

# SAR AND MAAD TL DATING OF "CAROSELLI" FROM THREE SITES IN CALABRIA, SOUTH ITALY

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# ABSTRACT

In this work three types of "Caroselli" from different sites in Calabria have been studied. The "Carosello" is a structural hollow element made of clay, placed in arches, in domes or even in the walls of buildings such as churches and houses, with the function of relieving vaults and main structures. Diagnostic tests have been conducted on the "Caroselli" in order to determine the presence of luminescent material, the final goal being the age assessment of such artifacts using thermoluminescence (TL) techniques. Of course the historical frame of the buildings, to which "Caroselli" belong, is known from their style and architectural considerations. The scope of our dating is to supply additional information to clarify whether "Caroselli" were used from the very beginning of building construction or employed in subsequent restorations. The dating process has been conducted using two protocols, the Multiple Aliquot Additive Dose (MAAD) and the Single Aliquot Regenerative-dose (SAR), both by applying TL. The correction obtained by the SAR TL method is preferred because it allows a more accurate estimate of the equivalent dose and avoids problems arising from sample sensitivity. The results show that the three "Caroselli" can be respectively dated back to 1844±11, 1825±40, 1803±30 A.D.. Such results suggest that for two sites ("Madonna del Buonconsiglio" church and "Torre dello Zuino" colonial house), the "Caroselli", as structural elements, were used since the beginning of the building construction, while for the remaining site ("*Pettoruto*" church) probably the "Caroselli" were added in a successive restoration of the building, since the historical context of this building belongs to the fourteenth century.

KEYWORDS: Thermoluminescence; Equivalent dose; SAR, MAAD, Caroselli.

#### INTRODUCTION

Dating clay artifacts is an important diagnostic tool towards classifying archaeological findings as well as explaining their historical context. The work of this paper aims to represent a successful conclusion to an interdisciplinary work performed by scientists and technicians in the Departments of Earth Sciences and Physics at the University of Calabria on some peculiar structural elements, termed as "Caroselli", which are made of clay. The "Carosello" is a structural hollow element made of clay, placed in arches, in domes, or even in the walls of buildings such as churches and houses, with the function of relieving vaults and main structures. In particular the scope of our dating is to supply additional information in order to clarify whether "Caroselli" were used from the very beginning of the construction or employed in a subsequent restoration.

sedimentary Clay is а rock, petrographically defined as clastic pelitic rock, obtained by the fragmentation and decomposition of feldspathic rocks in very small particles. It has a variable composition and can be divided into primary and secondary clay according to the formation process. Despite their variable composition, clays always show minerals such as quartz, feldspars, calcium carbonate, iron oxides and hydroxides and other minor components as well as organic substances and various random materials (Cuomo di Caprio, 1985).

Thermoluminescence is a technique that allows us to date pottery or terracotta in general, since their crystalline components, such as quartz or feldspars, are provided with intense luminescence signals (Murray, and Wintle, 2000). Some crystals like those included in ceramics, when heated, are able to emit light in a manner proportional to the dose of ionizing radiation accumulated over time by the crystals themselves. Since their formation the clay components accumulate a "geological dose" that they lose when heated at a temperature sufficient to empty all the electron traps. Once the object is deposited or is used, it begins again to accumulate a certain amount of absorbed dose ("archaeological dose") due to the natural radiation coming from the ceramic itself (U, Th, K40) and from the surrounding environment. The subsequent heating to high temperatures in the laboratory, results in the release of the light corresponding to the dose accumulated over time. By reading the light emitted, it is possible to calculate the time elapsed since the original heating in the oven, according to the well known equation age (Aitken, 1985; Murray, and Wintle, 2000; Murray, and Roberts, 1998; Furetta, and Gonzàlez Martinez, 2007, Liritzis et al 1994, 2002) expressed in years:

Age = Paleodose (Gy) / Dose Year (Gy / year).

In the following paragraphs we describe all the research phases specifying the methodological criteria, in particular this work represents the first application of the SAR/TL protocol to terracotta elements from Calabria. As successively reported, we used both SAR and MAAD methods by applying TL which give satisfactory agreement, but in our conclusions we use the results obtained by the SAR TL method because it allows a more accurate estimate of the Equivalent Dose (ED) and highlights problems arising from sample sensitivity thus enabling proper correction.

# SAMPLING AND HISTORICAL BACKGROUND

In the historic centers of many towns of Calabria we can still find examples of traditional buildings made with peculiar materials and unique manufacturing technologies. In this work we studied some specific, characteristic components used to make particular structures, which are produced from clay handworks. They have a cylindrical shape (see Figs.1-2) and are empty inside in order to render them very



Figure 1: Picture showing the typical structural element termed "Carosello".



