ORIGINAL PAPER

ANALYSIS OF ARCHAEOLOGICAL SWORD MATERIALS FROM BURIDAVA SITE

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Abstract. *In an attempt to obtain as much data as possible about the fragmentary sword discovered in 2017 in one of the most important Dacian sites (Ocnita, Vâlcea County), but also about the way in which the environment in which it was preserved for approximatively 2,000 years has influenced its conservation status, the methods of characterization applied aim to obtain results concerning, on the one hand, the composition of the material from which the weapon was made, and as far as possible the technique used by the blacksmith craftsmen, and on the other hand, the composition of the soil in which the weapon was deposited to see the way the soil influenced the conservation state in which the weapon was found. Several analytical techniques, such as optical macroscopy (OM), X-ray fluorescence spectrometry (XRF), X-ray CT scanning, X-ray diffraction (XRD), scanning electron microscopy with energy dispersive spectroscopy (SEM) were used in order to investigate the corrosion processes that aroused and affected the analysed parts of sword fragment.*

Keywords: *Dacian sword; archaeological soil; XRD; XRF; X-RAY CT; SEM-EDS.*

1. INTRODUCTION

The Dacian site from Ocnița, Vâlcea county, is among the most famous archaeological sites in Romania and, in the current stage of discoveries, it is considered as representing even the most important fortified settlement in the south of the Carpathians, for the I BC – I AD centuries [1-3]. It is located on the territory of Ocnele Mari, corresponding to the Ocnița and Cosota villages.

The complex from Ocnele Mari-Ocnița consists of three fortified areas (fortresses) and a civil settlement, located to the right of the Sărat brook and to the north of the Cosotei brook, in the immediate vicinity of the salt mines.

The archaeological discoveries from here have highlighted the special role played by the Dacian settlement, both in the trade with the Roman world and in the existence here of a

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possible centre of dynastic power. However, once the inscription *Basileus Thiamarkos epoiei* was discovered, the first contradictory points of view appeared among specialists. The discoveries from Ocnele Mari-Ocnița-Buridava allow us to speak of a dynastic centre. The inscriptions with Latin letters, the numerous objects of Roman origin, but also the chronology are elements which confer Buridava a special status throughout the first century AD [4]. To all these discoveries are added a series of pieces of military equipment, this kind of artefacts being in a continuous growth, year by year, with each archaeological research campaign.

To all these discoveries are added a series of pieces of military equipment, this kind of artefacts being in a continuous growth, year by year, with each archaeological research campaign. Thus, during the 2017 archaeological campaign, among other discoveries, M15 – a surface tomb – was identified in Section IV. During his research, it was possible to delimit a cyst made of river stone and tuff (whose superior part had been destroyed), directly on the rock, having a circular shape, with a diameter of 33 cm, given that the investigated section is on a slightly inclined landsliding area. The inventory of the M15 is quite special, the complex consists of a sword handle, a fragment of sword blade and a crease, a bronze buckle and a knife (Fig.1). All these are accompanied by numerous cinerary fragments and a bead made of translucent glass, slightly deformed (probably after strong burning) [5].

Figure 1. *In situ* discovery of the sword elements and bones

2. MATERIALS AND METHODS

2.1. HISTORICAL ASPECTS OF ARCHAEOLOGICAL MATERIALS FROM BURIDAVA SITE

In order to be able to correctly interpret the data obtained from the characterization methods applied on the evidence, one must take into account the current historical data regarding the Dacian armament, the technique of its making, but especially the way in which it was placed in the funerary context.

These last elements are of utmost importance, since there is a big difference in the evolution over time of iron weapons and objects, in general, when they were abandoned in the soil, and of the weapons deposited in the tombs in the context of the Dacian funeral rite and ritual, where the custom of burning and bending weapons began to be used.

As can be seen both from the analysis of the image made on the discovery (Fig. 1), but especially from the optical micrographs, the images made in the laboratory (Figs 2 and 3), the sword is fragmentary, only a piece of the blade and the handle are preserved from it, the weapon being thus intentionally damaged since ancient times.

Figure 2. Iron fragment of blade sword

Figure 3. Iron sword handle

Regardless of the fact that the bending of the swords was done as a result of a ritual or that, according to some theories, they were bent and folded simply from a pragmatic reasoning, because, due to their length, this type of weapons would not have fit in the funerary pits [6], from the point of view of our study, in the area where the weapons were folded, they show a more pronounced degradation.

