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# Studies

## ARCHAEOOMETRY

### FRAGMENTARY DISCOVERIES WITHOUT CONTEXT – WHAT CAN WE LEARN MORE? AN ARCHAEOMETRIC APPROACH ON A FORTUITOUS FIND FROM NORTH-EASTERN ROMANIA

**Abstract:** The ‘accidental’ discovery of archaeological material represents an ordinary fact in today’s archaeology. Unfortunately, most of these discoveries have no archaeological context, thwarting the possibility of integrating the objects in more complex studies. In this context, based on the type of data that the archaeometric study of metal objects provides (information regarding prehistoric metallurgy, especially the used alloys and technologies), we propose for analysis two metal pieces (a socketed axe and a sickle) fortuitously found in Dobrovăt (Iași County), in order to see how much information can we gather for two fragmentary objects, without archaeological context. The methods used are optical microscopy (OM), scanning electron microscopy coupled with energy-dispersive X-ray analysis (SEM-EDX) and micro-Fourier transform infrared spectroscopy ( $\mu$ FTIR) and our aim is represented by the obtainment of important information about patina and corrosion products, the metals used and how these objects were manufactured and utilised. As a result, the before mentioned methods illustrated a number of special features regarding the metallurgical practice of the Late Bronze Age in the Eastern Carpathian area, contributing to the expansion of the database in this chronological and geographical area.

**Keywords:** OM, SEM-EDX, FT-IR, Late Bronze Age, NE Romania

#### INTRODUCTION

The fortuitous discovery of artifacts without an archaeological context is, unfortunately, a fact more and more present in the archaeological world, these objects being considered as mere isolated discoveries and interpreted, most of the time, only from a statistical point of view. However, there are various methods of analysis that can provide a lot of information, even without knowing where the pieces were discovered. In this sense, in the present study we wanted to highlight a series of unique data, obtained as a result of the interdisciplinary analysis of a discovery with no archaeological context, from Dobrovăt area, Iași County (Fig.1). There were identified two metal fragments, from a socketed axe and a sickle, along with a ceramic fragment from a large vessel that, at first glance, suggests a

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**Fig.1.** Dobrovăț administrative unit and its location within the territory of Romania; source of DEM: LiDAR data from Romanian Water Administration, Prut-Bârlad branch, 10x10 m resolution (ArcGIS Pro 2.5; CorelDRAW 2020).

possible attribution to the Early Hallstatt period. Regarding the shard, it should be mentioned from the beginning that we do not have specific information regarding the place of discovery (either it was identified within the same context as the two metal pieces, or in Dobrovăț area, in general). The discovery of the two metal artifacts in a fragmentary state makes their typological classification difficult, as they are missing the upper part which, for Late Bronze Age and the beginning of the Iron Age, allows the differentiation of the subtypes and a more accurate chronological framing. Nevertheless, the proposed study allowed the identification of features that can facilitate the cultural and chronological attribution of the pieces.

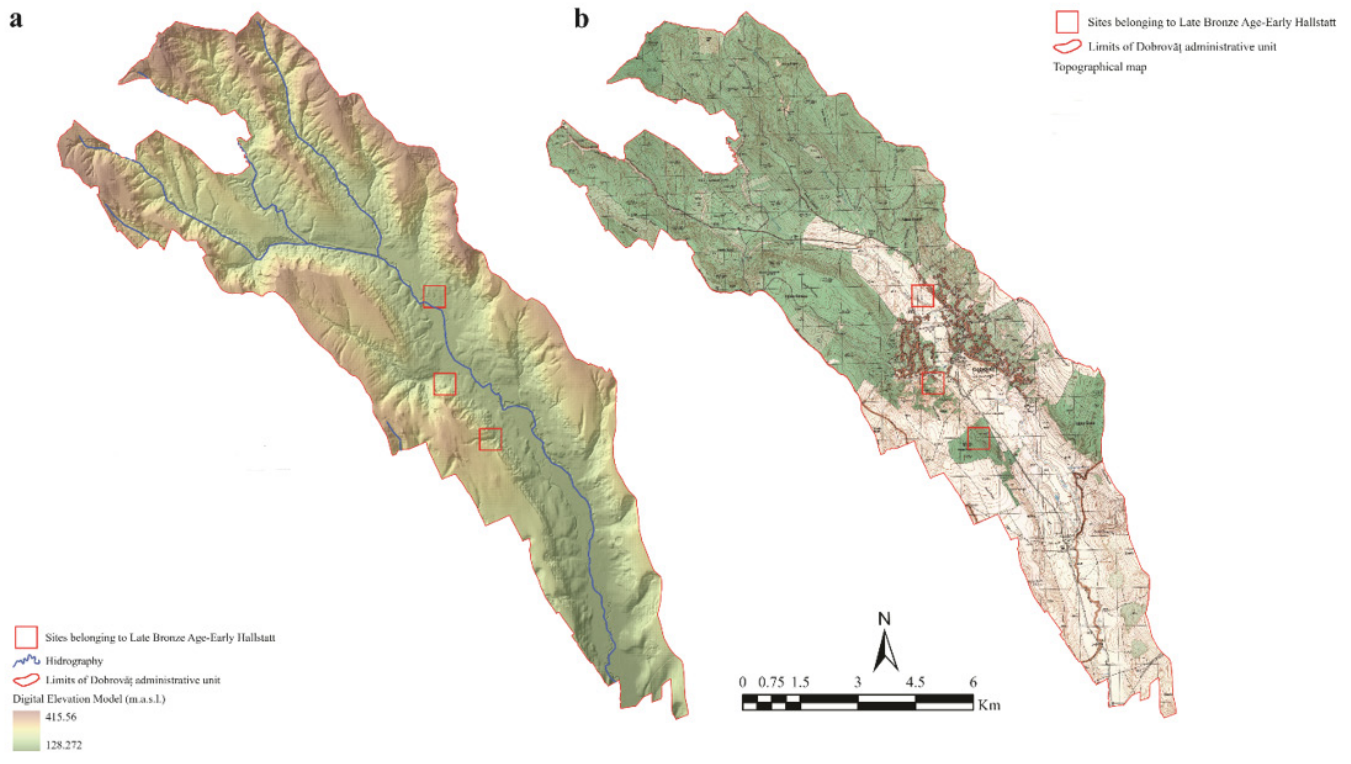
Despite the relatively high altitudinal average (260 m.a.s.l., max=415 m.a.s.l.) and the presence of heavily forested areas, the territory of Dobrovăț commune presented interest for prehistoric communities, being frequented since the Chalcolithic period. Regarding the discoveries from the chronological interval of interest (end of the Bronze Age - beginning of the Iron Age), three contexts were reported in the studied area: in the eastern extremity of the village was discovered a ceramic fragment from a large vessel with a simple clay strip, which had grog into paste, and was attributed to the end of the Bronze Age; in the place called *Buda Hill* a possible *krummesser* was identified, belonging to the same period; in the place known as *Tarlaua Jităria*, an intense habitation from the beginning

