ORIGINAL PAPER THE STRUCTURAL, MORPHOLOGICAL AND ELEMENTAL CHARACTERIZATION OF A SICA SCABBARD DISCOVERED AT BURIDAVA

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Abstract. In 2019, on one of the most important dacian archaeological sites (Ocnița, Vâlcea County, Romania), in a tomb dug into the rock, along with various weapons, ceramics, bones, etc. there was also discovered a fragmentary sica scabbard. As a consequence of the lack of information on solely such iron-made objects, the urgent need to obtain as much information as possible regarding this type of military equipment was mandatory. Thus we applied different characterization methods aimed at obtaining results regarding the inner structure assembly of the scabbard, the chemical composition and the elemental distribution of the material the scabbard was made of, in order to better understand the technique used by the dacian blacksmiths. With the help of optical macroscopy, energy dispersive - X-ray fluorescence spectrometry (EDXRF), X-ray CT scanning, scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS) we tried to reconstruct how the scabbard was made, and how it survived until present times.

Keywords: dacian weapons; scabbard; sica; chape; Buridava; XRF; X-Ray CT; SEM-EDS.

1. INTRODUCTION

Although the systematic research at the dacian Buridava from Ocnița, Vâlcea county started as early as 1961 [1], the resumption of the systematic archaeological research by the University of Pitesti starting from 2016 also led to the carrying out of the first interdisciplinary research on metallurgy [2]. Even if it is considered the most important dacian settlement south of the Carpathians for the 1st BC – 1st AD centuries [3], so far this has been reported for inventory items consisting mainly of household items (various pottery, tools), jewelry. Unfortunately, until now, the importance of this settlement has not been seen and implicitly not analyzed from the point of view of the dacian military equipment discovered here, and this passive attitude towards the research of this field must change.

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If until 6 years ago one could state that the number of weaponry pieces was not so large or varied (the vast majority of discoveries consisting of arrowheads), starting with the resumption of research in 2016, the pieces of military equipment have been discovered in increasing numbers, being represented by various items from both offensive and defensive military equipment

Considering the fact that pieces of military equipment were prestige items, which entailed very high costs, probably much higher than for certain items of adornment that are considered extremely valuable, the study of these weapons that could not be owned by common individuals can bring valuable information not only about the life of certain warriors, but also about the technology used to make them. A weapon with strong connotations in the dacian environment is represented by the *sica* dagger. For this reason, this weapon generally featured a number of representations and decorations that involved not only additional work on the part of the craftsman, but also a great deal of skill and probably corresponding costs. Due to the many iconographic representations - especially from the beginning of the 2nd century BC, the *sica* is considered to be an emblematic weapon of the dacians. This weapon, however, is found throughout the Thracian environment.

From a morphological point of view, the *sica* is a dagger with a curved blade, with a cutting edge on the inside and a blood drainage channel, generally between 25 and 35 cm in length, but it should be noted that there may be daggers which may deviate from this pattern [4]. Although no intact *sica* has been discovered in Buridava until now, the discovery of the metal part of a *sica* scabbard, namely a chape, was considered a great surprise, this piece also being the object of the present study. The fact that only the weapon protection was discovered, and not the weapon itself, is not an isolated case, as the same thing has been identified in at least five cases [5]. From a functional point of view, the scabbard that covered the *sica* had a double role: on the one hand, because it was attached to the owner through a system of straps and links, which prevented its loss, and on the other hand, as long as it was in the scabbard, the weapon could not accidentally injure the wearer [5].

This paper aim is to present a new discovered military artefact - sica scabbard discovered in Buridava site (2019) and to apply different advanced characterization methods in order to try answering important questions related to how the dacian *sica* scabbard was manufactured, and how it survived until present times.

