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ORIGINAL PAPER

Early metallurgy in Sardinia: characterizing the evidence from Su Coddu

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Abstract

This paper contextualizes analyses of a collection of metal artifacts and ostensible metallurgical slag from the prehistoric settlement of Su Coddu in south-central Sardinia (ca. 3400–2850 BCE). To characterize the types of metals and associated alloys utilized by the earliest residents of Su Coddu, two pins and an unshaped lump of unknown composition were analyzed using portable XRF spectrometry. In addition to metal artifacts, a large quantity of putative slag was discovered at the site that is consistently cited as the earliest evidence of in situ smelting in prehistoric Sardinia. To reconstruct firing temperatures and characterize mineral phases, four samples of the overfired material were selected for thin section petrography and powder XRD analysis. The results of this study indicate that the two pins were made of copper while the unshaped lump was composed of pure lead, making it the earliest lead-based artifact on Sardinia. XRD and petrographic analyses of the fired "slags" reveal that these samples are unrelated to metallurgical smelting and are likely burnt wall coatings whose mineralogical phases correspond with unfired plasters also recovered from the site. These results in combination contribute towards understanding early metallurgical practices in Sardinia and are relevant in reconstructing the events that have shaped the life history of Su Coddu.

Keywords Sardinia · Neolithic · Archaeometallurgy · Powder X-ray diffraction (XRD) · Thin section petrography · Portable X-ray fluorescence (pXRF) spectrometry

Introduction

This paper concerns analyses of an assemblage of metal artifacts and apparent slag from the site of Su Coddu/Canelles in south-central Sardinia (ca. 3400–2850 BCE; Fig. 1). The site is important because it contains some of the earliest evidence of metallurgy on the island and has been argued to provide clear indications of in situ smelting as early as the mid-to-late fourth millennia BCE (Dolfini 2013; Usai 2005). While Su Coddu is a well-established locus of early metal technology, there has been limited archaeometric testing of the associated artifacts to better characterize pyrotechnological practices at the site and to infer the technological capabilities of the earliest metalworking communities on the island. Despite the clear presence of metal technology in Late Neolithic Sardinia, the results of this study reveal that there is no unequivocal evidence for copper, silver, or lead-based smelting at this time.

Su Coddu is a prehistoric village that occupies an area of approximately 6 ha and is made up of both simple huts as well as more complex multi-room structures and associated wells and silos (Fig. 2). The site was originally excavated in the early to mid-1980s by Giovanni Ugas (Ugas et al. 1985) and later from 1994 to 2014 under the direction of Maria Rosaria Manunza in collaboration with the University of Cagliari, the University of Sassari, and the Archeological Superintendency