of the Iron Age has been reported<sup>1</sup>. Overall, it is observed that all three discoveries are located near the Dobrovăț River, the main source of water in this micro-area (Fig.2). With the exception of the Hallstattian settlement, the other two findings can be considered isolated and their location seems to fit the pattern already noticed for the Late Bronze Age communities<sup>2</sup>. During this period, the entire territory from the middle and upper Dniester, to the east of the Apuseni Mountains and from the sub-Carpathian region of Ukraine to the southern forest-steppe zone between Siret and Prut Rivers was occupied by the human groups of the cultural complex Noua-Sabatinovka-Coslogeni<sup>3</sup>. These communities practiced cattle shepherding, as evidenced by the presence of the so-called ashmounds (grey spots, visible on aerial photographs, that usually have diameters of 20-30 m). Unfortunately, we weren't able to identify such features on the available ortophotoplans so that, for now, we cannot tie the isolated discoveries to any Noua settlements. Regarding the characteristics of the material component, Noua culture is represented by double-handled kantharoi (present especially in funerary contexts), large vessels with simple clay strips, curved knives made of flint (*krummesser*), crenated scapulae and *tupik* sickles. Also, amongst the 'markers' of identification for these groups, we have to mention the high

<sup>1</sup> CHIRICA/TANASACHI 1984, 124-128.

<sup>2</sup> FLORESCU 1964, 146; DASCĂLU 2007, 78; SAVA 2014, 406; NICULICĂ 2015, 367.

<sup>3</sup> PETRESCU-DÎMBOVIȚA 2010, 276.



**Fig.2.** Spatial distribution of Late Bronze Age-Early Hallstatt discoveries on: a - hypsometric map; source of DEM: LiDAR data from Romanian Water Administration, Prut-Bârlad branch, 10x10 m resolution. b – topographical map, 1:25.000; source: Military Topographic Directorate (ArcGIS Pro 2.5; CoreLDRAW 2020).

number of animal bones, present in all settlements, due, most likely, to the type of economy practiced.

**MATERIALS AND METHODS**

A first step in the current approach was represented by the documentation of all similar discoveries from Dobrovăț area. After identifying three such contexts, they were visualized and mapped using ArcGIS PRO 2.5. Also, in the attempt to identify ashmounds characteristic to Noua communities, we used satellite images, ortophotoplans and LiDAR measurements (these features offer elevational responses, due to the aspect that these structures acquire over time, being similar to small mounds).

The main focus of the study is represented by the chemical analysis of the three pieces. The microscopic investigations allow the detailing of certain characteristics of the studied object, the establishment of data related to structure and microstructure, the disposition of corrosion products, as well as the identification of use-wear traces. SEM-EDX analysis provides data related to the microstructure and elemental composition of the studied samples.  $\mu$ FT-IR analysis offers information on the anions ( $Cl^-$ ,  $CO_3^{2-}$ ,  $SO_4^{2-}$ ,  $PO_4^{3-}$ , etc.) and functional groups ( $-NO_2$ ,  $-NH_2$ , etc.) identified in samples. Thus, the results of SEM-EDX and  $\mu$ FT-IR lead to the determination of the elemental compositions and chemical compounds in the analyzed samples.

