Metallography, Microstructure, and Analysis (2023) 12:327–348
https://doi.org/10.1007/s13632-023-00952-4

PEER-REVIEWED PAPER

Archeometrical Study of Metallic Remains from "La Ulaña" Archeological Site

J. Setién[1] · M. Cisneros[2]

Received: 16 September 2022 / Revised: 22 February 2023 / Accepted: 9 March 2023 / Published online: 31 March 2023

Abstract

This paper shows the results of the archeometallurgical study of 31 iron pieces, 23 bronze pieces, 1 silver, and 1 gold piece found in the archeological site of La Ulaña, which is located in the north of Spain in the foothills of the Cantabrian Mountains, and that lived its period of greatest splendor coinciding with the Second Iron Age. The basic chemical and microstructural analyses of the iron- and copper-based objects provide an overview of the use of these metals in the site. The results obtained are compared with those published from other archeological sites nearby. In addition, the most unique pieces are set in their archeological context to facilitate the understanding of their chronological ascription.

Keywords Archeometallurgy · Chemical analysis · Copper alloys · Metallography · Iron-based metals

Introduction: The Archeological Site

Peña Ulaña is located in the northwest of the province of Burgos, in the region of "Las Loras" in the municipality of Humada (Fig. 1). It is a 5-km-long hill with a flattened surface which is practically isolated from the surrounding topography, and with a variable width that ranges from 150 m at its northwest end to almost 1000 at its widest part. It has an altitude between 1150 and 1230 m and rises approximately 230 m in relation to the valleys that surround it, allowing the view of a vast area while making it visible from a great distance.

One of the peculiarities of the site is its extension: It covers an area of 586 hectares, of which 285 are located on the upper platform and the remaining 301 hectares belong to the natural moat or "Cinto" which surround it, giving rise to the largest settlement of the Second Iron Age of the Iberian Peninsula, and one of the largest in Europe [1].

Its defensive model is extraordinary due to its size and the use of nature: The natural moat, which is raised above the valley, uses the limestone ridges that surround the rock to act as a natural trench and channel all the accesses through it. This defensive system also had a discontinuous wall on the north side which protected the most easily accessible parts, that is to say, those areas that lacked outcrops, and there was another transversal wall that divided the *oppidum* into two uneven parts. The south side did not have any artificial defenses as they were not necessary given the height of the cliffs [2].

A total of 179 structures with different ground plans (circular, rectangular or oval) were found during the archeological works, which we have grouped into domestic spaces or dwellings based on the spatial analysis. This number indicates a low constructive pressure on the space, which reflects a partial occupation of the place which can be considered similar to that of other European *oppida*, such as Titelberg, Manching, Mont-Beuvray, or Heidengraben [3, 4].

The surfaces of the structures in La Ulaña range between 6.6 and 31.5 m^2. In this regard, it should be highlighted that

In memoriam Ramón Herce, member of University of Cantabria and of the La Ulaña Archeological Area Project.

This invited article is part of a special topical issue of the journal Metallography, Microstructure, and Analysis on Archaeometallurgy. The issue was organized by Dr. Patricia Carrizo, National Technological University – Mendoza Regional, and Dr. Omid Oudbashi, Art University of Isfahan and The Metropolitan Museum of Art, on behalf of the ASM International Archaeometallurgy Committee.

✉ J. Setién
jesus.setien@unican.es

[1] Laboratory of the Science and Engineering of Materials Division (LADICIM), Civil Engineering School, University of Cantabria, Cantabria, Spain

[2] Historical Sciences Department, Faculty of Humanities, University of Cantabria, Cantabria, Spain

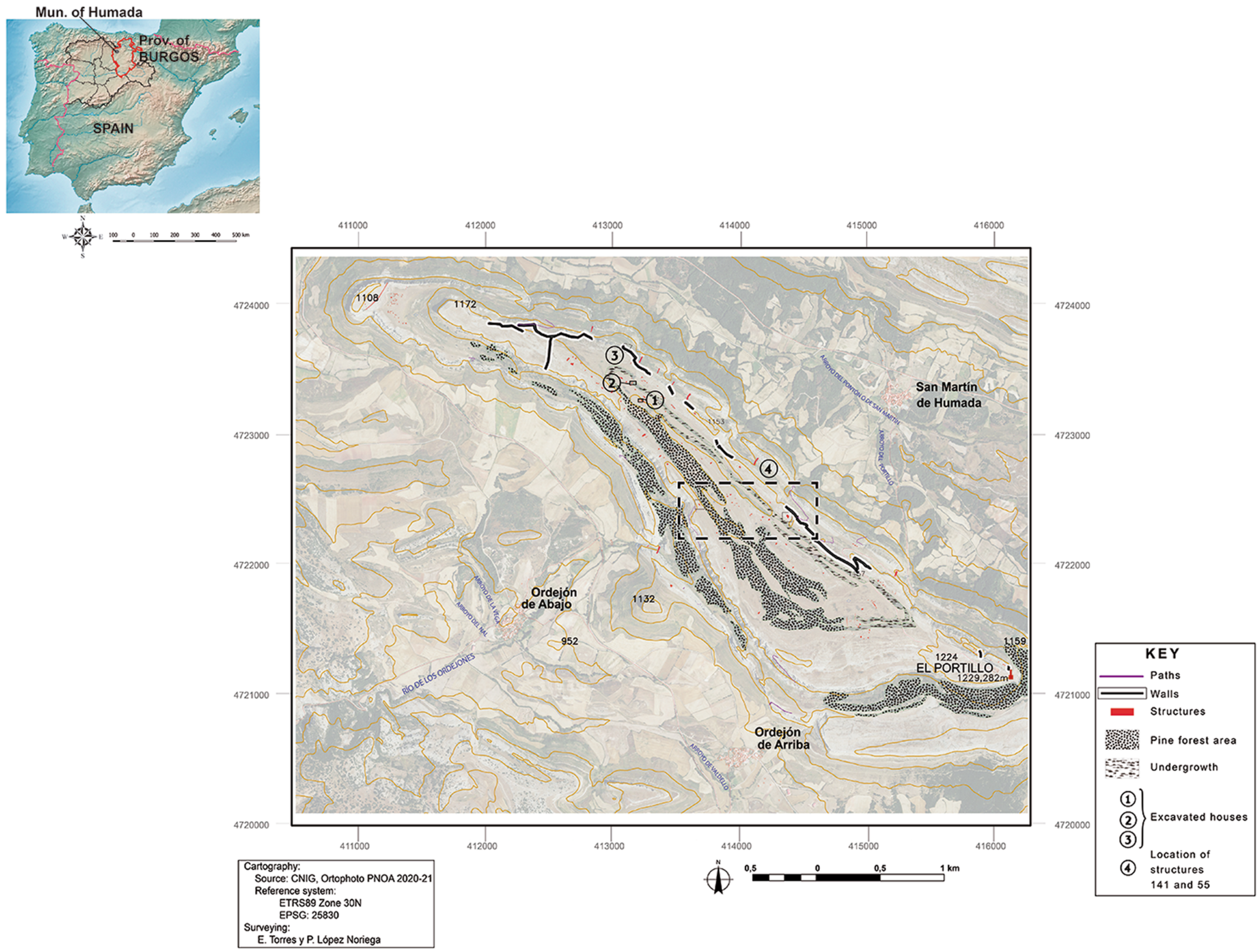

Fig. 1 Plan of La Ulaña superimposed on the orthophotograph (Proyecto Zona Arqueológica de La Ulaña, adapted by J. M. García Rodríguez, University of Cantabria)

the two living rooms (found in dwellings 1 and 3) are the ones that show the largest dimensions, while the rest are considered for the time being as different rooms or workplaces (Fig. 2) [5]. Likewise, this *oppidum* is the only one in the Cantabrian area that provides a completely dug out dwelling, number 3, with a useful area of 55.9 m^2.

The existence of two structures, numbers 55 and 141, must also be added to this. Their size and their morphological and topographic characteristics differentiate them from the rest of the documented structures, although only one of them has been excavated, number 55. Both have a similar shape with a rectangular ground plan, open to the southeast and finished off in one of their sides, the southwest one, in exedra. They are close to two accesses, one to the north, number 55, and another to the south, number 141 (Fig. 3). Furthermore, both have large dimensions: Number 55 is 20 m long on its northeast side and 16 m on its southwest side, the side finished off in exedra, while its width is of 18 m. Structure number 141 has an extension of 16 by 7 m. These characteristics have led us to hypothesize that these are two buildings that had a collective nature and which were connected to the entrance to the settlement, although their specific purpose is difficult to specify.

The excavation of structure number 55 has not provided any more information. This structure had a masonry plinth, of which between one and two layers still exist. Its walls could have consisted of wooden posts and a vegetation framework daubed with mud, based on some fragments that have been found, and the roof must have had wooden beams; fragments of those burned beams and wooden posts were found concentrated, mainly, in its northeast–northwest area. However, we do not know if the structure was totally or partially covered, since its surface was highly eroded, the rock level being visible in its central and southeast areas, and the depth of the excavated levels was not more than 50 cm from the ground level to the bedrock below, being concentrated precisely close to the walls. In addition, as shown in Fig. 3, the northeast and

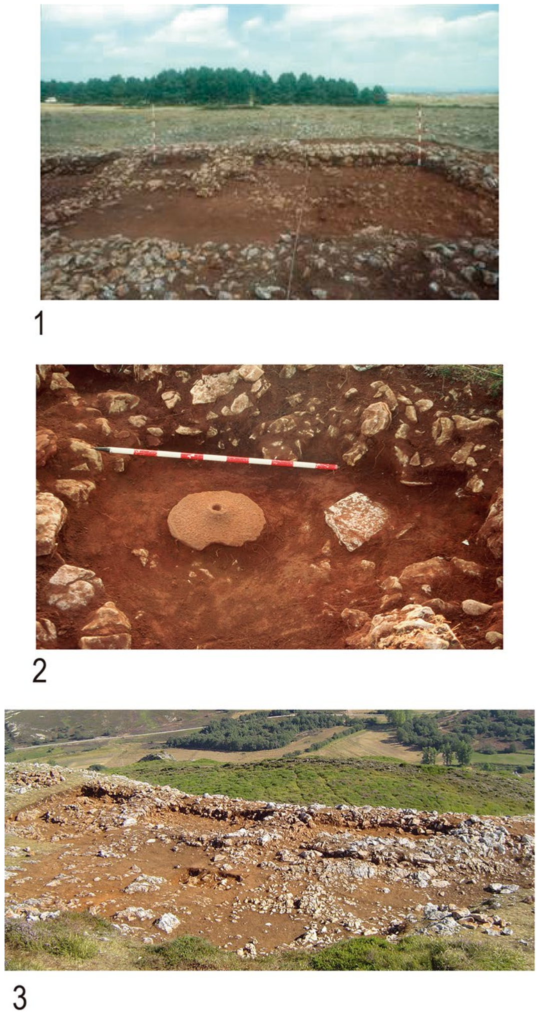

Fig. 2 (1) House 1: detail of structure 182. (2) House 2: detail of structure 78, (3) House 3: detail of structures 88, 89, and 89–1 (Proyecto Zona Arqueológica de La Ulaña. Photographs: M. Cisneros)

southwest walls are closed off with individual masonry walls, 3.5 and 3 m long, respectively, partially defining the space.

On the other hand, the materials found: ceramics (fragments of small handmade pots with incised decoration in the shape of a sprig, as well as large pots with a duck-head-shaped rim made using a potter's wheel), lithics (flint), metallic: bronzes (two articulated plaques or belt buckles, one possible scabbard decoration, one leaf shape decorated plate); irons (plate fragments and nails) and a fragment of molded thread, made of gold, and bone (*bos Taurus*, sheep and goat remains), do not allow us to be more explicit when it comes to specifying its purpose. However, we believe that their distinctive shape and location, the same as number 141, were not accidental, although at this time we can only suggest hypothesis.

Metallic Pieces

The site is not characterized by a great amount of materials, but both their chronologies and the absolute dating indicate an occupation during the Second Iron Age, as given in Tables 1 and 2. However, in Table 1 there is a date for dwelling 2, GX-27504, which places us in the First Iron Age, but only the following materials from that period were found in this house: handmade or turntable ceramics, which date from the transition from the First Iron Age to the Second and which are common in the latter, and a pendant made of slate, which has parallels in the so-called schist pendants that Mohen [6] dates between 550 and 400 BC in the Limousin–Perigordian group. Regarding the dates in which the lower range falls into moments after the Era, especially GX-30855, we can only mention that no material, which can be ascribable to Roman times [2], has been found at that level, level 52, as will be discussed later in the section dedicated to the denarius. In the same way, no other material ascribable to Roman times was found in the excavated areas either, unlike what happens in other nearby sites such as Peña Amaya (Fig. 4(1)) and Monte Bernorio (Fig. 4(2)). In Peña Amaya, the occupation during the Second Iron Age can be seen in a few materials, while the Roman occupation is documented from early stages in different types of ceramics, ornamental and work metallic elements, coins, and even in the appearance of marble, that continues until medieval times [7], while in Monte Bernorio in which the occupation during the Second Iron Age was very important, showing evidence of an attack by the Roman army from a camp located nearby, which is documented in the levels of destruction and in the remains of weapons found [8, 9].

2.1 The total number of iron pieces is 118, while the number of bronze pieces is 51. Those on their own do not provide us with a chronology, nor the ascription to a specific culture, since their shape varies little over time. For this reason, it is necessary to take their archeological context into account to date them. Among the findings, it is possible to highlight:

2.1.a Knives: they are common artifacts which are used for cutting or processing raw materials, and although their use is mainly in the household, they can also be found in other spaces, such as funeral and ritual spaces and therefore they are not considered as a weapon. The size of these objects may vary, although this does not affect their function, as can be seen, for example, in archeological sites as Numancia [10]. In La Ulaña, we have found seven knives: six coming from excavations and one from electromagnetic survey in the pine forest area (07.30.004).