Among the broken swords, of which only fragments are preserved, there are analogies discovered in the Geto-Dacian settlement from Poiana, Galaţi county [7], but also in Transylvania. Based on the analogies here, and on the basis of the classification made by Glodariu and Iaroslavschi, in which we can also fit the sword from Ocnița in the category of type II swords, this type which imitates the Roman *gladius*, has almost all the characteristics of the sword from Ocnița, respectively wide blade with the handle represented by a long tongue, rectangular in section and finished at the end with a hole [8]. The handle of this type of weapon is absolutely identical to the one which we studied.

Among the Dacian sites where bent swords were discovered, the leading place is occupied by the Dacian settlement of Mala Kopanja in Ukraine. Here, out of a total of 13 swords, they had all been folded 3, 5 or even 6 times [6]. From the desire to answer a series of questions, metallographic analyses were carried out on a number of 5 swords [9]. Using this method, can be able to conclude that not all the swords were made according to the same technique, one of them being of a more modest quality, considering that they are made in local workshops [9].

Even in the case of the swords whose bending did not lead to the complety breaking of the blade, in the torsion zone, the blade is much more affected by corrosion [10]. This type of corrosion, known as stress corrosion, affects metals which are subject to tensile or compressive forces and often takes the form of an intercrystalline but not generalised corrosion [11-17].

Besides the situation when pieces of military equipment are removed from their lattice environment and bear a real shock when they are brought to the surface and come into contact with moisture and oxygen [18], research has shown that when the warriors were incinerated, this was done together with a series of objects, including their weapons. This questions the result of some of the modern methods of characterization, given the temperature obtained on the funeral pyre, as well as the period the deceased was burned together with his weapons, which led to the annealing and then the hardening of the respective weapons.

3. APPROACHES OF METHODS USED FOR CHARACTERIZATION OF ARCHAEOLOGICAL MATERIALS

In an attempt to obtain as much data as possible both about the fragmentary sword discovered in 2017, but also about the way in which the environment in which it was kept for about approximatively 2,000 years has influenced its conservation status, the chosen methods of characterization have taken into account the obtaining of results regarding, on the one hand, the composition of the material which the weapon was made from, and as far as possible the elaboration technique used by the blacksmiths, and on the other hand, the composition of the soil where the weapon was deposited.

3.1. OPTICAL MACROSCOPY

In order to examine the external physical state, by macrography, but also the integrity of the corrosion layer of the military piece of equipment, a Nikon AF-S DX Micro-Nikkor 40mm 1:2.8G lens was used, with autofocus and fixed zoom, with the help of which images were captured which allowed the observation of the smallest details.

3.2. X-RAY FLUORESCENCE SPECTOMETRY

To determine the composition of the elements in sword blade and handle, the samples have been investigated with SPECTRO MIDEX M energy dispersive X-ray fluorescence spectrometer 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-Kα line.

For the point scan, two EDXRF spectra are acquired using the following set-up:

1. direct measurement in air with He purge (for light element detection), 1 mm collimation slit, U=44,70 kV working voltage, I=0,30 mA current intensity, 180 s acquisition time, and 2048 detector channels.
2. direct measurement in air with He purge (for light element detection), 2 mm collimation slit, U=18,87 kV working voltage, I=0,30 mA current intensity, 300 s acquisition time, and 1024 detector channels.

3.3. SCANNING ELECTRON MICROSCOPY WITH ENERGY DISPERSIVE SPECTROSCOPY

For the analysis of the soil in which the sword was deposited it was used HITACHI SU5000 scanning electron microscop (SEM) equipped with an energy dispersive spectroscopy (EDS) module. 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.

The soil samples have been homogenized by grinding and mixing. They were then pressed on carbon conductive tape and analysed by SEM-EDS. Images of backscattered electrons have provide information on the particle's morphology, while the area scans provided information abouth the chemical elemental composition and surface distribution in the case of mapping analysis.

3.4. X-RAY SCANNING USING THE TOMOGRAPH COMPUTER

With the aim to be able to pass through the consistent corrosion layer in order to obtain data on the unaffected core, as well as other data on the properties of the sword blade, the X-ray equipment Nikon XTH 225 ST was used to scan the samples. This was achieved in the laboratories of Top Metrology S.R.L., through the contribution of Mr. Florin Samoilă – application manager and Liviu Preda – sales manager, whom we also thank on this occasion.

So as to avoid any contamination of the samples with another type of material, they were fixed in a sponge holder. The low density of the sponge compared to that of the metal in the sample allowed us to scan it entirely without generating additional elements and without altering the results. For the parameterisation part, a power of 185kV with 185μA with an exposure of 0.708 seconds was chosen. During the acquisition, the equipment made 1440 projections (sections), for each projection making 2 pictures and keeping the picture with the highest quality.

All the resulting images were put together to make the 3D model, the alignment of these images being done in a separate program after the scanning was completed, with the help of the CTPro3D software, software developed by Nikon Metrology.

