



Examining cut mark residue with SEM to identify metal tool use: An experimental study

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ABSTRACT

In this manuscript, we explore the potential of studying metal residues in cut marks generated by copper and bronze knives. The method was developed in the forensic sciences for use with modern metals in order to identify microscopic particles of metal tools on bone surfaces. However, the study of residues in archaeological materials can be challenging due to the ways in which the bone remains may have been manipulated, both in the past and in more recent times. Using a scanning electron microscope (SEM), we detected microscopic fragments of bronze and copper knives along with contamination both inside and outside of the cut marks made by those knives. Copper and bronze residues were identified embedded in the bone inside the incisions and, in two cases, they left greenish stains caused by metal oxidation. In contrast, modern contamination of undetermined origin was found unattached to the bone and had a chemical composition not compatible with that of the knives. The amount of residue was influenced by the quantity of soft tissue between the bone and the knife during the butchering tasks. Bone cooking does not seem to influence the preservation of the residues. We anticipate that the approach used in this first exploratory study will emerge as a promising method for identifying the use of metal tools in archaeological bone remains.

1. Introduction

The analysis of residues in archaeological materials is a discipline that focuses on the identification of traces of a substance or an object in the archaeological record (e.g., Heron and Evershed, 1993). These studies use a wide variety of methods and techniques, including different types of microscopes like the scanning electron microscope (SEM) (Dominici et al., 2023; Monnier et al., 2012; Wang et al., 2022). Studies performed with SEM analyze the chemical composition of microscopic organic and inorganic residues recognizable on the surface of materials. The focus of this method has been the study of different contexts and raw materials in order to make inferences about the use and function of different objects during prehistory. The materials analyzed in this research tend to be lithic or metal tools (Caricola et al., 2022; Hayes et al., 2019; Hayes and Rots, 2019; Martín-Viveros and Ollé, 2020; Ollé et al., 2016; Sobolik, 1996), ceramics (Colombini et al., 2005;

Francés-Negro et al., 2021; Heron and Evershed, 1993; Mitkidou et al., 2007; Reber et al., 2019) and, to a lesser extent, bone remains (Abrams et al., 2014; Winnicka et al., 2020).

Current forensic studies have proven residue analysis as particularly useful for the identification of object traces on bone surfaces (Bai et al., 2007; Gibelli et al., 2012; Palazzo et al., 2018; Pechníková et al., 2012; Vermeij et al., 2012). Blunt and sharp force injuries produced with modern metal objects leave microscopic particles on the contact surface and are identifiable with SEM (Gibelli et al., 2012; Pechníková et al., 2012). The chemical composition of these residues makes it possible to associate them with specific objects. This method has also been employed for the detection of gunshot residues in soft tissues and bones (Amadasi et al., 2012; Berryman et al., 2010; Molina et al., 2007).

To date, metal residue analysis has never been used in the study of prehistoric skeletal remains. Despite evidence of the peri- and post-mortem manipulation of corpses during the Chalcolithic and Bronze

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Age, including funerary rites, the production of objects, violent events and/or human cannibalism (González-Rabanal et al., 2023; Jantzen et al., 2011; Marginedas et al., 2020; Pérez-Fernández et al., 2022), the role of metals in these contexts is poorly known. In fact, in these contexts, metal tools such as knives, daggers and axes are usually scarce, perhaps as a consequence of the recycling of these raw materials (Bradley, 1988; Pernicka, 2013). Currently, the only way by which the use of metal tools or weapons has been inferred is based on the morphology of the cut marks identified on the bones (Aramendi et al., 2023; Downing and Fibiger, 2017; Egeland et al., 2024; Greenfield, 1999; Maté-González et al., 2018).

In this study, we explore the method developed in the field of forensic medicine considering the limitations and problems that these analyses can have in the study of archaeological materials. The butchering process, bone cooking and past and recent manipulation are issues that must be considered. This research has two main objectives: 1) to establish whether cutting with copper and bronze knives during butchery activities leaves residues on fleshed and defleshed bones, and 2) to determine whether cooking the specimens can influence the preservation of knife residues on the bone surface.