The knives found in excavation have been: one (00.5.0004) in dwelling 1 on level 4 together with a

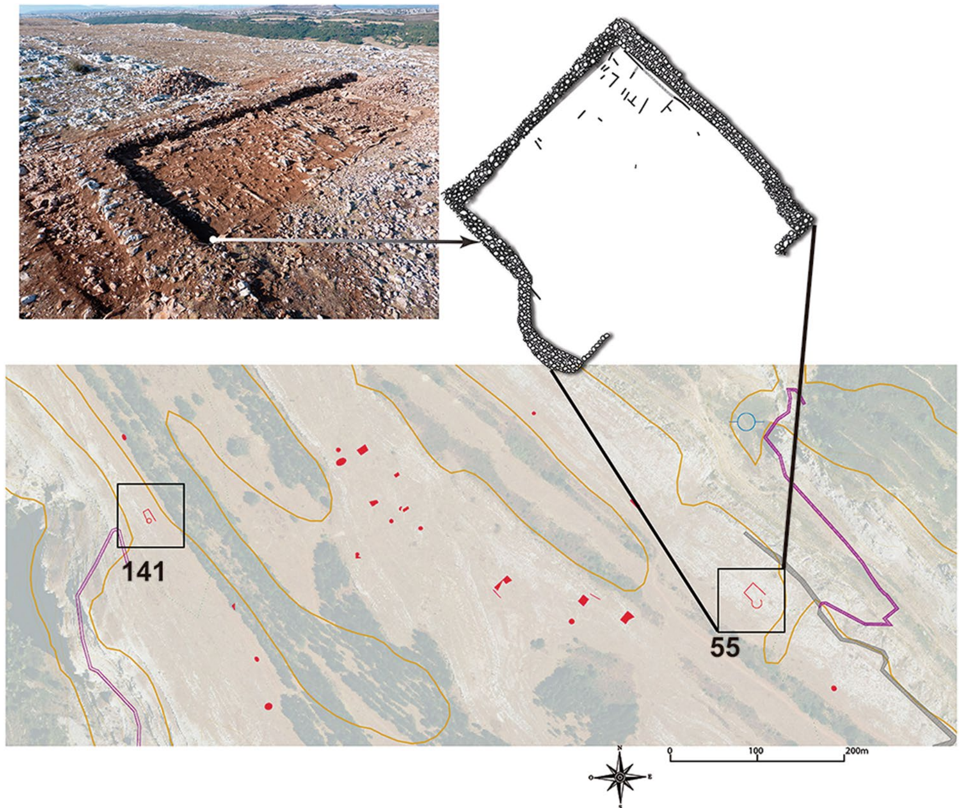

Fig. 3 Detail of the location of structures 55 and 141, and ground plan of structure 55 with an indication of the fragments of wooden posts and beams found and detail of structure (Proyecto Zona Arqueológica de La Ulaña. General plan: E. Torres and P. López Noriega. Plan of structure 55: P. López Noriega and M. Pardomingo, adaptation: J. M. García Rodríguez, University of Cantabria. Photograph: M. Cisneros)

Table 1 Radiocarbon dates calibrated using OxCal and the IntCal20 calibration curve (Author: M. Cisneros)

Laboratory reference	Origin	BP date	±	d13C	Material	BC cal Interval		%	Structure
GX-30247	E-6/N.37	2110	50	−25.8	Charcoal	−353	10	95.45	Transverse wall
GX-30248	E-6/N.37	2230	50	−25.8	Charcoal	−396	−171	95.45	Transverse wall
GX-30855	MN1-A/N.52	2060	100	−25.3	Charcoal	−362	202	95.45	Occupation inside north wall
CNA-564	MN1-B/N.48	2070	40	−20.37	Bone	−197	26	95.45	Occupation inside north wall
GX-27507	183 III/N. 10	2120	50	−24.4	Charcoal	−356	7	95.45	House 1
GX-27511	182 IV/N.18	2180	60	−23.4	Charcoal	−385	55	95.45	House 1
GX-27512	182 II/N. 18	2140	70	−25.3	Charcoal	−381	6	95.45	House 1
GX-27504	77-II/N. 35	2440	40	−27.3	Charcoal	−755	−406	95.45	House 2
Ua-32661	MN2-E/N.56	2075	35	−24.1	Charcoal	−176	17	95.45	House 3
CSIC-2036	MN2-I/N.69	2168	29	−24	Charcoal	−359	−103	95.45	House 3
Ua-32663	MN2-O/N.88	2090	40	−25.5	Charcoal	−341	9	95.45	House 3
CNA-2557	55-AF/N.42	2090	35	−22.23	Charcoal	−338	6	95.45	Structure 55
CNA-2558	55-AF/N.42	2250	35	−22.23	Charcoal	−395	−202	95.45	Structure 55
GX-30850	55-B/N.43	2070	40	−24.6	Charcoal	−197	26	95.45	Structure 55
GX-30851	55-B/N.43	2140	50	−24.7	Charcoal	−361	−43	95.45	Structure 55
GX-30852	55-B/N.43	2080	40	−22.8	Charcoal	−334	17	95.45	Structure 55
CNA-2561	55-G/N.47	2250	35	−23.59	Charcoal	−395	−202	95.45	Structure 55
CNA-2562	55-G/N.47	2115	35	−21.8	Charcoal	−346	−43	95.45	Structure 55
CNA-2559	55-T/N.115	2125	35	−24.23	Charcoal	−350	−45	95.45	Structure 55
CNA-2560	55-N/N.117	2175	35	−20.56	Charcoal	−363	−107	95.45	Structure 55

Table 2 Thermoluminescence datings (Author: M. Cisneros)

Laboratory reference	Origin	BP date	±	Material	Structure
Mad-2323	182-183-I/N.10	2417	21	Burnt clay	House 1
Mad-4682-BIN	MN2-Z/N.82	2313	149	Burnt clay	House 3
Mad-4683-BIN	MN2-Y/N.82	2296	179	Burnt clay	House 3
MadN-6320BIN	55-AF/N.42	2273	120	Burnt clay	Structure 55

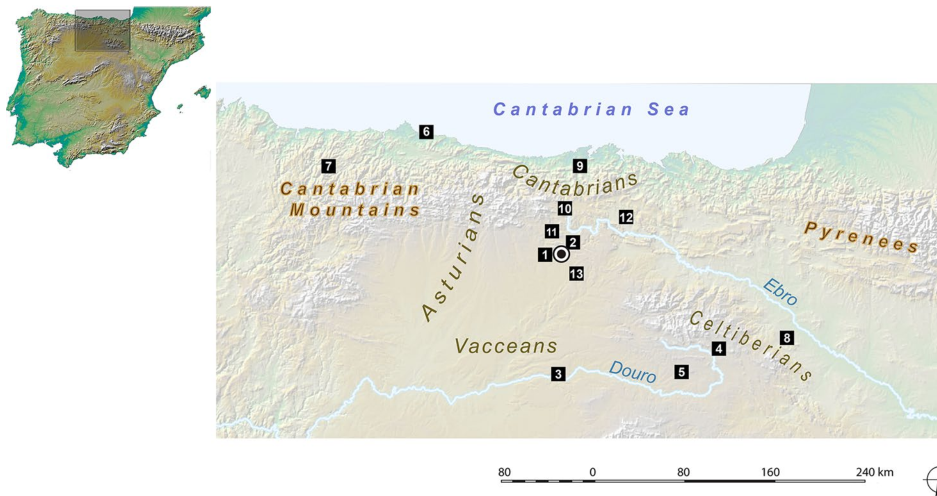

Fig. 4 Location of the sites and pre-Roman peoples mentioned: • La Ulaña (Humada, Burgos), (1) Peña Amaya (Amaya, Burgos), (2) Monte Bernorio (Villarén de Valdivia, Palencia) (3) Necropolis of Las Ruedas (Pintia) (Padilla de Duero, Valladolid), (4) Numancia (Garray, Soria), (5) La Mercadera (Rioseco, Soria), (6) La Campa Torres (Gijón, Asturias), (7) Hillfort of San Chuis (Allende, Asturias), (8) *Turiaso* (Tarazona, Zaragoza), (9) Soto-Iruz (Puente Viesgo, Cantabria), (10) Celada Marlantes (Cantabria), (11) Monte Cildá (Olleros de Pisuerga, Palencia), (12) Quintanalacuesta (Burgos), (13) Sasamón (Burgos) (adapted by J.M. García Rodríguez, University of Cantabria, from various sources)

rectangular iron plate (00.5.0001), a small iron bar (00.5.0005), a bronze dagger scabbard clamp (00.5.0002), fragments of a large vessel with an ovoid body—Castiella shape 19—fragments from a decorated large earthenware jar—Castiella shape 21—and fragments of a painted bowl or cup, dating from between the fourth and the first century BC [11]. Three knives have been found in structure 55: item 03.16.073 in level 42 with an iron punch (03.16.158), small indeterminate fragments of iron and bronze objects, and also of dark-colored handmade ceramic; radiocarbon dating CNA-2557, CNA-2558 (Table 1) and the thermoluminescence MadN-6320BIN (Table 2) are from this level. Item 11.22.050 in level 117 with flint fragments, an iron rod, two nails, a glass paste bead, a bronze articulated plaque or belt buckle (11.22.048), a fragment of molded thread, made of gold (11.22.026), ceramic fragments, similar to Celtiberian ceramic, but without traces of paint, and handmade pottery, some of the specimens with carved decoration, which have been dated from the end of the fourth–beginning of the third century BC; the radiocarbon dating CNA-2560 (Table 1) is from this level. Item 11.22.064 in level 44 with a possible bronze scabbard decoration (03.16.015), a bronze rivet (11.22.072), ceramic fragments with painted decoration which would indicate a chronology similar to the previous ones, and small indeterminate fragments of iron and bronze objects. And the two last knives have been found in dwelling 3: item 04.13.020 (Fig. 5(1)) in level 56 with dark-colored handmade pottery fragments and sheep bones; the radiocarbon dating Ua-32661 (Table 1) comes from this level. Item 05.25.029 (Fig. 5(2)) in level 82 with ceramic fragments that show turntable marks inside, similar to those dating from the fourth century BC. Bone fragments of indeterminate species, an iron rivet (05.25.015) and an iron riveted circular sheet (05.25.030); the Mad-4682-BIN and Mad-4683-BIN thermoluminescence dating come from this level (Table 2).

2.1.b Punches: In La Ulaña, we have found two (07.30.041, 07.30.031) using electromagnetic prospecting: the first one in the pine forest area and the second in the Cinto area. The use of this type of artifacts could have been multifunctional. Jimeno et al. [10] consider that it could be an awl-type instrument which has lost its wood or bone handle. This tool would be used to bore holes in

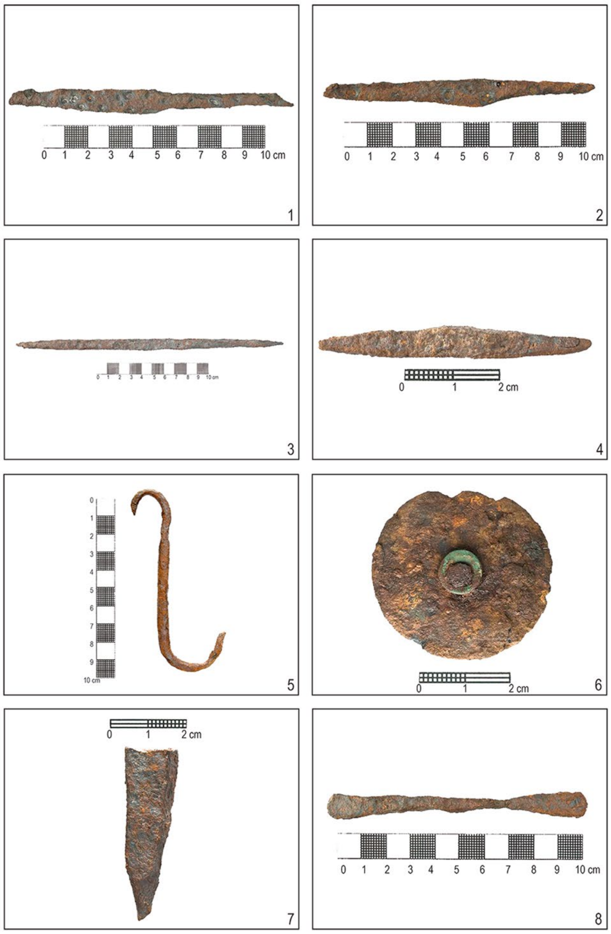

Fig. 5 Objects in iron: (1) Knife. Length: 126 mm; height: 10 mm at the top, 6 mm at the tip, 2 mm at the handle; thickness: between 1 and 2 mm; blade length: 69 mm. Inv. 04.13.020. (2) Knife. Length: 110 mm; height: 10 mm at the highest point, 5 mm at the tip, 4 mm at the handle; thickness: between 1 and 2 mm; blade length: 67 mm. Inv. 05.25.029. (3) Bi-pointed punch. Length: 227 mm, cross section: 9 × 10 mm. Inv. 07.30.041. 4. Bi-pointed punch. Length: 57 mm, cross section: 7 × 5 mm. Inv. 07.30.031. (5) Hook. Length: 100 mm, cross section: 6 × 6 mm. Inv. 07.30.044. (6) Circular iron plate with bronze rivet. Diameter: 39 × 36 mm, thickness between 1 and 2 mm; rivet: head, diameter: 10 mm, thickness: 12 mm, back: 12 × 13 mm. Inv. 05.25.030. (7) Ferrule. Length: 45 mm, cross section: 12 × 12 mm. Inv. 07.30.035. (8) Iron rod. Length: 103 mm, width: ends: between 9/10 mm; section: 6 × 6 mm. Inv. 05.25.122 (Proyecto Zona Arqueológica de La Ulaña. Photographs: J. Romeo)

clothing and belts, and therefore, it would be very useful in the domestic and military spheres without there being a need to link them to specific artisans. On the other hand, Sanz [12] considers that some of the punches found in the necropolis of Las Ruedas (Padilla de Duero, Valladolid) (Fig. 4(3)) could indeed have belonged to the grave goods of an artisan. Those punches found in Numancia (Garray, Soria) (Fig. 4(4)) do not exceed 80 mm, while in La Mercadera (Rioseco, Soria) (Fig. 4(5)) Lorrio [13] indicates a maximum length of 175 mm which is closer in size to one of our items (07.30.041) (Fig. 5(3)). Other bi-pointed punches found at the site are smaller and could have had a clear craft purpose (07.30.031) (Fig. 5(4)).