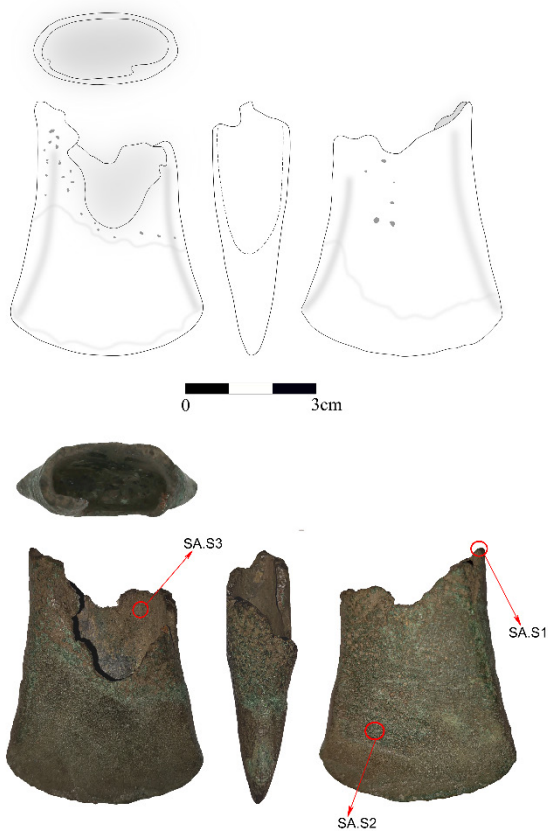
In this regard, the microscopic analysis was performed with a Zeiss Imager.a1M microscope with a built-

in AXIOCAM camera, which uses AxionVisionRelease 4.7.1 software. In this study was used an electron microscope with SEM scan, model VEGA II LSH, produced in the Czech Republic by TESCAN, coupled with an EDX detector type QUANTAX QX2, produced in Germany by BRUKER/ROENTEC. The spectroscopic analysis was recorded with an FTIR spectrophotometer coupled with a HYPERION 1000 microscope from Bruker Optic, Germany. The FTIR spectrophotometer is of the TENSOR 27 type, which is predominantly suitable for measurements in close IR. The standard detector is DLATGS and covers the spectral range  $7500-370\text{ cm}^{-1}$ , working at room temperature. For completely nondestructive measurements, the TENSOR 27 spectrophotometer is connected to the HYPERION 1000 microscope, and, usually, the solid samples are analyzed in reflection mode. Finally, the analyzed samples were noted SA.S (Socketed Axe Sample) and S.S (Sickle Sample), from 1 to 3, showing the different studied areas.

**RESULTS**

The first artifact that we will discuss is represented by the fragmentary socketed axe (Fig.3), that weighs 75.33 g. As stated before, only the lower part of the piece was identified, missing exactly the fragment that would have allowed the typological and chronological attribution of the artefact. However, the presence of the arched edge allows the issuance of possible judgments regarding this matter. Thus, this characteristic is found in two typological categories,

namely within the Ruginoasa<sup>4</sup> and Negrești<sup>5</sup> types, present in the area between the Carpathian Mountains and Prut River, from where they spread towards Transylvania, as well as to the East, until Dnieper River catchment, during the Bz D-Ha A period.



**Fig.3.** The analyzed socketed axe (drawing and photography) and the sampling area (Inkscape 0.92.1; AdobePhotoshop CC 2019).

The *microscopic analysis* highlighted the presence of several corrosion products in different areas. Of these, the carbonates are represented by the green malachite, which shows traces of a botryoid texture (Fig.4/a-e), the blue azurite<sup>6</sup> (Fig.4/b,d,e) being also visible, but only on some areas of the corrosion crust<sup>7</sup>. Also, the depositional environment determined the appearance of chlorides, represented by whitish areas. Moreover, at microscopic level, we were able to identify some defects, such as fine cracks and circular pores of variable dimensions (Fig.4/c). These indicate that the piece was casted using, most likely, a bivalve mould. In addition, there are visible fine scratches (Fig.4/g,h), representing use traces, indicating a functional aspect of the object<sup>8</sup>.