2. Materials and methods

The bones used for the experimental butchering process came from the processing of an intact carcass of an adult deer (*Cervus elaphus*) with two metal knives. The experimental session took place at the Boumort National Game Reserve (RNC) (Lleida, Spain). All the long bones were sliced so that they would fit inside the microscope camera for analysis. In addition, an adult bovid humerus and femur sliced into rectangular plates were also used (Fig. 1). Both knives were produced using the same model, which took the daggers from the bell-shaped tombs of Humanejos as a reference (Madrid; Spain; Muñoz-Moro et al., 2019). These were hafted with wooden handles to facilitate their use during butchery (Fig. 1d,e). The knives were made from copper (Cu) and bronze (CuSn) plates acquired from AmatMet© (El Papiol, Spain). The bronze alloy had a percentage of Cu above 75% and tin (Sn) of ≈ 10 –12%. The metal plates were cut, shaped and sharpened with the cutting, grinding and polishing discs of an angle grinder until the blades were obtained. During use, the blades were sharpened with a sharpening stone (Fig. 1a, b,c). The composition of the stone was unknown, which is why mapping was also performed with SEM.

In the first experiment, the entire butchering process was completed: half a deer (left hindquarter, forequarter and left trunk) was processed with the copper knife and the other half (right hindquarter, forequarter and right trunk) with the bronze knife. The butchering process was performed by two expert butchers, each working with a different raw material. After the complete processing of the carcass, including the disarticulation of the elements, the pieces were immersed in barrels of water for two months to accelerate the decomposition process and facilitate cleaning by maceration. They were strained through a metal sieve and washed under running water. Finally, bones larger than 10 cm were cut from the portions in which we documented cut marks in order to fit inside the SEM chamber (Fig. 1g). In the second experiment, cuts were made on the >10 cm rectangular plates of a previously defleshed cow humerus and femur using the same knives (Fig. 1f). We controlled the way in which the cut marks were made: they were spaced approximately 2 cm apart, and the bone surface was touched once per cut. These bones were boiled in a metal pot for one hour. They were then washed with water and dried.

Cut marks resulting from skinning, evisceration, defleshing and disarticulation of the carcass were identified in the taphonomic analysis of the bone surface from the deer specimens. These modifications were classified as incisions, scrapes and chop marks based on their morphology, which could also be related to the way the knives were used (Blumenschine et al., 1996; Lyman, 2008; Shipman and Rose, 1983). Incisions occur when the cutting edge of the tool is applied

parallel to the long axis of the bone. Perpendicular use of the cutting edge generates scrapes mainly associated with the removal of the periosteum and adhering flesh (Domínguez-Rodrigo, 2002) and finally, the application of dynamic force with the cutting edge produces chop marks (Lyman, 2008).

The cut marks were analyzed using a scanning electron microscope (SEM) model FEI Quanta 600 equipped with an energy dispersive x-ray spectrometer (EDX-EXL II System analytical Oxford) at the Scientific and Technical Resources Service of the Rovira and Virgili University (Tarragona, Spain) and with Oxford Instruments' INCA software (v 4.01). The specimens were observed at magnifications of 35–2000 (HFOV 8514 μm –149 μm) in low vacuum (LV) mode using the back-scattered electron detector (DualBSD) and, occasionally, the large field detector (LFD). The residues were identified through the spectra generated by SEM of the microparticles on the surface of the cut mark. These spectra showed their chemical composition by percentages of the elements that compose them, allowing to differentiate between microfragments of the metal knives and contamination (Gibelli et al., 2012; Pechníková et al., 2012).

3. Results

For this study, a total of 40 cut marks were considered. Of these, 17 (42.5%) were produced by a copper knife and 23 (57.5%) by a bronze knife (Table 1). These were all incisions located in the diaphysis, near the epiphyses of the long bones and in the spine of a thoracic vertebra. Based on their location and field notes, these cuts were associated with defleshing and disarticulation tasks.

The observation of the cut mark surfaces with SEM yielded different and complementary information depending on the detector used. On the one hand, the large field detector (LFD) showed the relief and morphology of the cut marks (Fig. 2a,b,c). These modifications exhibited a V-shaped cross-section and desquamation along the entire tool contact zone. At between 70 and 240 x (HFOV 4257 μm – 1241 μm) irregular bodies were documented alongside the incisions, although we were not able to identify their composition as they had the same coloration and texture as the bone (Fig. 2a). On the other hand, the image obtained with the back-scattered electron detector (DualBSD) at 100 x (HFOV 2980 μm) showed white-colored, shiny-appearing microparticles (metallic particles) inside and outside the cut marks (Fig. 2b,f). Between 70 and 240 x (HFOV 4257 μm – 1241 μm), we observed that these residues formed clusters inside the incisions and dispersions on the outside (Fig. 3). Notably, some of these particles were embedded in the bone (Fig. 3c,d). The greatest amount of residue was documented on the bovid samples, where incisions were made with little or no soft tissue (Fig. 4). Residues found on the outside of the cut marks were considered to be the product of contamination.

