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A study by thermal treatment and X-ray powder diffraction on burnt fragmented bones from tombs II, IV and IX belonging to the hypogeic necropolis of "Sa Figu" near Ittiri, Sassari (Sardinia, Italy)

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Abstract

In the course of an anthropological investigation of three tombs of the hypogeic necropolis of "Sa Figu" (near the village of Ittiri in North Sardinia, Italy), numerous burnt bone fragments were collected. In particular, from the whole necropolis we selected a tenth of long bones from tomb IV, a small fragment of femur from tomb II and four fragments belonging to tomb IX. The aim of this work is to determine a temperature range to which the bone fragments were heated, in order to assess a funerary cremation which was presumably used at that time to quickly and hygienically eliminate the flashy parts of deceased people rather than a purification rite with fires inside the sepulchres. We attempted to evaluate the range of temperature to which the bones were likely subjected, making a joint use of thermal treatment and powder X-ray diffraction investigations. From our X-ray line broadening results carried out with a modern approach (Rietveld method), it emerged that a group of five fragmented burnt bones (one specimen belonging to tomb IV, the other four from tomb IX) were subjected to a heat treatment in a temperature range from 600 and 750 °C. Conversely another group of bones (belonging to tombs II and IV) turned out to be subjected to temperatures not higher than 250 °C.

Keywords: Burnt bones; Hydroxylapatite; Funerary cremation; Thermal treatment; Powder X-ray diffraction; Rietveld method

1. Introduction

In Sardinia, burnt human bones are rarely found as it concerns an archaeological context that certainly refers to the Bronze age. In the site named "Anghelu Ruju" (Alghero, Sassari) a large number of cremation traces were found, presumably belonging to the Bronze Age (Germanà, 1984; Levi, 1952; Taramelli, 1909). Mixtures of ash and bones were invented in the site named "S'Isterridolzu" near Ossi (Sassari) (Germanà, 1980) and especially in tomb IX of Sa Figu (Melis, 2003a), in the context of the so-called Bonnanaro culture (see Table 1) (Melis, 2003b). Concerning our rare cases of apparently burnt bones, virtually no studies have been hitherto conducted in order to demonstrate or exclude the cremation rite in Sardinia at that period.

It is now accepted that in Sardinia, during the Phoenician colonization, the prevailing funerary practice was cremation while the subsequent Carthaginian domination stimulated the diffusion of the inhumation practice. This was of general use in the Vth and IVth century BC. In the late-punic age, perhaps due to the Greek influence, cremation was practised again. However, in the IIIrd and IInd century BC this funerary practice, even though frequently adopted, was not completely established (Barreca, 1988; Bartoloni, 1981; Fedele, 1977,

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Table 1
A proposed chronological sequence and classification of the nuragic civilization of Sardinia

1700–1500	Bronze Age	Middle 1	Nuragic IA	Sa Turricula (Bonnanaro 3rd)
1500–1350	Bronze Age	Middle 2	Nuragic IB	San Cosimo, metope ceramic
1350–1200	Bronze Age	Recent	Nuragic II	Comb ceramic, grey ceramic
1200–900	Bronze Age	Final	Nuragic III	Pre-geometric ceramic
900–730	Iron Age	1st Iron 1	Nuragic IVA	Geometric
730–600	Iron Age	1st Iron 2	Nuragic IVB	Oriental
600–510	Iron Age	1st Iron 3	Nuragic IVC	Archaic
510–238	Iron Age	2nd Iron	Nuragic VA	Punic
238 AC–476 AC	Historical period		Nuragic VB	Roman

1978, 1980, 1983; Martin Wedard, 1978; Mingazzini, 1948). On the other hand, it is not known if this practice was even used in the Bronze age.