2. MATERIALS AND METHODS

2.1. MATERIAL

The material used in this study was a sica scabbard discovered in Buridava site in 2019. The *sica* scabbard fragment presented in Fig. 1 was discovered in a tomb dug into the rock (M 28). It has the following size: actual length of 134.99 mm, button thickness of 16.88 mm, and metal sheet thickness of 3.52 mm. Measurements were taken on the unrestored piece.



Figure 1. Microscopy of the sica scabbard, Buridava.

The need to characterize this piece of military equipment occurred all the more so since, of all the pieces of dacian military equipment analyzed to date, no such scabbard has been subjected to these investigative methods [6]. The methods used to investigate the *sica* scabbard were: optical macroscopy – for a general view of the historical piece and details observation, energy dispersive X-ray fluorescence spectrometry – for chemical elemental analysis, X-ray CT scanning – for the investigation of the inner structure beyond the outer corrosion layer and scanning electron microscopy with energy dispersive spectroscopy – for chemical elemental micro-analysis and cross-section investigations.

2.2. METHODS

2.2.1. Optical macroscopy - In order to examine the external fixed state, by macrography, but also the integrity of the corrosion layer of the piece of the scabbard, a Nikon AF-S DX Micro Nikkor 40mm f/2.8G lens (Nikon, Tokyo, Japan) was used, with autofocus and fixed zoom.

2.2.2. X-ray fluorescence spectrometry (XRF) - To determine the composition of the elements of the fragmentary scabbard, the samples have been investigated with SPECTRO MIDEX M energy dispersive X-ray fluorescence spectrometer (Spectro Analytical Instruments GmbH, Kleve, Germany) having the following characteristics: 30 W-Molybdenum X-ray tube generating a 2 mm spot diameter, SDD detector with a resolution of 160 eV measured on Mn-K α line.

2.2.3. Scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS) - For the analysis of the scabbard sample it was used HITACHI SU5000 scanning electron microscope (SEM) equipped with an energy dispersive spectroscopy (EDS) module (Hitachi, Tokyo, Japan). The system allows analysis at ambient temperature or low temperatures (-25°C) on large samples (200 mm diameter and 60 mm height), in high vacuum but also at variable pressures (10-300 MPa) for poorly conductive samples and even plastic materials. The qualitative and quantitative analytical determinations can be performed by energy dispersive spectroscopy (EDS) module.

2.2.4. X-ray scanning using the tomograph computer (XR-TC) - In order to virtualy pass through the consistent corrosion layer, to obtain data on the unaffected core, as well as other data on the properties of the scabbard, the X-ray equipment Nikon XTH 225 ST (Nikon, Tokyo, Japan) was used to scan the samples.

3. RESULTS AND DISCUSSION

3.1. MACROSCOPIC CHARACTERIZATION

The macroscopic analysis provides a series of important data regarding both the manner in which the scabbard was made and the factors that led to its degradation. In Fig. 2 we can notice that the scabbard has a textile pattern. Originally, it was covered with a consistent layer of corrosion products that were removed by the restorer. In fact, the discovery of the textile pattern led to the decision not to go any deeper with the cleaning procedure. The

macroscopic analysis of this side of the scabbard was able to capture in detail the textile pattern in Fig. 2.



Figure 2. Macroscopic image: a) Textile pattern; b) the joining area of the metal sheet.

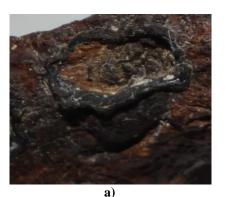
We can also see that it displays strong traces of burning, being corroded over the entire surface, with approximately half of the upper part missing. The discovery of the textile pattern confirms the rule according to which precious elements such as metal scraps or traces of organic fibers can be found inside the layer with corrosion products [7, 8]. A prominent crack can be observed longitudinally on the scabbard. Given the large thickness of the metal sheet, most likely that crack that exceeded 1.5 mm is where the sheet was joined at the ends.