The spectrum performed on the metal knives showed 90.9% copper (Cu), 8.1% carbon (C) and 1% aluminum (Al) for the copper knife and 75.3% Cu, 14.6% C, 4.9% tin (Sn), 3.6% oxygen (O) and 1.5% Al in the bronze knife (Table S1) (Fig. S1). These data contrast with the whetstone, which was found to include a wide variety of elements, such as O (33.1%), silica (Si=29.3%) and C (27.8%), as well as minor percentages of Cu (0.3), Al (2.4), iron (Fe=2.5%), sodium (Na=1.5%), calcium (Ca=1.2%), potassium (K=0.8%), chlorine (Cl=0.6%), magnesium (Mg=0.2%), chromium (Cr=0.1%), sulfur (S=0.1%) and phosphorus (P=0.1%) (Fig. S1). In addition, the chemical composition of the bone was found to contain O (30.7%), Ca (28.9%), C (23.2%), P (11.5%) and nitrogen (N=4.2%) in practically all spectra, and very low values of Al (0.1%), Na (0.1%), Mg (0.5%) and S (0.6%) (Table S1).

Only 38.5% (n=15) of the microparticles documented inside the cut marks were consistent with the knives (Table S1). However, of the 39 spectra performed, 61.5% (n=24) corresponded to contamination. Copper residues had Cu percentages of 7.7–37.6%, four of them showed very low values of Al (0.1%–0.3%) and six of them had Fe levels of between 0.8% and 5.6% (Fig. S2; Fig. S3). The bronze residues were



Fig. 1. Sharpening stone (a,b,c), bronze knife (d), copper knife (e), bovine humerus sliced into a rectangular plate (f) and deer humerus with sections in which cut marks were localized (g).

Table 1

Number of marks analyzed according to whether they were boiled or not, presence of residues and spectra of the particles analyzed with SEM.

Sample	Boiled	Copper	Bronze	Cu* residues	CuSn* residues	Spectrum residues	Spectrum contamination
Cut marks	Yes (<i>Bovidae</i>)	4 (23.5%)	10 (43.5%)	4 (100%)	10 (100%)	15 (38.5%)	24 (61.5%)
	No (<i>Cervidae</i>)	13 (76.5%)	13 (56.5%)	9 (69.2%)	9 (69.2%)		
Total		17 (42.5%)	23 (57.5%)	13 (76.5%)	19 (82.6%)	39 (100%)	
		40 (100%)	32 (80%)				