In order to investigate these controversial aspects, we have undertaken a study by X-ray powder diffraction of some selected burnt bones from three Sa Figu tombs and have made use for comparative purposes also of a cremated reference bone coming from a cinerary urn of the Punic and Phoenician archaeologic area of Monte Sirai (Amadasi and Branconi, 1967; Bartoloni, 1989), near the village of Carbonia (Cagliari). This was decided in order to have a reliable reference of the microstructure properties of bones induced by a cremation ascertained at that age. To perform the very accurate investigation required to answer properly the problem in hand, this work takes full advantage of the latest progress in the powder diffraction technique both in terms of instrumentation and data evaluation. In particular, our approach makes extensive use of the Rietveld refinement technique (Rietveld, 1967), combined with precise thermal treatments of the archaeological bones.

Shipman et al. (1984) investigated the microscopic morphology of varied osteological materials and used X-ray diffraction to assess whether specimens of unknown taphonomic history were burnt and the maximum temperature reached by those specimens. These investigations are based on the fact that heating the bones causes a sharpening of diffraction patterns, attributed to increased crystallite size and decreased lattice strain of osteological phases.

Alternatively, Bartsiokas and Middleton (1992), with the aim of characterizing and dating recent and fossile animal and human bones, suggested measuring a so-called crystallization index from their diffraction patterns, which can be mainly ascribed to the presence of natural apatite phase. The crystallization index, which is strictly related to the peak sharpening effects, was actually defined as the intensity ratio of (300)/(202) line profiles of hexagonal apatite, which normally occur as shoulder of the most intense (211) line in the 2θ angular range from 31° to 35° when using CuKα radiation. A linear correlation between crystallinity index and bone age was reported over a period length of more than 10^6 years. It should also be noted that another crystallinity index was almost concomitantly defined by Person et al. (1995) using more peaks belonging to the same angular range.

It is clear that the two above-mentioned lines of work may be conflicting, especially if the bones were subjected simultaneously to both physical effects of burning and annealing across very long periods of time. However, in a sufficiently short period of time it seems that the two effects can be assumed to be well distinct. As a matter of fact the time period investigated by Bartsiokas and Middleton involves some million of years to observe significant changes in the crystallization index, which is a quantity determined with an error bar of a few percent. This means that the crystallinity index may be regarded as virtually unchanged in a period as short/long as ten thousand years, which certainly is the case with the Sa Figu bones.

2. The archaeological context

The hypogeic necropolis of Sa Figu is located in the northern side of the Coros plateau, north-east from the Ittiri village (Sassari) and at a short distance from another important hypogeic necropolis, the so-called ‘‘domus de janas’’ (meaning House of the Fairies in the local language), i.e., a place named Ochila.

At the beginning of the 1960s, the presence of four tombs in this area which were previously violated was reported by Contu (1961). The excavations of tombs labelled as I and III brought to light materials of the Middle Bronze age. Starting from 2001, new archaeological excavations were undertaken which have so far involved tombs II, IV, V, VIII, IX and X (Melis, 2002, 2003c,d).

Thus, the hypogeic necropolis of Sa Figu currently includes 11 existing tombs and only traces of some others previously destroyed. At least six periods of use were envisaged in this funerary area, starting from the neolithic period and ending with later sporadic reuse during the Roman age.

In the first period, corresponding to the excavation of the domus de janas hypogea, which is dated back to the late Neolithic age, tombs I, II, III, VI, IX, X and XI were likely realized.

In the second period, corresponding to the copper age, analogously to what happened in other Sardinian domus de janas, the hypogeic tombs of Sa Figu were again used as burial grave, without any meaningful change in the funerary rite.

The third phase of the necropolis use involves the Early Bronze Age, with the local ‘‘Bonannaro Culture’’. Evident traces of this period emerged during the excavation of tomb IX.

Nevertheless, the most interesting monumental aspect is represented by the fourth occupation phase of the area, occurring at the beginning of Nuragic era (middle Bronze age, 1700–1300 BC). In this period, the so-called ‘‘giant tombs’’ appear in central northern Sardinia, which are characterized by orthostatic structures and arched steles in the middle of the ceremonial exedra. On the north-western side of Sardinia, within a restricted area, this kind of giant tomb has been mainly excavated in limestone rocks, providing hypogeic funerary rooms (called hypogea with ''architectural prospect''). This was frequently done reusing previous domus de janas. In the necropolis of Sa Figu the evidence suggests that three preexisting domus de janas (tombs II, III and VI) were enlarged and transformed into ''domus with architectural prospect'', with the addition in the frontal façade of the typical elements of the giant tombs: arched stele, semicircular hexedra and upper tumulus. Conversely, tombs IV, V, VII and VIII were excavated ex novo.