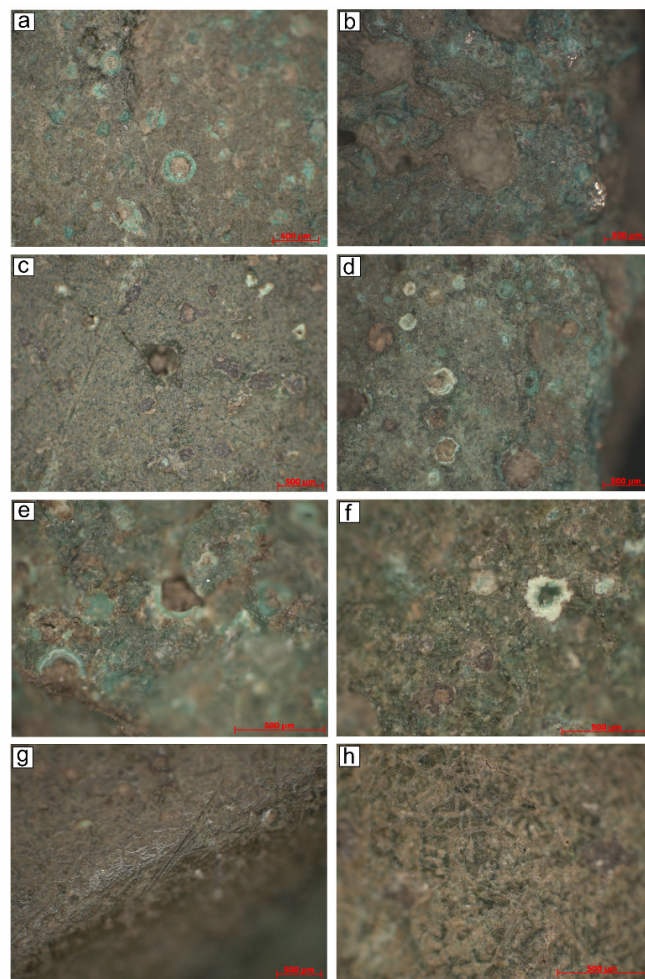
<sup>4</sup> RUSU 1966, 24-26; DERGAČEV 2002, 143-144; DIACONU 2014, 199-200; LAZANU 2016, 54.

<sup>5</sup> RUSU 1966, 26; DERGAČEV 2002, 137-141; DIACONU 2014, 199; LAZANU 2016, 54-55.

<sup>6</sup> MIRCEA *et alii* 2012, 1467.

<sup>7</sup> BARON/MIAZGA/NOWAK 2014, 334.

<sup>8</sup> BARON *et alii* 2020, 7/Fig.5.



**Fig.4.** Optical microscopy of the analyzed socketed axe. a-c. corrosion products (50× magnification); e-f. corrosion products (10× magnification); g-h. Surface use-wear (g-50×; h-100×) (Inkscape 0.92.1; AdobePhotoshop CC 2019).

During the depositional processes, metal artifacts undergo changes caused by the depositional environment, leading to a series of physical and chemical changes<sup>9</sup>. In this sense, in order to identify some archaeometric characteristics, the metallic core and the corrosion crust of the studied objects must be investigated<sup>10</sup>.

Thus, the *SEM-EDX analysis* was performed both on the metal core and on the surfaces, in the interest of identifying the elements of the alloy and the corrosion crust (Fig.5/Table 1). In this sense, for the basic alloy, the analyzes were performed on three distinct areas of the metallic core (Fig.3/SA.S1), with unitary results, their average being calculated and used. The socketed axe was manufactured from a Cu (86.35%), Sn (5,87%), Pb (6,19%) and O (1,57%) alloy, no other trace elements being identified. Alloying Cu with a 5-10% Sn<sup>11</sup> concentration, leads to the obtaining of a yellow-gold color, making the object eye-catching, aspect that could indicate the symbolic function of the artefact, besides its utilitarian role.

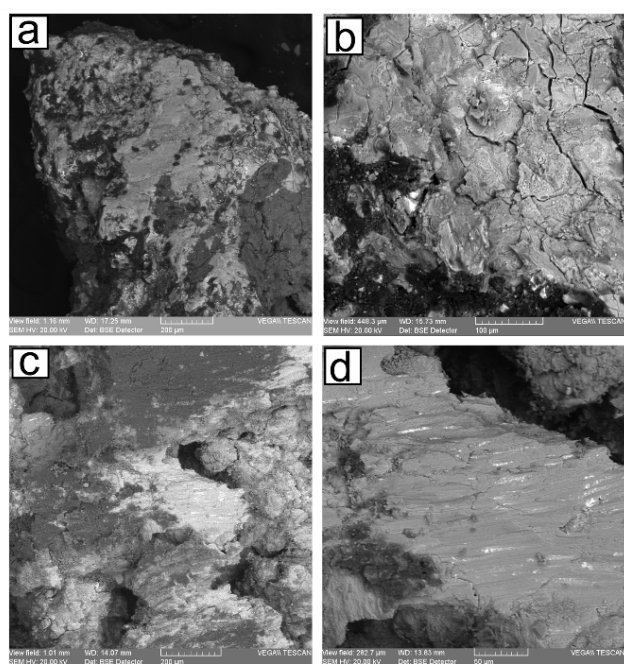
<sup>9</sup> MIRCEA *et alii* 2012, 1467.

<sup>10</sup> SANDU *et alii* 2012, 1651.

<sup>11</sup> CORNACCHIA/ROBERTI/FACCOLI 2020.

**Table 1.** Elemental composition of the analyzed socketed axe (AdobePhotoshop CC 2019).

Sample	Elemental Composition in Weight Percent (%)														
	Cu	Sn	Pb	P	Al	Si	K	Ca	Na	Mg	Fe	Cl	S	C	O
Socketed axe (core 1)/SA.S1	84,31	6,71	7,16	0	0	0	0	0	0	0	0	0	0	0	1,82
Socketed axe (core 2)	86,01	5,37	6,66	0	0	0	0	0	0	0	0	0	0	0	1,95
Socketed axe (core3)	88,74	5,55	4,75	0	0	0	0	0	0	0	0	0	0	0	0,95
Socketed axe (corrosion 1)/SA.S2	15,99	9,93	0,67	1,43	1,45	3,12	0	0	0	1,04	8,80	0,67	0,29	13,05	43,54
Socketed axe (corrosion 2)/SA.S3	19,48	4,50	0,66	0,26	4,87	13,14	2,23	0,33	0,60	0,89	5,43	0,46	0,18	9,15	37,78

**Fig.5.** SEM micrograph of the analyzed socketed axe. a-b. Corrosion crust; c-d. Metallic core (Inkscape 0.92.1; AdobePhotoshop CC 2019).