The alignment consisted of configuring an axis on the body of the part, the axis which the program used to identify the resulting elements in all 1440 projections. After the alignment was made on a single axis, another alignment was made after 2 axes to have a high accuracy and the certainty that the scanning is the correct one.

3.5. X-RAY DIFFRACTION

The XRD characterization was performed in order to establish how the weapon had been influenced by the elements in the soil, especially in the case of corrosion. All the X-Ray diffraction measurements were done on Rigaku Ultima IV equipment. The working conditions were: CuKα radiation at 45 kV and 40 mA, Bragg-Brentano geometry with focused beam, D/teX Ultra detector with graphite monochromator, continuous mode scan with a speed of 1 deg./min and a step width of 0,05 degrees in the 2θ range of [18^0-60^0]. PDXL2 software from Rigaku and PDF4+ 2020 database from ICDD [19] were used to perform qualitative phase analysis.

4. RESULTS AND DISCUSSION

4.1. RESULTS OF SWORD ANALYSIS

Optical macroscopy

With few exceptions, the macroscopic analysis (Fig. 4) reveals that the blade has several cracks in the corrosion layer, especially in the edge area and the handle, the details of which can be seen in Fig. 5, shows traces of slag, a sign that it has felt a combustion.

Figure 4. Macroscopic image of the blade sword

Figure 5. Macroscopic image of the sword handle

The visually identified corrosion products are either stratigraphically or randomly disposed, they have colours from yellow (goethite, akaganeite, lepidocrocite) to black (wurtzite, magnetite, pyrite) and reaching up to brown (siderite, iron oxychloride) [20]. The red colour on both the blade and the handle indicates the presence of hematite (Fe_2O_3).

Compared to the blade, the corrosion layer of which is in the form of a relatively compact and rather thick crust, the handle does not show cracks in the corrosion layer, which, moreover, is much thinner.

The good state of preservation of the handle is most likely also due to the arrangement of the bones of the deceased around it. The fact that certain elements of the environment can migrate into iron objects deposited in the soil certainly validates the theory that phosphates and carbon derived mainly from bones, along with other compounds of an organic nature produce a protective layer for archaeological pieces of iron [21].

At the same time, the slag marks on the handle indicate that this was subjected to burning at a high temperature, most likely even on the funeral pyre.

X-Ray fluorescence spectometry

As shown in Fig. 6, EDXRF qualitative analysis for the blade sword shows iron (Fe) as major element, and other minor elements: Si, Pb, Cr, Ni, Cu, while for the sword handle shows iron (Fe) as major element, and other minor elements: Si, Pb, Cr, K, Ca.

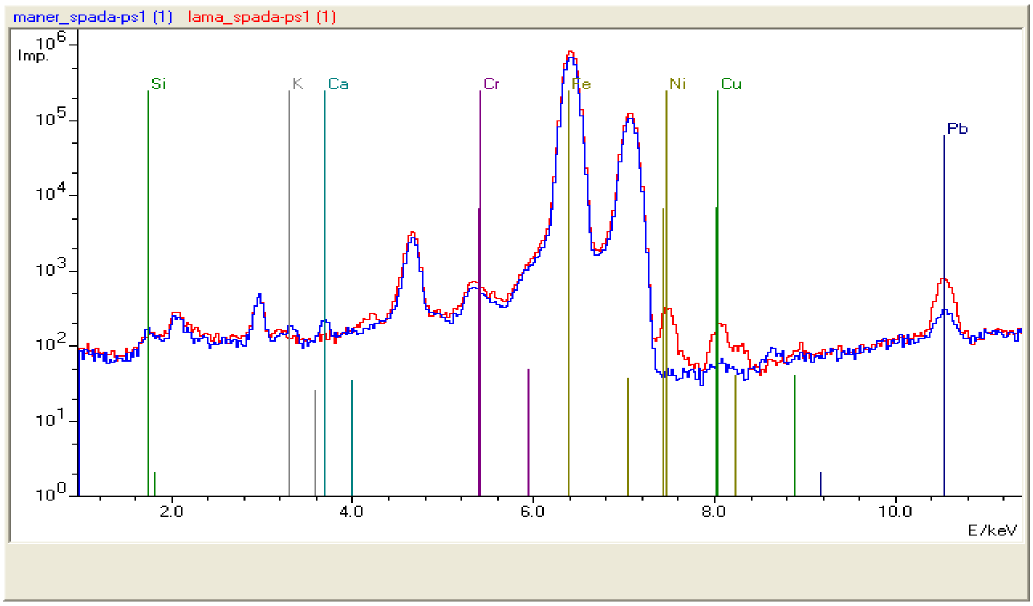

Figure 6. The overlapped EDXRF spectra for the iron blade (red) and sword handle (blue)