Despite the fact that previous study of some *sica* scabbards revealed that they were made of iron sheet, slightly curved to the shape of the blade, with the edges bent inward lengthwise until they are welded together at the end in a specific discoid button [9], the Buridava scabbard does not obey this rule. As can be seen in Fig. 2 on the longitudinal side of the scabbard it was discovered that the edges of the metal sheet were only close. The rather large gap, visible with the naked eye, between the two edges of the rolled metal sheet is mainly explained by its large thickness. If in general *sica* scabbards are made of a thin metal sheet to be able to make specific designs and inlays, the Buridava scabbard is made of a massive iron sheet, which implies an extremely difficult malleability.

Considering the fact that this item of military equipment was subjected to burning on the pyre at a high temperature, it could be observed that in the curvature area of the scabbard, the latter was curved unnaturally to the side. The burning on the pyre at a high temperature also generated the physical modification of the scabbard, during which, most likely, the edges of the metal sheet moved away, creating the impression of a longitudinal crack. The white color of most of the bones discovered in the Buridava tombs [10] determines the approximate temperature at which the deceased and their equipment, including the scabbard which was subjected to the present analysis, were cremated, this color indicating a burning temperature between 650-800°C [11].

As can be seen in Fig. 3, the corrosion layer shows cracks, but also black craters, very prominent, indicating that the object was subjected to combustion. Among the external factors that lead to the degradation of iron objects, the most important is the pH, or considering that in the immediate vicinity of the tomb where the object was discovered, soil pH measurements were made, its value being 7 (neutral) [12], it follows that this was not the main cause of the damage, but, more importantly, the cause was the high burning temperature which the scabbard fragment was subjected to.

In this situation, one of the factors that led to favoring corrosion is incineration, considering that high temperatures lead to deformations, changes in structure and the acceleration of the corrosion process until the total transformation of the metal into corrosion products [13]. The macroscopic analysis also allowed the identification of some identifiable compounds due to their color. A number of black craters could be observed, while most of the piece shows a reddish corrosion crust [8, 14, 15].



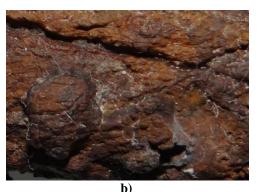


Figure 3. Details with traces of melting and cracks.

3.2. X-RAY FLUORESCENCE SPECTROMETRY EDXRF

Because of the non-destructive principle of the EDXRF [16-21], in order to determine the elemental composition of the scabbard, which can provide information about the process of making the material, a point scan was performed (Fig. 4a) in the cross section of the metal sheet from which the scabbard was made, without interfering with the surface of the section by polishing. The obtained EDXRF spectrum is represented in Fig. 4b and highlights the presence of the following elements: Si, P, Pb, Ca, Cr, Fe, Cu, and Zn.

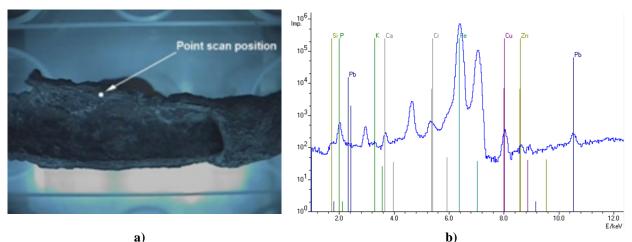


Figure 4 EDXRF Analysis a) the image of the sample and the position of the scanning point b) the EDXRF spectrum obtained on the sica scabbard.

The quantitative analysis of the elements is presented in Table 1.

