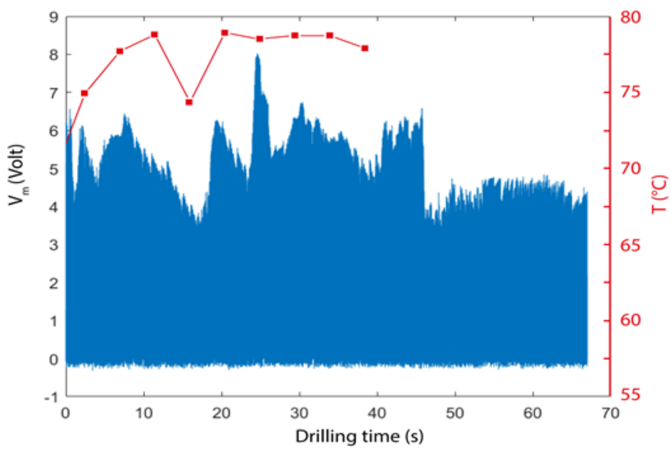



**Fig. 9.** Comparison between the electrical data ($V_m$ vs drilling time) and the temperature data elaborated by thermal imaging software and plotted as a function of the time.

The figure evinces that the trend of the proposed method (see $V_m$) is in accordance with the measurements perfomed through the IR camera. In particular, it must be observed that the temperature values are stabilized while the $V_m$ presents variable values. This is because the IR acquisition are superficial instead with electrical measurements gave us values of $V_m$ coming from a greater depth. This evidence is important from an applicative point of view. The electrical measurements in the collection phase could allow monitoring the temperature reached by the powder collected as sample. Starting from this result, we applied the equation 2 (see section measurement strategy) in order to verify the proposed model. In absence of a mechanical load of the drill, the energy dissipated regards all the aforementioned steps without $E_T$ ($E_T$=0 J). During the drilling, there is a load and $E_T$≠0 J. In fact, this condition will change the value of the torque C, and as consequence $I_a$ and $V_m$ respectively. The model has been compared with the obtained results by adopting the IR camera, in particular a time window of 5 seconds has been used. Good agreement has been obtained and, as shown in the Figure 9, coherence has been also detected in terms of voltage and temperature as function of time. A resolution of about 1 K has been experimentally estimated.

Table 2 shows the parameters of the model.

**Table 2.** Parameters of the model

| Parameter | Value | Unit of measurement |
|---|---|---|
| L | 1 | mm |
| Ψ | 31.8 | - |
| λ | 0.8 | W/mK |
| S | 7.07 | $mm^2$ |
| R | 0.68 | Ω |

It should be also noted that the electrical measurements and IR thermography gave information respectively about the rate of absorbed heat and the temperature at fixed time intervals.

From equation 2, known the diameter of the bit, it is possible to obtain the ratio between the thermal conductivity λ and the penetration depth (L) as a function of the temperature (T), that represents the power per unit of surface and of temperature ($W/m^2K$) supplied during drilling. The experimental results highlight that this last quantity is inversely proportional to the bit diameter and it decreases with the drilling time (see Figure 10 a,b,c). This is due to the greater pressure applied to the sample in a smaller surface areas, related to the diameter of the drill bit, and also to the dynamic friction between the bit and the sample decreasing with the drilling time.

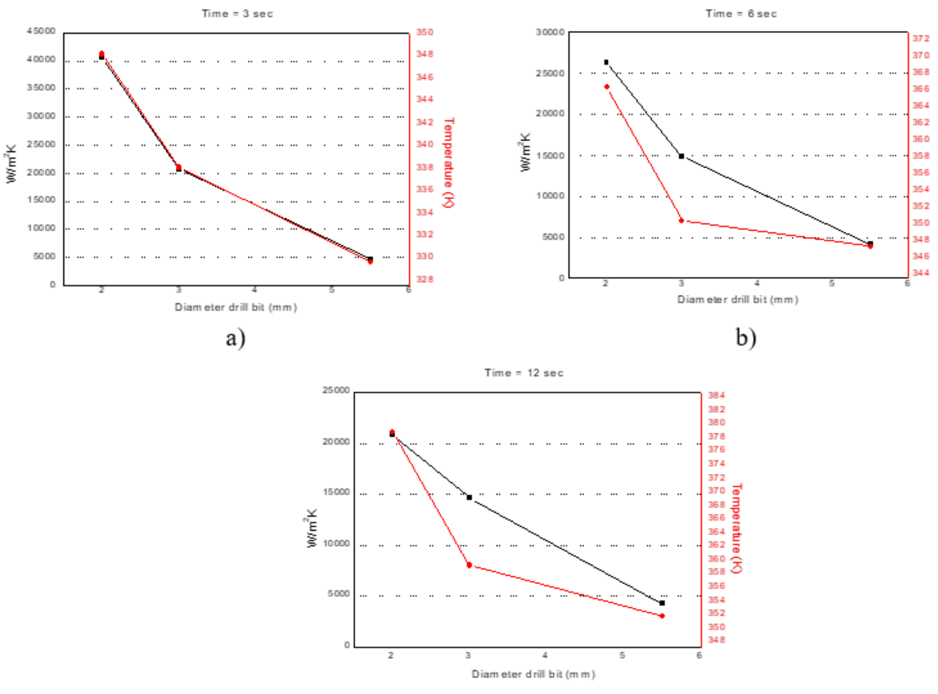



**Fig. 10.** Trends of power per surface unit for each kelvin ($W/m^2K$) and of temperature (K) values as a function of the drill bit diameter: a – time = 3 seconds, b – time = 6 seconds, c – time = 12 seconds.

This evidence assumes considerable importance for the procedure aimed to perform a TL authenticity test. If the minor invasiveness of sampling leads to the use of smaller diameter bit, the experimental measurements show instead a greater invasiveness in terms of maximum temperature reached. The results, therefore, suggest the use of bit with larger diameter but this is applicable only for sampling in hidden areas of the artefact.

## Conclusions

The present paper starts from the importance of the temperature monitoring during the sampling in the authenticity tests by ThermoLuminescence (TL). The authors proposed a model aimed at implementing the procedure used to perform these tests with the measurement of the temperature reached by the sample during the drilling.

For this purpose, a sampling by a terracotta sherd is simulated and the temperature is recorded by IR thermography imaging coupled with electrical measurements. By the comparison of the results obtained thanks the two techniques, it is possible to confirm the validity of the model. The experimental results in fact connect the electrical measurements with the thermal conductivity. The correlation between these results with the related IR images shows a good agreement and allowed establishing the basis for the model implementation and the optimization of the procedure used for TL authenticity tests.

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*Received: December 1, 2019*
*Accepted: February 29, 2020*