* Cu=copper, CuSn=bronze

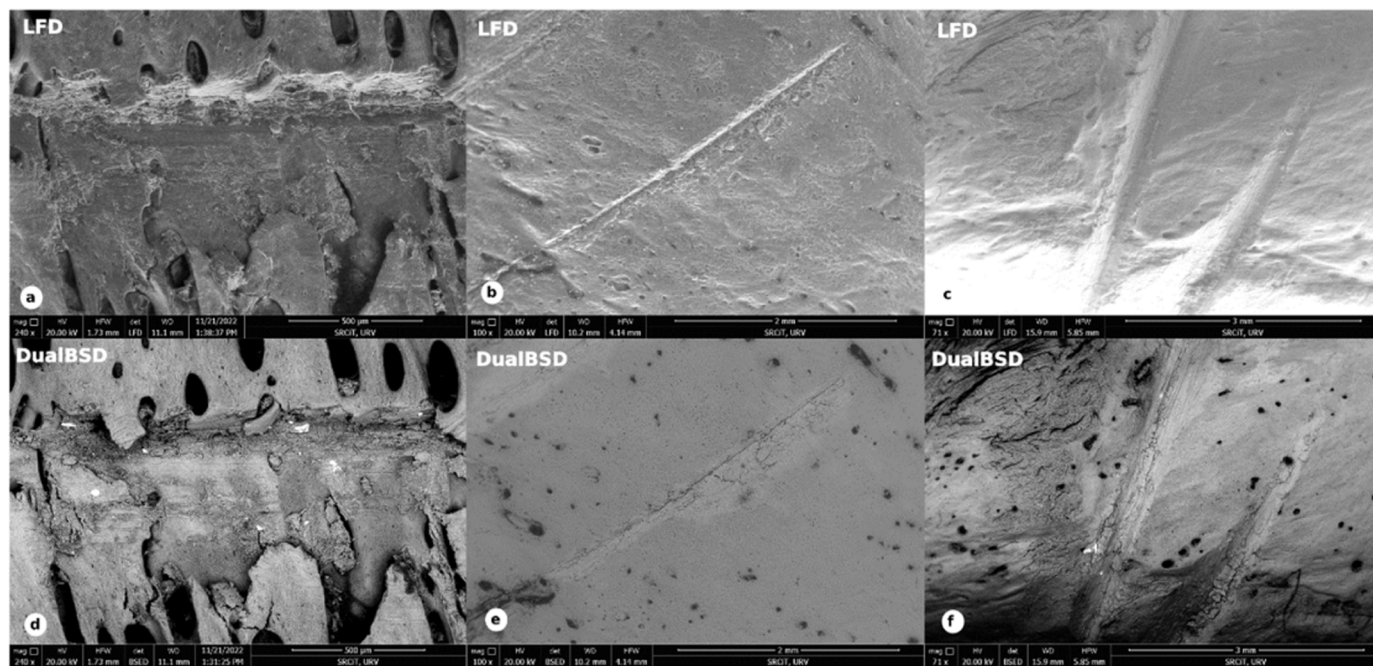


Fig. 2. Comparison of the results obtained on the same sample at different magnifications by LFD (a,b,c) and DualBSD (d,e,f) where metallic residues can be seen in bright white.

between 5.8% and 43.1% Cu, between 0.7% and 8.3% Sn and 0.1%–0.4% Al (Fig. S2; Fig. S3). Similarly, Fe (4%) was documented in one of these spectra. In contrast, in the contamination we found elements not present in the composition of the knives, the sharpening stone or the bone (Table S1). High values of lead (Pb=81.1%), Fe (24.4%), cesium (Ce=16.7%), nickel (Ni=12.2%) and titanium (Ti=7.3%) were detected in the contamination, as well as lower values of zinc (Zn=3.1%), manganese (Mn=1.5), barium (Ba=0.8), bromine (Br=0.4) and scandium (Sc=0.3) (Fig. S4).

Knife residues were identified in 80% of the cut marks analyzed. Residue was detected in 69.2% of the incisions on bone with tissue, while metallic residues were found in 100% of the cut marks on bone without soft tissue (Table 1). The size of the residue particles in the first experiment showed some variability, ranging from 20 to 100 µm (Fig. 3). In contrast, the second experiment showed both a greater accumulation of residue and larger particle sizes (<300 µm), which gave rise to greenish staining within the cut marks in two cases (Fig. 4; Fig. 5). This greater residue accumulation was documented on the boiled remains.

4. Discussion

Among the cultural modifications in the archaeological record, cut marks have received the most extensive research attention as a means of identifying human activity (Fisher, 1995). They provide information regarding cultural practices and the management of animal resources by past human groups (Bertolini and Thun Hohenstein, 2016; Egeland et al., 2024; Galindo-Pellicena et al., 2017; González-Rabanal et al., 2023; Taylor et al., 2020). In recent years, several studies have focused

on determining the raw materials that generated these modifications in order to better understand the cultural impact of the metal age (e.g., Greenfield, 1999, 2000; Maté-González et al., 2018; Olsen, 1988; Pierce et al., 2019). This is an important period in prehistory as the appearance of metal represented a technological turning point for prehistoric societies and led to a significant decrease in the use of stone tools (Greenfield, 1999, 2000).

From a functional perspective, the use of tools provokes constant wear, which requires them to be sharpened (metals) or retouched (stones) (Sáez and Lerma, 2015). This wear is produced by direct contact with a surface (flesh, skin or bone) that causes the detachment of microscopic particles from the tool itself. These particles/residues remain deposited along the cut surface (Gibelli et al., 2012). Although a method for identifying residues left by metal objects on a body has been developed in the field of forensics (Gibelli et al., 2012; Palazzo et al., 2018; Pechníková et al., 2012), it is difficult to apply the same parameters to archaeological materials. First, archaeological remains may bear superficial cut marks in which the tool barely and only accidentally comes into contact with the bone (Braun et al., 2008). This is in contrast to the modifications that occur in violent events in which greater force is applied during the blow or cut (Gibelli et al., 2012; Pechníková et al., 2012), during the butchering process, and/or in funerary contexts in which bone manipulation may take place at an advanced state of decomposition or desiccation (Bello et al., 2016; Wallduck and Bello, 2016). Secondly, the preservation of the residues may be conditioned by factors related to post-processing manipulation, such as cooking and the presence of external contamination.

