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Anthropological and Physicochemical Investigation of the Burnt Remains of Tomb IX in the 'Sa Figu' Hypogeal Necropolis (Sassari, Italy) – Early Bronze Age

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ABSTRACT Excavations carried out in Tomb IX of the hypogeic necropolis of 'Sa Figu', near the village of Ittiri (Sassari, Italy), supplied burnt human bone remains and pottery unambiguously referred to the Early Bronze Age (characterised by the local culture of 'Bonnannaro'). Besides the anthropological study, we have investigated and evaluated the possibility of a funerary cremation practice in Sardinian pre-history, a subject that has previously not been considered from a scientific point of view. Making use of a calibration procedure based on X-ray diffraction (XRD) line-broadening analysis, related to the microstructural properties, it was possible to estimate the combustion temperature to which the fragmented bones were subjected. It was found that the studied bones reached temperatures varying from 400°C up to a maximum of 850°C. This spread of values suggested inhomogeneous combustion of the bones, which seems compatible with funerary cremation practices. Copyright © 2007 John Wiley & Sons, Ltd.

Key words: hypogeal necropolis; Early Bronze Age; anthropological study; funerary cremation practice; X-ray diffraction (XRD)

Introduction

On Sardinia (Italy), cremation rites of the Aeneolithic Age (Copper Age) were suspected in the tombs of Serra Cannigas (Villagreca-Nuraminis) and in the 'domus de janas' of Filigosa (Macomer-Nuoro) (Melis MG, 2000).

However, burnt human bones are rarely found. The only known examples of apparently burnt human bones ascribed to the local 'Bonnanaro' culture (2200–1800 BC) (Melis P, 2003a) were found in Tombs IV, XX, XX bis and XXVI of the Anghelu Ruju necropolis (Alghero) (Levi, 1952; Germanà, 1984). Also, ashes mixed with inhumated individual remains were found in Tomb III of the same site. Other cranial remains showing ash incrustations were found in a nearby burial site called 'S'isterridolzu' (Ossi) (Germanà, 1980).

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Further evidence of fire was also observed in Tomb IX of 'Sa Figu' (Melis P, 2003b), which is the subject of the present work.

Apart from visual inspection, no scientific studies have hitherto been conducted in order to demonstrate or exclude cremation rites in Sardinia during the Bronze Age, probably because these hypothetical incinerations are very rare (Lilliu, 1988). In order to investigate the controversial occurrence of cremation on Sardinia in the Bronze Age, we present here a study by X-ray powder diffraction of the temperature to which some burnt bones from Sa Figu Tomb IX were subjected (Enzo *et al.*, 2007). The methodology consists of a calibration procedure of the X-ray line broadening which is evaluated in a powder pattern of the bone biomineral hydroxylapatite. In the case of bones not subjected to any thermal treatment, the hydroxylapatite X-ray line broadening appears to be considerable but regularly diminishes as a function of the treatment temperature. Thus, from a careful evaluation of the X-ray line sharpening/broadening, the temperature can be estimated for the bones, which is of paramount importance even in forensic studies (Thompson, 2004, 2005), being related to the energy distribution of the firing process. With this procedure it is possible to estimate temperatures of treatment from 300°C up to ca. 1000°C with an uncertainty of ±30°C.

For comparative purposes, cremated reference bones from Sardinia (Amadasi & Branconi, 1965; Bartoloni, 1989) and Spain (Hernàndez *et al.*, 1998) were also investigated (see Table 1). This allowed a close evaluation of the X-ray line-broadening properties of ancient bones which are known to be certainly cremated.

To perform the very accurate investigation required to answer properly the problem in hand, this work took full advantage of the latest progress in powder diffraction techniques both in terms of instrumentation and data evaluation.

Archaeological context of Sa Figu Tomb IX

The prehistoric and protohistorical hypogeal necropolis of 'Sa Figu' is located on the northern side of the Coros plateau of Sardinia, NE of Ittiri village. Today the necropolis is made up of 11 existing tombs with traces of others now destroyed. Present archaeological excavations involve tombs II, IV, V, VIII, IX and X.