Fig. 6 Hemispherical tinned iron rivet. Inv. 07.30.008 (Proyecto Zona Arqueológica de La Ulaña. Photograph: J. Romeo)

2.1.c Hooks: They can have very different uses. Of the two that have been found at the site, we can highlight the one (07.30.044) (Fig. 5(5)) which was found in structure 90-1, of dwelling number 3, next to handmade and wheel-made ceramics, bones, and other metallic elements and next to a small hearth [5]. The other item was found in construction number 55 in level 118 together with flint, sheep, and goat bone and other undefined fragments and a bronze necklace bead (11.22.075). Hooks with a similar shape, although of very different sizes, have been documented in Numancia (Fig. 4(4)) [14].

2.1.d Circular iron plate with bronze rivet: One item (05.25.030) (Fig. 5(6)) has been found in the site in dwelling number 3 in level 82. The materials have already been mentioned in the knives section. It was probably part of the decoration of a scabbard. A parallel to these could be found among some artifacts from Numancia (Fig. 4(4)) [10].

2.1.e Ferrules: Two ferrules have been found at the site. One of them has already been reported [1], and the other one is presented in this paper, which was found in the electromagnetic survey of the Cinto area (Fig. 5(7)). It is an iron tip with a pyramidal body and quadrangular section, which could be the lower part of an iron ferrule like the one that was found in grave 28 of the necropolis of Las Ruedas (Fig. 4(3)) [12].

2.1.f Other pieces also in iron which have already been previously published should be added to these materials, such as nails and scissors and some items of unknown use, a rectangular sheet, a small iron rod, or a rod fragment, which are analyzed in the following section [15]. And there are other unpublished items, such as a hemispherical rivet, which would be part of the decoration of some unspecific piece (Fig. 6), or a square section iron rod with flat ends (Fig. 5(8)) which was found during an electromagnetic survey carried out at the Cinto area. All this is in addition

Fig. 7 Objects in bronze: (1) necklace bead. Diameter: 9 mm, thickness: 7 mm. Inv. 03.16.099. (2) Necklace bead. Diameter: 16 mm, thickness: 11 mm. Inv. 11.22.075. (3) Leaf shape decorated plate. Length: 24 mm, width: 8 mm, thickness: 1 mm. Inv. 03.16.115. (4) Articulated plaque or belt buckle. Length: 27 mm, height: 21 mm, thickness: 3 mm Inv. 11.22.048. (5) Articulated plaque or belt buckle. Length: 18 mm, height: 16 mm, thickness: 1 mm. Inv. 11.22.091. (6) Ring. Diameter: 12×12 mm, thickness: 3 mm. Inv. 03.16.156 (Proyecto Zona Arqueológica de La Ulaña. Photographs: J. Romeo)

to the remains of slag which are frequently found in the excavations.

2.2 The number of bronze pieces is 51 and most of them are incomplete decorative elements. Among them, it is worth highlighting:

2.2.a Necklace beads: In La Ulaña, we have found two bronze specimens that come from structure 55. They have different shapes (Fig. 7(1) and (2)) and were threaded through the center with a cord. The first item (03.16.099) (Fig. 7(1)) was found in level 47, together with three iron plate fragments (03.16.049, 03.16.109, 03.16.118), a bronze ornament (03.16. 177), an unknown bronze fragment (03.12.144), a bronze rivet (11.22.010), large vessel fragments with duck-head-shaped rim, which are dated in the late fourth–early third centuries to the first century BC [16], handmade ceramic fragments with incised decoration in the shape of a sprig, which could be dated from the late fourth–early third century BC, a bronze leaf shape decorated plate (03.16.115), a bronze articulated plaque or belt buckle (11.22.091), in addition to an iron rod and nails. The radiocarbon dating CNA-2561 and CNA-2562 (Table 1) come from this level. The second item (11.22.075) was found in level 118, and the materials have been previously indicated in the hook section.

Metallic beads with similar characteristics have been sufficiently confirmed in the Celtiberian area [13]. Among the most important nearby sites which have received an exhaustive publication, we can mention the discovery of this type of beads in the pre-Roman levels of La Campa Torres (Gijón, Asturias) [17], in Pintia in the necropolis of Las Ruedas [12], and in the necropolis of Numancia (Fig. 4(3), (4), and (6)), in which both bronze and glass necklace beads have been found, and although it cannot be asserted that they were part of the same necklaces, this idea cannot be ruled out either [10]. The same approach can be carried out for the necklace beads found at the site, as glass ones have also been found.

2.2.b Leaf shape decorated plate: It was found in structure 55 in level 47 and the materials have already been mentioned in the necklace beads section. It is fragmented and was part of a decorative ornament (Fig. 7(3)). It could be a pendant, since these have very different shapes, as documented by Lorrio [13] in the Celtiberian region.

2.2.c Articulated plaques or belt buckles: two fragmented elements (Fig. 7(4) and (5)) which could correspond to the so-called articulated plaques or belt buckles, although this possibility given their size seems more remote, but it cannot be ruled out observing the bibliography. Both pieces were found in structure 55. The first (11.22.048) (Fig. 7(4)) was found in level 117, and the materials have been previously mentioned in the knives section. The second one (11.22.091) (Fig. 7(5)) was found in level 47, and the materials have already been mentioned in the necklace bead section.

Articulated bronze plaques similar to ours have been found in the necropolis of Las Ruedas (Fig. 4(3)) [12], although, as Sanz points out, they could be plaques and plates of doubtful ascription, as they are fragmented and they are not usually found complete. They could also be ornamental elements that lack the organic support that would give them coherence which makes their interpretation difficult [12]. These Celtiberian plaques that could be belt buckles, not always entirely metallic, are made up of several rectangular plaques which are articulated with each other through a hinge system. Their development corresponds to Celtiberian age which even extends to the first century AD. They are richly decorated elements based on simple incised and parallel lines, concentric circles, bands of triangles, etc., which may also have round headed rivets [12].

These pieces could also be similar to some of the belt buckles published by Lorrio [13] for the Celtiberian area. In particular, Inv. 11.22.091 (Fig. 7(5)) is similar to the ones published by Maya [18] from the hillfort of San Chuis (Allende, Asturias) (Fig. 4(7)), although this author points out his strangeness about the appearance of an item that he considers fully pre-Roman in a settlement of Roman chronology.

However, Jimeno et al. [10] consider that the label of articulated plaque is not correct, as anything from pectoral ornament plaques to decorative pendants is put into this classification, including wrongly identified plaques. In fact, in the necropolis of Numancia (Fig. 4(4)) these plaques are considered as decorative elements.

2.2.d Other pieces also in bronze such as rings which may have different functions should be added to these materials (Fig. 7(6)). These pieces could correspond to open chain links, but their use cannot be specified. Other bronze items previously published can also be added to these pieces, such as sheets of disks, a dagger scabbard clamp, rivets, a triangular plate, a possible fragment of a weighing scale, a fragment of a cauldron handle [15], or a needle [1].

2.3 An object that stands out for being the first remains of gold work found in the territory of the ancient Cantabrians is a small gold thread (Fig. 8(1)) which appeared in structure 55 in level 117, and the materials have already been mentioned in the knives section. It is a fragment of molded thread, made of gold (the analysis reveals an alloy of gold and silver, as will be discussed in the following section) and which, perhaps, was welded at the base to another object, as can be seen at one of its ends. It is an ornamental element that could have been part of a complete jewel or some other type of ornament accessory.

However, the small size of our item, which is 10 mm in diameter, makes it difficult for it to have been part of the bezel of a ring, despite the fact that its more or less circular layout brings to memory a type of ring known in the Second Iron Age in the North Plateau, which could be made in gold or silver and whose bezel usually consisted in three concentric threads (two molded and one smooth, for example) although its diameter is bigger [19].

It could be part of an ornamental element of a pendant earring, either from the body area (the earring hooks) or from some part of the extension [19]. Although, it could also be a piece of thread used for any other purpose, such as body or clothing adornment. Any of these interpretations is feasible.

2.4 Another noteworthy finding took place at the ground level of a possible occupation inside one of the excavated parts of the North wall in level 52 showing a ferrule (03.16.123), fragments of small handmade pots which are dated from the transition of the First to the Second Iron Age [20], *bos Taurus* and *Capra hircus* bones and goat and sheep teeth. The radiocarbon dating GX-30855 comes from this level.

It is a denarius from the Turiaso mint (Tarazona, Zaragoza) (Fig. 4(8) and Fig. 8(2) and (3)), which shows a bearded head looking to the right on the obverse, with curly hair, a necklace around the neck, and the legend *Kastu*, in Iberian characters around the head [2]. On the reverse side, there is a horseman holding a spear, with the four legs of the horse on the legend *Turiasu*, also in Iberian characters. It would belong to group V, established by Gonzalbes [21], when studying the coins of that mint, and which the chronology would date between 125 and 80 BC as possible group deadlines.

Fig. 8 (1) Molded gold thread Inv. 11.22.026. (2) and (3) *Denarius* from *Turiaso* mint. Observe and reverse. Inv. 03.16.122 (Proyecto Zona Arqueológica de La Ulaña. Photographs: J. Romeo)

Archeometallurgical Analyses

As mentioned in archeological section, the number of metallic pieces found is small (169 records) regarding the total of materials recorded (1092), out of which a total of 54 metallic objects from the site have been analyzed; all of them are based on the use of iron and of copper and their alloys, with some notable exceptions that will be discussed about later. Similarly, various fragments of iron foundry metallurgical slag have been collected.

The pieces found, in general, show a very incomplete state of conservation, their uses in daily life being very different, as we have already described in the previous section.

Iron Pieces

A total of 31 iron or some type of iron-based metallic objects have been registered, which makes up a 55.4% of the total number of metallic objects recorded, while the remaining 41.1% and 3.5% correspond to different quality bronze pieces and other noble metals (gold and silver), respectively. The iron objects are very damaged by the action of corrosive processes and the layers of corrosion products reach very significant thicknesses.

Analytical Methodology

The compositional analyses have been carried out on the exposed surfaces of the base metal after locally eliminating the superficial patinas of corrosion products using abrasion methods. Energy-dispersive x-ray microanalysis by microprobe of a scanning electron microscope (SEM + EDX) has been used for the analytical characterization of the pieces. The equipment used is a ZEISS model EVO/MA 15 scanning electron microscope equipped with a lanthanum hexaboride (LaB_6) emitter. The x-ray detector is the X-Act model from Oxford Instruments and the x-ray dispersive energy microanalysis software is INCA from the same company. The ZAF-4 analytical correction was used in the quantifications. The operating voltage used in the analyses has been 15 kV and the working distance has been around 9.5 mm, operating at different magnifications depending on the specific needs of each sample. The calibration of the x-ray detector is carried out annually using conventional reference standards from RAVE SCIENTIFIC.

On the other hand, the sample extraction for the metallographic analyses has been carried out using a precision saw with a diamond abrasive disk. The surfaces of interest of the selected samples have been polished with abrasive paper and then with a cloth impregnated with diamond paste until the surface had a mirror-like finish, after which they have been etched with Nital 3 and analyzed using a metallographic or a scanning electron microscope depending on the needs.

Finally, microhardness determinations have been performed on the selected samples using a Vickers-type indenter, with the applied load ranging between 20 and 500 g depending on the microstructural features and a load application time of 20 s in all cases.