Table 1. Quantitative EDXRF analysis (element %) of the principal elements of the blade sword

Element	Fe	Si	Pb	Cr	Ni	Cu
Weight [%]	98.04	0.4440	0.1878	0.01990	0.0782	0.0367

Table 2. Quantitative EDXRF analysis (element %) of the principal elements of the sword handle

Element	Fe	Si	Pb	Cr	K	Ca
Weight [%]	97.95	0.6490	0.0382	0.0217	0.0782	0.030

As can be observed in both Tables 1 and 2, the share of iron, 98.04%, respectively 97.95%, in the total elements is the majority. The very large amount of iron identified in the analysed samples is not a surprise, because this has already been encountered in the case of iron objects made and discovered in Dacian environment, especially in the metallurgical area of Sarmizegetusa Regia, where they reached the same value as in the case of the Buridava sword. Basically, the weapons and tools from Sarmizegetusa Regia, where the other elements summed up (excepting Fe) represented 2% of the total [22].

Furthermore, on the characterization of the iron blooms from which the weapons and tools were made, regarding the other elements accompanying the iron, the same results from Sarmizegetusa Regia, highlight the fact that, apart from carbon, these elements had reached the bloom at the same time with the iron, they had come from the ore, not being intentionally added [22].

It is very likely that the same thing happened in the case of the raw material of which the sword from Ocnița was made, with the mention that, in the case of the handle, Ca could have come from the bones of the deceased which were in the vicinity, as can be seen in Fig. 1.

Regarding the presence of silicon, just as it has been argued in other cases where Dacian iron weapons and tools, from the same period as the sword from Ocnița, were subjected to metallographic analysis, it is confirmed that it is used in the technological process by the craftsmen. Eighteen samples taken from Dacian weapons and tools from Malja Kopanja thus presented inclusions coming from the sand used by blacksmiths to dissolve the limestone when the surface of the semi-finished products or finished parts was heated, the presence of these inclusions proving the use of heating, respectively forging and welding [23].

Concerning the other elements represented by Pb, Cr, K, Cu, Ni, considering their small weight, they come either from the ore from which the iron was obtained or from the process of reducing the iron made in special furnaces used for this purpose, with the introduction into the furnace of rocks with a fondant role [16], or from the reactions occurring during the process of reducing the ore inside the furnace. This is also the case of copper discovered alongside the iron slag recovered from the ore-reduced furnaces discovered at Grădiștea Muncelului [22].

X-ray scanning using the tomograph computer

X-ray scanning performed under special technical conditions involved the realisation of a number of 1440 frames, which were later integrated with the help of CTPro3D software, software developed by Nikon Metrology, thus obtaining the 3D images from Figs. 7 and 8.

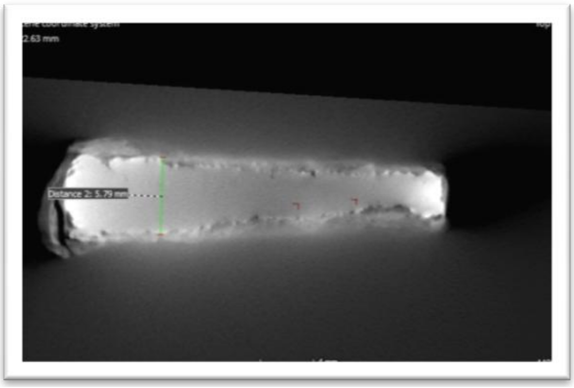

Figure 7. Cross-section image for the iron blade sword

Figure 8. Overview for the iron blade sword

After the virtual penetration through the corrosion layer, a first piece of information obtained when viewing the 3D images consisted in obtaining the dimensions of the sword fragment. Thus, the total length of the blade, with the remainder from the broken handle, is 100.72 mm, the width of the blade is 28.51 mm, and the maximum thickness is 5.79 mm.

The massive image of the intact iron core shows that originally, the weapon was intended to be an extremely robust one, which fits perfectly into the La Tène era, an era when swords, due to the evolution of combat tactics, had to become more solid and rather heavy [24]. Another valuable piece of information obtained after penetration through the corrosion layer is that the blade was not sharpened. It can also be seen that the iron core is very robust even today, the virtual penetration of the entire blade showing that the massive core does not contain any imperfections, which implies a thorough and long-lasting hammering and a high level of knowledge of the craftsman who created the blade.

The swords from the La Tène period have several characteristics, among which are that they are double-edged weapons, or as seen in Fig. 7, the blade analysed with the help of X-rays was made by the blacksmith as if it had to be sharpened only on one side, which raises a series of questions about the role of the blade discovered next to the handle. We are going to use the same analogies from Malaja Kopanja, because there, during other metallographic analyses, it was found that several two swords had only one edge [25], which means that the sword from Buridava is not the only one of its kind.

