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Indirect Temperature Measurements for TL Signal Loss during Drilling

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***Abstract* – Sampling represents an important phase of the procedure to perform authenticity test by thermoluminescence. The test could be compromised if this step is not properly carried out. In particular, if the local heating of the area subjected to the sampling by drilling reaches high temperatures, the signal results underestimated. In order to evaluate the increasing temperature during the sample collection in authenticity test, in this paper we propose a procedure useful for temperature in situ measurements using an indirect method. This method allows estimating the temperature dissipated during the drilling of the samples as consequence of the drill rotation. The temperature variation is obtained through the evaluation of an output voltage measured across a known reference resistor, in presence of a current.**

I. INTRODUCTION

As described by the Oxford dictionary, authentication is useful *to prove or show something to be true, genuine or valid* [1]. The increased interest in collecting ancient materials brought about a growth in the number of reproductions for general sale as well as in the creation of forgeries intended to deceive the most avid collector. The desirability of art and archaeological materials caused a significant increase in prices during the last centuries. The high demand and high prices for artefacts and artworks meant collectors required firm confirmation that the pieces were authentic. With the increase and more accurate presentation of fakes and forgeries, accurate studies of artefacts were necessary. A combined knowledge of art history, techniques and materials is needed to full access an artwork, encouraging the connection with the scientist [2].

The Thermoluminescence (TL) is the most important methodology used for the authentication of potteries, ceramics and other materials heated at high temperature (> 500°C). The heating causes the bleaching of the luminescent signal accumulated by the crystalline inclusions (feldspars and quartz) from geological formation thanks to the environmental radioactivity. Starting from this moment, the crystals store a luminescence signal proportional to the passed time until to a new thermal bleaching in laboratory [3].

The TL represents also the best methodology to date archaeomaterials that were heated at high temperature during their "life". In general, the age was calculated considering the time elapsed from the first bleaching in kiln during manufacture and the last heating in laboratory. In order to obtain this time, the TL age calculation requires the measurement of the two quantities: the Equivalent Dose (ED) and the Dose Rate (DR). The ED is the total dose accumulated from the last firing of terracotta samples evaluated trough luminescence signals while the DR is the quantity of the dose absorbed in one year. The ratio between the ED and DR represents the age of the analysed sample.

In laboratory, the ED is obtained recording the luminescence signal emitted during the heating of the material. The intensity of this light is plotted against temperature to produce the so called "natural glow curve". This curve is in fact considered the TL signal accumulated by the sample since the last bleaching (heating) event corresponding to the total absorbed dose.

To calculate the elapsed time, the natural dose rate needs to be evaluated by additive or regenerative techniques [4-5]. As aforementioned, for the most precise dating, the relationship between exposure to different sources of radiation must be accurately measured to obtain the rate of dose absorption. The irradiation comes from the sample itself and from the environment. The DR determination requires the evaluation of each contribution.

The lack of information about the environmental contribution to dose rate does not allow obtaining chronological information [3]. Unfortunately, this situation is typically in all authenticity testing of ceramic art objects because the environment radiation is unknown. In this case, the TL measurements are aimed at establishing whether the expected age of the object is consistent with the stylistic attribution. In authentication, in fact, the TL test is aimed at solving the problem of distinguishing between old (= original) and new (= faked) art objects. In particular, if the natural luminescent signal is comparable with the age estimated by archaeologists on stylistic and typological criteria, the TL test result indicates that the studied object is authentic.

The TL methodology is destructive and the related procedure requires the collection of a portion of the sample that is normally obtained by drill. The sampling procedure is fundamental for performing TL test and specialists must carry it out according to the artwork owner. Each sampling phases is documented by photographs and by written documentation includes the sample location, the rational for sampling, the drilling technique, and type of drill bit, size of holes, date and person taking the sample. Usually, the procedure is carried out both to cause as little damage as possible to the object and to ensure that the sample is not contaminated as it is being removed. In general, the drilling consists in a withdrawal of about 100 mg from the underside of the object with a drill that usually produces the form of small holes of around 5 mm in diameter [6-18]. Because bright light will cause loss of the TL signal, drilling should be undertaken in a room that can be darkened, using a safelight such as a dim red-light. Considering that TL gives information only for the sample tested, the results generally reflect the age of the entire object, but certain conditions can render a TL test invalid or give a misleading result. For this reason, it is important to understand the structure of the object, in order to avoid drilling in fragile areas and to avoid contaminating the sample with restoration or mounting materials. In fact, if the object has been subjected to extreme heat at any time after its manufacture, the TL result will reference that firing. This can occur when the object has been involved in a fire, or if it has been heated, as sometimes occurs in restoration.