The results obtained in this study show that the cut marks made by

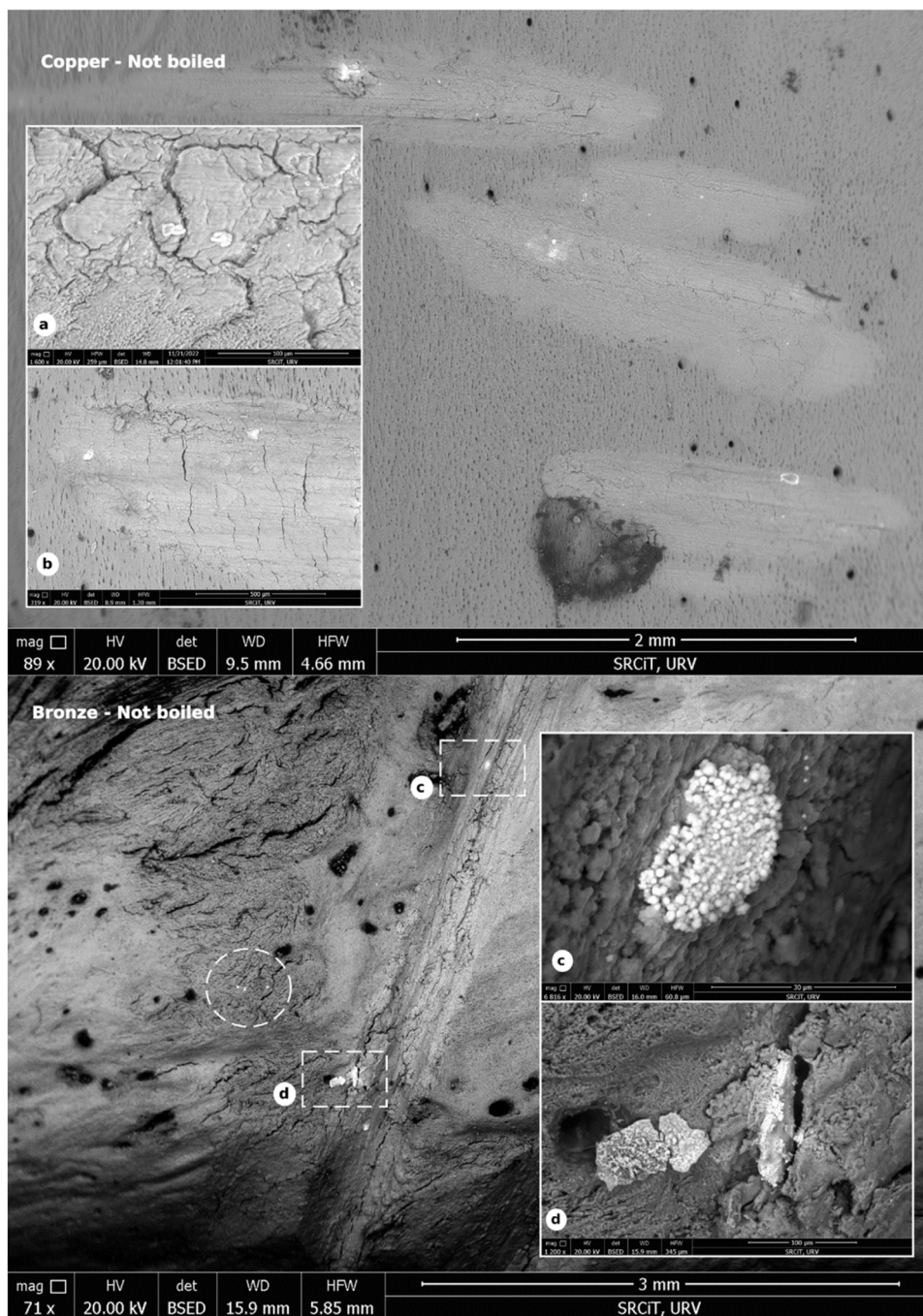


Fig. 3. Copper (a) and bronze (b,c) residues embedded in the bone surface within the experimental cut marks (unboiled samples). External contamination near the cut mark (dashed circle). Oxidized bronze residues (c,d).

both copper and bronze knives leave residues on bones with and without flesh. These residues were mainly found embedded in the bone surface, on the walls and on the floor of the incisions (Fig. 2b). The attachment to the bone causes them to remain invisible during LFD analysis with SEM, and they can be mistaken for bone flakes, organic matter or contamination (Fig. 3e,f). As an alternative, the back-scattered electron detector loses definition of the morphology of the incision; however, the number of back-scattered electrons provides contrast with the grayish and dark background of the bone and organic matter (lower atomic number), and the metallic microparticles (higher atomic number), which appear as bright white bodies (Fig. 2). The spectra of the chemical composition of these microparticles showed that 38.5% of them could be associated with the knives, while 61.5% corresponded to contamination (Table 1).

Despite having performed a similar number of spectra, our results depart from forensic studies in which $\approx 60\%$ of the residues are associated with knives (Gibelli et al., 2012). This difference may be related to the handling of the remains during and after processing, including sieving in wire mesh, washing with water and drying on paper. Additionally, a metal pot was used to boil the bones, which could be associated with most of the contamination on the boiled specimens.