4.2. RESULTS OF SOIL ANALYSIS

Scanning electron microscopy with energy dispersive spectroscopy

In order to see how the sword was influenced by the soil in which it was deposited, 2 samples of compact soil were taken, respectively dacitic tuff (sedimentary rock based on pyroclastic material coming from volcanic eruptions) and their characterization by SEM-EDS. The weapons, including the sword that is the subject of this study, were deposited either in the tombs represented by pits dug in the dacitic tuff, or directly on this type of rock. It was thus proceeded to characterise a sample of tuff coming from the archaeological excavation and used to fence the tomb, respectively a sample taken from the depth, from the compact layer of tuff at the base of the hill on which the sword was discovered. Backscattered electrons SEM images from Figs. 9 and 10 shows particles morphology for the two types of soil samples.

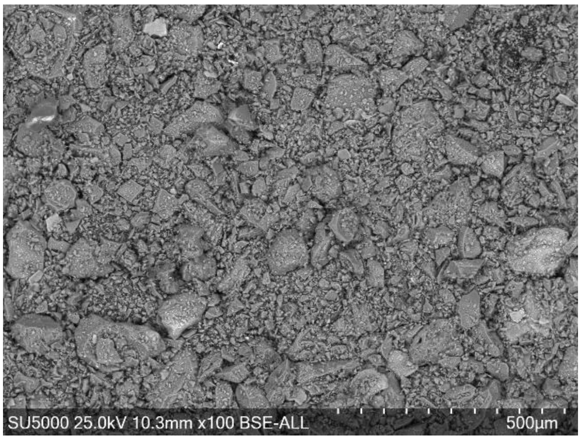

Figure 9. Backscattered electrons SEM image at x100 magnification for the dacitic tuff sample collected during the archaeological research

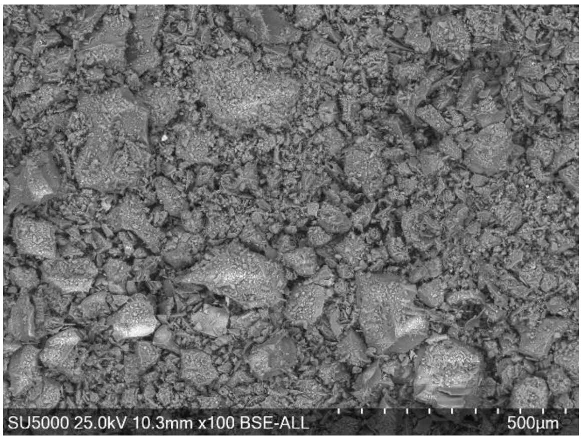

Figure 10. Backscattered electrons SEM image at x100 magnification for the dacitic tuff sample collected at the base of the hill

The qualitative EDS chemical elemental analysis for the two soil samples obtained by area scanning is presented in Fig. 11, while the quantitative data is presented in Table 3.

Table 3. The EDS quantitative analysis for the two soil samples

Element	dacitic tuff sample collected during the archaeological research		dacitic tuff sample collected at the base of the hill	
	Wt%	Wt% Sigma	Wt%	Wt% Sigma
O	53.53	0.08	54.01	0.09
Na	1.31	0.02	2.00	0.03
Mg	0.60	0.02	0.56	0.02
Al	7.34	0.03	6.19	0.03
Si	30.31	0.06	26.98	0.06
P	0.07	0.01	0.09	0.01
S	0.05	0.01	0.07	0.01
Cl	0.05	0.01	0.34	0.01
K	2.61	0.02	2.23	0.02
Ca	0.49	0.01	4.87	0.02
Ti	0.21	0.01	0.15	0.01
Cr	0.04	0.01	-	-
Fe	3.39	0.03	2.51	0.03
Total:	**100.00**		**100.00**	