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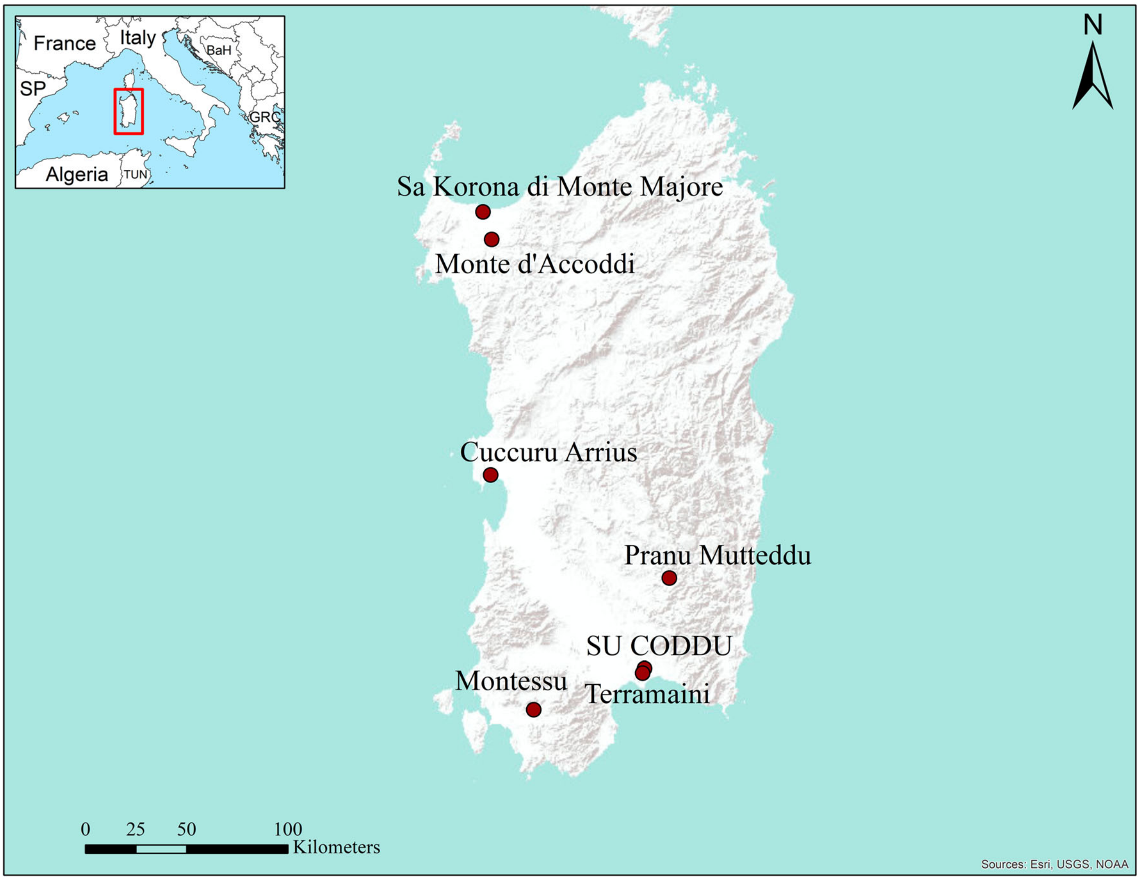

Fig. 1 Map displaying the location of Su Coddu as well as other contemporaneous Ozieri/Sub-Ozieri sites with metal technology

of the provinces Cagliari and Oristano (Manunza 2007–2012; Manunza et al. 2005–2006). A series of radiocarbon dates situate the earliest occupation of the site at the Late Neolithic Ozieri phases of the mid-fourth millennium cal BC. This is an era of significant cultural transformation in Sardinia as represented by island-wide settlement expansion, new pottery typologies, and the adoption of metal technology (Malone 2003: 262). Indeed, Su Coddu gradually grew in size through the Sub-Ozieri phases of the early third millennium cal BC to occupy an area of roughly 1.5 ha known as Canelles (Melis 2009: 85; Melis et al. 2007). Later phases of site occupation through historical times have also been recognized, which includes the discovery of slag that points to bloomery iron smelting (Charlton et al. 2010; Humphris and Rehren 2013; see Online Resource 1 for analytical data).

To appreciate the types of metals and associated alloys utilized by the earliest residents of Su Coddu, two pins and an unshaped lump of unknown composition were elementally characterized using portable X-ray fluorescence (pXRF) spectrometry. A sample of four ostensible slag artifacts was also characterized using thin-section petrography and powder X-ray diffraction (XRD) to reconstruct mineral phases, firing temperatures, and to clarify these artifacts' association with metallurgical smelting. The results of this study in combination contribute towards understanding early metallurgical practices in Sardinia and are relevant to broader debates about the origins of metalworking within the wider central Mediterranean region.

Situating the Su Coddu study

The earliest metalworking communities of the central Mediterranean appear in northern Italy during the Late Neolithic phases of the mid-fifth to early fourth millennia BCE, where locally available sources of copper and silver in the Alps made smelting possible (Dolfini 2013: 145; Kassianidou and Knapp 2005: 219). Sardinia also sees evidence of early metal technology dating to the mid-to-late fourth millennium BCE (Dolfini 2014; Melis 2009). Early metals on Sardinia consist of copper and silver pins, daggers, and rings found at a range of sites across the island, including Cuccuru Arrius, Grotta Sa Korona di Monte Majore, Montessu, Monte d'Accoddi, Pranu Muteddu, and Terramaini (see Fig. 1; Usai 2005; Lo Schiavo 1989). Locally available sources of copper and silver facilitated the adoption and proliferation of metalworking on the island from the Copper Age onwards (see