Element	Si	Р	K	Ca	Cr	Fe	Cu	Zn	Pb
Concentration (wt%)	0.221	> 1.475	N.C.*	< 0.030	0.031	96.77	0.072	0.011	0.032
*NC - Element not augmentified by the spectrometer algorithm (Eurodemontal parameters, analysis method)									

Element not quantified by the spectrometer algorithm (Fundamental parameters, analysis method)

The quantitative analysis of the elements shows that iron is found in a very high percentage, of 96.77%, accompanied by minor elements such as Si, P, Ca, Cu, Zn, and Pb. Iron, which is the majority element, fits perfectly into the context of dacian metallurgy, considering that the iron blooms obtained with the help of ore reduction furnaces reached a percentage of iron of up to 99.96% [22]. Regarding the minority elements, their existence can be explained, in the case of Ca and P, in terms of coming from the natural environment where the scabbard fragment was deposited. Practically, in the tomb, along with the inventory items of the deceased, traces of ash, coal, and fragments of calcined bones were also identified. Regarding the other minor elements, Si, Cr, Cu, Zn, and Pb, they come either from the soil, respectively from the ore, or from the metallurgical process.

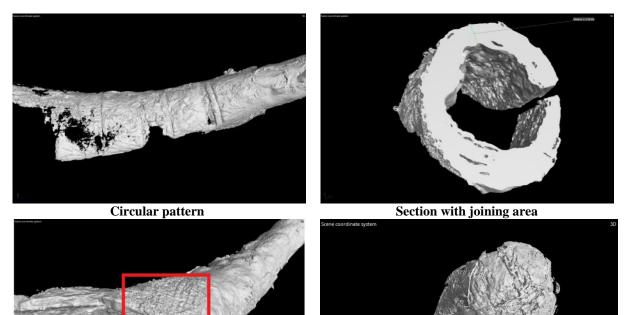
3.3. X-RAY SCANNING WITH THE HELP OF THE COMPUTER TOMOGRAPH

Out of the desire to obtain new data, considering the fact that the scabbard had strong traces of burning and corrosion, it was scanned with the help of the X-ray computer tomography. The main reason was that in scientific literature, in the case of *sica* scabbards preserved in a better state of conservation, it is recorded that, in most of them, they are provided with decorations.

As can be seen from the 3D images in fig. 5, after the virtual penetration and after the data was sent to the detector [23] on the material from which the scabbard was made brought new data regarding the size and pattern of the scabbard and the bump, the way the metal sheet was joined, but also a better visibility of the textile pattern printed on the scabbard.

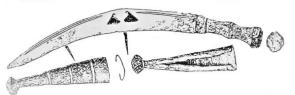
Another essential thing that regards obtaining new data is related to the identification of the original surface [24], which is achieved according to Fig. 5b.

With the help of this method, it was possible to see that the scabbard was provided with a series of circular patterns made both in the negative of the material and in the positive (Fig. 5a). For this type of pattern, it was possible to identify some analogies with other scabbards belonging to private collections in Germany (Fig. 6a) [9], or obtained through archaeological research in the area of Bulgaria (Fig. 6b) [25] or belonging to other private collections from Bulgaria (Fig. 7) [26].



Textile pattern Figure 5. CT scans. Scabbard details under the corrosion layer.





Chape with pattern Sica / Chape with pattern Figure 6. a) Analogy with a scabbard from a private collection Germany, [9], b) Analogy with a sica scabbard from Tarnava, Bulgaria, [25].

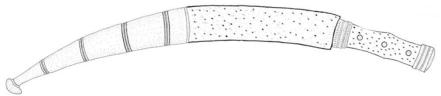


Figure 7. Analogy with a sica scabbard, private collection, Bulgaria, [26].

Not only that the decorative elements applied to the blade, handle and scabbard personalize the weapon, but they also conferred a strong spiritual character on it [27]. The circular shape of the decoration on the scabbard may be related to the belief of the geto-dacians. From this point of view, it should be specified that on the *sica* blades, along with the representation of some zoomorphic forms, in many situations there are representations of the sun [28, 29], or the shape of the circles on the scabbard may even represent the solar disk.

In Fig. 5b, respectively in Fig. 5d it is noted that this portion of the scabbard was made by hammering the sheet of metal until its edges were joined, and in the case of the button, the sheet of metal was cut and its edges were joined by hammering. This way of working would later favor the corrosion process because the mechanical actions of an object is subjected to produce changes in the orientation of the dendrites and create tensions in the material, tensions that are the basis of electrochemical corrosion processes by creating pitting microcells that lead to cracking materials in that area [29].