Some tombs belong to the 'domus de janas' class: the typical rock-cut tomb spread throughout the Sardinian prehistoric settlements and in use from the Late Neolithic to the Early Bronze Age. Other tombs of the same necropolis (IV, VII, VIII) were of the Nuragic period (Middle Bronze Age) as hypogeic burials of the 'Giant's Tomb' shape (so-called *architectonic prospect domus*). Tomb IX is a 'domus de janas' hypogeum which is excavated in a slightly sloping limestone area and was found without the original ceiling. The original plan included a small corridor (so-called *dromos*) into the first chamber (*anti-cella*). In the front wall, a little port-hole leads to the main

Table 1. Details about provenance and chronology of Sardinian and Spanish bones used in the comparative study

Specimen	Provenance	Chronology
Phoenician urn	Necropolis of Monte Sirai (Carbonia, Sardinia, Italy)	900–510 BC Phoenician culture
IP- A 1962–2	Necropolis of S'Illot des Porros (S. Margalida, Majorca, Spain)	VI–II century BC talayotic culture
IP- A 10	Necropolis of S'Illot des Porros (S. Margalida, Majorca, Spain)	VI–II century BC talayotic culture
IP- A 11	Necropolis of S'Illot des Porros (S. Margalida, Majorca, Spain)	VI–II century BC talayotic culture
IP- B 13	Necropolis of S'Illot des Porros (S. Margalida, Majorca, Spain)	VI–II century BC talayotic culture
SM '03 urna iberica 1	Necropolis of Sta Madrona (Riba-roja, Ribera d'Ebre, Spain)	IV–III century BC. Iberic culture
SM '03 urna iberica 2	Necropolis of Sta Madrona (Riba-roja, Ribera d'Ebre, Spain)	IV–III century BC. Iberic culture

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Figure 1. A plan view of the Sa Figu Tomb IX showing the absence of the ceiling. This figure is available in colour online at www.interscience.wiley.com/journal/oa.

room which has not yet been excavated by archaeologists (see Figures 1 and 2).

Excavations have involved only the *dromos* and the *anti-cella*. No significant materials were found in the *dromos*, whereas a meaningful context was discovered in the *anti-cella*. Remains of the Neolithic and Copper Age graves had already been removed in ancient times; only the latest burials (in the Early Bronze Age) were found and at that time it was possible that the tomb was already without the original rock ceiling. The *anti-cella* is 1.52 m wide and 1.22 long; the maximum remaining wall height is 0.65 m. The archaeological fill height was about 0.50 m.

From the excavation emerged a single significant cultural level, immediately below the surface, characterised by a bone concentration that was extremely fragmented and burnt. The depositional layer sat on a thin layer of ashes in contact with the basal pavement. The bones were accompanied by numerous pottery fragments whose shape could not be completely reconstructed. Only two very small pots (i.e. a little cup and a very small jar) were discovered almost intact, except for a little damage due to compaction. The collection of bones and pottery was surmounted by fragments of a calvarium that was partially reconstructed in the laboratory (see Figure 3).

Chronological evidence from the materials assign this burial to the Early/Middle Bronze Age (around the 18th century BC) in a final phase of