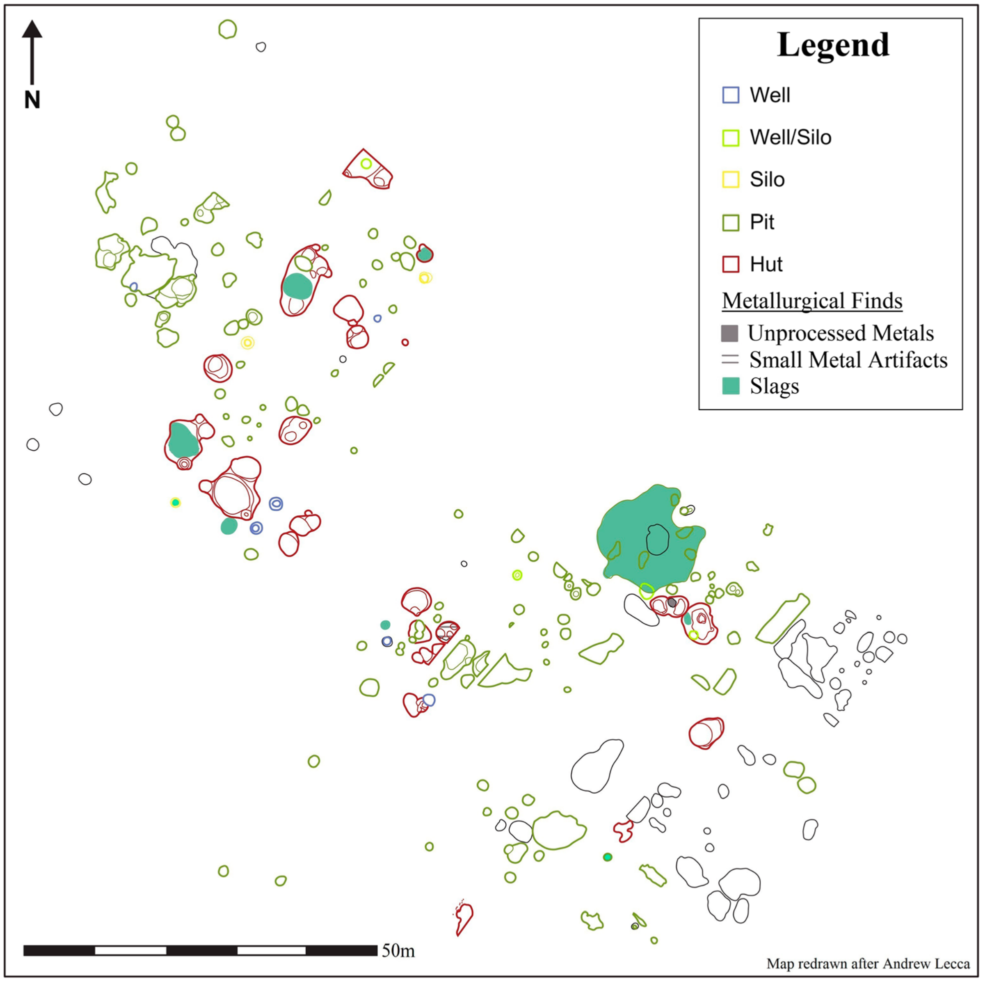

Fig. 2 Plan of the excavation of Su Coddu. For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article

Valera and Valera 2005), but metal artifacts predate this period and it is currently unclear when these raw materials first began to be utilized.

At present, the earliest evidence for in situ metalworking on Sardinia is solely testified by ostensible slags found at Su Coddu and possible crucible fragments from Monte d'Accoddi and Terramaini (Manunza 2005; Melis 2000: 83, Melis 2009: 87; Skeates 1993; Ugas et al. 1989). Prior studies such as Manunza et al. (2005–2006) could not provide convincing evidence of early smelting processes at Su Coddu. This study contributes to these debates by providing a much needed archaeometric assessment of the metallurgical evidence (cf. Dolfini 2013: 140; Melis 2005: 558).

Methods

This study combines several analytical techniques to analyze a total of seven artifacts from Su Coddu (Table 1).