The fifth phase of the necropolis use is relevant to sporadic usage in the late nuragic era (first iron, VIII–VIIth century BC). The final period of necropolis use —we as yet do not understand if undertaken with a funerary scope—is ascribed to the Roman age.

3. Experimental

After the excavations carried out in the hypogeic necropolis of Sa Figu, bone fragments from tombs II, IV and IX were collected and examined, whose demographic and paleo-pathological features are still under investigation.

Little evidence of fire was observed inside tombs II and IV. As a matter of fact, only ten burnt fragments were found in tomb IV with various degrees of colour and just one burnt femur fragment in tomb II.

The situation inside tomb IX was even more interesting. From immediately below the surface it emerged on a single significant cultural level, characterized by concentrated bones extremely fragmented and burnt, along with numerous pottery fragments. The deposition layer was sitting on a thick layer of ashes in contact with the basal pavement. The whole of the bones and pottery fragments was surmounted by a cranium, located in the south-western side of the cell (see Fig. 1), according to the rite already observed in ''Su Crucifissu Mannu'', Porto Torres (Sassari) (Ferrarese Ceruti, 1972–74) and in other sites near the Sassari area.

From the entire collection, some samples were selected for a thorough physico-chemical investigation. The samples are hereafter labelled as:

- heated tibia, young tibia, burnt tibia (see Fig. 2), grey femur, belonging to tomb IV;
- bone tomb II, belonging to tomb II;
- cranium (see Fig. 3), cranium II, teeth (see Fig. 4), mandible, (see Fig. 5), belonging to tomb IX.

Thus, we investigated the structure and microstructural features of the bones collected in the grave and compared our results after calibrating a sample bone as a function of the temperature, in order to surmise the thermal treatment involved and to lend support to possible cremation practices.

Thermal treatments were performed using the furnace of a SETARAM instrument from room temperature up to 900 °C in air at a heating rate of 20 °C/min.