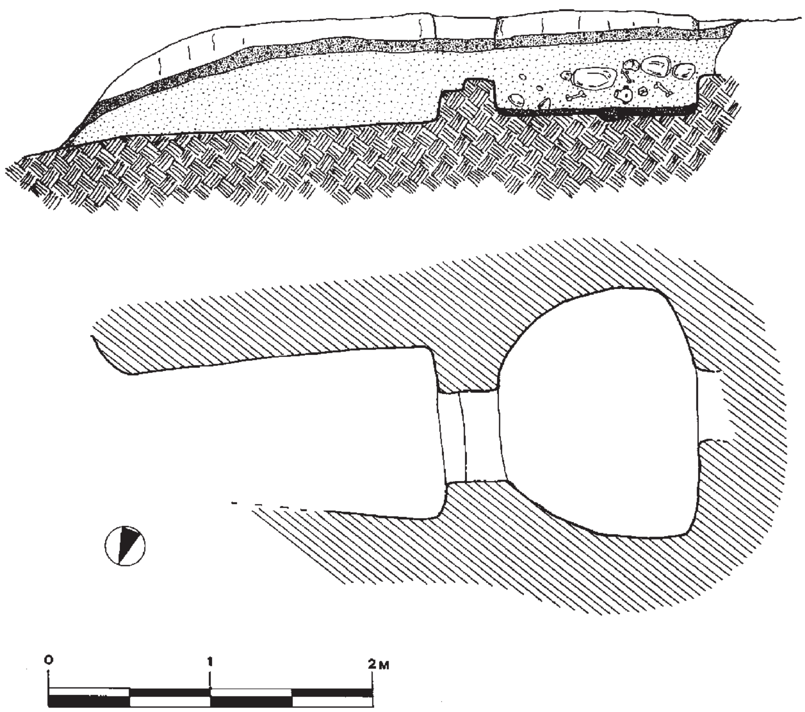

Figure 2. Plan and section of the Sa Figu Tomb IX.

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Figure 3. Calvarium partially reconstructed in the laboratory. The unit size is 5 cm. This figure is available in colour online at www.interscience.wiley.com/journal/oa.

the 'Bonnanaro' culture, the so-called 'Sant'Iroxi' from the hypogeum near Decimoputzu (Cagliari) excavated by Ugas (1999).

Methods

We have investigated all the skeletal remains found in Sa Figu Tomb IX. Where possible, the osseous and dental remains have been reconstructed and classified in order to evaluate the characteristic features and the minimum number of individuals in the tomb. The latter was determined based on the occurrence of repeated skeletal elements or disagreements in size and age.

In order to establish the sex of single individuals, given the impossibility of observing the morphological features of the pelvis or cranium, nor to conduct the classic determinations in long bones, we follow the techniques of Ferembach *et al*. (1980) and Iscan & Kennedy (1989). The estimation of age at death was essentially based on the diaphysis size and, for subadults, from teeth formation and eruption according to Ubelaker (1989). For the dental identification we used the FDI World Dental Federation notation.

Powder X-ray diffraction (XRD) patterns were recorded with a Bruker D8 diffractometer in the Bragg-Brentano geometry using Cu K_{α} radiation ($\lambda = 1.54178$ 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 antiscatter slits of 0.5°) was determined using α-SiO_2 and α-Al_2O_3 standards free from the effects of reduced crystallite size and lattice defects. The powder patterns were analysed according to the Rietveld method (Rietveld, 1967) using the programme MAUD (Lutterotti *et al*., 1998) running on a personal computer. It is worth noting that the methodology followed is based upon the same assumptions made by Bonucci & Graziani (1975), Shipman *et al*. (1984), Person *et al*. (1995) and Rogers & Daniels (2002), with the only difference being that in our work we have introduced the Rietveld method as the most suitable tool to analyse the whole diffraction patterns.

This approach involves a suitable physical and chemical model which is able to assess with the best precision and accuracy the broadening of the X-ray diffraction peak profiles, separating the effects due to the reduced crystallite size from the lattice strain.

Results and discussion

General considerations on the bone collection

During excavations, the bones were clearly combusted, highly fragmented, and piled up randomly. The specimens were of rather homogeneous colours, with prevailing tones of brown, while some other fragments were dark grey and shades of violet. Looking to the exterior of the bones, we observed early fractures, mostly longitudinal, which are typical of fire treatment on dry bones (Holck, 1986; Etxeberria, 1994).

The combustion probably eliminated the collagen from the bones, causing distinct fragmentation of skeletal remains. However, it is not possible to exclude totally the role of diagenetic factors (Denys, 2002), manipulation and collection of remains (McKinley, 1994) (see Figure 4).