Portable X-ray fluorescence (pXRF) spectrometry

The use of pXRF to characterize metal alloys has been applied throughout the Mediterranean, including Sicily (Caponetti et al. 2017; Vianello and Tykot 2017), Cyprus (Charalambous et al. 2014), and Israel (Shalev et al. 2014). Major advantages of these analyses are that they are non-destructive and can provide results both quickly and cost-effectively. Nevertheless, they are surface analyses whose readings can be skewed due to the presence of superficial contamination, often in the form of increased Fe, Mn, or Zn readings that must be accounted for in any interpretation; testing multiple spots across the surface of an artifact is also necessary to detect erroneous measurements (Liritzis and Zacharias 2011; Tykot 2016). For quantitative analysis, proper calibration standards in the form of pure and alloyed metals analyzed by the same instrumentation are needed to account for matrix effects and obtain valid results (Orfanou and Rehren 2015; Speakman and Shackley 2013: 1439).

Table 1 Contextual information about artifacts analyzed in this study. Inv, inventory number

Sample number	Dating	Material	Provenience
IRSC 58	Ozieri/Sub-Ozieri	Metal pin	Bag 196 US 671 Inv. 200,189
IRSC 59	Ozieri/Sub-Ozieri	Metal pin	Bag 173 US 546 Inv. 200,188
IRSC 60	Ozieri/Sub-Ozieri	Unshaped metal	Bag 83 US 346
IRSC 93	Ozieri/Sub-Ozieri	Overfired material	Pozzo 23A Strada D US 95
IRSC 94	Ozieri/Sub-Ozieri	Overfired material	Bag 173 US 567 Inv. R2663
IRSC 95	Ozieri/Sub-Ozieri	Overfired material	Bag 173 US 542 Inv. R2093
IRSC 96	Ozieri/Sub-Ozieri	Overfired material	Bag 173 US 528 Inv. R1599

For this study, a Bruker Tracer III-SD pXRF machine was used to analyze three metal artifacts from Su Coddu, in turn taking three separate measurements along the length of each artifact and averaging the results (Fig. 3a, b). Each run lasted 60 s at 40 kV/4 mA with no vacuum. A filter composed of 12 mm Al and 1 mm Ti was placed directly into the machine to enhance results for the elements of interest (Ag, As, Cu, Fe, Pb, Sn, and Zn). The raw data were calibrated using an established program for copper-based metals whose standards were analyzed by the same instrumentation (see Vianello and Tykot 2017). In addition to elemental characterization, each artifact was analyzed typologically, and the maximum length, width, and thickness of each artifact were recorded.

Fig. 3 Artifacts from Su Coddu, including **a** copper pin, **b** unshaped lead, **c** fired wall coatings

Powder X-ray diffraction (XRD)

Powder XRD allows for the characterization of minerals that cannot be recognized in a thin-section petrography due to their small size, such as clay minerals or new phases formed during firing (Maggetti 1994). XRD measurements of minerals present in ceramics can also help identify the interval of temperatures at which ceramics were fired, since particular minerals are indicators of changes that occur during the firing process; examples include hematite, magnetite, cristobalite, mullite, calcite, montmorillonite, illite, vermiculite, and feldspars (Maggetti 1982; Maritan 2004; Nodari et al. 2007).

As part of excavations at Su Coddu, a large quantity of light and porous overfired material was discovered (Fig. 3c), and four of these samples were selected for analysis using a Bruker D8Advance powder diffractometer with a Cu-X-ray tube running at 40 kV/20 mA, Goebel mirror optic, 0.2-mm divergence slit, fixed knife edge to suppress air scatter, rotating sample, and VÅNTEC-1 detector. Before being powdered, inner parts of three artifacts were separated and cleaned with an ultrasonic finger using deionized water to reduce contamination from the ground. For the fourth artifact (IRSC-93), this procedure was not possible due to the small sample size.

Thin section petrography

Pottery petrography consists of the description, classification, and interpretation of ceramic thin sections, adopting techniques from geology and soil micromorphology to gain insights into technological aspects of ceramic materials (see Quinn 2013). Four samples of the overfired material were selected to be analyzed with this technique. These sections, after having been consolidated with an epoxy resin, were lapped with silicon carbide powder (600 grain size) and pasted over a glass slide. The samples were then ground to approximately 40 μm using a Buehler PetroThin thin sectioning system. Finally, they were brought to ca. 20–30 μm thickness, again using silicon carbide powder (600 to 900 grain size) and covered with a removable transparent varnish. The thin sections were analyzed in XP and PPL with a Leica DM 2500P.