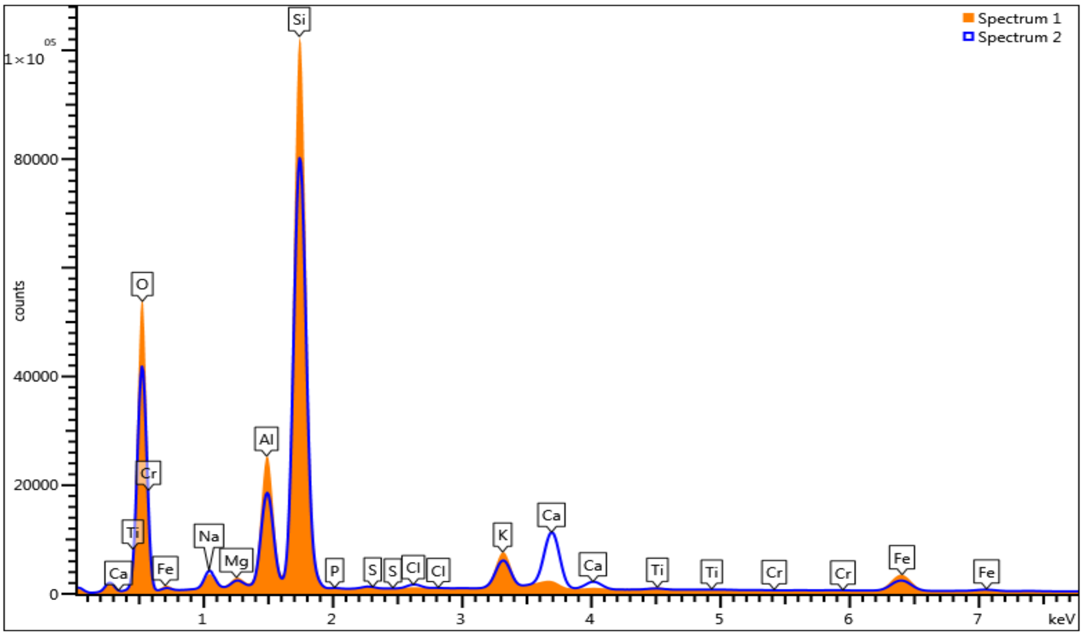

Figure 11. The overlapped EDS spectra for the dacitic tuff sample collected during the archaeological research (Spectrum 1 – orange) and for the dacitic tuff sample collected at the base of the hill (Spectrum 2 – blue)

The EDS analysis shows a Si based matrix with oxides formation and significant quantities of Al, Fe, K, Na, Ca and some other minor elements. It can be observed that the dacitic tuff sample collected at the base of the hill has higher amonts of Ca, Na, Cl and less Al, Si, Fe comparing to the dacitic tuff sample collected during the archaeological research.

Figs. 12 and 13 shows the EDS mapping performed on the two types of soil samples. It shows a relative homogen dispersion for Si, O, Al for both soil samples some large concentrations of K, Fe, Na, Ca, Mg for the dacitic tuff sample collected during the archaeological research and small area concentrations of Fe, Ca, Mg for the dacitic tuff sample collected at the base of the hill.

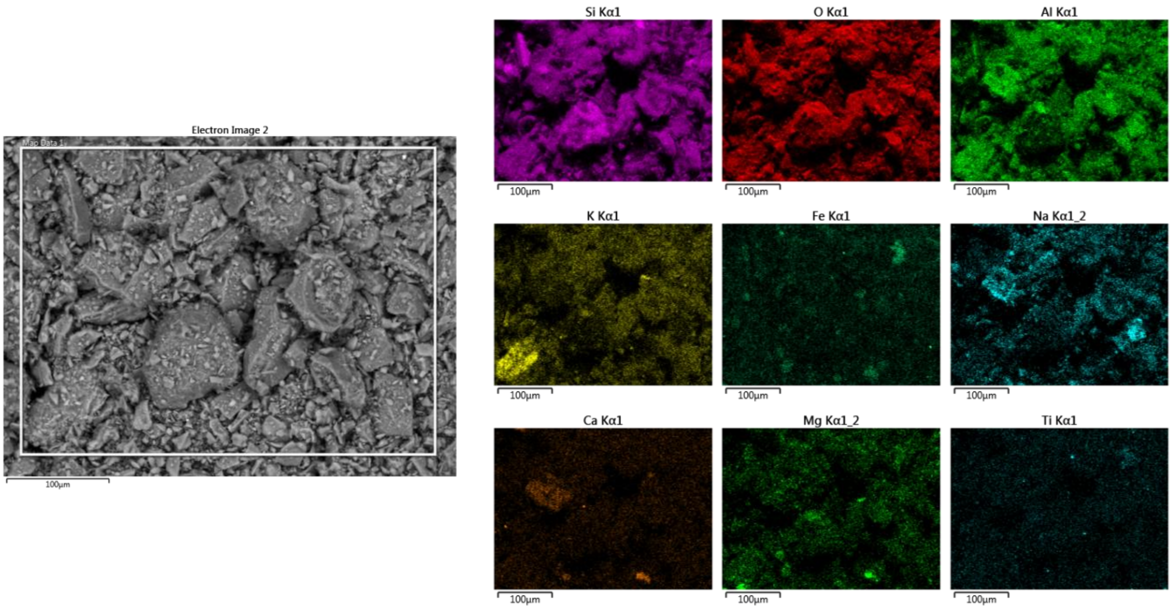

Figure 12. Electron image (left) and EDS mapping (right) of Si, O, Al, K, Fe, Na, Ca, Mg and Ti obtained for the dacitic tuff sample collected during the archaeological research