The over 5% Pb concentration of the alloy shows an intentionality of the process<sup>12</sup>, being accepted that the addition of lead in small quantities decreases the melting point of the alloy and increases the fluidity of the metal, making the casting more efficient. However, alloying with an appreciable amount of lead (> 20%) conducts to a possible breakage of the object due to the low solubility of lead in copper, which causes segregation in multiple areas, causing the heterogeneity of the alloy<sup>13</sup>. Although the concentration of Pb in the alloy is small, in the SEM images of the metal core the segregation process of Pb is visible in the form of whitish areas (Fig.5/c,d), their dimensions having no negative effect on the quality of the object.

The analysis of the object's surface (Fig.3/SA.S2, SA.S3) revealed elements specific to corrosion products<sup>14</sup>

<sup>12</sup> PERNICKA 2014, 256.

<sup>13</sup> MONTERO *et alii* 2003, 39.

<sup>14</sup> MIRCEA *et alii* 2012, 1473.

such as Cl and S, belonging to atacamite ( $\text{Cu}_2\text{Cl}(\text{OH})_3$ ) and brochantite ( $\text{Cu}_4\text{SO}_4(\text{OH})_6$ ), being also present elements of soil contamination during the depositional processes (C, P, Al, Si, K, Ca, Na, Mg, Fe)<sup>15</sup>.

The FT-IR analysis performed on the surface (Fig.6) led to the identification of some compounds resulted from the corrosion processes and the contamination from the depositional environment. The water region between 4000-3000  $\text{cm}^{-1}$  is well represented by the OH groups in the corrosion crust, the absorbed water<sup>16</sup> being present also at 1629  $\text{cm}^{-1}$ . The intense peaks at 2928  $\text{cm}^{-1}$  and 2859  $\text{cm}^{-1}$  are attributed to organic carbon<sup>17</sup> resulted by the decomposition of organic matter in the soil, and those at 701  $\text{cm}^{-1}$  and 636  $\text{cm}^{-1}$  are specific to the silicates<sup>18</sup> from the same environment, these compounds being the result of post-depositional contamination. The peaks at 3583  $\text{cm}^{-1}$ , 3548  $\text{cm}^{-1}$ , 3512  $\text{cm}^{-1}$  and 2429  $\text{cm}^{-1}$  are attributed to lead carbonates<sup>19</sup> ( $\text{PbCO}_3$ ), present in the specific region from 1300-1500  $\text{cm}^{-1}$ . Other carbonate corrosion products<sup>20</sup> identified are the azurite<sup>21</sup> ( $\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$ ) present at 3454  $\text{cm}^{-1}$  and the malachite<sup>22</sup> ( $\text{Cu}_2[(\text{OH})_2 | \text{CO}_3]$ ) identifiable at 806  $\text{cm}^{-1}$ . From the category of sulphates was identified the brochantite<sup>23</sup> ( $\text{Cu}_4\text{SO}_4(\text{OH})_6$ ), at 1156  $\text{cm}^{-1}$  and the atacamite type chloride<sup>24</sup> ( $\text{Cu}_2\text{Cl}(\text{OH})_3$ ) at 925  $\text{cm}^{-1}$ . All these corrosion products have also been identified by optical microscopy as well as by elemental composition.

Next, we will turn our attention to the sickle fragment (Fig.7), that weighs 15.15 g. Although, unfortunately, we do not possess the upper part of the sickle, namely the handle, the shape of the blade and the place of discovery could suggest a possible attribution to the so-called East-Carpathian series, more precisely Ghermănești type<sup>25</sup>, belonging to Noua-Sabatinovka-Coslogeni cultural complex.

<sup>15</sup> MIRCEA *et alii* 2012, 1472.

<sup>16</sup> PAPADOPOULOU/VASSILIOU 2021, 236/Tabel 4.

<sup>17</sup> COLUMBINI *et alii* 2005; MARITAN 2020.

<sup>18</sup> VASILACHE *et alii* 2013; VASILACHE/KAVRUK/TENCARIU 2020.

<sup>19</sup> RAMPAZZI *et alii* 2017, 146.

<sup>20</sup> KOTLAR *et alii* 2021.