Results (Table 3)

The results shown in Table 3 are the average of three analytical determinations carried out in three different points of each of the samples. In most cases, the analyses

Table 3 Chemical analysis of the iron pieces (wt%) (Author: J. Setién)

Inventory	Description	Fe	Si	P	Al	Mn	Sn
00.5.0001	Rectangular plate	99.2	0.1	0.7			
00.5.0005	Prismatic ingot	99.9	0.1				
00.5.0093	Circular section rod	99.8	0.1	0.1			
03.16.073	Knife	100					
03.16.158	Punch	99.0	1.0				
03.16.123	Ferrule	99.4	0.6				
03.16.118	Plate fragment	100					
03.16.049	Plate fragment	99.0	1.0				
03.16.109	Plate fragment	98.6	1.4				
03.16.161	Slag	95.8	4.2				
03.16.102	Plate fragment	98.6	1.4				
03.16.092	Nail	98.6	1.4				
03.16.184	Slag	92.4	5.2		2.4		
04.13.020	Knife	100					
05.25.015	Rivet	100					
05.25.029	Knife	100					
05.25.030	Riveted circular sheet	98.6	1.4				
05.25.122	Square section iron rod with flat ends	100					
07.30.001	Rivet	100					
07.30.004	Knife	97.4	1.7		0.9		
07.30.007	Rivet	96.9	2.0		1.1		
07.30.008	Hemispherical tinned iron rivet	Base metal	98.1	1.4	0.5		
		Coating	0.1	0.1			99
07.30.012	Bent sheet	100					
07.30.016	Rivet	100					
07.30.017	Rivet	96.4	1.4		2.2		
07.30.030	Rivet	85.5	10.5			4.0	
07.30.033	Possible key	100					
07.30.035	Ferrule	98.9	1.1				
07.30.037	Ring	100					
07.30.041	Bi – pointed punch	100					
07.30.044	Hook	99.0	1.0				

reveal that they are practically pure iron pieces (as the knives—04.13.020 and 05.25.029, the rod—05.25.122, and the bi-pointed punch—07.30.041—among the items mentioned in the previous section), and the associated presence of peaks corresponding to carbon is also detected in the spectra. The presence of variable silicon contents (as the forged sheet—00.5.0001, the small rod—00.5.0005, the circular section rod—00.5.0093, the ferrules—03.16.123 and 07.30.035, the riveted circular sheet—05.25.030, and the hook—0.30.044, among the aforementioned pieces) and small amounts of phosphorus (as the forged plaque—00.5.0001, the small rod—00.5.0005, the circular section rod—00.5.0093-) are not uncommon either. In some very specific cases, the minor presence of other elements such as aluminum or manganese, which can have a very diverse origin, has also been reported.

However, the small hemispherical rivet shown in Fig. 6 stands out within this fairly homogeneous set of analyses due to the singularity of its result.

As can be seen in the image, there are two well-differentiated areas in the piece: one with a brownish hue that typically corresponds to the dark reddish and ocher tones of the ferric oxides, which reveals an iron core. On top of this layer, there is a more superficial overlapping layer grayish in tone and, in some cases, whitish. The analysis of this overlapping layer reveals that it is a tin coating, so it would be a type of tinplate iron, precursory to modern tinplate [22].

Metallographic Analysis

Some detailed metallographic analyses have been carried out with the aim of studying in detail the knowledge of the underlying technology in the production and manufacturing of these objects made of iron-based metals. Given the intrinsically destructive nature of the preparation of the pieces for the microstructural analysis and given the need to preserve the integrity of those pieces that show the greatest archeological value, it was decided to choose among all the pieces a subset of three indeterminate typology and lesser archeological value objects aiming to limit the impact that the characterization methodology could have on them. However, these objects are considered representative enough to provide significant results that may be applicable to the rest of the pieces, with the due precautions that must always be taken into consideration in this type of extrapolation, as the craftsmanship of each piece is unique.

In this sense, the selected pieces have been a 2-mm-thick rectangular sheet, quite possibly worked on by forging (Inv. 00.5.0001), a small rod (27 × 12 × 7 mm) (Inv. 00.5.0005), and a fragment of a rod with a circular section of about 9 mm in diameter (Inv. 00.5.0093).

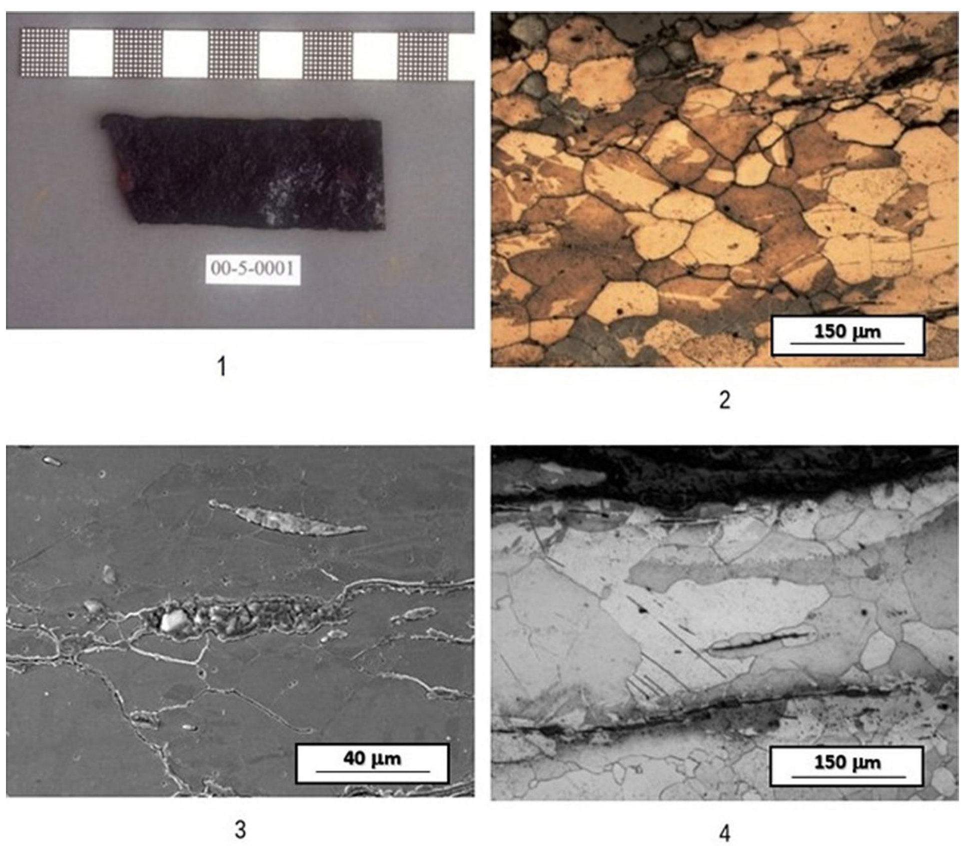

Fig. 9 (1) Rectangular sheet worked by forging. Inv. 00.5.0001 (Proyecto Zona Arqueológica de La Ulaña. Photograph: J. Romeo). (2) General appearance of the ferritic microstructure of the sheet (× 100). (3) Detail of an inclusion in the ferritic matrix. (4) Detail of the microstructure showing the heterogeneity of the ferritic grain size (× 100) (Photographs: J. Setién)

Figure 9 shows the original aspect (Fig. 9(1)) of the rectangular sheet that was worked on by hammering until the desired shape was obtained, the traces left by the tool on its surface being visible to the naked eye. Figure 9(2) offers a general view (× 100) of the aspect of the microstructure of this piece. Basically, they are polygonal ferrite grains with an acceptable equiaxiality and very dark boundaries in those grains which are close to the edges. The presence of inclusions (Fig. 9(3)) and, above all, very elongated cavities (voids) along the longitudinal orientation of the piece, are evidence that the piece has been shaped through severe forging which has caused their crushing and their orientation according to the preferential direction defined by the forging process itself. On the other hand, the fact that the ferrite grains are equiaxial and do not show this preferential orientation shows that the metal has been recovered through an annealing heat treatment that counteracts the most harmful effects of work hardening, specifically the excessive brittleness that is a consequence of the forging process, which in this case is increased by the presence of a relatively high content of phosphorus in the metal (0.74%).

On the other hand, the fact that the boundaries of the ferrite grains close to the outer surface of the piece are burnt, with a typical strong intergranular corrosion, is an indication that at some point in the manufacturing process the piece was subjected to excessively high temperatures, which produced a localized corrosion in the edges of the grains which later, when cooled, show a low cohesion, a mishap that usually receives the name of "burnt metal." The appearance of this phenomenon in iron-based metals with such a low carbon content as this would need, in the absence of phosphorus, temperatures ranging over 1300 °C. However, the presence of phosphorus in significative amounts, which is segregated in the intergranular zone, favors the fact that this phenomenon can take place at lower temperatures, in the range of 1100 °C.

Figure 9(4) shows another different part of the piece (× 100) in which a marked heterogeneity in the grain sizes after the recrystallization can be seen quite clearly, with an alternation of large crystals with much smaller ones and even with some that have been interrupted at an early stage of the crystallization process.

The microhardness values recorded for this piece, for a total of 10 measurements carried out applying a load of 100 g, ranged between 178.9 HVN and 258.2 HVN, with an average value of 219.6 HVN, which is higher than normal for the ferrite phase (approximately HVN160). The hardness of the material varies inversely with the grain size, and therefore, the variability shown in the hardness values is consistent with the heterogeneity observed in the microstructure. On the other hand, the fact that the average hardness is higher than what is expected for the ferrite could be attributed to a slight plastic strain after recrystallization, as the incipient slip lines which are present in some of the bigger grains from Fig. 9(4) seem to indicate.

Figure 10 shows the microstructures corresponding to the small cast bar retrieved from the site. From a microstructural point of view, this piece shows two well-differentiated areas. What mostly can be seen is a typical isothermal transformation structure (Fig. 10(2), × 100), characterized by the presence of a bainitic phase and locally showing signs of a pronounced characteristic intergranular cracking in certain areas, predictably following the original austenite grain boundary, as a consequence of some process of deterioration by corrosion. In this regard, it is well known that treatments such as carburizing or quenching were known in the late Iron Age, although not always did they have a successful result [23]. Evidence of recrystallization, carburizing, and quenching treatments has been observed in the Iberian Peninsula in pieces dating from the beginning of the Iberian culture (seventh century BC) which were found in excavations carried out in the Catalan Mediterranean coast [24] and also in pieces dating from Roman times in the peninsular north [25].

Figure 10(3) (× 500) and (4) (× 1000) shows two details of the microstructure and the massive presence of nonmetallic inclusions stands out, which undoubtedly implies a significant decrease in the mechanical properties of the material, and the hardness is established at a relatively low value for a bainitic microstructure: 241 HVN on average from a total of 15 measurements (maximum value = 268.7 HVN). On the other hand, the chemical analysis reveals that it is a higher purity iron than the one used in the manufacturing of the aforementioned studied sheet, as it contains only 0.013% phosphorus in its composition. In addition, the fact that the piece does not show decarburization in its microstructure reveals that its carbon content must be relatively low.

To finish, the presence of a fairly extensive decarburized area can be observed close to one of the side edges of the piece (Fig. 10(5), × 500) (accounting for approximately 15% of the total section of the bar, Fig. 10(1)). The decarburization on the contour of pieces is something that happens quite recurrently and is usually associated with hot forging work [26]. This area shows a microstructure which is characterized by the presence of ferrite that locally acquires the typical form of the so-called Widmanstätten ferrite, which is quite frequent in very low carbon iron. Its origin would be attributed to an insufficient austenitization in the part which is most depleted in carbon, in which there are typical casting structure remnants. This phenomenon would occur in the decarburized area even when the austenitization temperature has been correct in the rest of the piece, with a higher carbon content, and the heat treatment has been efficient in most of the piece. The characteristic Widmanstätten ferrite structure is so more pronounced the larger the austenitic grain size is and the faster the cooling rates are. In addition,

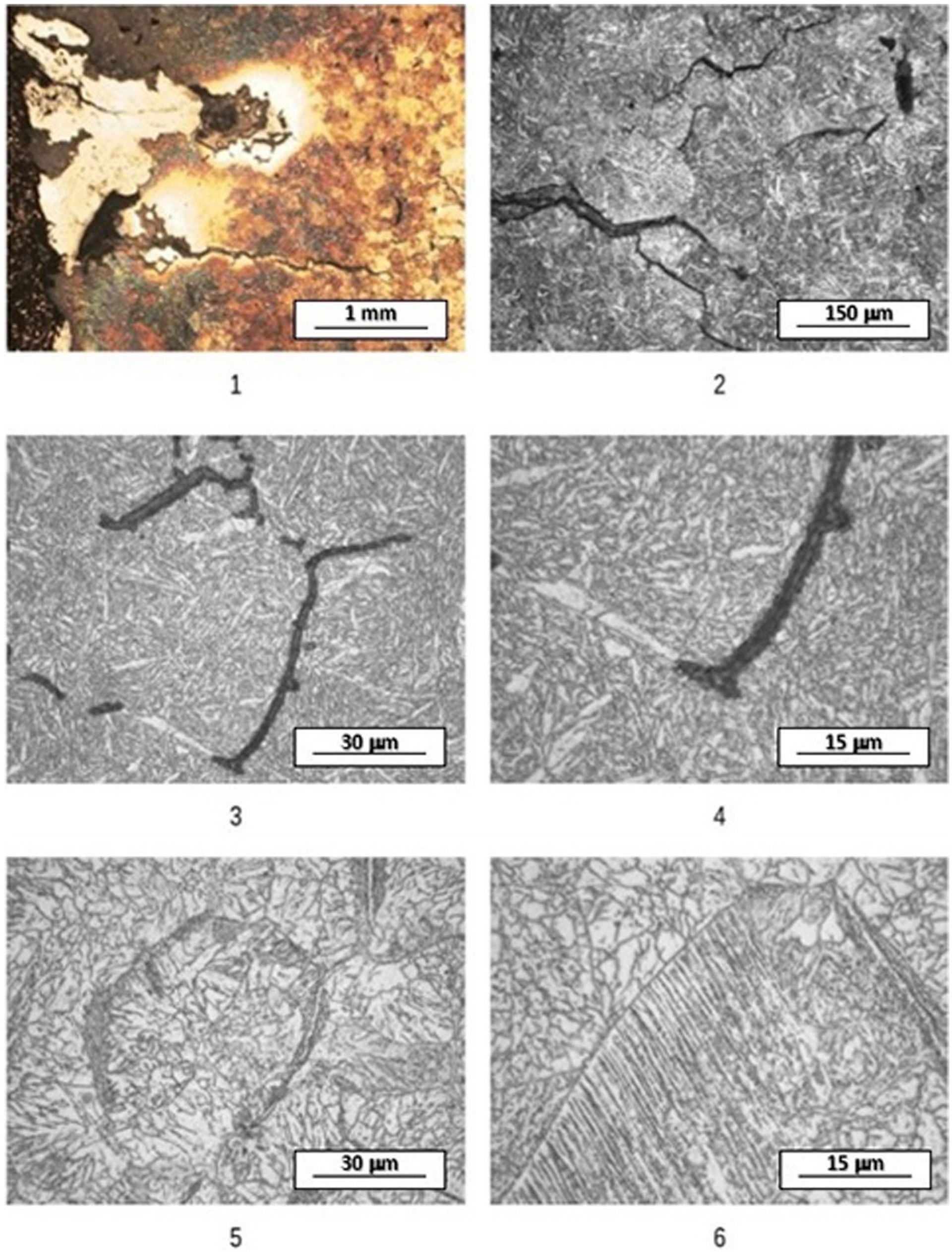

Fig. 10 (1) Decarburized area in the ingot section (×16). (2) General appearance of the bainitic microstructure of the ingot (×100). (3) and (4) Details of the bainitic microstructure revealing the presence of inclusions (×500) and (×1000). (5) Detail of the microstructure in the decarburized area in which the presence of Widmanstätten ferrite can be seen (×500). (6) Bainitic growth from the previous grain boundary (×1000) (Photographs: J. Setién)

needle-like shape growth of bainite can be observed in this same microstructural area arranged in parallel bands and following a preferential orientation within the previous austenitic grain from its boundary (Fig. 10(6), ×1000). In this part of the piece, the microhardness determinations made with an applied load of 100 g yield values that range between 175.0 HVN and 199.3 HVN, with an average value of 187.8 HVN.