For Tomb IX we reject the hypothesis of body transportation from one sepulchre to another. If this was the case, then we should observe the

Figure 4. A view of humerus remains representative of the entire bone collection. The high fragmentation may be ascribed to fire treatment combined with taphonomic processes. This figure is available in colour online at www.interscience.wiley.com/journal/oa.

absence of some phalanges (see Figure 5), which are unlikely to be collected and integrally transported to another sepulchre after the body decomposition process. Also, a fine ash layer on the pavement demonstrates unequivocally that the skeletal remains were subjected to fire treatment in the place where they have been brought to light.

The absence of a sensible number of bone elements with a high percentage of compact texture (vertebrae, carpus and tarsus bones) may be justified by the extreme fragility and possible pulverisation of the spongy texture after fire treatment. Thus, fire in the same burial is the most probable hypothesis, although the relationship between burial rites and fire is not known. One possibility supports the use of fire as a purifying process in the mortuary rites.

Figure 5. Phalanges retrieved from Tomb IX of "Sa Figu". The occurrence of these short bones strongly support the hypothesis that the bodies were burnt in-situ and were not transported from another sepulchre. This figure is available in colour online at www.interscience.wiley.com/journal/oa.

Since the ceiling of the tomb was found destroyed and the bone remains could have been subjected to weathering for a long time, we should not disregard the hypothesis of an occasional fire, unrelated to any burial practices.

After separating the mostly fragmented and undefined remains from the rest of the bones of larger size, we have considered and analysed the collection according to Table 2. The minimum number of ascertained individuals derives from the analysis of 16 mental regions. The results are reported in Table 3.

Of the 16 identified persons, nine are adults and seven subadults. We were able to recognise the sex of seven individuals by observing the morphological and metric features of the mental region; we recognised four male and three female individuals, but the remaining bones were too small to proceed further.

In spite of the high number of subadults–about half of the total–such an evaluation does not agree with the post-cranial evidence because a large part of the diaphysis fragments belong to adults. In any case, an important bias emerges for children, including infants as well as children aged 1 to 5 years. According to the typical indices of mortality, particularly those from ancient settlements (Hoppa & Vaupel, 2002), it is singular to find a high percentage of infants belonging to class II (4/7, 57%) and class I (1/7, 14%) without any individual less than a year old or perinatal. The absence of infant remains may be interpreted either as a census error due to taphonomic reasons, or to different funerary practices involving the separation of baby bodies.

In this context, it is likely that the small bone size, combined with weathering processes, prevent their correct recovery and recognition to the point that, in practice, they may not be included in the archaeological record. The absence of significant quantities of infant individuals may also be related to the fact that they were not

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Table 2. Classification of fragmented bones belonging to the Sa Figu tomb IX

Skeletal bones	Right side	Left side	Not classified	Total	Adult	Sub-adult			Minimum likely number of individuals
Cranium				321					9
Mandible				38	9	7	3	4	16
Vertebra				5					3
Teeth	30	24	22	76					
Rib			31	31					
Clavicle	6	7	13	26					7
Humerus	5	9	13	27	4	2		4	10
Radius	4	7		11	6	1	1	4	7
Cubitus	9	9		18	9		4	6	9
Carpus	3	1		4					2
Maniphalanx	1	3	37	41					2
Pelvis			13	13					
Femur	7	5		12	4		2	2	4
Patella	2	2	1	5	3				3
Tibia									
Fibula									
Tarsus	6	3		9	6				6
Pediphalanx	2	6		17	5	1			6
Unrecognised fragments (<2 cm)				423					
Total number of examined bones				1075					

buried with the other individuals of the population (Barrial, 1989).

Nevertheless, keeping in mind the infant class values determined for ancient necropoli (Alesan *et al.*, 1999), the infant mortality of greater than 50% in the Sa Figu Tomb IX appears to be very high (Weiss, 1973), possibly on account of hard life conditions for this community.