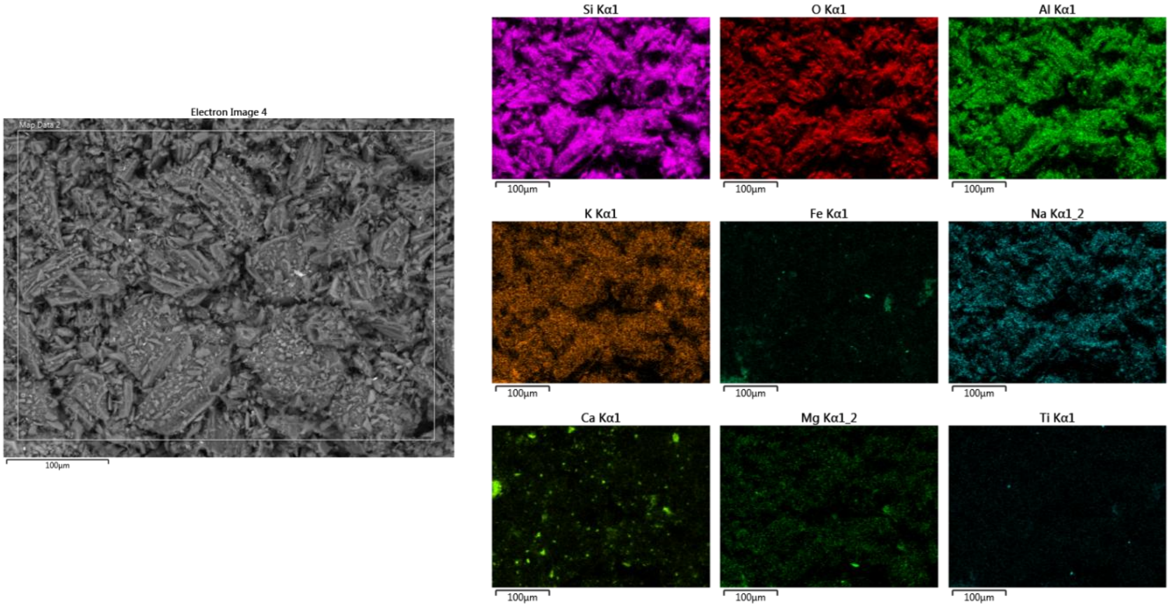

Figure 13. Electron image (left) and EDS mapping (right) of Si, O, Al, K, Fe, Na, Ca, Mg and Ti obtained for the dacitic tuff sample collected at the base of the hill

X-ray diffraction

For the same reasons as EDS, the XRD analysis of the dacitic tuff was made both on a sample taken from the archaeological excavation and on a sample taken at the base of the hill on which the archaeological site is located.

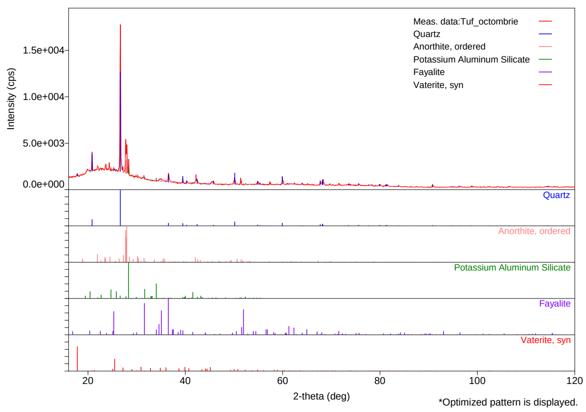

Figure 14. Qualitative Analysis of the XRD dacitic tuff from the archaeological research

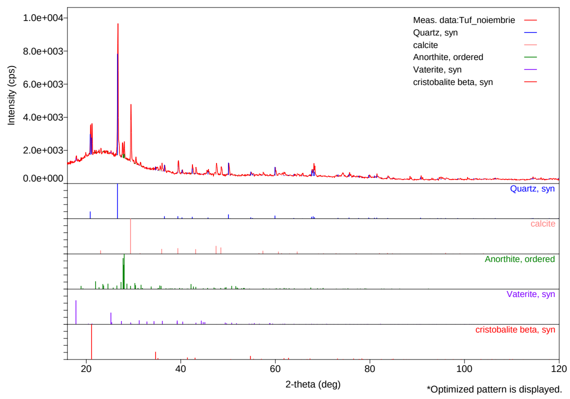

Figure 15. Qualitative Analysis of the XRD dacitic tuff from the base of the hill