For its part, Fig. 11 shows the microstructures corresponding to the circular section rod. Figure 11(1) (×50) shows the general appearance that the micrograph of this piece reveals, which basically consists of a typical balanced structure of ferrite and pearlite. The morphology and characteristic elongation of the grains, which is shown clearly in Fig. 11(2) (×200), reveal that the piece would have been hot forged, although there is no preferential forging direction since there are different well-defined orientations. In this regard, it should not be forgotten that the piece has a more or less cylindrical geometry and that the micrographic analysis has been carried out in a cross section of the same and therefore does not show the longitudinal section which, quite probably, would reveal a more pronounced directionality. In any case, the idea that the piece is simply a direct casting product, as could be assumed at first, is ruled out.

However, the presence of ferrite islands within the pearlite (Fig. 11(3), ×500) seems to indicate that the forging was carried out without receiving enough heat. After the forging

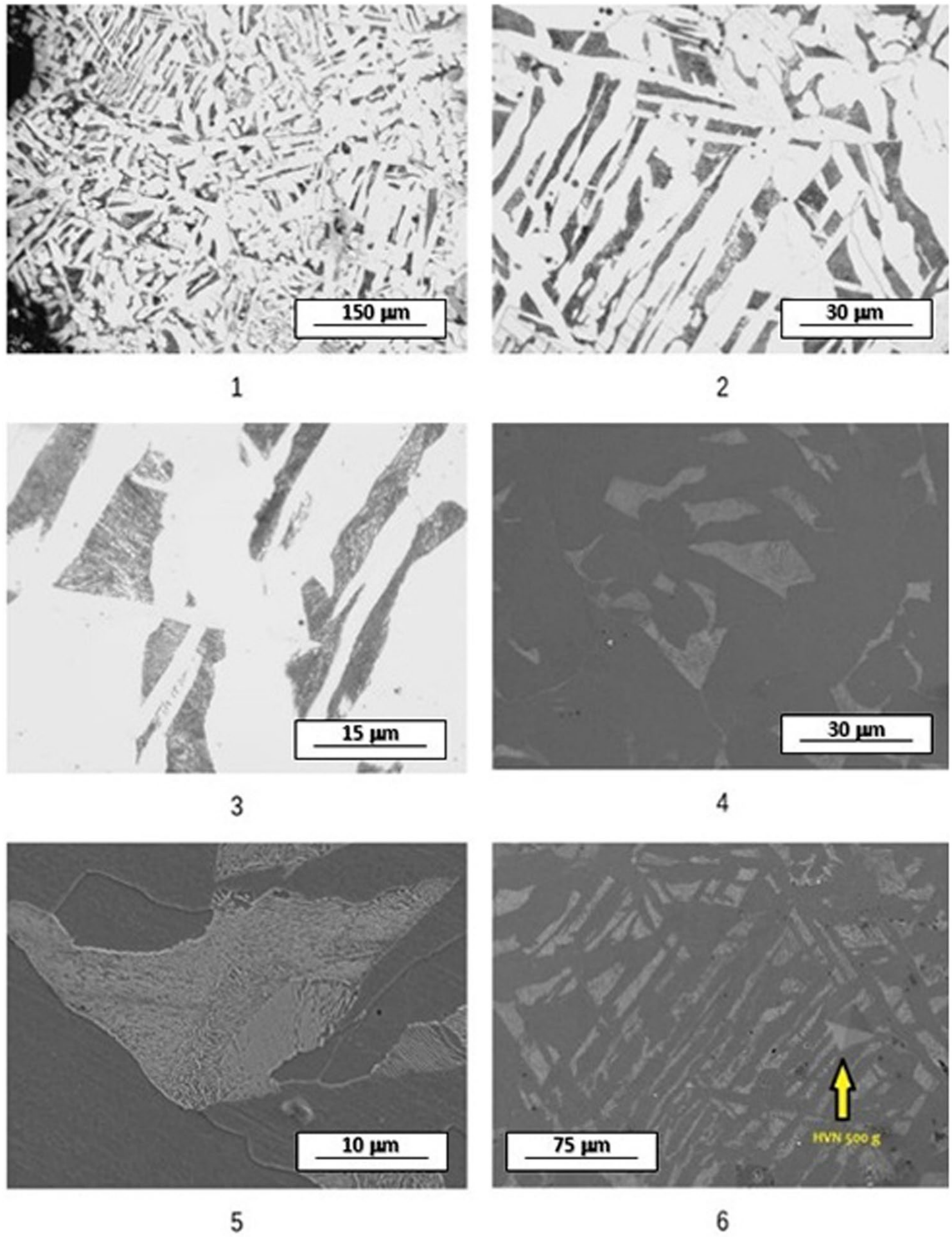

Fig. 11 (1) General appearance of the ferritic–pearlitic microstructure of the piece (×100). (2) and (3) Details of the microstructure showing the elongation of the ferritic grain and its imbrication with pearlite (×500) and (×1000). (4) Pearlite distribution in the ferritic matrix (×500). (5) Detail (SEM) of the pearlite structure and its fineness (×1500). (6) Image showing a Vickers hardness imprint (×200) (Photographs: J. Setién)

process, which was not too intense in any case, the piece slowly cooled down to room temperature.

The distribution of pearlite in the ferritic matrix (×500) is shown in Fig. 11(4), and Fig. 11(5) shows a detail (×1500) of a pearlitic grain with its characteristic banded structure. It is a fine pearlite, with good quality and resistance. This eutectoid component consists in alternate bands of ferrite and cementite (iron carbide, CFe_3) in a theoretical proportion of 86.5%–13.5%. Considering that the carbon content in the ferrite can be considered negligible, and that cementite contains a 6.67% of this element, it is possible to estimate the carbon content of the alloy indirectly by measuring the proportion of pearlite it contains. Classical quantitative metallography techniques based on planimetric methods allow us to establish a 26.2% of pearlite for this piece compared to the remaining 73.8% ferrite. The total ferrite content in the piece would then be of $73.8\% + (0.262 \times 86.5\%) = 96.463\%$, while the cementite content would be its complement up to 100% ($0.262 \times 13.5\% = 3.537\%$). Therefore, the carbon content of this piece will be estimated considering that practically all the carbon, as already mentioned, is in the form of cementite, while the carbon dissolved in the ferrite is negligible. Consequently, there will be a percentage of $0.0667 \times 3.537\% = 0.236\%$ of this element, which is a value that is certainly acceptable for an alloy without excessive mechanical resistance claims.

To finish, the ferritic phase has shown on average a microhardness value of 114.4 HVN (applied load of 100 g), with an estimation of 269.6 HVN for pearlite (applied load of 50 g). The global microhardness of the material, assessed under applied loads of 500 g (Fig. 11(6), × 200), is on average 148.7 HVN, which is a completely normal value for an alloy with these microstructural and compositional characteristics (%Si = 0.136 and %P = 0.075).

In conclusion, it has been observed that there is a certain microstructural variety in the iron pieces, which in turn suggests a certain degree of selectivity that would vindicate the high qualification of the craftsmen, with a production that optimizes the treatments according to the needs of each object that comes out of their forges [26].

Bronze and Copper Pieces

A total of 23 objects made of some type of copper-based alloy have been registered. Typologically, they are all small objects except for one of them, with a maximum dimension of 85 mm, which could correspond to a fragment of a weighing scale obtained by hammering from a thin sheet (approximately 1 mm thick) (Inv. 00.5.0092) [15]. The maximum dimension of the rest of the objects does not exceed 55 mm, and their functionality is, in some cases, uncertain. However, in other pieces it is possible to identify pieces of weapon sheaths, rivets or fragments of rivets, riveted sheets, plaques, disks, or a fragment of a cauldron handle, as mentioned in archeological section.

Analytical Methodology

The same as with the iron pieces, scanning electron microscopy (SEM) techniques with an energy-dispersive X-ray electron microprobe (EDX) have been used for the analytical characterization of these objects, and the working conditions have been similar to those used for the iron-based objects. However, due to their specific characteristics, this technique only provides compositional information about the surface of the sample. Therefore, given the effect that the corrosion patinas can have on the results of this analytical route, attempts have been made to take samples in clean areas in which the base metal may have become exposed. Despite this precaution, those analyses that have provided abnormal contents of certain elements have been discarded. In this sense, it is well known that a certain overestimation of the tin and lead contents can happen in alloys which are rich in such metallic elements. For instance, the analysis of the sample 11.22.091 provided abnormally high contents of tin (45–60%) and lead (10–20%) being too low the copper contents (approximately 20%), and consequently, these results were discarded for further discussion. In the same way, the detection of high contents of iron, sodium, phosphorus, sulfur, or chlorine is indicative of the presence of typical corrosion products.

On the other hand, identical protocols to those already described for the ferrous-based pieces have been followed for the metallographic analyses, with the exception that in this case the etching to reveal the microstructure has been performed with a ferric chloride-based reagent.

Results Obtained (Table 4)

As with the analyses performed on the iron-based objects, the results in Table 4 for the bronzes are the average of three analytical determinations carried out in three different points of each of the samples. From the chemical point of view of the alloys, there is a clear prevalence (19 pieces) of leaded bronzes (copper/tin/lead), alloys in which other associated elements, such as arsenic, antimony, iron or silicon, and to a lesser extent phosphorus and sulfur (such as the dagger scabbard clamp—00.5.0002, the rivets—00.5.0003, 00.5.0072, 00.5.0073, the sheet of riveted disks—00.5.0071, a triangular plate—00.5.0074, the fragment of a cauldron handle—00.5.0082, and the needle—03.16.130, among the materials mentioned in the previous section) appear frequently but with uneven significance. The contents of these aforementioned elements are highly variable and probably were part of the mineral gangue [27]. Only in one case has the presence of a copper/tin binary bronze (88.7% Cu–9.8% Sn, with small amounts of sulfur, silicon, and iron) been reported (it is a fragment which is impossible to identify—03.16.114, as it underwent a recasting process when it was buried under the wooden beams in a part of structure 55, in which a fire must have broken out). In addition, one of the pieces has no trace of tin, it has a 77.7% copper content, and the remaining 22.3% is lead (articulated plaque or belt buckle—11.22.048, which was also mentioned in the previous section), being the only example of a copper alloy with another element that is different from tin.

As for the tin contents, these range between 8.6% and 16.8%, with an average value of 13%. In short, these are pieces that fall inside the range of those bronzes which are classified as good quality bronzes, and are in line with those pieces found in other chronologically related archeological sites, such as the Las Ruedas necropolis [28]. Figure 12 shows the histogram of the frequency of objects depending on the distribution of tin content in the pieces analyzed. The results obtained are characterized by the high frequency of bronzes rich in tin, although the small number of pieces analyzed does not allow statistically significant conclusions to be drawn. In this regard, it should also be remembered that the successive recycling processes of bronze pieces can cause a gradual depletion of the tin content of the alloys. This factor should be considered in relation to the simultaneous coexistence of excellent quality bronze pieces with

Table 4 Chemical analysis of the copper and bronze pieces (wt%) (Author: J. Setién)

Inventory	Description	Cu	Sn	Fe	Pb	Sb	As	S	Si	P
00.5.0002	Dagger scabbard clamp	86.3	13.4		0.1	0.2				
00.5.0003	Rivet	83.4	11.2	0.2	3.7	0.4	1.1			
00.5.0008	Trapezoidal ornamental plate	79.2	11.8	0.1	6.2	0.4	2.3			
00.5.0010	Rivet fragment	82.7	16.6	0.2	0.2	0.2	0.1			
00.5.0071	Sheet of riveted disks	81.0	16.8	0.2	1.1	0.5	0.4			
00.5.0071R	Sheet rivets	83.8	14.3	0.2	0.7	0.5	0.5			
00.5.0072	Rivet	80.4	14.2	0.3	3.9	0.2	1.0			
00.5.0073	Rivet	78.0	14.6	1.0	4.5	0.4	1.5			
00.5.0074	Triangular plate	84.2	10.4	0.5	3.8	0.3	0.8			
00.5.0082	Fragment of a cauldron handle	88.8	9.8	0.2	0.8	0.3	0.1			
00.5.0084	Ornamental disk	80.2	14.4	0.1	4.2	0.2	0.9			
00.5.0092	Fragment of a weighing scale	97.9	11.8	0.3						
03.16.015	Possible scabbard decoration	86.9	11.6	0.1	0.7		0.5		0.2	
03.16.114	Unknown fragment	88.7	9.8	0.2				1.0	0.3	
03.16.130	Needle	82.9	12.0		2.7	0.8	0.3	0.2	1.1	
03.16.177	Ornament	85.0	14.1		0.5		0.3	0.1		
07.30.079	Hemispherical rivet	81.9	13.5		3.9	0.6		0.1		
11.22.007	Rivet	76.7	14.1	3.3	2.5	0.2	0.6	0.2	2.4	
11.22.010	Rivet (body area)	76.3	15.0	0.3	5.8	0.5	0.6	1.3	0.2	
11.22.010	Rivet (rim)	84.3	12.4	0.2	2.1	0.5	0.3		0.2	
11.22.048	Articulated plaque or belt buckle	77.7			22.3					
11.22.072	Rivet	89.5	8.7	0.3	0.4	0.2		0.2	0.7	
11.22.075	Necklace bead	90.2	9.8							
11.22.091	Articulated plaque or belt buckle	24.0	50.7	6.7	15.2					3.4