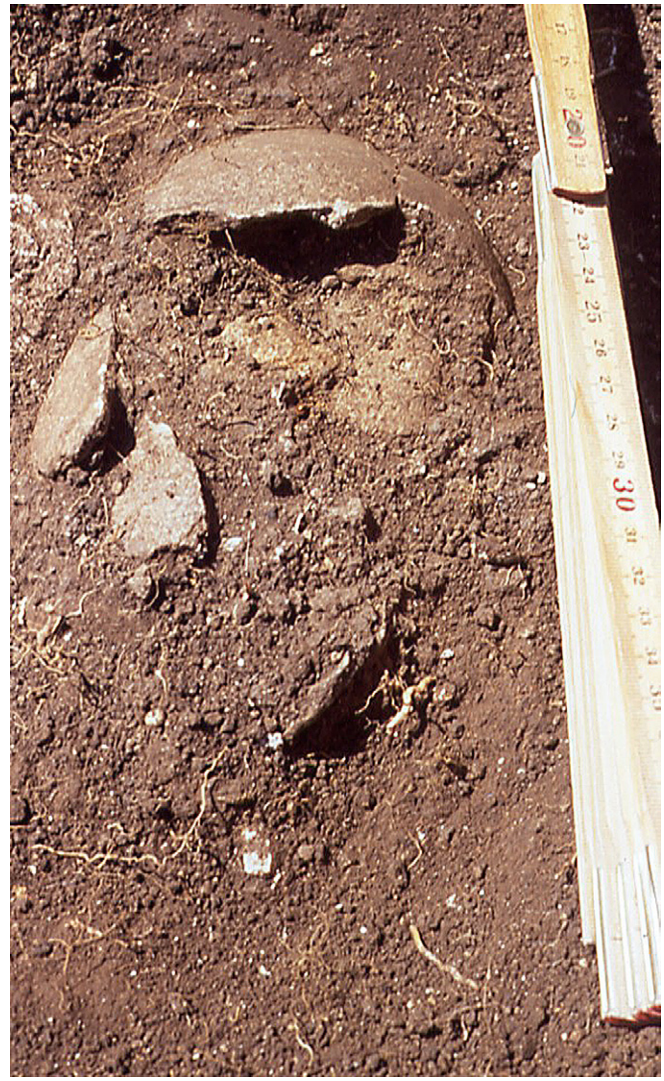

Fig. 1. A crane fragment emerged during excavation of tomb IX which was found later to surmount a collection of burnt bones and pottery.

Fig. 2. A fragment of burnt tibia bone retrieved from tomb IV. The scale unit is 5 cm.

Fig. 3. A fragment of burnt cranium bone retrieved from tomb IX.

Fig. 5. A fragment of burnt mandible retrieved from tomb IX.

The samples used for calibration were heat-treated at various temperatures in air using a NEY muffle at an heating rate of 20 °C/min.

Powder X ray diffraction (XRD) patterns were recorded with a Bruker D8 diffractometer in Bragg–Brentano geometry using CuKα radiation ($\lambda = 1.5418$ Å). The X-ray generator worked at a power of 40 kV and 40 mA and the goniometer was equipped with a graphite monochromator in the diffracted beam. The resolution of the instrument (divergent and anti-scatter slits of 0.5°) was determined using α-SiO_2 and α-Al_2O_3 standards free from the effect of reduced crystallite size and lattice defects. The powder patterns were analyzed according to the Rietveld method using the program MAUD (Lutterotti et al., 1998) running on a personal computer.

4. Results and discussion

To follow the structural evolution of bone as a function of temperature, we started from an ancient femur fragment in the bone collection of tomb IV, which was certainly unaffected by any fire. This will be hereafter coded ''sample bone''.

Fig. 6 shows the Thermo-Gravimetric (TG) curve of the bone used as a standard in the range from room temperature up to 900 °C. After an initial oscillation of the balance, one sees a continuous weight loss of the bone with a first quick decay between 300 and 400 °C. The whole weight loss for the range of temperature here investigated amounts to ca. 17% of the initial mass. This pattern has similarities but also differences with the TG plot reported by Bigi et al. (1997) from a sample of trabecular rat bone. The weight loss appears limited in our case and this may be ascribed to the fact that our human bone is quite ancient and actually refers mainly to the cortical zone. Nonetheless, the quick weight decay at 350 °C is common in the observation of Bigi et al. and in ours.

Fig. 7 shows the diffraction patterns of sample bone as a function of the indicated treatment temperatures, where dots are data points and full lines represent the fit after the Rietveld refinement. The bar sequence at the bottom is

Fig. 4. A fragment of burnt teeth retrieved from tomb IX.

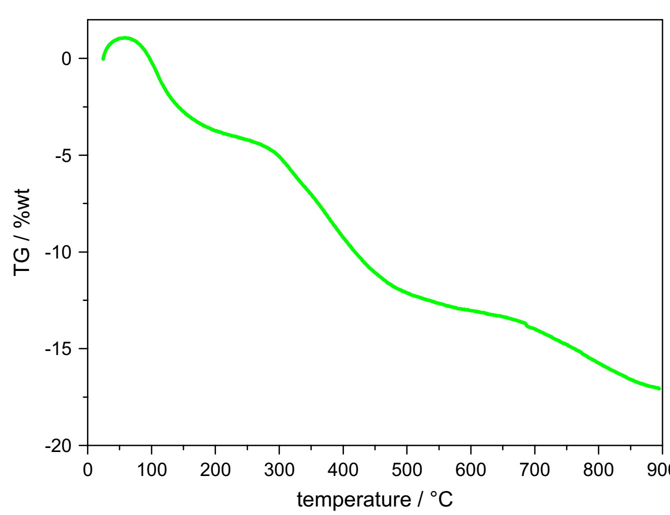

Fig. 6. Weight loss curve of a sample bone coming from tomb IV of Sa Figu and not subjected to fire, which was used as a reference material.