XRD study of specimens from Sa Figu Tomb IX

For the comparative study of structural and microstructural properties of burnt bones, 12 bone fragments, which were numbered progressively, were chosen from the Sa Figu remains of Tomb IX. This selection was made taking into account the diversity of colours, shades and textures. Bones certainly subjected to cremation from Spanish and Sardinian sites were used for reference (see Table 1).

The XRD patterns of our specimens from Tomb IX are reported in Figure 6. On the left-hand side, starting from the bottom we can observe that specimen no. 4 is made up of two phases: hydroxylapatite $Ca_5(PO_4)_3(Cl,F,OH)$ for more than 60 wt%, while the remaining 40 wt% is calcite $CaCO_3$. We do not possess a credible explanation for the high percentage of the latter mineral. At the moment we attribute calcite to an exogenous origin with respect to the osseous material, since the presence of carbonate units CO_3^{2-} that may substitute for phosphate groups PO_4^{3-} in the apatite structure amount to not more than 7–8 wt% (Wopenka & Pasteris, 2005).

Concerning the apatite mineral, we have verified that the monoclinic structural phase is better than the hexagonal phase in order to account for the experimental data points for all samples here investigated (Wilson *et al.*, 1999). As can be seen from the patterns, the apatite peak profiles are considerably broadened due to a marked nanocrystalline structure with high lattice disorder in the sample.

Sample numbers 3, 7 and 10 have very similar patterns. All diffraction peaks are attributable to hydroxylapatite and no traces of calcite are discernible. We may note that in this case the line

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Table 3. Detailed analysis of mandible fragments that determined the minimum number of individuals

Individual identification code	Age	Sex	Notes
SF.T9.M1	Adult	Male	Left hemimandible. Alveolus pathology. The 41 and 42 alveolus are undergoing reabsorbtion; there is a likely reabsorbtion of 43.
SF.T9.M2	Adult	Male	Left hemimandible Reabsorbtion of 36 and 37; root of 33 and alveolus of 31,32, 34, 35.The 41 is undergoing reabsorbtion.
SF.T9.M3	Adult	Not determined	Two separate fragments of left and right hemimandible from the same person. In the right side there are the alveolus of 41 and 47. 42, 43, 44, 45, 46 are maintained; likely infection below 43. Alveolus from 34 to 37 are preserved in the left side.
SF.T9.M4	Adult	Not determined	Right hemimandible. Alveolus from 41 to 47 are found; 45 is maintained
SF.T9.M5	Adult	Female	Mental region between alveolus 35 and 45
SF.T9.M6	Adult	Female	Mental region between alveolus 32 and 33
SF.T9.M7	Adult	Male	Mental region
SF.T9.M8	Adult	Male	Mental region between alveolus 33 and 42
SF.T9.M9	Adult	Female	Left hemimandible. The alveolus from 43 to 37 are found; only the roots of 32 and 36 are also present
SF.T9.M10	9–10 years old	Not determined	Left hemimandible The 33 and 34 are not erupted. 32, 35, 36, 37 are maintained.
SF.T9.M11	9–10 years old	Not determined	Left hemimandible. 34 and 35 are not erupted
SF.T9.M12	10–11 years old	Not determined	Left hemimandible. It shows 46 and 85 roots, the 37 is not erupted. The alveolus of 34 and the crown of 35 are maintained.
SF.T9.M13	3–4 years old	Not determined	Right hemimandible. The root of 83 is maintained.
SF.T9.M14	5–7 years old	Not determined	Mental region. Crowns of 34 and 44 are maintained.
SF.T9.M15	Not determined	Not determined	Mental region. The individual is certainly more than 1 year old.
SF.T9.M16	10–11 years old	Not determined	Mental region. Alveolus 81–74 are maintained.

broadening is slightly less than in the previous pattern. This effect is clearly assigned to a wider extension of the coherent domains of diffraction and/or to a lower amount of lattice strain.