Direct observation of the SEM images showed that 100% of the residues of the knives analyzed were lodged in the bone, probably as a consequence of pressure, while contamination residue was loose (Fig. 3c,d; Fig. 4a,c). It is noteworthy that Fe peaks were recorded in seven of the spectra performed on knife residues, in addition to their main components (copper=CuAl, bronze=CuSnAl). The unexpected

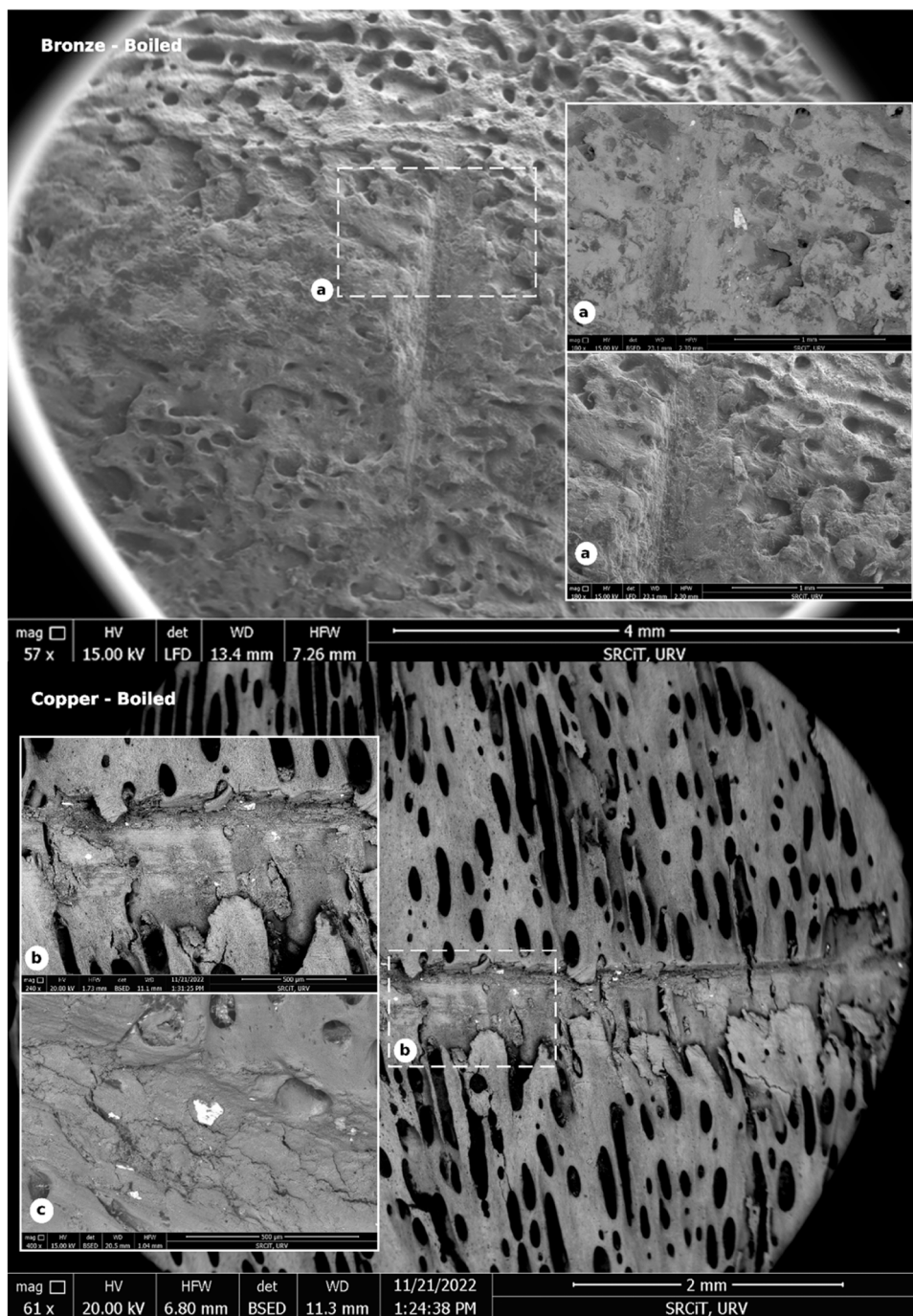


Fig. 4. Detail of metal residues in boiled bones shown as bright white dots within the cut marks (a,b,c). These cut marks were made without soft tissue.

presence of iron has also been documented in other studies, where it has been associated with environmental dust particles (Bai et al., 2007; Pechníková et al., 2012). However, no dust was detected in our samples. In fact, the presence of Fe may be related to the whetstone, which was used to sharpen iron knives prior to our experiment. Traces of that use are visible as orangish oxidation on the different faces of the stone, overlapping with the oxidation of the copper and bronze knives (Fig. S5). The common presence in all spectra of C, O, Ca, P, N, Mg and S is related to the composition of the bone (Table S1). High oxygen percentages of over 35% in five residues may be the product of metal oxidation, and the S percentages, which in some cases exceed 13%, stand out. Although sulfur is present in the bones (0.6%), we believe that the contribution in this spectrum may be related to the water used to

clean the samples.