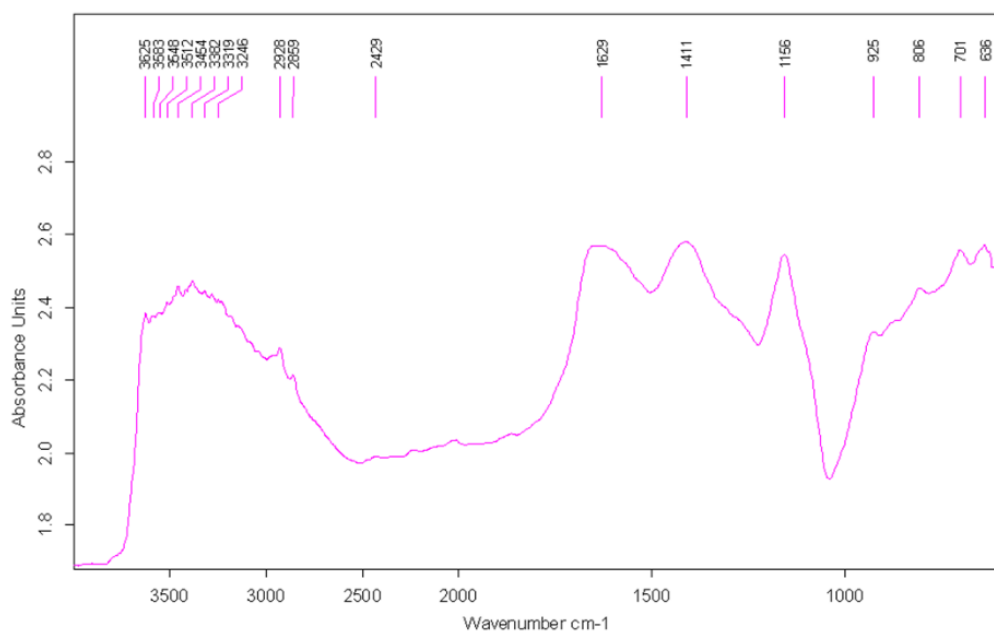
<sup>21</sup> VASILACHE *et alii* 2013.

<sup>22</sup> BARON/MIAZGA/NOWAK 2014, 335.

<sup>23</sup> PAPADOPOULOU/VASSILIOU 2021, 236/Tabel 4.

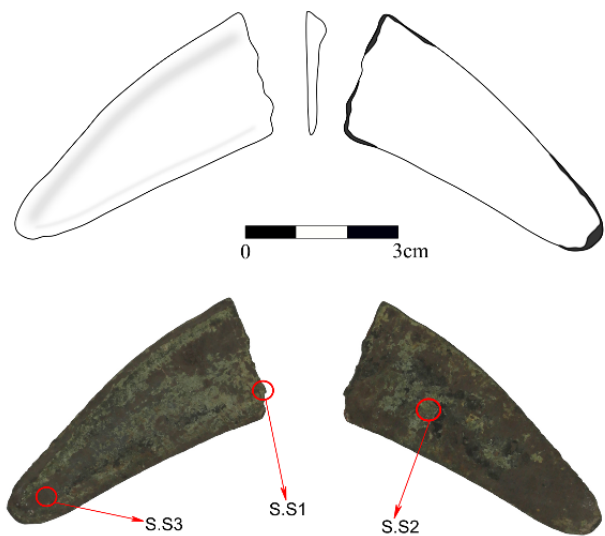
<sup>24</sup> PAPADOPOULOU/VASSILIOU 2021, 236/Tabel 4.

<sup>25</sup> DERGAČEV/BOČKAREV 286-229, 2006.



**Fig.6.** FT-IR spectra of the socketed axe patina (Inkscape 0.92.1; AdobePhotoshop CC 2019).

The *microscopic analysis* of the sickle’s surfaces revealed the presence of several types of corrosion products, such as the green ones represented by malachite (Fig.8/a,d), the black ones known as tenorite (Fig.8/c) and the reddish called cuprite<sup>26</sup> (Fig.8/b). Tenorite and cuprite appear as a result of the degradation or oxidation of bodies or deposits of copper sulfur, an aspect that indicates the presence of sulfur in the artefact’s composition. Also, the analysis revealed the presence of use traces<sup>27</sup> (Fig.8/e-h) in the form of depths/grooves on the blade edge, which appear to have resulted from hitting a hard object<sup>28</sup>.