Figure 2: This picture clearly illustrates the use of "Caroselli" as structural element in the wall structure.

light in weight; the so-called "Caroselli".

The juxtaposition of such elements gives origin to simple or complex construction systems such as arches, domes, floors and partitions, and these peculiar construction methods appear in modest domestic contexts, as well as in important historicalarchitectural buildings both in civil and religious contexts. Moreover, the structures containing "Caroselli" showed numerous advantages such as protection against earthquakes, thermal excursion, humidity



Figure 3: Schematic drawing of a wall cross section showing the spatial allocation of "Caroselli".

or accidental fires, in addition to their good grip to mortar plaster. A picture along with a schematic sketch showing the usual allocation of "*Caroselli*" in a building are presented in figs. 2, 3 respectively. Such elements are employed as "fillers" to lighten the structure and it can be seen that they are "shielded" by an external wall on one side and by a thick plaster layer, being also embedded in cement mortar.

In historic buildings the use of "*Tubuli*" is known, similar to "*Caroselli*", concatenated to each other, that gave rise to architectonic structures of considerable size. Their use has been known since the Byzantine time, when also different artifacts, like amphorae and jars, were employed with the same purpose of lightening the structures [Gattuso, C.(1996); Crisci, G.M. (1997); Arsalan, E.A. (1965)].

The "*Caroselli*" look like a hollow cylindrical body, closed at one end. The top face has a convex shape and is characterized by the presence of a hole that is often located along the sides, which has a small diameter of about 0.5 cm. There is a large variety in size and shape of such artifacts, so that short and flat "*Caroselli*" or long and slender ones can be found. On the external surfaces we can often observe manual work traces, due to finger pressure of the craftsman in order to allow a better

adhesion to the cement mortar. Moreover, it must be pointed out that the presence of the hole had a very precise function: during the annealing process in the oven, the hole allows an easier circulation of air, avoiding its thermal expansion due to heat; the thermal expansion can cause damage or deformation, and, on the other hand, the passage of hot air inside the carousel, allows a more uniform annealing (Gattuso, et al. 2000; Rutigliano, 1996).

The samples used for the present analysis have different sizes and weights: the cylindrical body height ranges between 14 to 19 cm and from 9.5 to 12 cm in diameter; the side average thickness is 6 mm, while the weight (obviously depending on size, shape, working process and type of clay used) ranges from a minimum of 550g to a maximum of 950g.

Such clay elements have been used in different geographical areas, in particular in southern Italian regions (Puglia, Molise, Abruzzo and Calabria), and they are named or termed by different names ("*Pigniatelli*", "*Caccavelle*", "*Caroselli*" and "*Bubbole*").

Specifically in Calabria various kinds of "Caroselli" have been found and classified,



Figure 4: Map of Calabria displaying the geographical distribution of towns and villages where "Caroselli" were recovered.

mainly along the ionic coast of the region (Fig.4).

The "Caroselli" objects of this study come from three different sites in Calabria: the Sanctuary of "Madonna del Buonconsiglio" in San Giacomo di Cerzeto, Cosenza province (the samples referring to this building will be termed as CER here after); the Church of "Pettoruto" in Lattarico, Cosenza province (termed as LAT hereafter); and the Colonial "Torre dello Zuino" in Santa Caterina dello Ionio, Catanzaro province (termed as SCI hereafter).

The Sanctuary of the "Madonna del Buonconsiglio" is located in San Giacomo, an area which belongs to the town of Cerzeto, a small village in the inner part of the Cosenza province. The Church of "Madonna del Buon Consiglio" was built between 1730 and 1748 in memory of the venue in Italy of a "Madonna del Buon Consiglio" painting (April, 25 1468), a painting coming from Albany after the Turkish invasion of 1468. Since March 15, 2002 the church has been promoted to diocesan Sanctuary of "Madonna del Buon Consiglio" under the care of the brotherhood "Maria SS.ma del Buon Consiglio". Numerous were the alterations made over the centuries and during one of the recent interventions in 1996, on the occasion of the dome removal above the main altar, it was possible to preserve and then to study some of the "Caroselli" which escaped the destruction.