Another problem that could be compromised the TL authenticity test is represented by the underestimation of the luminescent signal caused by local heating of the area subjected to the sampling by drilling. TL emission derives from thermal release of electrons trapped in localized energy levels, called traps, with different associated escape probability values [19]. The amount of light is proportional to the number of trapped electrons, which in turn is proportional to the amount of absorbed dose. The probability of escape from a trap rises very rapidly with temperature [3].

All local eating due to drilling step during the sample collection can partially bleach the signal of interest causing an underestimation of absorbed dose. This possible lack could be evaluated considering the relationship between a trap depth and the electron mean life entrapped in it for a specific average temperature of the system.

The present research work represents a contribution to solve this issue by the assessment of a methodology useful to indirect measurements of the temperature reached during drilling. Other procedures documented in literature until today [6-18] have been take in consideration some of aspects dealt with but our paper is aimed at presenting a method for measurement the temperature in situ during the sampling campaign by using an indirect method. After the description of the measurement method and the experimental set-up, we present the waveform obtained and an estimation of the thermal dissipation during the operation of the drill on a terracotta sample.

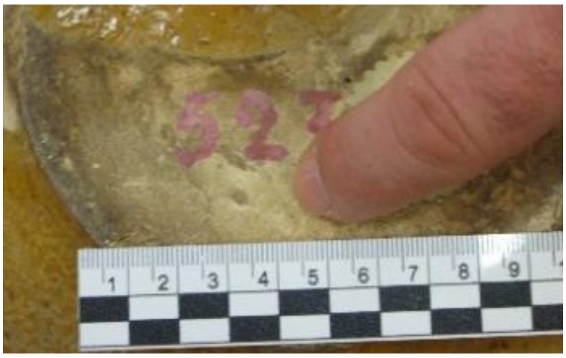
II. MATERIALS AND METHODS

A. Sampling

Standard procedure used in the PH3DRA laboratories is based on the sampling by drill at low speed using tips of increasing diameter (5 mm maximum) up to obtain the desired quantity of powder. Each step of the procedure is documented. The Fig. 1 shows an example of the documentation regarding a terracotta tale before (a) and after (b) and a bowl before (c) and after (d) sampling phase.

(a)

(b)



(c)

(d)

Fig. 1. Sampling area before (a) and after (b) drilling for a terracotta tile and before (c) and after (d) for a bowl.

B. Measurement methodology

A non-invasive measurements methodology was used to estimate the temperature during the drilling of an archaeological pottery sherds object of a TL authenticity test.

Fig. 2 shows the principle of the measurement architecture in order to estimate the temperature dissipated during the sampling by drill. The methodology consists in five steps (S#*). We start from the assumption that the sampling area is interested by a temperature increase as consequence of the drill rotation and that it is possible to evaluate this warming through the evaluation of an output voltage (V_m) measured across a known reference resistor R, in presence of a current I_a (see step S#5 of Fig. 2).

The nature of the current I_a is described considering the S#4, in particular, as can be observed, it is the consequence of a power supply applied to the sample-drill. It is worth noting that, in this condition, it starts the rotation and as consequence a current I_a will flow and its value (see equation at the S#4) is correlated with the torque C, at the moment in absence of mechanical load.

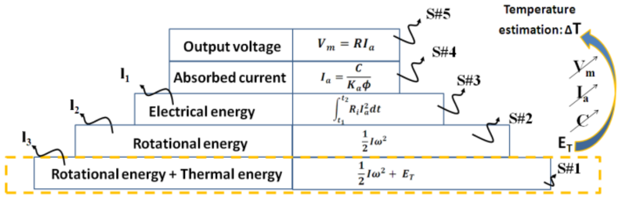


Fig. 2. Diagram of the measurement architecture useful to estimate, through an indirect method, the value of the temperature increase during the sampling by drill. The diagram includes several leakages which can be found during power conversion and transduction mechanisms [20-21].