As to how the button was made, the cross shape indicates the procedure used in this case. Basically, the metal sheet was cut in the form of four triangles (after the metal sheet was rolled), which by hand forging were brought together and possibly welded by heating the metal sheet during this operation. The approach and union of the four triangles also implied the slight flattening of the button, and implicitly obtaining the relatively round shape of the tip of the scabbard. Although it seems very ingenious, the method of making the scabbard by bending, rolling and cutting iron is known to have been carried out in the workshops of the La Tène period [30], the period to which the present piece also belongs.

3.4. SCANNING ELECTRON MICROSCOPY WITH ENERGY DISPERSIVE SPECTROSCOPY

In order to carry out the morphological characterization of the sample taken from the scabbard, the latter was cleaned with the help of ultrasound in ethyl alcohol, using the Pulsatron KC2 ultrasound bath equipment to remove oxides. With the help of SEM, we were able to analyze the surface of the sample, while also being able to measure the thickness of the metal sheet from which the scabbard tip was made. Thus, if before the restoration, taking into account the consistent layer of corrosion, the scabbard measured 3.52 mm, this time, it was observed that, after cleaning, the thickness of the metal sheet is on average about 2 mm (Fig. 8). For the elemental qualitative and quantitative characterization of the chape, 4 point scans were applied, numbered: Point scan 1-4 shown in Fig. 9.

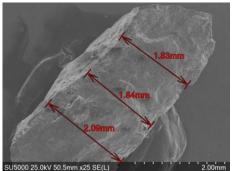


Figure 8. SEM micrograph of metal sheet cross section at x25 magnification.

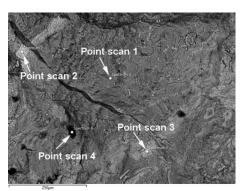


Figure 9. Position of the point scans.

The elemental analysis of the EDS spectra confirms the presence of the following elements: O, Mg, Al, Si, P, S, Cl, K, Ti, Fe, and Zn, according to Fig. 10. The quantitative EDS analysis from Table 2 reveals the weight % of each element for the 4 scan points.

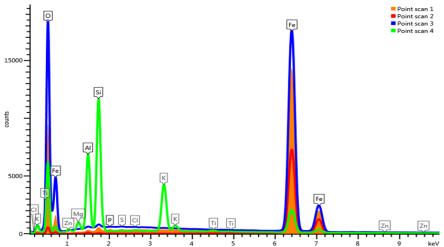


Figure 10. Qualitative EDS analysis with the overlap of the 4 point scans.

Spectrum Label	Concentration [wt.%]							
Spectrum Label	Point scan 1	Point scan 2	Point scan 3	Point scan 4				
0	25.92	4.45	33.83	41.38				
Mg	-	-	-	1.74				
Al	0.34	0.87	0.24	12.46				
Si	0.68	0.27	0.36	21.98				
Р	0.23	-	-	-				
S	-	-	-	0.18				
Cl	-	-	-	0.15				
K	-	-	-	8.96				
Ti	-	-	-	0.39				
Fe	72.82	94.41	65.57	12.54				
Zn	-	-	-	0.22				
Total	100.00	100.00	100.00	100.00				

Table 2. The quantitative EDS analysis on the investigated points.