The "*Pettoruto*" *Church,* located in the town of *Lattarico* within the Cosenza province, belongs to the XIV century. Even if only the monument ruins are remaining today, after a recent earthquake at the beginning of the twentieth century, it is possible to recover "Caroselli" in various part of the building. It is important to note that, besides the aforementioned earthquake, this building was completely destroyed by another earthquake in 1783 and rebuilt in 1834.

The Colonial farm, named *"Torre dello Zuino"*, is situated in the town of *Santa Caterina* within the Catanzaro province. The farm belongs to a complex of several

separated buildings, some of which are stables and mills located in peripheral zones, while the other buildings are situated around a courtyard fenced by high walls. The farm house is located on the north side of the courtyard unfortunately in an advanced state of neglect and degradation. The "*Caroselli*" have been found in the arches, in the walls and elsewhere in the building.

#### INSTRUMENTATION AND ANALYSIS

The diagnostic plan includes a first phase for the cognitive context in which the *"Caroselli"* were found and then a physicalchemical characterization of constituents followed by thermoluminescence analysis to date the artifact using the Single Aliquot Regenerative-dose (SAR protocol). Preparatory diagnostic tests have been conducted on the samples to achieve a sufficient knowledge about the materials which are the basic constituents of terracotta (Renzelli, 2010).

Surveys were conducted using а Scanning Electron Microscope (SEM) equipped with chemical microanalysis probe (SEM-EDS) to highlight the morphological characteristics the samples and the chemical characterization of inorganic components. Our SEM has a spatial resolution of about 1µm2 and it has been modified (Pingitore, et al 2008) to combine usual SEM investigations with Cathode-Luminescence (CL) analysis on the same surface. Moreover, XPS (X-ray Photoelectron Spectroscopy) analysis, performed on the samples, allowed to determine not only the chemical composition, but also the nature of bonds involved, through the analysis of the photoelectronic peak positions (Chemical Shift). Finally the dating has been obtained via thermoluminescence measurements using a TL reader from Risø, by applying the SAR protocol and comparing it with traditional methods (MAAD). For the

artificial doses we use a beta source characterized by a dose rate of 0.1 Gy/sec. In this context we have considered as Annual Dose a value of  $2.7\pm0.02$  mGy/year. This value is a mean of the annual dose rate data reported in (Troja, et al. 1996), which has been used to analyze some findings from Sicilia, a region that is geographically near our archaeological area. Generally we used a heating rate of 5 °C/s up to a temperature of 450 ° C and the samples were irradiated with seven regeneration doses.

#### SAMPLE PREPARATION

Sample preparation took place in condition of darkness with the aid of a single soft red light (> 600 nm). We have chosen the quartz inclusion technique, since with this method we obtain grains of larger size which can then be submitted to acid solution to eliminate surface carbonates. The preparation consisted of the following sequential steps. The first stage of preparation involved sampling (using a low speed drill) in the sample bulk, in order to avoid external contamination (such as sources of light mostly, heat or radiation to which the sample surfaces may have been exposed after the discovery). To select grains of the same size, the powder has been sieved using sieves with meshes between 63 and 125 microns. The second step involved a chemical treatment with 10% HCl to eliminate any carbonates present. Then we applied treatment with 30% H<sub>2</sub>O<sub>2</sub>, which is necessary to eliminate organic material. The so obtained different crystalline fractions were separated by using a heavy liquid solution. The quartz grains were then extracted, dried and used for the dating process. These grains were fixed with silicone oil on stainless steel discs of 9.7 mm in diameter and then loaded on the spectrometer.

It is known in literature that the conventional regeneration procedure

Step	Sequence		label
1	NTL 450°C,5°C/s		NTL
2	Irrβ 1Gy + TL	Test dose	Test NTL
3	Irrβ 2Gy + TL	First regenerative dose	First TL 2Gy
4	Irrβ 1Gy + TL	Test dose	Test TL 2Gy
5	Irrβ 3Gy + TL		TL 3Gy
6	Irrβ 1Gy + TL	Test dose	Test TL 3Gy
7	Irrβ 3.5Gy + TL		TL 3.5Gy
8	Irrβ 1Gy + TL	Test dose	Test TL 3.5Gy
9	Irrβ 4Gy + TL		TL 4Gy
10	Irrβ 1Gy + TL	Test dose	Test TL 4Gy
11	Irr $\beta$ 4.5Gy + TL		TL 4.5Gy
12	Irrβ 1Gy + TL	Test dose	Test TL 4.5Gy
13	Irrβ 5Gy + TL		TL 5Gy
14	Irrβ 1Gy + TL	Test dose	Test TL 5Gy
15	Irr $\beta$ 2Gy + TL	Last regenerative dose	Last TL 2Gy
16	Irrβ 1Gy + TL	Test dose	Test TL 2Gy

produces changes in sensitivity of the quartz grains during repeated TL measurements (Nakagawa, et al 2003; Przegietka, et al 2005), so that one must take into account these changes in sensitivity in order to get a reliable estimate of the equivalent dose. In this work the modified SAR protocol has been adopted, where the