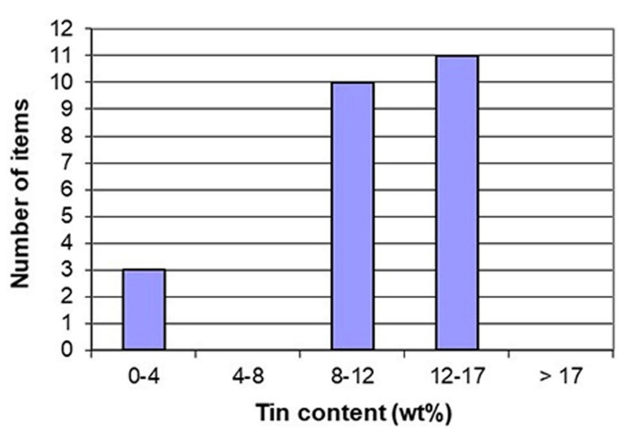

Fig. 12 Histogram of the frequency of objects depending on the tin content (Author: J. Setién)

other pieces that show a poorer mechanical performance as they are made from alloys that are significantly poorer in tin.

Regarding the more or less significant presence of the third alloying element, lead, it should be remembered that leaded alloys began to be more frequent in Western Europe late in the Middle Bronze Age, although the amount of lead, which initially was small, only becomes prominent in the Iberian Peninsula well into the Late Bronze Age, after 900 BC [29]. It is not uncommon to find leaded bronzes in the archeological record with a lead content that sometimes is higher than that of tin, and can even reach relative values higher than 20% by weight [28].

A total of 12 of the 19 leaded bronze pieces analyzed contain lead concentrations greater than 1% by weight in their composition. The maximum percentage found in one piece is that of an ornamental plate, and it rises to 6.2% (trapezoidal sheet 00.5.0008). Another piece with a lead content that exceeded 22% but did not contain tin was excluded from this analysis, as it cannot be considered as a leaded bronze. The average lead content for all the pieces analyzed is 2.51%. The heterogeneity in the values of lead concentrations is typical of pieces containing a high content of this element, and it is justified based on the tendency of this heavy metal to emulsify when it is mixed with other metals as a result of its characteristic insolubility, in both liquid and solid states.

It is interesting to analyze the results obtained for the lead and arsenic contents jointly, as it is observed that both are related in such a way that there is a clear tendency that the higher the lead content, the higher the arsenic content and

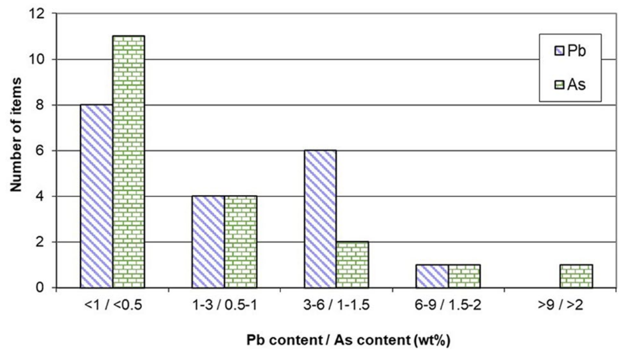

Fig. 13 Histogram of the frequency of objects depending on the lead and arsenic content (Author: J. Setién)

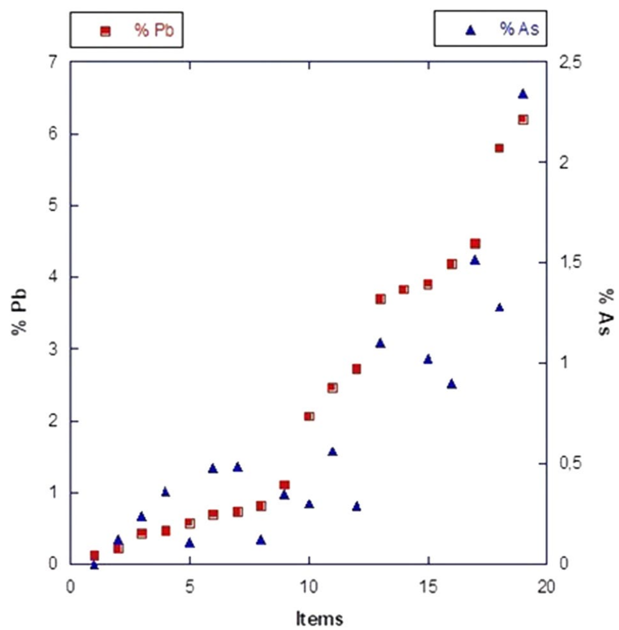

Fig. 14 Correlation between lead and arsenic content for the studied pieces (Author: J. Setién)

vice versa (Fig. 13). This seems to indicate that both metals are found together in the ore deposits and are incorporated into the alloys during the metal reduction process in the furnaces [30].

In this sense, it should be remembered that for many centuries bronze was obtained by direct reduction in the smelting furnace, jointly processing copper minerals (malachite or azurite), tin (cassiterite), and occasionally the intentional alloying with lead to improve the casting ability of the metal [31, 32]. The ideal working conditions would require reaching high temperatures (1000–1100 °C), highly reducing environments, and a high fuel consumption per load for the complete reduction of the ore to metallic tin. Considering how minerals behave in the furnace, the non-fulfillment of any of these strict conditions would lead to their partial reduction, thus justifying the coexistence of coppers, poor bronzes, good quality binary bronzes and slightly leaded ternary bronzes [29].

Figure 14 shows precisely the existing correlation between the lead and arsenic content values. This correlation could indicate the same source of mineralogical raw material for most of the analyzed pieces, although this is a risky hypothesis and it is difficult to prove based on the currently available data.

To finish, it should also be noted that those pieces that contain lead and arsenic generally also have significant concentrations of antimony (17 pieces; some of them are: the dagger scabbard clamp—00.5.0002, the rivets—00.5.0003, 00.5.0072, 00.5.0073, the sheet of riveted disks—00.5.0071, a triangular plate—00.5.0074, the fragment of a cauldron handle—00.5.0082, and the needle—03.16.130, among the materials mentioned in the previous section), although this element does not correlate with lead as clearly as it does with arsenic. In any case, this metal also appears as a common impurity of copper as a result of the polymetallic associations in the ore deposits. Most of this element is usually lost in the fumes during the process of reducing the ore to metal and in refining or re-smelting, so high contents of this element are not usual, although the opposite is not surprising either [29]. In our sample population, the maximum content of antimony is 0.80% (needle—03.16.130), with an average concentration for all the pieces of 0.37%.

Metallographic analysis

As was done with the collection of iron pieces, some selective metallographic analyses have been performed on bronze pieces. In this case, a plaque of leaded bronze (Cu: 87.8%–Sn: 11.2%–Pb: 1.0%) and a piece showing a very similar composition to this plaque, but in this case the result of a typical casting process (Cu: 87.5%–Sn: 11.0%–Pb: 1.5%) have been selected. Figure 15 shows the results of the metallographic analyses carried out on each of them.

As could be expected, the microstructure of the cast that is shown in Fig. 15(1), (3), and (5) at different magnifications (×100, ×200, and ×400, respectively) reveals the presence of dendritic growths which are typical of crystalline solidification from casting, with large dendrites that leave behind significant unfilled areas that have as a consequence the high porosity in the final material. The ample presence of these voids, which represent an important weakening in the mechanical resistance as they are discontinuities within the core of the material, may be a consequence of degassing problems in the mold that reveal a certain degree of primitiveness in the bronze casting process, which is not totally controlled by the craftsman. The structure also shows a large number of oxide inclusions and a reduced number of

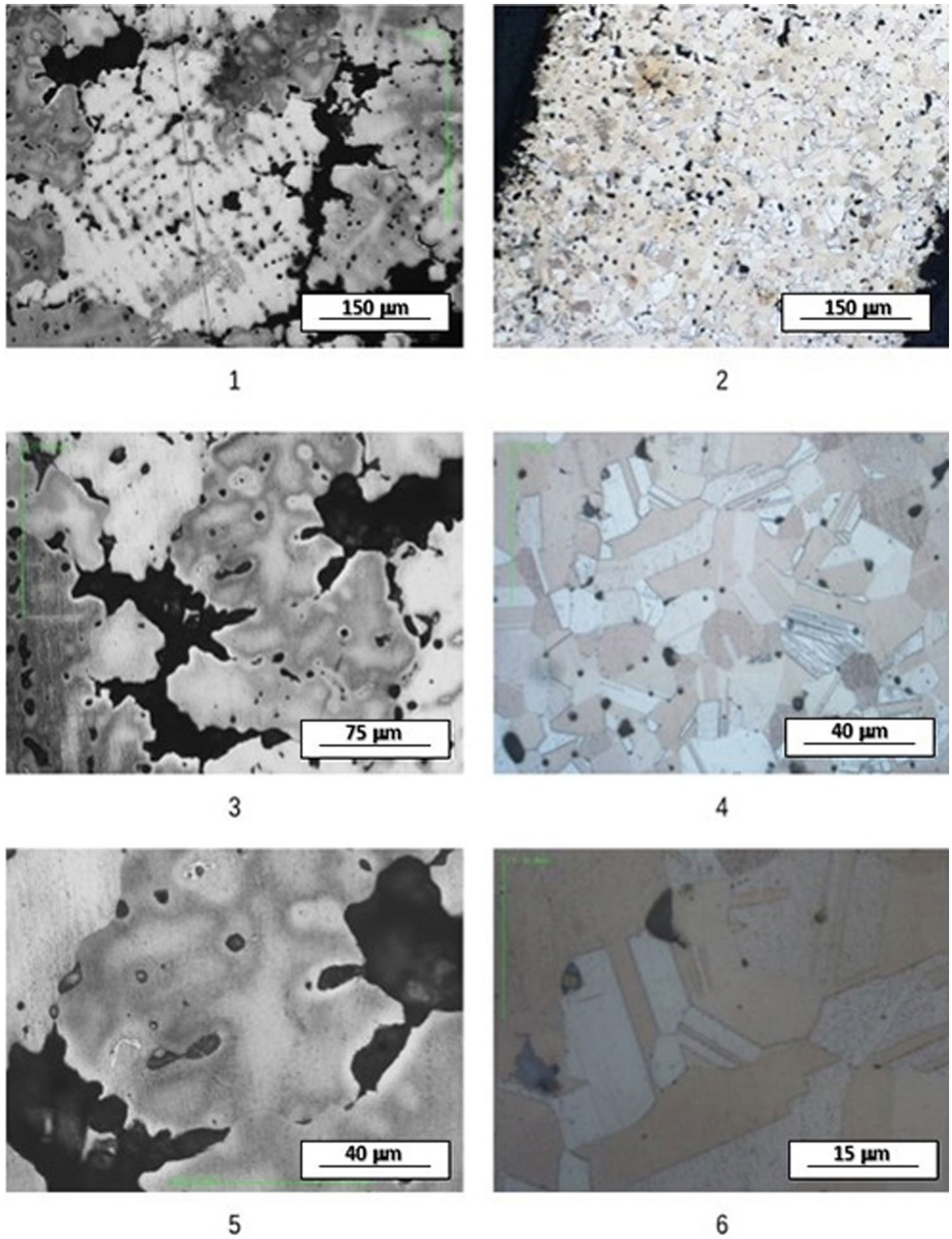

Fig. 15 (1) General appearance (× 100) and 3 and 5 details (× 200) and (× 400), respectively, of the microstructure of the cast piece showing dendritic crystal growths. (2) General appearance (× 100) and 4 and 6 details (× 400) and (× 1000), respectively, of the microstructure of the leaded bronze sheet, revealing a typical microstructure of highly twinned equiaxed grains. In both microstructures, the high internal porosity within the material can be seen (Photograph: J. Setién)

dispersed lead globules, in line with the low lead content of alloys. The porosity and the oxide inclusions suggest that the temperature of the crucible may have been too high and that an open mold was possibly used in the casting [33].

The same happens with the microstructure of the plaque that is shown in Fig. 15(2), (4), and (6) at different magnifications (× 100, × 400 and × 1000), also showing a high porosity. In this case, the microstructure is typical of quasi-equiaxed grains and they are strongly twinned. It is possibly a piece which was briefly annealed and worked on by intense hammering, as can be gathered from the reported recrystallization structure which is characterized by the presence of very small and twinned grains. This structure is consistent with other structures reported by other authors for pieces from the same period [33].

Unique Objects: Precious Metals

As mentioned above, two unique pieces made of noble metals were found at the excavation site: a goldsmithing piece, shaped as a molded gold thread, and a silver denarius. Both objects have been described in detail in the archeological section and are shown in Fig. 8. On the other hand, Fig. 16 shows two details of their appearance under the electron microscope, Fig. 16(1) and (2), and their characteristic EDX spectra, Fig. 16(3) and (4), respectively.