From the quantitative EDS analysis, it can be seen that the majority element is represented by Fe. Its value differs depending on the area of the point scans. The highest Fe value of 94.41 wt% is measured in the median area of the transversal section, and the lowest of 12.54 wt%, even though it is also measured in the median area, the point scan overlaps a surface indicating a cavity containing various inclusions (most likely aluminosilicates). It is

also the reason why, from the total concentrations in this point scan (Point scan 4), O, Si and Al were identified as having a major weight, respectively 41.38 wt%, 21.98 wt% and 12.46 wt %. Regarding the minority elements represented by Mg, P, S, Cl, K, Ti, and Zn, it is worth noting that they appear in only one point scan. It should be noted that, except for Zn, all other minority elements were identified as being part of the native soil from Buridava [31], a soil in which the scabbard chape was discovered and analyzed. Moreover, the minority elements with a higher value than the others such as Mg, Al, Si and K had a higher weight among the minority elements in the native soil, as in the present situation. Regarding Zn, it is generally found in archaeological soils in small quantities, being relatively mobile in soils and easily exchanged by other trace metals [32], as is the case here.

The other minor elements such as O, Al, and Si are the only ones found in all four point scans. The high presence of O, up to 41.38 wt%, is explained to the greatest extent by the presence of oxides, the piece being extremely affected by the corrosion process, Al is one of the elements found in quite large quantities in the native soil of the archaeological area [31] from which the scabbard fragment was taken, while Si, apart from the fact that it represents the main element in which the weapon was deposited, it could be used in the technological process of obtaining iron and making the scabbard, as is also mentioned in other situations regarding the making of dacian weapons and tools [33]. By means of the EDS *line scan* one can see in Fig. 11 the cross section evolution of the concentration of each element (O, Al, Si, P, and Fe) within the investigated section.

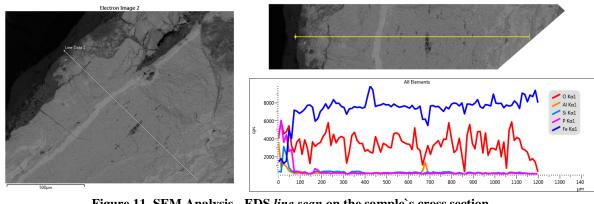


Figure 11. SEM Analysis - EDS *line scan* on the sample's cross section.

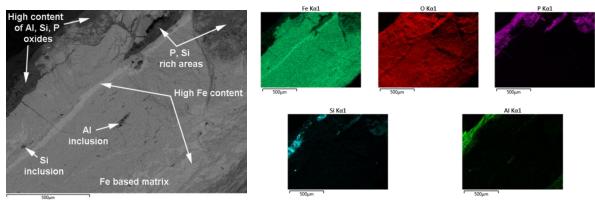


Figure 12. SEM – EDS Analysis on the surface in transversal section.

The EDS *line scan* analysis complement the results previously obtained by 4 points scan. Thus, the majority elements Fe and O appear both relatively evenly distributed throughout the sample's cross section and it can be observed that Si, Al, and P appear mainly in the extremities of the scabbard, towards the outside, while Al has a high concentration point in sample's median area. The concentration distribution of the elements (Fe, O, P, Si,

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and Al) is also observed as a result of the EDS mapping analysis on the cross section sample's surface, according to Fig. 12. SEM-EDS mapping analysis from the same area together helps in the interpretation of how metal alloys and coatings were fabricated [34] and shows a relatively compact cross-section material where it can be noticed high concentrations of Al, Si and P oxides towards the scabbard's surface; inner areas with rich P and Si content, inner Al and Si inclusions, a strip and inner areas with high Fe concentrations.

4. CONCLUSIONS

A first conclusion is related to the shape and size of the *sica* scabbard. It is obvious that the fragment studied represents only the chape, respectively the lower part of the scabbard. If we refer to the fact that a *sica* blade, which the scabbard was supposed to cover, was on average 20-23 cm long [35] and to the fact that the chape analyzed measures only about 14 cm, it is obvious that it only protected the tip of the blade, and in the upper part it had to be continued most probably with an organic material, namely wood or leather.