Analogously, sample numbers 5, 2 and 1 (in the right-hand plot) may be envisaged in a third group of similar structure and microstructure properties, with a further increase in crystallite size and reduction of lattice disorder. Even in this case, hydroxylapatite is the only phase present. The same is also true for fragments of specimen numbers 11, 12, 9 and 6, where we can note further peak sharpening with respect to the previously examined series.

This process of peak sharpening (i.e. further growth of the coherent domain of diffraction and a decreased lattice strain) is even more evident for sample no. 8. We may notice in this specimen a weak additional contribution from calcite, which was estimated at 5.0 wt% using the Rietveld method. The relatively good agreement between experimental and calculated models constitutes our starting point for the next correlations for the possible thermal treatment to which the bones were subjected.

The evolution of these growth processes for the crystallite size of apatite of human and animal bones is normally followed in the archaeological environment making reference to an angular range of the 2θ diffraction angle from 30° to 39°. In this angular range, Person *et al.* (1995) have defined a crystallinity index of the bone specimens, which was used to establish a correlation with the temperature of treatment.

Table 4 reports the crystallite size and lattice strain values for the series of samples here examined. As expected from the line-broadening evolution, for sample no. 4 the average crystallite

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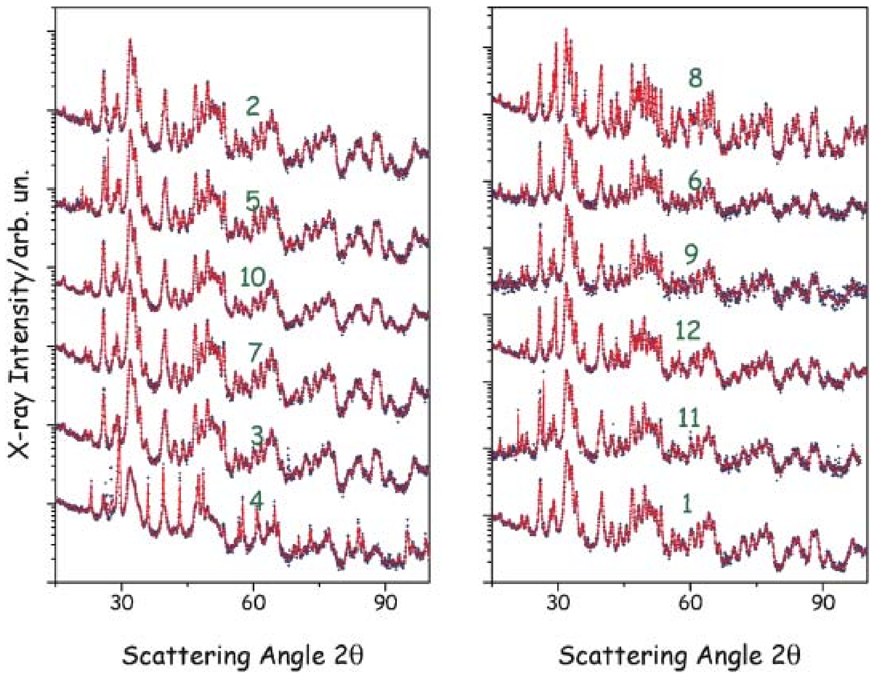

Figure 6. The XRD patterns of the 12 specimens from Sa Figu Tomb IX, ordered by increasing average crystallite size from bottom to top and from left to right respectively. The large peak broadening associated with the hydroxylapatite phase of sample no. 4 evolves progressively through the various specimens ending with sample no. 8, where a considerable sharpening of the X-ray peaks is evident. This figure is available in colour online at www.interscience.wiley.com/journal/oa.

size turns out to be 180 Å, and it grows progressively up to 540 Å for sample no. 8. Conversely, there is not such a definite regular trend for the lattice strain, which in any case seems to diminish progressively according to the way the samples are ordered.