Results

Table 2 provides a breakdown of the elemental composition of each of the three metal artifacts analyzed with pXRF in this

Table 2 pXRF results of analyses of three metal artifacts from Su Coddu. Note that each artifact was analyzed three times and the average weight percentages and associated standard deviations are reported

Sample number	Length (in mm)	Width (in mm)	Thickness (in mm)	Type	Chemical composition (wt% ± std)							Total (%)
					Ag	As	Cu	Fe	Pb	Sn	Zn	
IRSC 58	62	2	1	Pin	0	0.2 ± 0	98.8 ± 0.2	0.2 ± 0.1	0.1 ± 0	0	0	99.3
IRSC 59	72	3	2	Pin	0	0.3 ± 0.1	97.2 ± 0.2	1.6 ± 0.2	0.1 ± 0	0	0	99.3
IRSC 60	25	19	8	Unshaped	0	0	0	0	99.9 ± 0	0	0	99.9

study. The low standard deviations represented in the three separate analyses per artifact suggest a uniform consistency. The first two artifacts are copper pins of 62 and 72 mm in length respectively. They are almost pure copper, with less than 1 % arsenic. A minor quantity of iron was present in the samples at 0.2 ± 0.1 and 1.6 ± 0.2 % respectively, which indicates an overall lack of iron in the copper ores or that no iron oxides were utilized during the slagging process. The copper pins are typologically consistent with finds from the contemporaneous sites of Cuccuru Arrius and Monte d'Accoddi. While the functions of these artifacts are unknown, their square profiles and tapering along their lengths have led scholars to suggest their use as awls or possibly pins used to fasten fabric or clothing; their use in flint pressure flaking has also been suggested (Dolfini 2014; Pearce 2000).

The third metal artifact analyzed has no recognizable shape and is composed of pure lead, measuring 25 × 19 × 8 mm in size. Lead has a low melting point, and possible functions of an artifact of this type include its use as a sealer for filling spaces in masonry, clamps for repairing pottery, ammunition for slings, and as a possible alloy (Atzeni et al. 2005: 164).

The combination of petrographic and XRD analyses of the four overfired samples reveal that they are composed of quartz, cristobalite, calcite, and feldspar (Figs. 4 and 5). The identification of feldspar only in the non-ultrasonically cleaned sample, IRSC 93, likely points to contamination from the soil. The cristobalite within all of the fired samples suggests that these materials were heated to temperatures above 1000 °C. This interpretation is also supported by the clearly visible glass hump in all of the diffractograms, which is a clear indication of a certain amount of a former melt now existing as a glassy amorphous matrix. In addition, the absence of optical activity (isotropic behavior) within the matrix and the presence of bloating pores in the thin sections is an indication of the high temperatures to which these materials were exposed (Quinn 2013: 199). The high temperatures involved in combination with the overall lack of copper or mineral phases typical of copper smelting (e.g., cuprite, spinel, and delafossite) or related to metallurgical wastes (wustite, fayalite, or magnetite) suggests that these samples are unrelated to metallurgical smelting (see Bachmann 1982; Radivojević et al. 2010). The clearly identifiable presence of calcite in samples IRSC 94 and IRSC 95 is likely due to soil contaminants within the pores of the artifacts that were not completely removed during ultrasonic cleaning. This interpretation is confirmed by the petrography showing that calcite is deposited in voids as micritic mass (Fig. 4; see Quinn 2013: 2017).

Discussion

Metal technology first appears on Sardinia several centuries after its emergence in mainland Italy, and current discourse highlights the influence of mainland communities in facilitating the spread of knowledge of these practices to the island (Dolfini 2014: 497). This is further supported by the existence of contemporaneous Neolithic exchange networks connecting