Table 1: Sequence illustrating the procedure for S.A.R. measurements adopted in this study. Refer to this table to identify the measurement labels.

thermal treatment is excluded with respect to the original SAR protocol (Hong, et al 2006). The sequence of measurements is summarized in Table 1, the identification labels used throughout the text and figures are also reported for each measure.

## **RESULTS AND DISCUSSION**

The luminescence signal in quartz crystals, related to the Equivalent Dose (ED) in the age equation, can be released from traps by both thermal stimulation (Thermo Luminescence, TL) or Optically Stimulated Luminescence (OSL).

Regarding the determination of the

Equivalent Dose, the traditional method involves normalization procedures due to various irradiation rates or to difference in sample masses, or to sample sensitivity problems which render such methods more complicated and time consuming. Instead the procedure based on a Single Aliquot Regenerative-dose (SAR) has the advantage, over the traditional methods (MAAD), of avoiding standardization or sensitivity problems and reduces the effort required in sample preparation.

The SAR protocol was extensively discussed by Murray and Wintle (Murray, and Wintle, 2000) who proposed a modified regeneration method with respect to the conventional one. The conventional regeneration procedure has the advantage of being simpler and easier; however, bleaching and heat treatments carried out repeatedly in laboratory produce changes in the sensitivity of the luminescence signal characteristic of the sample. Initially, the SAR protocol, using optical stimulation, was applied mainly for dating sediments, the field that has inspired the development of the technique. Subsequently in literature the SAR protocol has been applied to the case of quartz by TL measurements and the results on the equivalent dose have been compared with those measured by the OSL technique giving satisfactory results and

similar accuracies (Hong, et al 2006). In our work we decided to use the protocol proposed by Hong et al.

In this work we performed several measurements for each sample using and comparing both methods, the SAR-TL and the Multiple Aliquot Additive Dose protocols (MAAD). Additional applications of comparison between Additive Dose and Single Aliquot techniques can be found in other works (Tso, et al 1994; Michael, et al 2006). The determined equivalent doses are reported in Tables 2 (SAR protocol) and 3 (MAAD protocol). The value of the equivalent dose is the mean value over all the measurements for which the sensitivity correction has been successful. We just

Sample	Mean ED (Gy)	Annual Dose (Gy/year)	Years ago	Age
CER	0.45±0.03	0.0027	~ 166±11	~1844±11 A.D.
LAT	0.5±0.1	0.0027	~185±40	~1825±40 A.D.
SCI	0.56±0.08	0.0027	~ 207±30	~1803±30 A.D.

SAR Protocol

#### Table 2: Equivalent Dose values as obtained using TL data based on the S.A.R. protocol.

MAAD Protocol					
Sample	P(Gy)=ED+I	Annual Dose (Gy/year)	Years ago	Age	
CER		÷	â	÷	
LAT	0.7±0.2	0.0027	~ 262±75	~1748±75 A.D.	
SCI	0.7±0.1	0.0027	~262±37	~1748±37 A.D.	

Table 3: Equivalent Dose values as obtained using TL data based on the AD protocol.







Figures 5a, 5b, 5c: Examples of Glow Curves for the CER (fig.5a), LAT (fig.5b), SCI (fig.5c) samples respectively with correction for sensitivity as described in the text. The TL response to regenerative doses First 2Gy and Last 2Gy is almost the same, indicating that the correction is successful.

mention that, as reported in Table. 3, for the sample labeled CER, the AD protocol was unsuccessful because of sensitivity



Figure 6: Example of plateau test.



Figures 7: Determination of the Equivalent Dose, based on the Single Aliquot Regenerative-dose (SAR) protocol, using sensitivity corrected TL signals from quartz in SCI sample. The experimental points are fitted by a linear function. Note that at 2Gy the two experimental points, representing the success of sensitivity corrections, coincide.

problems, probably because the quartz quality of this sample is different from that contained in the other samples. It can be seen that we obtain a good agreement between the two used protocols within the estimated errors.