The high degree of contamination inside the cut marks is a factor that needs to be studied in greater depth. These randomly distributed particles within the cut marks stand out because, unlike the knife residues, they were not embedded. Their chemical composition differed from that of the knives and contained industrial metals such as lead, titanium, zinc and cesium, some of them with percentages above 80% (Table S1). Although the origin of this contamination may be difficult to identify, it is probably related to the handling of the bones after the butchering process. However, the contamination that the knife may contribute based on what it cut prior to processing is something worthy of further research in the future. In fact, tools with an irregular cutting edge, such as a serrated knife, would retain a greater amount of contamination on

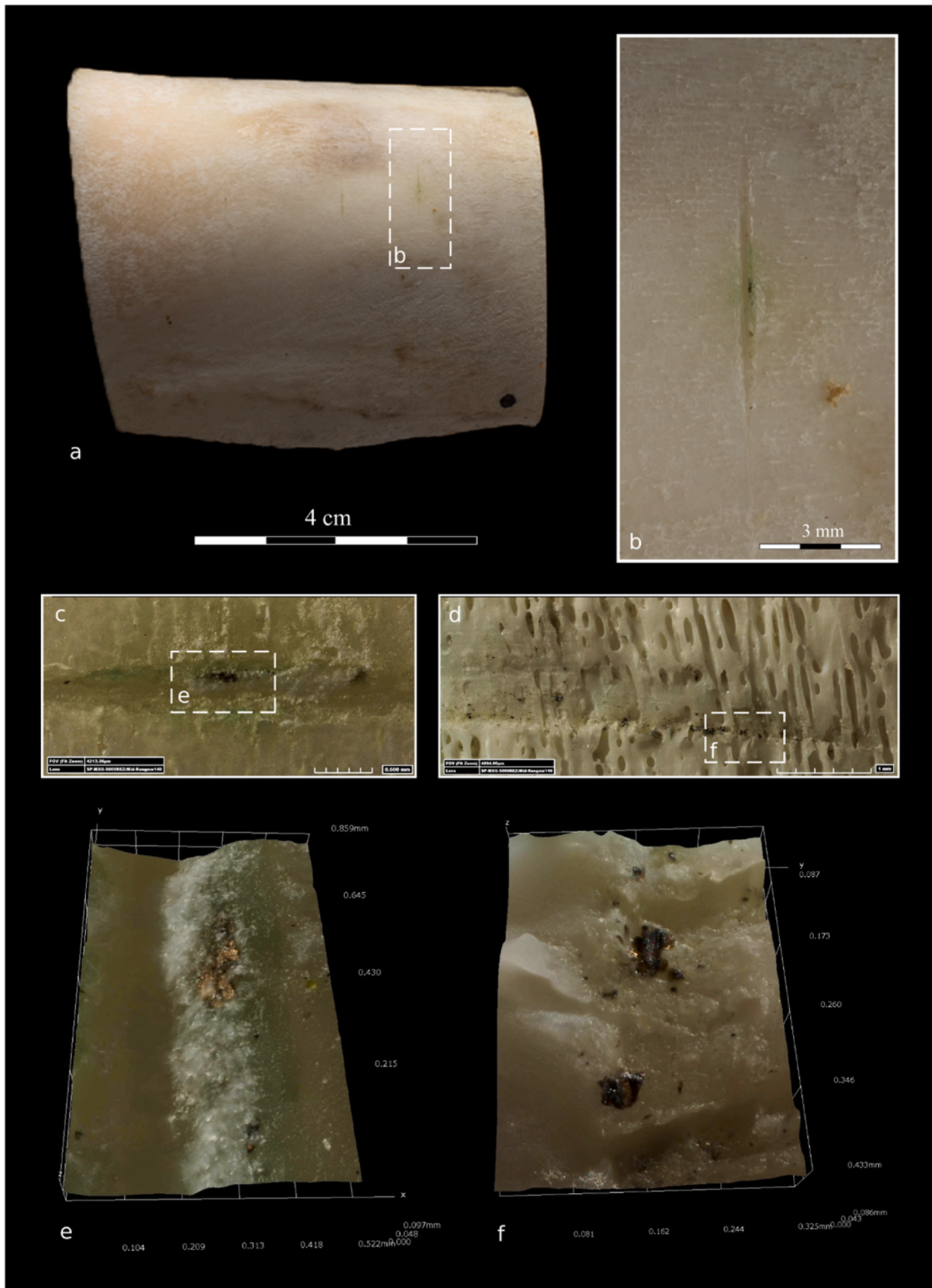


Fig. 5. Greenish staining within the cut marks (b). Embedded residues in the bone surface within the cut mark produced with bronze knife (e) and copper knife (f).