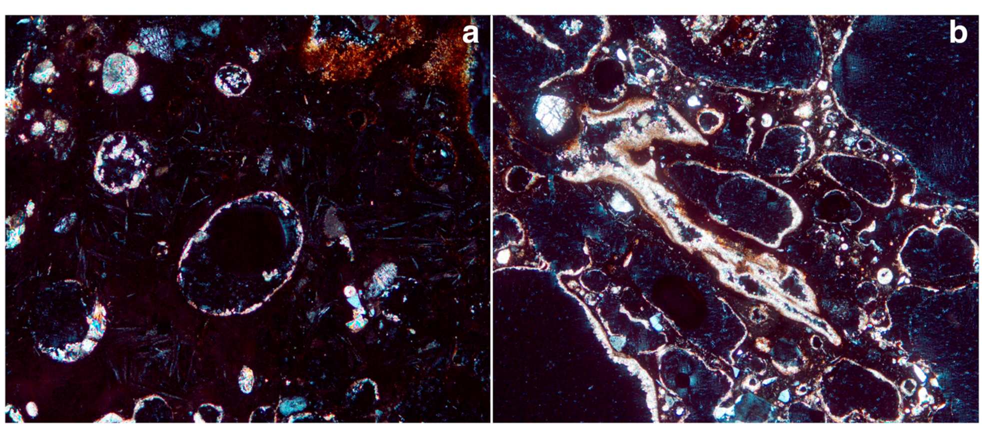

Fig. 4 Thin section micrographs of **a** IRSC 96, XP field of view 4 mm, **b** IRSC 96, field of view 2 mm. Inclusions of quartz, bloating pores, and secondary calcite deposited in voids as micritic mass are visible in both images

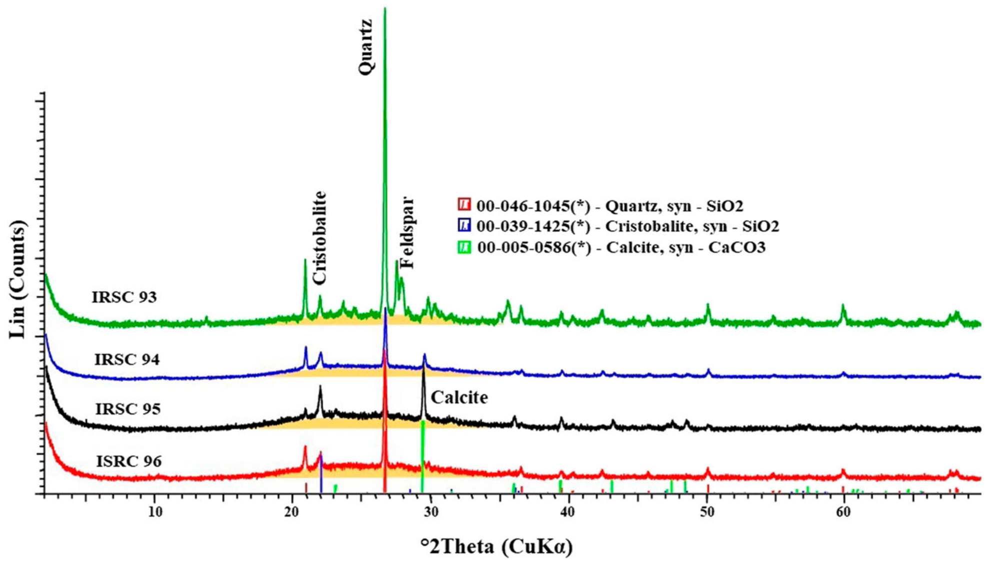

Fig. 5 XRD diffractograms (uncorrected background) of the four analyzed samples. Theoretical peak positions are from the JCPDS-PDF-database of quartz, cristobalite, and calcite, and the glass hump in the range of 18°–34°2Θ is marked in orange. In the diffractogram of IRSC 93, the main peak positions of feldspar are indicated. For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article

the islands of Sardinia and Corsica to the mainland, structured by the flow of obsidian and other goods and information less visible in the archeological record (Freund and Batist 2014). Nonetheless, there is no evidence for the movement of metal objects through these networks, and evidence of insular production has taken form at several sites, including Terrina IV in east-central Corsica, where locally sourced copper artifacts, socketed crucibles, and slags have been dated to the mid-to-late fourth millennium BCE (Camps 1988; Pearce 2013).

On Sardinia, evidence for local production is tentative, and further archaeometric testing of apparent crucibles from Monte d'Accoddi and Terramaini as well as lead isotope analyses of Late Neolithic artifacts are warranted, building on a large body of discourse about Sardinian Bronze Age copper sourcing and connections with Cyprus and the wider Mediterranean world (e.g., Stos-Gale et al. 1997; Begemann et al. 2001). A broader point to be made is that if in situ smelting did occur on Sardinia, it would have been relatively simple, involving processes carried out under poorly reducing conditions that would have resulted in little subsequent slag (Roberts 2014: 430). During these early phases, metal artifacts were not widespread and likely had little cultural relevance beyond a few interspersed communities. However, over the course of the Chalcolithic from the third millennium BCE onward, the proliferation of these practices across the island and the development of distinct traditions of production would eventually have a profound impact on prehistoric lifeways.