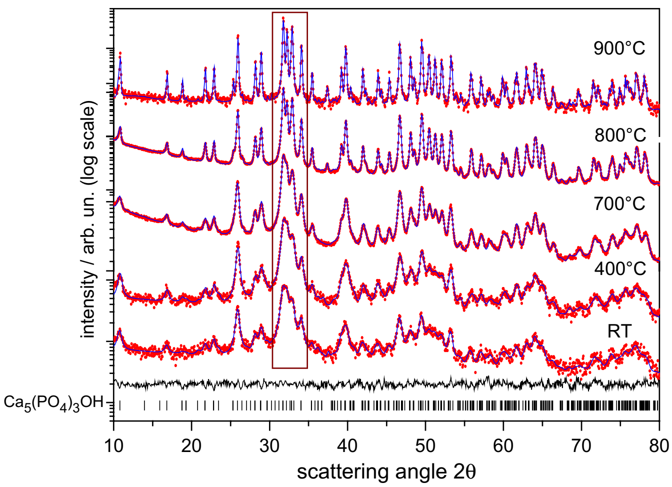

Fig. 7. XRD powder patterns of the sample bone treated at the quoted temperatures.

a fingerprint of the monoclinic hydroxylapatite structure factor and marks the expected position of each peak. Moreover, the band above marks and below the first pattern is the difference between square root of calculated and experimental intensities (residuals). The trend shown by our sample bone is similar to that published by Rogers and Daniels (2002) in the case of a 67-year old male bone specimen heated to 1200 °C. After deconvolution of the instrument resolution function, these authors performed a very accurate line-broadening analysis across ca. 30 hkl peaks in each of the eight specimens analysed and applied the Scherrer equation to determine the average crystallite size (Scherrer, 1918).

From the methodological point of view, the Rietveld refinement that we have adopted stands on approximations similar to those involved by Rogers and Daniels, but has the inherent advantage of carrying out automatically a global structure and microstructure refinement analysis, supplying the required quantitative estimations with a straightforward and fast procedure.

As can be seen, all the patterns are described satisfactorily with the monoclinic structure factor of apatite which turns out to be more advantageous with respect to the hexagonal form, for which the resultant agreement indices were not comparatively so good. Apart from the goodness-of-fit, from the bone pattern evolution a clear peak sharpening emerges as a function of the treatment temperature, which is normally ascribed to a concomitant increase of average crystallite size and decrease of (static) lattice disorder. In particular, the considerable line broadening of the as-received bone at room temperature is indicative of a nanocrystalline condition which was recently evidenced also by Meneghini et al. (2003) in human foetal bones using synchrotron radiation, together with the presence of an amorphous fraction. However, we do not need to include any amorphous contribution in the room temperature specimen in order to describe the experimental pattern and to retrieve a satisfactory uniform distribution of residuals across all of the inspected diffraction range. Moreover, little strain (below 10^{-4}) was measured with our refinement procedure, suggesting that all the line broadening is constant across the peaks and that it can be ascribed just to very small crystallite size. This held also for the other heat-treated specimens, for which a slow sharpening of the peaks (increase of the average crystallite size) was found up to 700 °C while, at higher temperatures, the sharpening process appeared accelerated. Of course at 900 °C and above the line broadening approaches the physical value determined by optical geometry and slits of the instrument, so not too much can be deduced under these conditions about the growth process. The lattice parameters and the related cell volume of the monoclinic phase do not change appreciably with the temperature treatment apart from a very slight decomposition of the hydroxylapatite phase to calcium oxide in the specimens heated above 700 °C, suggesting that the apatite may contain carbonate units. This aspect may also be related to the behaviour of the TG weight loss in Fig. 6.