In order to evaluate the temperature to which the bones were presumably subjected, it is necessary to make reference to a calibration that was worked out with a sample bone subjected to selected temperatures of cremation (Enzo *et al.*, 2007). Applying this calibration and keeping in mind the uncertainty involved in the procedure, we have estimated the temperature to which our bones were presumably subjected, as reported in the right-hand column of Table 4. As can be seen,

Table 4. The domain size and lattice disorder of hydroxylapatite microcrystals and the corresponding temperatures of treatment, according to our calibration procedure reported in Enzo *et al.* (2007)

Sample code	Average crystallite size/Å (±20)	Lattice disorder/10^{-3}	Temperature/°C (±30°C)
4	180	2.0	No burning
3	240	1.2	>400
10	240	1.8	>400
7	260	1.7	550
5	270	1.5	600
2	280	1.5	670
1	285	1.2	690
11	290	2.0	710
12	300	0.8	750
9	340	1.3	800
6	355	1.4	820
8	540	0.5	>850

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the temperature associated with sample no. 8 is ca. 850°C which is of a certain relevance, while for the other groups of bones the temperatures of treatment appear to be lower, except for sample no. 4 which is not burnt at all.

Comparative study of cremated bones

A comparison of XRD patterns was undertaken with other bones for which previous studies (Enzo *et al.*, 2007) concluded that they were very likely subjected to cremation. Figure 7 shows the six XRD patterns of the IP and SM series of bones and the pattern of a Phoenician urn bone (top curve). We should note that sample IP-A1962-2 (a cranium fragment) originally contained a considerable amount of exogenous calcite that we have removed from the bone. The main mineral is of course hydroxylapatite, but for SM 1 and 2 samples, small amounts of calcite and perhaps dolomite were also found (<10.0 wt%). As can be seen, the lines for specimen IP-B13 (curve at the bottom) show considerable broadening, on account of the small crystallite size and large lattice strain. This is typical of bones which have not been subjected to significant cremation or heat treatment, as can be deduced by making reference to the previously cited calibration (Enzo *et al.*, 2007) – see Table 4. Moreover, this result is also in accordance with morphological characteristics of the bone (dark brown colour and non-crystalline texture). In any case, the present level of broadening could still be the

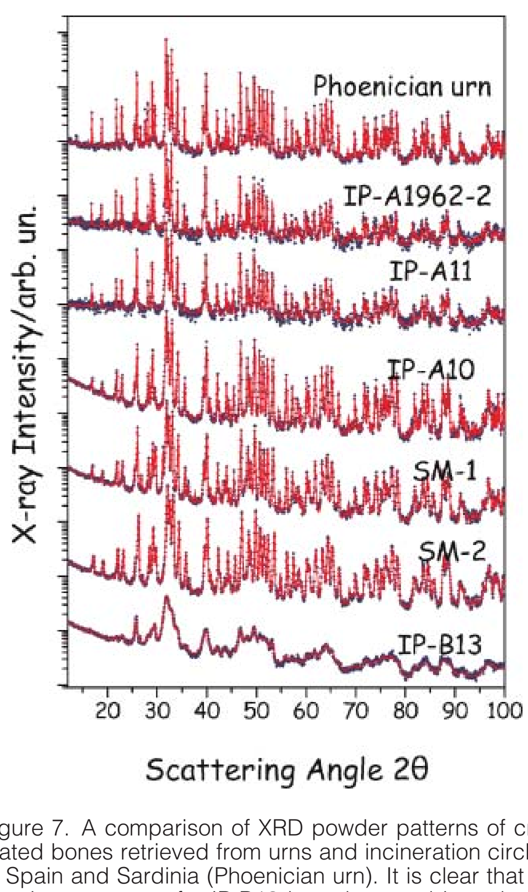

Figure 7. A comparison of XRD powder patterns of cremated bones retrieved from urns and incineration circles of Spain and Sardinia (Phoenician urn). It is clear that all specimens except for IP-B13 have been subjected to a high-temperature thermal treatment, as shown by the sharp diffraction peaks. This figure is available in colour online at www.interscience.wiley.com/journal/oa.