Following the qualitative analysis of the dacitic tuff with the help of XRD, it was found that in the sample taken from the archaeological site, according to Fig. 14, the identified phases are: quartz, anorthite, potassium aluminum (kalsilite), fayalite, vaterite. Regarding the qualitative analysis of the dacitic tuff with the help of XRD of the sample collected at the base of the hill, so from the native rock on which the necropolis from where the sword was extracted is located, it was found that in the sample taken, according to Fig. 15, the identified phases are represented by quartz, calcite, anorthite, vaterite, cristobalite. If the quartz comes from the soil, the other chemical or mineral elements in the tuff should be analysed according to the impact they have on the iron weapons buried in it. Thus, the anorthite, $Ca(Al_2Si_2O_8)$, which is found, moreover, in both samples, is a fairly common element, especially in areas with volcanic eruptions [26], as is the case with the Buridava site, located in an area affected by volcanic eruptions. As for the presence of potassium aluminum (kalsilite)- $KAl(SiO_4)$, this is explained by the fact that it is a mineral found in the groundmass of some potassium-rich and silica-deficient lavas and tuffs [27].

The presence of fayalite, $Fe_2(SiO_4)$, along with fosterite, Mg_2SiO_4, is confirmed in many volcanic rocks where sodium is more common than potassium, and forsterite-fayalite minerals also occur in dolomitic limestones, in marble and in transformed sediments, rich in iron, with the mention that these minerals are relatively infusible, do not melt below 1500°C and are sometimes used in the manufacture of refractory brick [27].

The presence of the vaterite, $Ca(CO_3)$, can be explained as it is a major constituent of a carbonated calcium silicate hydrogel complex formed from larnite, a rock-forming mineral formed at low temperatures by hydration of metamorphic calc-silicate rocks in the presence of atmospheric CO_2, in slightly metamorphosed marls and conglomerates, and in weathering crusts [27].

As for the sample collected from the base of the hill, new phases of calcite and cristobalite appear in it. The presence of the calcite, $Ca(CO_3)$, is based on the fact that it is a major rock-forming mineral found in limestones, marbles, chalks, as a common cement in clastic sedimentary rocks [27]. Furthermore, cristobalite, SiO_2 represent a late-crystallizing phase in basaltic to rhyolitic volcanic rocks, from acid-sulfate-type hydrothermal alteration of volcanic rocks [27].

5. CONCLUSIONS

The macrographic and microscopic analysis of the sword fragments reveals, at least in the case of the handle, that it has suffered a strong burning, as demonstrated by the molten slag deposited on it. The fact that, unlike the blade fragment, which is covered with a consistent layer of corrosion products, the handle is in a better state of preservation, and the corrosion layer is not so deep, is due to the calcium from the bones which the handle was in direct contact with. Even if the blade fragment seemed more affected by the corrosion process, the analysis of the results obtained with the help of X-ray scanning proved that the iron core is a very robust and healthy one, without imperfections, which denotes an extremely rigorous hammering performed by the craftsmen. The large amount of iron resulting from the characterization with the help of EDXRF of 97.95% in the case of the handle, respectively 98.04% in the case of the blade represents a large amount of iron, which implies that the weapon in question is comparable from this point of view with the products made at Sarmizegetusa Regia where there was perhaps one of the most important metallurgical centres in Europe of the I BC – I AD centuries. Moreover, the fact that we are dealing with the existence of calcium only concerning the handle, this is probably due to the fact that, unlike the blade, only the handle was surrounded by fragments of bones.

The CT analysis reveals an important series of new data for the study of Dacian military equipment. Thus, the way in which the blade is made (with a single edge) denotes that we are dealing with a type of sword that is rare in archaeological discoveries, and which although it can be classified among the type II swords in terms of the handle, the entire categorisation of swords must be reconsidered, based on the latest discoveries, this type of weapon being part of a subcategory of this type.

Furthermore, the fact that it is deposited in the tomb without being sharpened, a fact observed very clearly with the help of CT, implies a number of theories regarding the funeral ritual. The characterization of the dacitic tuff which represents, on the one hand, the rock which the necropolis was made on and where the sword was discovered, and, on the other hand, the constructive material used to fence the tomb was made both by scanning electron microscopy with energy dispersive spectroscopy and by X-ray diffraction. The results obtained revealed that, for the most part of them, the minerals which make up the tuff are of volcanic origin and, generally, they have positive properties in maintaining a state of good conservation. This can also explain the fact that not only the sword fragments which are the subject of the study, but also many other artefacts of iron, bronze, copper have been preserved sometimes in excellent conditions given their age of about 2,000 years.

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