As expected from the line-broadening analysis, the initial value of the average crystallite size around 200 Å at room temperature slightly and smoothly increases up to 300 Å corresponding to a thermal treatment up to 700 °C. From this value onward, the average size quickly increases.

With these refined data we have made a calibration curve where rhombic symbols are interpolated with full lines (Fig. 8). The twofold trend appears to be in relative agreement with that of Rogers and Daniels (2002). It should be noted that these authors, from the broadening of the (002) reflections, evaluated larger average crystallite size and larger average microstrain with respect to our whole powder pattern fitting approach.

Fig. 9 shows the diffraction patterns of our collected bones plus, as stated in Section 1, one more bone as a further reference which was coded "urn bone" (see Fig. 10). We can see that also in the case of the collected bones, the monoclinic structure factor was well suited to describe the experimental data. The line-broadening analysis of the sample from the Phoenician urn confirmed the absence of lattice microstrain

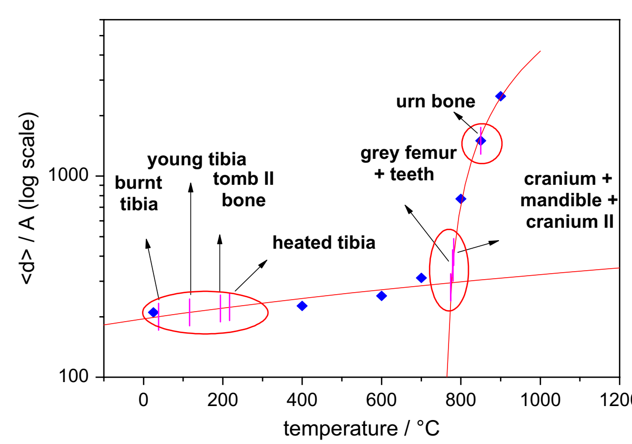

Fig. 8. Calibration curve of the reference sample in terms of average crystallite size as a function of temperature treatment. The full line is the interpolation to the rhombic symbols. The average crystallite size of investigated bones are reported as bars, whose width is associated to the estimated standard deviation of the quantity.

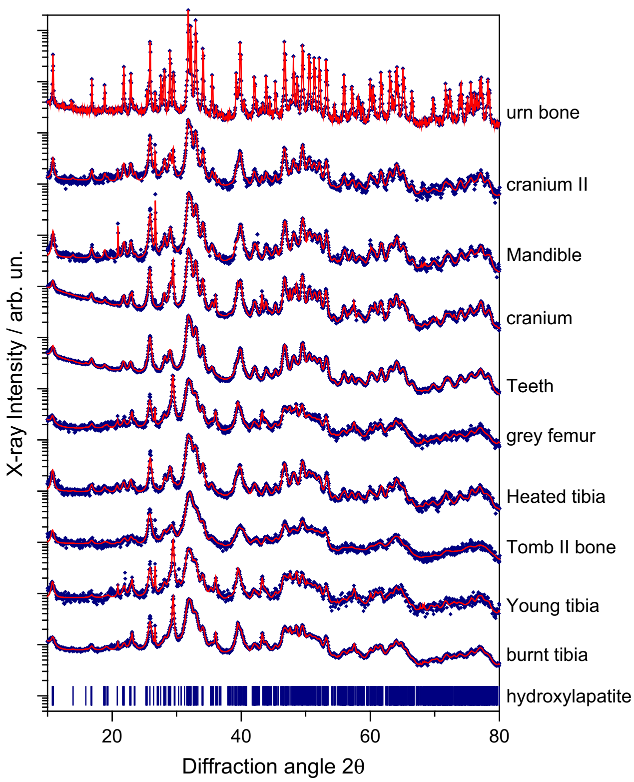

Fig. 9. XRD powder patterns of the bones collected in the three Sa Figu tombs, together with an additional bone retrieved in a Phoenician urn of archaeological area of Monte Sirai, Carbonia (Cagliari) named urn bone.

and supplied an average crystallite size value of 1100 Å. Making reference to our calibration curve permits us to conclude that this bone was heat treated at a temperature of ca. 850 °C.