the blade and therefore contribute more contamination than those with a smooth cutting edge (Gibelli et al., 2012).

We detected a relationship between the quantity of residues and the amount of soft tissue between the bone and the tool during processing. The action of cutting skin, muscle tissues and tendons left at least one instance of residue in 69.2% of the incisions, while cuts made on bone with no or little soft tissue left large amounts of residues in variable sizes in 100% of the cut marks. The low presence of residues on bones with tissue is similar to that found in forensic studies (Gibelli et al., 2012; Pechníková et al., 2012). However, this similarity should be treated with caution as modern metals and their alloys are harder than copper or bronze. This suggests that direct bone contact with archaic metals leaves a high quantity of residues, some with sizes between 250 and 300 µm (Fig. 5), which can give rise to greenish staining produced by macroscopically visible oxidation (Fig. 5b,c,e). Metallic oxidation staining is not unknown and is often associated with copper or bronze objects such as jewelry (e.g., Schultz and Dupras, 2022) or with postdepositional modifications (Winnicka et al., 2020).

Beyond the residues left by human activity and contamination, these studies have the challenge of identifying metallic residues in bones that were manipulated in the distant past. In fact, food cooking, which was a common practice during the Bronze Age (Hernando et al., 2022; Martín et al., 2014), does not seem to influence the preservation of residues. In fact, various sizes of microfragments (<20–300 µm) embedded in the bone were detected in the boiled bones from the second experiment, probably due to their anchorage to the bone surface.

Furthermore, the study of residues in cut marks can complement the morphometric analysis of these modifications in contexts where unexpected results have been obtained, such as in Ulaca (Spain), a Late Iron Age assemblage, where the morphometry of the cut marks is associated with lithic industry and not with metal implements (Maté-González et al., 2023).

We believe that this line of research can provide relevant data on the use of metal implements in Chalcolithic, Bronze Age and Iron Age populations in relation to their daily lives, cultural traditions and inter and intra-personal conflicts, and even in the practice of ceremonial or ritual acts. However, we do not know the degree to which the formation processes of the archaeological record influences the preservation of residues. In addition, a careful procedure is necessary for those archaeological materials with cut marks to be analyzed with SEM. These should be carefully washed, not brushed, or wrapped with aluminum or covered with silicone to make molds of the cut marks, in order to avoid contamination and removal of metal residues. These studies should be undertaken in greater depth with both archaeological and experimental materials, and a possible cleaning protocol developed to eliminate contamination without affecting knife residues.

5. Conclusion

The use of copper and bronze knives leaves cut marks on bone which contain microfragments identifiable by DualBSD with SEM. The microfragments are embedded in the bone and their preservation is not affected by cultural activities like bone boiling. We observed that the amount of identifiable residue is influenced by the presence or absence of tissue when making the cuts with more residue documented on bones without flesh. The presence of old and new contamination within the cut marks is important and needs to be further investigated. However, from what we have observed so far, contamination is not embedded in the bone and has a different chemical composition than the knives. Even so, we do not know what role archaic contamination may play and how post-depositional processes over thousands of years may affect the preservation of microscopic metal fragments. The study of residues in cut marks is a line of research with great potential to be applied in different contexts where cultural modifications have occurred, either on human or faunal remains, thus adding to the current understanding of the management of raw materials and the socio-cultural implication of

metals in these prehistoric societies.

CRediT authorship contribution statement

Antonio Rodríguez-Hidalgo: Writing – review & editing, Writing – original draft, Visualization, Supervision, Data curation, Conceptualization. **Palmira Saladié:** Writing – review & editing, Writing – original draft, Visualization, Supervision, Data curation, Conceptualization. **Josep Maria Vergès:** Writing – review & editing, Writing – original draft, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **Francesc Marginedas:** Writing – review & editing, Writing – original draft, Resources, Methodology, Investigation, Formal analysis, Data curation, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at [doi:10.1016/j.micron.2024.103614](https://doi.org/10.1016/j.micron.2024.103614).

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