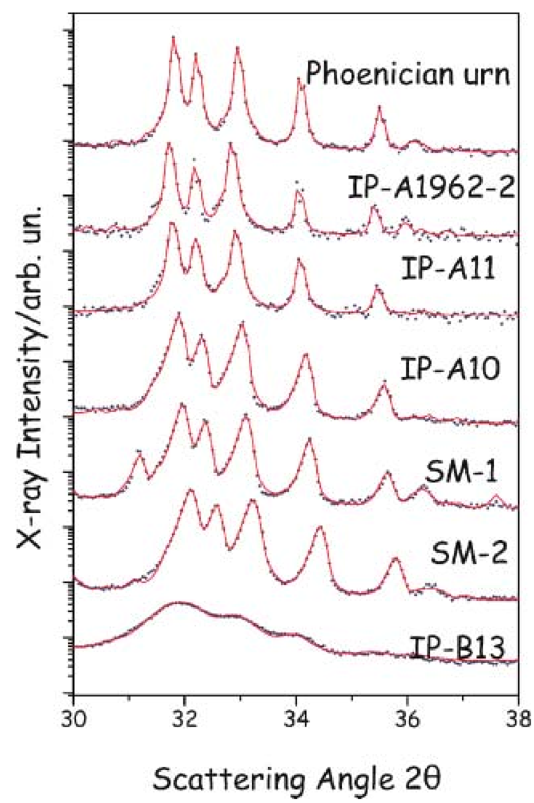

Figure 8. The XRD patterns of cremated bones from Sardinia and Spain as in the previous figure, when plotted in the 'typical' angular range from 30° to 38° in 2θ, where it is customary to calculate a so-called crystallinity index. Again, it is evident that sample IP-B13 displays significant peak broadening with respect to the remaining specimens on account of the weak thermal treatment received. This figure is available in colour online at www.interscience.wiley.com/journal/oa.

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result of inhomogeneous cremation of the body (Etxeberria, 1994)

The rest of specimens (IP-A10, IP-A11 and IP-A1962-2, SM1, SM2 respectively) show noticeable sharpening of the peaks, which is comparable to that of the Phoenician urn reported by us recently. Other authors (Bonucci & Graziani, 1975; Person *et al.*, 1995) inferred the temperature of treatment from the sharpening of the X-ray diffraction pattern, expressed as a crystallinity index from analysis in the angular range from 30° to 38° in 2θ. The whole patterns are zoomed into this range in Figure 8 just for comparison with this method. It may be concluded that even the 'classic' approach conveys the same degree of information as for the cremation. The imperfect alignment of corresponding hydroxylapatite peaks for various specimens may be due to imperfect positioning of powdered specimens in the diffractometer, and/or slight differences in the lattice parameters of the monoclinic phase. Even for these two specimens, the temperature of treatment is estimated as between 800 and 850°C.

Conclusions

The bone remains found in Sa Figu Tomb IX are part of a Sardinian human group composed of at least 16 individuals. It was established that the bones correspond to individuals of both sexes and different ages. The colour and texture of the remains show that the bones were burnt.

Absence of irregular lengthwise splitting, transverse fractures, and the marked warping typical from green-burnt bones, are concurrent observations suggesting that the fire occurred on dry bones.

The bones from Sardinian and Spanish cinerary urns (with just one exception) that were used for reference show quite homogeneous temperatures of firing (exceeding 800°C), intuitively suggesting that a cremation involves a high-temperature treatment. These values are presumably reached when the body is reduced to just a few fragmented bones and ashes The temperature treatment determined on selected bones from Sa Figu Tomb IX, in the range of 400 to 850°C according to XRD, does not appear homogeneous.

It is hard to state absolutely whether the fire in Sa Figu Tomb IX was used for crematory rites. Certainly the high temperatures determined especially for samples 8, 6 and 9 are compatible with a crematory treatment. The observed range of temperature suggests the occurrence – although imperfect – of an *in situ* fire treatment. On the other hand we cannot exclude entirely the possibility of occasional fires occurring after the destruction of the hypogeum ceiling.

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