In Fig. 5 we report examples of TL glow curves corrected by using the sensitivity correction procedure described by Hong, et al. (2006). The correction for sensitivity changes was made by monitoring the luminescence response of the sample to a subsequent test dose of 1 Gy, given to the sample after measuring the natural dose and

after each regeneration step, as resumed in Table 1. For both, the natural dose and the subsequent regeneration dose, the correction procedure consists in normalizing each measurement to the intensity of the relative test dose. Because the first regeneration dose equals the last regeneration dose, the TL intensities for these two regeneration doses should be identical if the correction is successful. As shown in Fig. 5 the TL responses for the first and the last regeneration doses are indistinguishable.

Based on the result of the plateau test, all the glow curves have been integrated in the temperature range 290–350 °C. An example of plateau test is reported in Fig. 6.

Fig. 7 shows an example (sample SCI) of the sensitivity-corrected TL signal versus



Figures 8: Determination of the Equivalent Dose, based on the MAAD protocol, using data from sample LAT.



Figures 9: Typical examples of supra linearity correction to the Equivalent Dose in the case of sample SCI. The correction, labeled by "I", has been extrapolated to "0" intensity.

radiation dose. The data of the growth curve, corrected for sensitivity, were fitted by a linear function. The equivalent dose was calculated from the growth curve extracting its value at the corrected NTL (Natural Thermoluminescence).

The equivalent doses determined for each sample are listed in Table 2 where we report only mean values over the measurements for which the sensitivity correction was successful. For some samples, even if extracted from a single "Carosello", the sensitivity correction was not successful. We can attribute such failure to the non homogeneity of "Caroselli" as handmade artifacts, since they are used as "filler" to lighten the building structure and so their manufacture is very rough without attention to clay selection and refinement. To determine the equivalent dose, a comparative study has been carried out between the TL measurements obtained using the SAR protocol and those obtained by the MAAD method (Furetta, and Gonzàlez Martinez, 2007; Duller 1992). The results for the ED obtained by the MAAD method are shown in Fig. 8 (sample LAT).

Concerning the CER sample no satisfactory results for MAAD have been obtained. Plotting the so obtained intensities as a function of the given dose, we get a linear behavior from which we calculate the equivalent dose. The natural TL intensity is then extrapolated in correspondence to zero given dose. Supralinearity corrections have been considered only for the samples for which the MAAD method has been satisfactory (samples SCI and LAT). The magnitude of this correction is reported in Fig. 9 (sample SCI), where we show the TL intensity as a function of the added dose. The obtained corrections are included in Tab. 3.

It was evident from the beginning of this work that we are faced with a TL signal which is low since the artifacts are presumed to be very recent. But the aim of our work is to state if the "*Caroselli*", as structural elements, had been employed from the beginning of the three constructions or if they are much more recent than the buildings and so employed in a subsequent restoration.

From our results reported in tables 2-3, we can see that the obtained signal is as low as expected (below 1 Gy) and the associated error can be estimated between 7 and 28%. So the age of the artifacts is recent and from our results we can argue that this age ranges between 160 and 260 years before the present day. This result is perfectly compatible with the location and the historical background of two buildings, the church of "Madonna del Buonconsiglio" and the colonial house of "Torre dello Zuino". While the dating of "Caroselli" belonging to the third site ("Pettoruto" church) is evidently more recent than the historical collocation of the building.

## CONCLUSIONS

This work represents the first use of the TL for dating Caroselli artifacts from Calabria. The values obtained by measuring the equivalent doses by both SAR and MAAD methods by applying TL give satisfactory agreement and similar results (Tables 2,3). However, the correction obtained by the SAR TL method is preferred because it allows a more accurate estimate of the ED and avoids problems arising from

sample sensitivity, which, in this way are highlighted and corrected. From our results, even if applied to very recent artifacts, we can presume that, in the case of "Madonna del Buonconsiglio" and "Torre dello Zuino", the "Caroselli" are contemporary to the age of the buildings and so it is possible to suggest that such structural elements were used since the beginning of the building construction. In the case of "Pettoruto" church, our dating suggests that the "Caroselli", as structural elements, have been added in a successive restoration of the building, since the historical context says that this building belongs to the fourteenth century. In fact it is known that this church was completely destroyed by an earthquake in 1783 and rebuilt in 1834: this historical event is consistent with our results and supports our suggestion about the use of "Caroselli" in a subsequent restoration.

## **ACKNOWLEDGEMENTS:**

This article is co-financed with support from the European Commission, European Social Fund and the Region of Calabria. The author is solely responsible for this article and the European Commission and the Region of Calabria decline any responsibility for any use which may be made of the information contained therein.

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