The excitation flux Φ and the constant K_a which depends on the construction parameters of the machine (length, diameter, number of poles, type of windings and etc.) [22]. This current will flow through an excitation circuit of the drill, which presents a resistance R_i. Through the equation presented in S#3, it is possible to evaluate the electrical energy absorbed in the time interval t_2-t_1. This time interval represents in our case the duration of the sampling phase. The leakages l_1 (i.e. Joule effect of the excitation coil) are neglected. This energy, neglecting the leakages l_2 (i.e. electro-mechanical dissipative effects), will be converted into a rotational energy which can be expressed as the inertial moment multiplied by the square of the angular velocity ω (see step S#2). In presence of a mechanical load, such as the drilling procedure of the samples, a thermal energy E_T will be dissipated in accordance with the equation (1). Other effects of leakages (l_3) are also neglected.

$$\mathbf{E_T = K\frac{S\Delta T}{L}\Delta t} \qquad (1)$$

where, K 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.

The indirect estimation of ΔT can be accomplished through the inversion of the over mentioned model and imposing that the thermal power corresponds with the electrical power ($E_T/\Delta t = V_m^2/R$):

$$\mathbf{\Delta T \approx \frac{V_m^2 L}{KSR}} \qquad (2)$$

we suppose that in absence of load, the energy E_T is equal to 0 J. By using equation (2) it is possible to estimate the value of the temperature variation during the drilling. In fact, this condition will change the value of the torque C, as consequence the value of the absorbed current I_a and, finally, the voltage V_m. In the next section, the setup and some experiments will validate the working principle here described.

III. EXPERIMENTAL SETUP AND RESULTS

The implementation of the measurement method has been accomplished by using a DAQ board NI-6009 to measure the voltage V_m [23]. In order to perform the analyses a resistor (R) of 0.7 Ω-20 W has been connected to the DAQ board. A differential configuration has been selected in order to decrease the noise level of the measurement system increasing the performance in terms of resolution. A laptop with a LabVIEW routine has been used to acquire the voltage across R (V_m) and to save the signal. The LabVIEW routine is composed of a DAQ assistant block used to manage the signal coming from the resistor R. A virtual oscilloscope has been used to show the V_m waveform. A block write to measurement file has been used to save the data in a text file. The routine has been inserted inside a loop in order to acquire the data for several seconds.

The validation of the principle has been performed acquiring the signal of the voltage V_m in order to estimate the temperature during the drilling of a terracotta sample. Fig. 3 shows the evolution of V_m as function of the acquired samples. The signal presents several spikes correlated with the drilling procedure. In particular, ΔT_1 is the variation of temperature, which corresponds to about 0.15 V, is the step between the no-load and a mechanical load condition (mean value during the working condition). The other fast spikes with higher amplitudes represent two transient contributions induced by the drill.

Considering the equation 2 and assuming the following values of the parameters: K=0.7 W/mK, $S=\pi\cdot 10^{-6}$ m^2, L=0.003 m, an average thermal variation of about 44 K has been detected by using the proposed method.

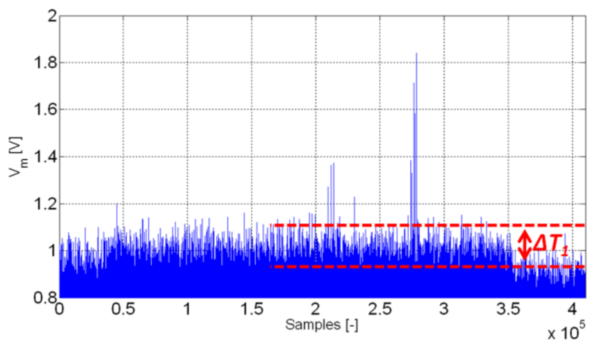


Fig. 3. Measured V_m used to estimate, through an indirect method, the temperature dissipated during the drilling procedure.

IV. CONCLUSIONS

In this paper, we discussed about a non-invasive measurements methodology to estimate the temperature during the sampling performed by drill for TL authenticity test. The proposed method is indirect and it is useful to estimate the temperature dissipated during the drilling of the samples (consequence of the drill rotation) through the evaluation of an output voltage measured across a known reference resistor, in presence of a current.

The results obtained on a pottery sherds put in evidence that the methodology allows controlling the temperature increasing. In the examined case, the average temperature variations resulted lower than 50 K. This value of temperature not implies any bleaching of the TL signal of interest.

The perspectives regard the validation of the proposed model both improving the measurement methodology and performing infrared thermal imaging in order to evaluate the method accuracy. Furthermore, in order to quantify the probable underestimation of the TL signal due to the heating by drill, a relationship between reached temperature during sampling and signal loss must be studied.

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