Moreover, the fact that only the metal part was discovered in the tomb strengthens the assumption regarding its continuation with organic material that did not withstand the fire to which it was subjected during the incineration, on the one hand, and on the other hand, the organic remains of the incinerated material could hardly have resisted the passage of almost 2,000 years. The macroscopic analysis of the chape highlighted the fact that it showed traces of burning, so the piece was placed next to the cremated person on the funeral pyre. At the same time, a textile pattern could be observed printed in the layer of corrosion products. Also, the longitudinal crack observed on the scabbard fragment seems to indicate that this occurred due to the high temperature during the incineration which led to the expansion of the material.

The quantitative EDXRF analysis highlights the fact that iron is found in a very high percentage of 96.77%, which is accompanied by minor elements such as Si, P, Ca, Cu, Zn, Pb among which it can be observed that P holds his highest share of over 1.475%. The provenance of P, but also of the other minority elements, is related both to the technological process of making the weapon in the workshop, but especially to the soil in which it was deposited. The existence of Ca alongside P may be an indication of the past presence of organic material (bone). X-ray scanning revealed that the *sica* scabbard was provided with a series of circular incised and excised patterns in the iron sheet. For this type of model, some analogies could be identified, but the model captured with the help of X-ray scanning highlighted the existence of decorations that seem to be more complex.

Regarding the way in which the scabbard was made, it was possible to highlight, as was also observed during the macroscopic analysis, that the metal sheet is cracked along the entire longitudinal distance. Most likely, the fact that the welding of the metal sheet did not occur properly is due to its thickness, and the high temperature to which the scabbard was subjected during the incineration process led both to the disappearance of the organic material from which it was made, and to widening the area where the two ends of the sheet metal should have joined. Also, the trace of textile material that appears to be printed on the scabbard and which was observed following the macroscopic analysis was captured with even greater clarity following the analysis of the 3D images. Last but not least, starting from the information known in the specialized literature according to which some of the scabbards discovered have a button-shaped termination provided with decorations, the careful analysis of the button of the scabbard from Buridava revealed the fact that it was provided with an incised ornament in the form of a cross. But that shape is obtained by cutting in the form of triangles, free forging and bringing them together until the shape of the rounded button is obtained.

Using SEM-EDS it was possible to observe the concentration distribution for the identified chemical elements. Interestingly, P was located towards the outside of the material together with the oxide layer, which leads to the idea that it originates from an organic material near the scabbard rather than from the technological process (bones). Regarding the thickness of the metal sheet from which the scabbard was made, even if, according to the SEM calculations, it had a current thickness of about 2 mm, the original thickness cannot be known precisely, considering the fact that the analyzed part is strongly affected by corrosion. The only certainty in this situation is that it must have been thicker originally.

The experimental analysis of the iron scabbard fragment was a necessity considering that in specialized literature we could not identify any detailed analysis of a *sica* scabbard made of iron. In general, the attention of specialists was drawn to the study of sword scabbards, which are much larger and were, unfairly, considered much more spectacular. As we have seen in the present case, the *sica* scabbards, although they are much smaller in size, present the most varied decorations and elements.

The analysis of the scabbard fragment discovered in Buridava in 2019 opens up new perspectives regarding the understanding of the technological process by which this type of object was made. Until now, no such object has been subjected to such detailed characterizations, on which occasion it was possible to observe that the scabbard fragment analyzed is unique at the current stage of discoveries. In general, iron *sica* scabbards are made from a thin sheet that is easily malleable, while the Buridava fragment is made from a massive sheet of iron, the making of which requires advanced knowledge of the blacksmiths. Also, the fact that the decorative models made of bronze, an easy-to-process alloy, were also discovered on the iron scabbard from Buridava confirms again that the craftsmen had a more advanced level of knowledge than the one promoted in the specialized bibliography.

The characterization of scabbard chape opens a new horizon regarding the research of such equipment; this is primarily due to the increasingly advanced modern research methods that bring detailed information on the elaboration technology or elemental composition. The model applied in the present study requires its application to similar objects discovered in the past, but which benefited from rudimentary analyses specific to the last decades or century.

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