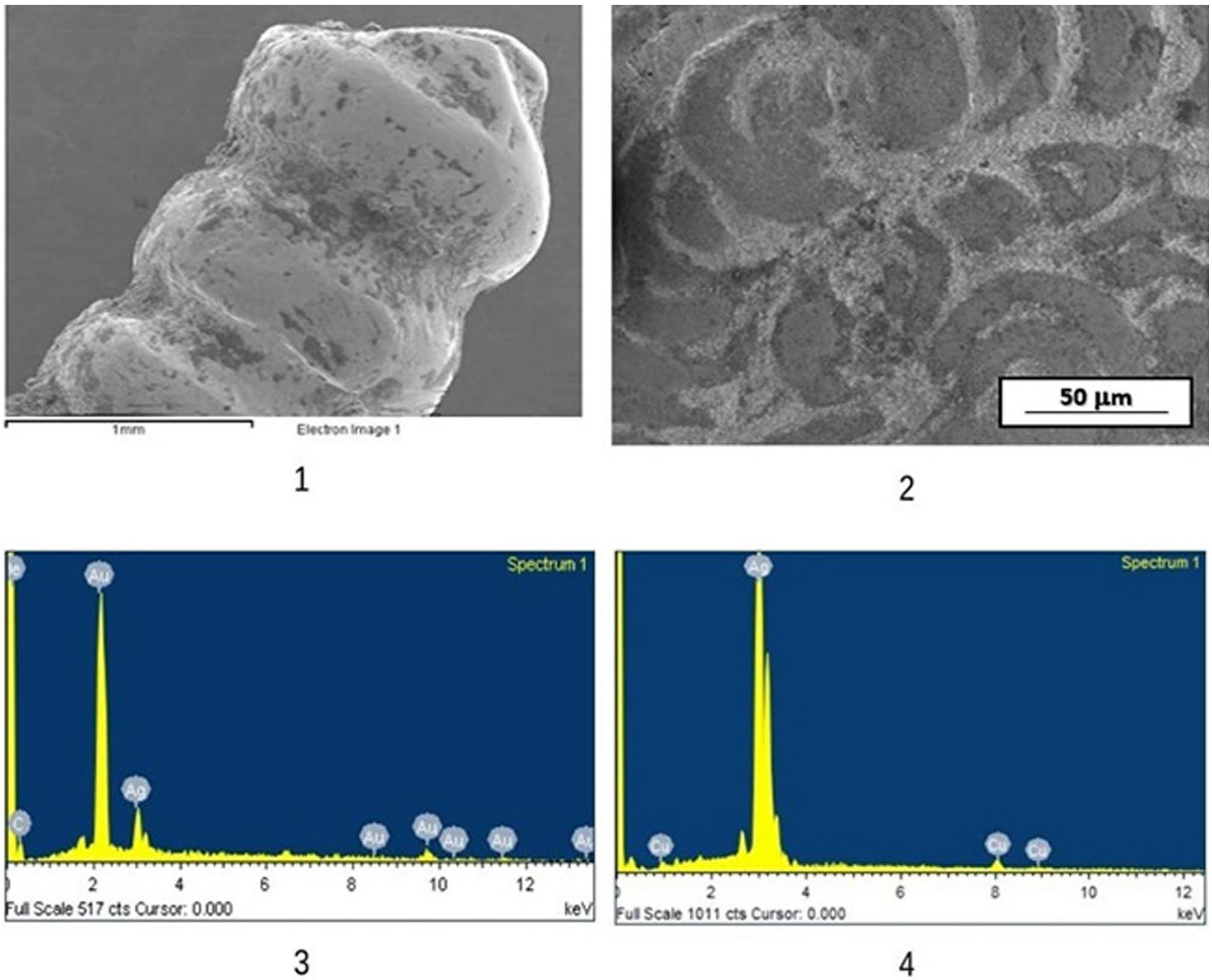

Fig. 16 (1) Detail of the gold thread molding work. (2) Detail of the mint on the obverse of the denarius. (3) EDX spectrum of the analysis carried out on the gold thread, revealing that it is a gold/silver alloy. (4) EDX spectrum of the analysis carried out on the silver denarius (Photograph: J. Setién)

The analyses carried out on the gold thread reveal that the constituent material is actually a binary gold/silver alloy (78.2% Au–21.8% Ag) both in the area of the main body of the thread and in that of the welding that can be seen at one of its ends, although in this union point, possibly to another object, the alloy has a higher gold content (81.9% Au–18.1% Ag). The use of a filler material, usually called welding alloy, with similar compositional characteristics to those of the base metal indicates that it is a strong brazing process [34]. In no case is the presence of copper as the third alloying element reported, although the use of these ternary alloys in pre-Roman goldsmithing work in the Iberian Peninsula was quite frequent and is well documented [34, 35]. In relation to the silver content, the results obtained are comparable to those of other thread fragments analyzed and attributed chronologically to Periods II and III in the "Au Project" of the Iberian Peninsula [34], in which, in general, an important and significant increase in the amount of silver present in all the pieces can be seen throughout the chronological framework, which also comes with a progressive reduction in the amount of gold employed in the alloy.

On the other hand, the analyses carried out on the denarius show an average silver content of 97.1%, while the remaining 2.9% is copper. As mentioned in archeological section, this coin can be assigned to Group V of the classification established by Gonzalbes [21], although most of the pieces in this group show silver contents in the range of 85%—95%, and therefore, this piece would be slightly above the upper limit.

Discussion

A great part of the iron tools is related to woodworking—nails, kitchen work, and textile crafts—scissors, knives, and bi-pointed punches, this last one being related to the exploitation of livestock [36]. The items found at the site could have been used for these functions regardless of their size, as has been observed in the case of knives at other sites such as Numancia, as we have already mentioned. The specimens found in La Ulaña range from 156 mm (00.5.0004) to 110 mm long (05.25.029), both found in domestic contexts, and their use is reflected, at least, in the cut marks found mainly in some bovine, sheep, and goat bones, although incisions corresponding to the removal of the flesh have also been found in a horse jaw (03.16.056), which would indicate that this animal was eaten by the inhabitants of the settlement, although it cannot be asserted that it was a systematic consumption, since we only have this remain [36]. Some weapons have also been documented, including the two ferrules (03.16.123, 07.30.035). Likewise, the iron tools must be related to the slag found in the excavation [15], which indicates the existence of a metallurgy based on the abundance of this mineral in the north of Spain and the presence of small furnaces in the settlement itself [27], and that mainly used the technology of forging. Although they have not been thoroughly analyzed in this paper, iron slags are rich in ferric silicates (mainly fayalite and kirstenite, which are not seen in modern iron and steel slag analyses) but also in iron oxides (wüstite and maghemite). This datum is an indicator that the technique used to reduce the iron ores in the furnace, producing this type of slag as a result, corresponds to an iron process which has not been yet perfected. Indeed, although the formation of fayalitic slags was obtained, the furnace itself was not quite adequate to reduce all the iron present in the ore, hence producing slags which were very rich in iron oxides as a result.

On the other hand, metallographic analyses are evidence of the technological mastery of certain isothermal transformation treatments that give rise to the formation of bainitic structures, as well as the normal use of the annealing treatments for recrystallization and recovery of the metal microstructure that has been strongly altered in the forging processes.

To finish, it is worth mentioning the remarkable finding of a piece of tin-plated iron (07.30.008), which although it is a technique that was used in antiquity, it is more commonly used with copper or bronze pieces [37].

The bronze pieces found in La Ulaña only document the craftmanship activities linked to foundry, hammering, and riveting among the techniques which are typical of this craft in the workshops of origin. The fact that most of these pieces are incomplete makes their interpretation difficult, and it is not possible to offer a precise dating beyond placing them in the Second Iron Age based on some established parallels. These pieces lead us to consider that the workshops that produced these alloys worked with an already manufactured raw material, producing goods which were widespread. Based on our current knowledge, it is not possible to locate any of these workshops at the site as the excavations have not permitted us to document remains of bronze processing. However, this possibility cannot be ruled out either, since the production of the metal artifacts found at the site could be an indicator of the existence of a local craftsmanship that could have used the same workshop for both activities, as has been documented in other sites in northern Spain [27], although each technique must have required its own specialists.

The analyses carried out on these pieces show that, in general terms, they are good quality bronzes, with a 13% average tin content and ternary composition in which lead is a frequent alloying element, although its relative presence is very variable. However, there is an acceptable correlation between the lead and arsenic content of the bronzes, which suggests that both metals would have originally been found in the ore deposits and were assimilated into the alloys during the metal reduction process in the ovens. Although the associated presence of antimony is also frequent in these cases, the contents of this element do not show such a correlation with lead as clear as that of arsenic.

Metallographic analyses confirm that the annealing treatment as a means of recovering the structure and mechanical properties of copper which had been severely cold-worked and which consequently was highly embrittled was also fully established in copper metallurgy.

Regarding the gold thread, of the usual types found in pre-Roman goldsmithing (smooth, twisted, and molded), the latter is frequent in the Second Iron Age in the North Plateau, although it can also be found in orientalizing and in Roman goldsmithing. It is not uncommon that the three types of thread can be found in the different elements that make up a jewel. The manufacturing of molded threads is relatively simple and can be made from a thicker bar or thread of gold hammering it on an anvil. Another technique is by means of a matrix with the desired shape or even by rolling a smooth thread between a smooth surface and a cutting tool that gradually deforms the section of the thread [38]. On their own, these types of threads have no chronological value and much less do they have an ethnic or cultural value [19].

From the archeometric point of view, it should be noted that binary gold/silver alloys have been used both in the main body of the piece and in the filler material of the welding area but there is no trace of copper as the third alloying element, although the use of these ternary alloys was quite widespread in pre-Roman goldsmith work in the Iberian Peninsula, and there are well-documented examples in nearby geographical areas [39].

The denarii from the *Turiaso* mint spread mainly to the west of the mint, this is, in the area of the Cantabrian coast—south of the Pyrenees, the upper Ebro valley and north of the Duero River, although their distribution also reached other areas of Spain as treasures or isolated findings. Thus, sporadically, as in our case, denarii have been found in other sites which belong to the territory of ancient Cantabria: Soto-Iruz (Puente Viesgo, Cantabria), Celada Marlantes (Cantabria), Monte Cildá (Olleros de Pisuerga, Palencia), and, perhaps, Quintanalacuesta (Burgos) [21] (Fig. 4(9)–(12)).

Regarding the analytical results, the purity of this piece is quite remarkable, with a silver content slightly above 97% (while the rest is exclusively copper), which places it above the upper limit of purity (95% silver) of the group of coins from the same mint to which it is ascribed based on its chronological and minting characteristics [21]. This purity is compatible with that of the republican Roman denarii of the second–first centuries BC, which did not change and was around 97% [40]. On the other hand, the fact that copper is the only metal with a significative presence in the analyses carried out, complementing the lack of silver, goes in line with what was reported for all the Group V pieces analyzed by different authors. It even seems that it was intentionally mixed and that this action was not done correctly, producing a non-homogeneous metal as verified in the analyses which show different amounts of silver when analyzing different points of the same piece [41].

In the current state of our research, everything indicates that the small number of artifacts found is the direct effect of the set of taphonomic alterations that the deposit has suffered. A little more than 1000 m^2 have been dug up, finding evidence of fire in only about 30 m^2, to be precise, in a structure of dwelling 1, actually in the area of the hearth, and in a part of structure 55. There are no other signs of fire in the remaining dug up areas. That is to say, the traces of fire are very localized and not widespread. No level of destruction has been found either. Therefore, it is possible to hypothesize that the *oppidum* was intentionally abandoned, although this does not mean it was done voluntarily, and that the fires could have different causes, as accidents of this type must have been very common at the time as a consequence of the materials used in the construction, among other reasons. But quite probably this was not the cause that made its inhabitants leave. Without doubt the reason has to do with the Roman presence in the area, although it must not be understood exclusively from the military perspective of the Cantabrian wars as this Roman presence dates back more than a century before these events. In times of conflict, the inhabitants of the *oppidum* had to know that its extension (285 hectares) was a guarantee of security against equal forces, but that it was of little use against an army like the Roman one, which they undoubtedly saw maneuvering on the Burgalese moors, since they set up their camp in Sasamón (Burgos) (Fig. 4(13)) from where the conquest of this central sector of northern Spain began. It seems more logical that in the case of wanting to face an army like the Roman army they would have looked for a more favorable environment, such as the one provided by the mountainous areas of the Cantabrian Mountains in which a traditional army has more problems to maneuver and the population can protect itself in more abrupt places [42]. This is the reason why we think that the settlers either abandoned the *oppidum* before the Roman military advance took place and took refuge in more inaccessible and easier to defend areas, or they reached some type of agreement with the invader, which implied the abandonment of the place where they lived, since no occupation attributed to these military events has been documented. In either case, this means that the inhabitants were able to take their most essential and appreciated belongings, discarding broken objects and losing some on their way. This is the reason why the archeological record is so scarce and fractured.

Acknowledgments The archeological works carried out at the site were all authorized by the Junta de Castilla y León (Spain) and financed by this institution between 2001 and 2011, by Adecco-Camino, between 1998 and 2000, and by the Humada City Council (Burgos, Spain) between 1997 and 2000 and in 2003.

Funding Open Access funding provided thanks to the CRUE-CSIC agreement with Springer Nature.