Contextualizing metallurgy at Su Coddu

This study demonstrates the presence of both copper and lead-based metal artifacts at Su Coddu as early as the mid-to-late fourth millennium BCE. The copper pins analyzed in this study lack any appreciable quantity of arsenic. Arsenical copper alloys are known from sites in later time periods; however, Atzeni et al. (2005: 118) argue that in many cases they were inadvertently smelted together as a result of arsenic minerals being present in the ores. If this is true, then the copper artifacts from Su Coddu are not derived from the same ore sources often utilized in later time periods.

The presence of an unshaped piece of lead at Su Coddu is unique because it predates the earliest known artifact of its type on the island, which comes from the Abealzu Copper Age site of Corte Noa several centuries later, consisting of a gallery grave with both silver rings and "scraps of lead" (Melis 2000: 83; Usai 2005: 262). The precocious presence of lead at Su Coddu could be related to the site's close proximity to available sources of ore, taking form as lead-zinc Hercynian vein deposits roughly 30 km northeast of the site (see Valera and Valera 2005).

While metal artifacts are well documented during the Ozieri phases of the Late Neolithic, it is not well understood how and where these artifacts were first produced. Su Coddu has consistently been cited as possessing evidence of in situ smelting during the Late Neolithic based on the recovery of numerous pieces of metallurgical slag, but XRD analyses in this study reveal that these "slags" are unrelated to metallurgical activities and are best characterized as ceramic pyrotechnology. The mineralogical composition of the fired artifacts matches well with similar archaeometric analyses of ceramic wall coatings within mud-brick structures from the Canelles area of Su Coddu (Fig. 6). These wall coatings consist of a combination of multiple layers, including (a) a coarse coat of locally sourced Tertiary fossiliferous marly clay high in quartz and K-feldspars, (b) a fine coat of the same marly

Fig. 6 Photograph of unfired wall coatings from Su Coddu (from Melis and Santacreu 2017: 193)

clays, and (c) a very thin limewash patina rich in calcite (Melis and Santacreu 2017; Santacreau et al. 2016). The large quantity of overfired material throughout the residential structures of the site further supports the conclusion that they could be burnt wall coatings related to residential construction; since they are found across multiple structures, a single destruction event is possible.

Conclusions

This study characterizes the metallurgical and pyrotechnological evidence from the site of Su Coddu/Canelles in south-central Sardinia (ca. 3400–2850 BCE), and in doing so reveals the presence of copper and lead-based artifacts. While Su Coddu has long been thought to provide evidence of in situ smelting practices in its earliest phases of occupation, the material analyzed in this study shows that the evidential bases on which these claims were made are unreliable.

Despite the presence of metal artifacts in Late Neolithic Sardinia, the results of this study further highlight the lack of contemporaneous, unequivocal evidence for copper, silver, or lead-based smelting anywhere on the island. Additional isotopic and elemental sourcing work on pre-Nuragic artifacts is therefore of critical importance in addressing questions about the nature of early metallurgical practices and their effects on the social and cultural development of prehistoric communities (cf. Pearce 2016). These results in combination contribute not only to understanding early metallurgical practices in prehistoric Sardinia but are also relevant in reconstructing the events that have shaped the life history of one of the most important prehistoric sites on the island.

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Author Contributions. Kyle Freund: conceptualization, funding acquisition, investigation, methodology, project administration, pXRF analysis, writing (original draft); Silvia Amicone: conceptualization, investigation, methodology, thin section petrography, writing (petrography and XRD methods and analysis), reviewing and editing the original draft; Christoph Berthold: funding acquisition, methodology, XRD analysis, reviewing and editing the original draft; Umberto Veronesi: SEM-EDX analysis, reviewing and editing the original draft; Robert H. Tykot: methodology, pXRF equipment and calibration; reviewing and editing the original draft; Maria Rosaria Manunza: conceptualization, sampling, excavation, reviewing and editing the original draft.

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