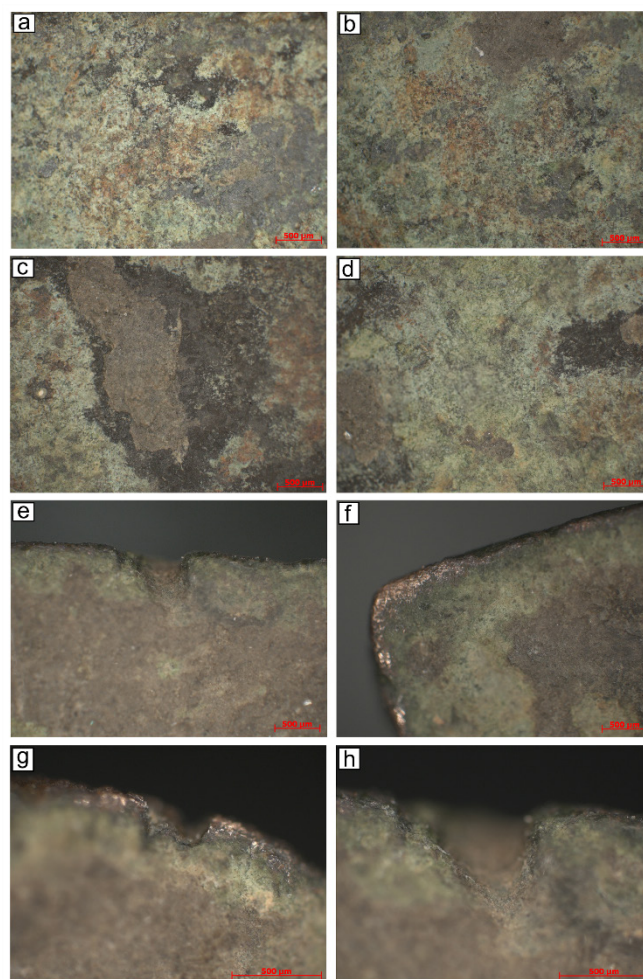


**Fig.7.** The analyzed sickle (drawing and photography) and the sampling area (Inkscape 0.92.1; AdobePhotoshop CC 2019).

<sup>26</sup> BARON/MIAZGA/NOWAK 2014, 334; MIRCEA *et alii* 2012, 1472.  
<sup>27</sup> SYCH *et alii* 2020.  
<sup>28</sup> AMKREUTZ/FONTIJN/GENTILE 2019.

The chemical composition obtained (Table 2) following the three analyzes performed on the metal core of the sickle (Fig.7/S.S1) led to uniform results, the average of which indicated the manufacturing of the object from Cu (92.76%), Sb (2.69%), Fe (1.34%), S (1.25) and O (1.96%).

The presence of these elements indicates the processing and use of tetrahedrite ores ((Cu,Fe)<sub>12</sub>Sb<sub>4</sub>S<sub>13</sub>)<sup>29</sup> during the manufacturing of the object, no other trace elements being identified. In the SEM micrographs (Fig.9/c,d) of the metal core, darker circular areas are observed, represented

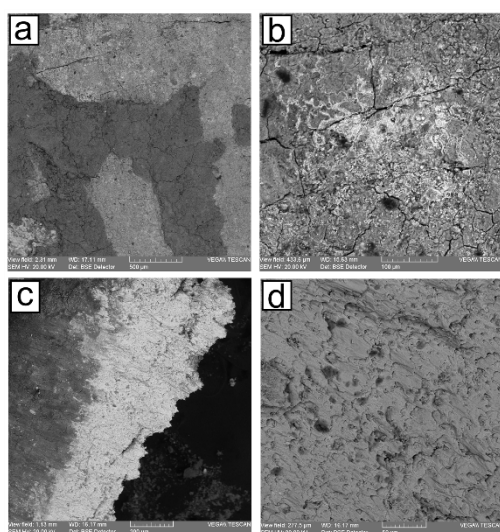


**Fig.8.** Optical microscopy of the analyzed sickle. a-c. corrosion products (50× magnification); e-h. blade use-wear (e, f-50×; g, h-100×) (Inkscape 0.92.1; AdobePhotoshop CC 2019).

<sup>29</sup> KOWALSKI *et alii* 2019, 53.

**Table 2.** Elemental composition of the analyzed sickle (AdobePhotoshop CC 2019).

Elemental Composition in Weight Percent (%)													
Sample	Cu	Sb	Al	P	Si	K	Ca	Mg	Fe	Cl	S	C	O
Sickle (core 1)/S.S1	93,06	2,69	0	0	0	0	0	0	1,35	0	0,64	0	2,25
Sickle (core 2)	93,10	2,75	0	0	0	0	0	0	1,31	0	1,47	0	1,36
Sickle (core 3)	92,13	2,60	0	0	0	0	0	0	1,36	0	1,64	0	2,27
Sickle (corrosion 1)/S.S2	45,79	0	1,83	2,50	7,04	1,52	2,59	0,98	2,65	0,87	2,60	2,99	28,63
Sickle (corrosion 2)/S.S3	9,23	0	7,06	0,88	21,07	2,43	4,16	1,83	5,65	0,27	0,21	0,89	46,31



**Fig.9.** SEM micrograph of the analyzed sickle. a-b. Corrosion crust; c-d. Metallic core (Inkscape 0.92.1; AdobePhotoshop CC 2019).

by sulfur, which in concentrations higher than 1% tends to form inclusions in the metal mass<sup>30</sup>.

The analyzes performed on the exterior areas of the sickle (Fig.7/S.S2, S.S3) revealed the presence of corrosion products such as malachite<sup>31</sup> ( $\text{Cu}_2[(\text{OH})_2|\text{CO}_3]$ ), tenorite ( $\text{CuO}$ ) and cuprite ( $\text{Cu}_2\text{O}$ ), products such as atacamite ( $\text{Cu}_2\text{Cl}(\text{OH})_3$ ) or brochantite ( $\text{Cu}_4\text{SO}_4(\text{OH})_6$ ) being also identified, in smaller quantities<sup>32</sup> (Table 2). In this case, also, were identified elements resulting from soil contamination, such as C, P, Al, Si, K, Ca, Mg, Fe<sup>33</sup>. Although in the elemental composition was identified iron in a significant concentration, the values obtained for the surfaces are much higher,

<sup>30</sup> KOWALSKI *et alii* 2019, 52.