References

1. M. Cisneros, La Ulaña (Prov. Burgos). Ein Oppidum der Jüngeren Eisenzeit im Norden der Iberischen Halbinsel (La Ulaña (Prov. Burgos). An Oppidum of the Second Iron Age in the Northern Iberian Peninsula). Madr. Mitt. **49**, 209–228 (2008). **(in German)**
2. M. Cisneros, P. López Noriega, El sistema defensivo del castro de La Ulaña (Humada, Burgos). (The defensive system of La Ulaña hillfort (Humada, Burgos). Arch. Esp. Arqueol. **77**, 3–22 (2004). **(in Spanish)**
3. S. Fichtl, *La ville celtique. Les oppida de 150 av. J.-C. à 15 ap. J.-C. (The Celtic city. The oppida from 150 BC to 15 AD)* (Éditions Errance, Paris, 2000) **(in French)**

4. T. Knopf, M. Leicht, S. Sievers, Die grossen süddeutschen Oppida Heidengraben, Manching und Kelheim. (The large South German oppida of Heidengraben, Manching and Kelheim), in *Les Processus d'urbanisation à l'âge du Fer. Eisenzeitliche Urbanisationsprozesse*. ed. by V. Guichard, S. Sievers, O.H. Urban (Centre archéologique européen du Mont Beuvray, Glux-en-Glenne, 2000), pp.141–147. **(in German)**
5. I. Martínez Casas, M. Cisneros, J.M. Sánchez Espeso, Aplicación de los SIG al análisis microespacial del yacimiento arqueológico de La Ulaña (Humada, Burgos). (Application of GIS to the microspatial analysis of the archaeological site of La Ulaña (Humada, Burgos), in *Técnicas de Información Geográfica y Análisis Arqueológico del Territorio. Actas del V Simposio Internacional de Arqueología de Mérida*. ed. by V. Mayoral, S. Celestino (Instituto de Arqueología-CSIC, Mérida, 2011), pp.809–818. **(in Spanish)**
6. J.P. Mohen, *L'Age du fer en Aquitaine (The Iron Age in Aquitaine)* (Centre National de la Recherche Scientifique, Paris, 1980) **(in French)**
7. J. Quintana, *El castro de Peña Amaya (Amaya, Burgos): del nacimiento de Cantabria al de Castilla (The hillfort of Peña Amaya (Amaya, Burgos): from the birth of Cantabria to the birth of Castile)* (Instituto de Prehistoria y Arqueología Sautuola, Santander, 2017) **(in Spanish)**
8. J.F. Torres-Martínez, M. Fernández-Götz, A. Martínez-Velasco, D. Vacas-Madrid, E. Rodríguez-Millán, From the Bronze age to the roman conquest: the oppidum of Monte Bernorio (Northern Spain). Proc. Prehist. Soc. **82**, 363–382 (2016)
9. J.F. Torres-Martínez, M. Fernández-Götz, S. Domínguez-Solera, A. Martínez-Velasco, D. Vacas-Madrid, M. Serna-Gancedo, G. Cabanillas de la Torre, M. Galeano, R. Fernandes, "Invisible burials" and fragmentation practices in the iron age Europe: excavations at the Monte Bernorio necrópolis (Northern Spain). J. Field Archaeol. **46**(6), 399–413 (2021). https://doi.org/10.1080/00934690.2021.1924435
10. A. Jimeno, J.I. de la Torre, R. Berzosa, J.P. Martínez Naranjo, *La Necrópolis Celtibérica de Numancia (The Celtiberian Necropolis of Numantia)* (Consejería de Cultura y Turismo de la Junta de Castilla y León, Valladolid, 2004) **(in Spanish)**
11. A. Castiella, *La Edad del Hierro en Navarra y en Rioja (The Iron Age in Navarre and Rioja)* (Diputación Foral de Navarra, Institución Príncipe de Viana, Consejo Superior de Investigaciones Científicas, Pamplona, 1977) **(in Spanish)**
12. Sanz, C. : Los vacceos : cultura y ritos funerarios de un pueblo prerromano del valle medio del Duero. La necrópolis de Las Ruedas, Padilla de Duero (Valladolid) (The Vacceans: culture and funerary rites of a pre-Roman people from the middle Duero valley. The necropolis of Las Ruedas, Padilla de Duero (Valladolid). Consejería de Educación y Cultura de la Junta de Castilla y León-Ayuntamiento de Peñafiel, Valladolid (1997) (**in Spanish**).
13. A. Lorrio, *Los Celtíberos (The Celtiberians)* (Editorial Complutense, Madrid, 1997) **(in Spanish)**)
14. M.A. Manrique, *Instrumentos de Hierro de Numancia (Iron Tools from Numantia)* (Ministerio de Educación, Cultura y Deporte, Madrid, 1980) **(in Spanish)**
15. R. Erice, J. Setién, Los metales (Metals), in *El Castro de La Ulaña (Humada, Burgos). La Documentación Arqueológica (1997–2001)*. ed. by M. Cisneros, P. López Noriega (Universidad de Cantabria, Santander, 2005) **(in Spanish)**
16. J.D. Sacristán, *La Edad del Hierro en el Valle Medio del Duero. Rauda (Roa, Burgos) (The Iron Age in the Middle Douro Valley. Rauda (Roa, Burgos)* (Universidad de Valladolid, Valladolid, 1986) **(in Spanish)**
17. J.L. Maya, F. Cuesta, Excavaciones arqueológicas y estudio de los materiales de La Campa Torres (Archaeological excavations and study of materials from La Campa Torres), in *El Castro de La Campa Torres, Período Prerromano*. ed. by J.L. Maya, F. Cuesta (Ayuntamiento de Gijón, Gijón, 2001), pp.11–277. **(in Spanish)**
18. J.L. Maya, *La Cultura Material de los Castros Asturianos (The Material Culture of the Asturian Hillforts)* (Universitat Autònoma de Barcelona, Barcelona, 1988) **(in Spanish)**
19. G. Delibes, A. Esparza, R. Martín Valls, C. Sanz, Tesoros celtibéricos de Padilla de Duero (Celtiberian treasures from Padilla de Duero), in *Arqueología Vaccea Estudios Sobre el Mundo Prerromano en la Cuenca Media del Duero*. ed. by F. Romero, C. Sanz, Z. Escudero (Consejería de Cultura y Turismo de la Junta de Castilla y León, Valladolid, 1993), pp.397–470. **(in Spanish)**
20. F. Fernández Gómez, *Excavaciones Arqueológicas en El Raso de Candeleda (Archaeological Excavations at El Raso in Candeleda)* (Institución Gran Duque de Alba Diputación Provincial de Ávila, Ávila, 1986) **(in Spanish)**
21. M. Gonzalbes, *La Ceca de Turiazu. Monedas Celtibéricas en la Hispania Republicana (The Mint of Turiazu. Celtiberian Coins in Republican Hispania)* (Museo de Prehistoria de Valencia-Diputación de Valencia, Valencia, 2009) **(in Spanish)**
22. E. Ottenwelter, J. Ded, J. Hosek, Experimental tinning of iron, an attempt to identify techniques of tinning used by ancient craftsmen, in *Eksperymental'na Archeolohija: Zavdannja, Metody, Modeljuvannja/Експериментальна археологія: завдання, методи, моделювання*. ed. by O.P. Mocja (Kiev, Lira-K, 2011), pp.204–217
23. J. Lang, Roman Iron and Steel: A Review. Mater. Manuf. Process. **32**(7-8: Ancient Metallurgy), 857–866 (2017)
24. J. Simón, J. Tartera, M. Marsal, J. Auladell, De los íberos al imperio romano. Evolución tecnológica del hierro (From the Iberians to the Roman Empire. Technological evolution of iron). Rev. Metal., Vol. Extraordin, 507–512 (2005) (**in Spanish**).
25. M. Urteaga, El hierro en época romana. Tradición e innovación en los territorios vascos (Iron in Roman times. Tradition and innovation in the Basque territories), in *Historia del Hierro en Bizkaia y su Entorno*. ed. by X. Orue-Etxebarria, M.E. Apellaniz, P.P. Gil-Crespo (Servicio Editorial de la Universidad del País Vasco, Bilbao, 2016), pp.77–118. **(in Spanish)**
26. J. Barrio, A. Criado, J.A. Martínez, L. Cancho, L.M. Rodríguez, Estudio arqueometalúrgico de objetos de hierro de la necrópolis celtibérica de Numancia: aproximación a la siderurgia celtibérica (Archaeometallurgical study of iron objects from the Celtiberian necropolis of Numantia: an approach to Celtiberian iron and steel production), in *la Necrópolis Celtibérica de Numancia*. ed. by A. Jimeno, J.I. de la Torre, R. Berzosa, J.P. Martínez (Consejería de Cultura y Turismo de la Junta de Castilla y León, Valladolid, 2004), pp.460–465. **(in Spanish)**
27. S. Rovira, P. Gómez Ramos, La metalurgia prerromana de La Campa Torres (Gijón, Asturias) (The pre-Roman metallurgy of La Campa Torres (Gijón, Asturias), in *El Castro de La Campa Torres*. ed. by J.L. Maya, F. Cuesta (Período Prerromano. Ayuntamiento de Gijón, Gijón, 2001), pp. 375–384. (**in Spanish**)
28. S. Rovira, Apéndice C. Análisis de los metales de Las Ruedas por espectrometría de fluorescencia de rayos–X (Appendix C. Analysis of the metals from Las Ruedas by X-ray fluorescence spectrometry), in: *Los vacceos : cultura y ritos funerarios de un pueblo prerromano del valle medio del Duero. La necrópolis de Las Ruedas, Padilla de Duero (Valladolid)*, Ed. Sanz, C. (Consejería de Educación y Cultura de la Junta de Castilla y León-Ayuntamiento de Peñafiel, Valladolid 1997), pp. 541–546 (**in Spanish**).
29. S. Rovira, Estudio Arqueometalúrgico. Capítulo 5. La evolución de las aleaciones y la metalurgia de taller (Archaeometallurgical study. Chapter 5. The evolution of alloys and workshop metallurgy.), in *Metalurgia de la Edad del Bronce en el piedemonte meridional de la Cordillera Cantábrica–La Colección Fontaneda,* eds. Delibes, G., Fernández Manzano, J., Fontaneda, E., Rovira,

S. (Consejería de Educación y Cultura de la Junta de Castilla y León, Zamora, 1999), pp. 123–149 (**in Spanish**).
30. P. Gómez Ramos, Hornos de reducción de cobre y bronce en la pre y protohistoria de la Península Ibérica Copper and bronze reduction furnaces in the pre- and protohistoric Iberian Peninsula. Trab. Prehistori. **53**(1), 127–143 (1996). **(in Spanish)**
31. S. Timberlake, New ideas on the exploitation of copper, tin, gold and lead ores in Bronze Age Britain: the mining, smelting and movement of metal. Mater. Manuf. Processes. **32**(7–8), 709–727 (2017)
32. R.F. Tylecote, *The Prehistory of Metallurgy in the British Islands* (The Institute of Metals, London, 1986)
33. F.J. Sarabia-Herrero, J. Martín-Gil, F.J. Martín-Gil, Metallography of Ancient Bronzes: Study of Pre-Roman Metal Technology in the lberian Peninsula. Mater. Charact. **36**(4–5), 335–347 (1996)
34. A. Perea, O. García Vuelta, C. Fernández Freire, *El Proyecto Au. Estudio Arqueométrico de la Producción de Oro en la Península Ibérica (The Au Project Archaeometric Study of Gold Production in the Iberian Peninsula)* (CSIC, Madrid, 2010) **(in Spanish)**
35. I. Montero, S. Rovira, El oro y sus aleaciones en la orfebrería prerromana (Gold and its alloys in pre-Roman goldsmithing). Arch. Esp. Arqueol. **64**, 7–21 (1991). **(in Spanish)**
36. A.B. Marín Arroyo, M. Cisneros, Consideraciones económicas sobre el oppidum de La Ulaña (Humada. Burgos). La explotación ganadera (The economic framework of La Ulaña oppidum (Humada, Burgos): the livestrock exploitation). Zephyrus. **62**, 151–162 (2008). **(in Spanish)**
37. A. Giumlia-Mair, Plating and Surface Treatments on Ancient Metalwork. Adv. Archaeomater. **1**, 1–26 (2020)
38. G. Nicolini, *Techniques des ors Antiques: la Bijouterie Ibérique du VII au IVe Siècle (Ancient Gold Techniques: Iberian Jewellery from the 7th to the 4th Century)* (Picard, Paris, 1990) **(in French)**
39. O. García-Vuelta, I. Montero, A. Villa, Orfebrería castreña en el Museo Arqueológico de Asturias (Oviedo): aproximación a su caracterización arqueométrica y problemas de estudio (Castro Culture gold objects from Archaeological Museum of Asturias (Oviedo): an approach to their archaeometric characterization and to the complexities of their study). Trab. Prehist. **77**(1), 163–183 (2020). **(in Spanish)**
40. G. Sejas, Análisis físico–químicos (Physical - chemical analysis), in *La Ceca de Turiazu: Monedas celtibéricas en la Hispania republican* Gonzalbes, M. (eds.). (Museo de Prehistoria de Valencia-Diputación de Valencia, Valencia, 2009), p. 125–131 (**in Spanish**)
41. W. Hollstein (ed.), *Metallanalytische Untersuchungen an Münzen der Römischen Republik (Metal Analytical Studies on Coins of the Roman Republic)*. (Gebr. Mann Verlag, Berlin, 2000) **(in German)**
42. A. Menéndez Blanco, J. García Sánchez, J.M. Costa-García, J. Fonte, D. González-Alvárez, V. García, Following the Roman Army between the Southern Foothills of the Cantabrian Mountains and the Northern Plains of Castile and León (North of Spain): archaeological applications of remote sensing and geospatial tools. Geosciences. **10**(12), 485 (2020). https://doi.org/10.3390/geosciences10120485