Our data are in agreement with the recent observation using X-ray diffraction reported by Chakraborty et al. (2006), who treated four bioapatites of different origin up to 1100 °C. Moreover, in our samples there is no evidence of the transformation process of hydroxylapatite into β tricalcium phosphate reported by Bonucci and Graziani (1975).

Concerning the other bones from the three Sa Figu tombs, the relevant average crystallite size have been refined and reported in Fig. 7 as bars. One sees that a first group of specimens (heated tibia, young tibia, burnt tibia and tomb II bone) coming from tombs II and IV which appeared at a first visual inspection to have been subjected to fire, do not show substantial peak sharpening with respect to the sample bone at room temperature. This suggests that the temperature to which these samples were subjected could not be larger than 250 °C. There is a second group of collected bones (cranium, cranium II, mandible, teeth and grey femur) from tomb IX, except for the grey femur of tomb IV, which can be located in a range of temperatures between 600 and 750 °C. This is certainly due to thermal treatment in a more intense fire, compatible with a crematory rite.

5. Conclusions

From the present diffractometric analysis of the bone fragments from the Sa Figu site, after a calibration curve of the X-ray line broadening of apatite mineral of bones as a function of the temperature it emerges that a first group of bones belonging to the rests of tombs II and IV have been subjected to temperature not higher than 250 °C. The estimated temperature obtained may be related to a cremation of short duration. As a matter of fact, after considering the few burnt fragments found with respect to the many not burnt, this hypothesis can also suggest the occurrence of a weak occasional fire inside the sepulchre. For the second group of five bones (four of them from tomb IX, one from tomb IV) the case seems

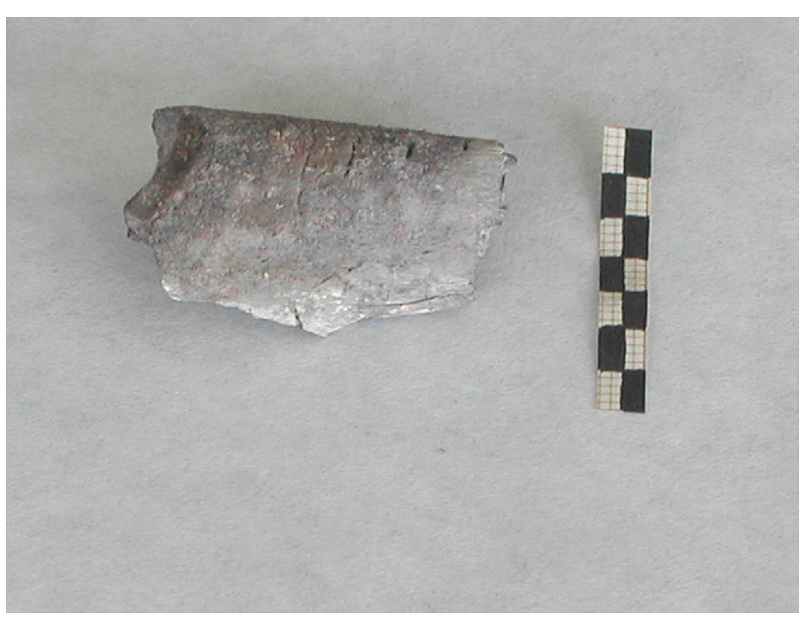

Fig. 10. A direct view of the bone subjected to a cremation rite which was used as a reference, coming from the Punic and Phoenician archaeological area of Monte Sirai, Carbonia (Cagliari).

to be different since it was assessed that these bones were subjected to temperatures as high as 600–750 °C.

The odd presence of the grey femur in this group of bones (from whose provenance we have excluded a cremation) can be explained as due to the direct contact of the bone fragment with the core fire inside the sepulchre.

The temperature range of 600–750 °C is compatible with a ritual combustion of bodies that occurred in tomb IX. On the other hand we cannot totally exclude ritual fires inside the hypogeic site that have all involved dead bodies.

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