<sup>31</sup> BARON/MIAZGA/NOWAK 2014, 335.

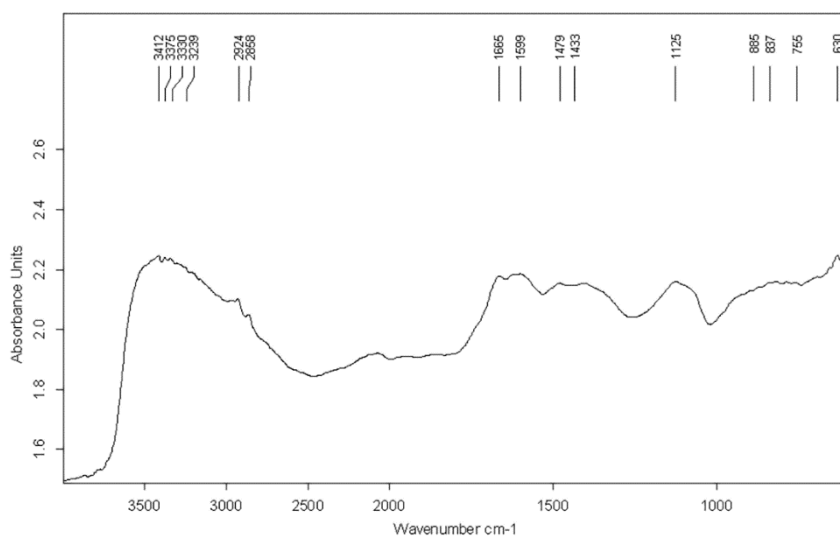
<sup>32</sup> MIRCEA *et alii* 2012, 1473.

<sup>33</sup> MIRCEA *et alii* 2012, 1472.

reaching up to 5%, these representing the result of contamination with the depositional environment.

The FTIR investigations performed on the surface (Fig.10) revealed the presence of chemical compounds from the basic alloy or as a result of soil contamination.

The band assigned to the waters between  $4000\text{-}3000\text{ cm}^{-1}$  is representative for the OH groups of the corrosion crust, and through the  $1665\text{ cm}^{-1}$  peak the presence of the absorbed water is also highlighted<sup>34</sup>. As in the case of the socketed axe, the presence of intense peaks at  $2924\text{ cm}^{-1}$  and  $2858\text{ cm}^{-1}$  is attributed to organic carbon<sup>35</sup>, the silicates<sup>36</sup> in the soil being present at  $755\text{ cm}^{-1}$ , these compounds representing the result of post-depositional contaminations. The region between  $1300\text{-}1500\text{ cm}^{-1}$ , attributed to carbonates<sup>37</sup>, is represented by the obvious peaks of tenorite<sup>38</sup>, cuprite<sup>39</sup> (which is also identifiable at  $630\text{ cm}^{-1}$ ) and malachite<sup>40</sup>, the latter being visible also at  $837\text{ cm}^{-1}$ . Sulphate-type corrosion products



**Fig.10.** FT-IR spectra of the sickle's patina (Inkscape 0.92.1; AdobePhotoshop CC 2019).

<sup>34</sup> PAPADOPOULOU/VASSILIOU 2021, 236/Tabel 4.

<sup>35</sup> COLUMBINI *et alii* 2005.

<sup>36</sup> VASILACHE *et alii* 2013; VASILACHE/KAVRUK/TENCARIU 2020.

<sup>37</sup> KOTLAR *et alii* 2021.

<sup>38</sup> VASILACHE *et alii* 2013.

<sup>39</sup> VASILACHE *et alii* 2013.

<sup>40</sup> BARON/MIAZGA/NOWAK 2014, 335.



**Fig.11.** The analyzed pottery shard (Inkscape 0.92.1; AdobePhotoshop CC 2019).

are represented by brochantite<sup>41</sup> ( $\text{Cu}_4\text{SO}_4(\text{OH})_6$ ) at  $1125\text{ cm}^{-1}$  and the chlorides<sup>42</sup> by atacamite ( $\text{Cu}_2\text{Cl}(\text{OH})_3$ ) at  $885\text{ cm}^{-1}$ , the peaks identified having a low intensity. This can be explained by the low concentrations of S and Cl detected in the EDX analysis. Some of these corrosion products were identified by optical microscopy and compositional investigations, and the presence of all compounds was determined by  $\mu\text{FT-IR}$  analysis.

Last but not least, the ceramic fragment belongs to a large biconical vessel, with a maximum diameter of 46 cm, with an auxiliary element (strip of clay decorated with impressed notches) applied in the supramedian area (Fig.11). The inner and outer color is 10YR6/6 (brownish yellow), the pot being manufactured by coiling technique, well smoothed, but carelessly finished. In this sense, there are visible traces from applying the clay strip and small lumps of clay on the outer surface. The wall thickness of the vessel is 1.06 mm, the paste from which it was made being a coarse one, with inclusions such as grog of appreciable dimensions, angular and sub-angular shape, and a frequency of 20-25%. The exact chronological and cultural framing is difficult due to the size of the ceramic fragment and the lack of archaeological context, but the shape and decoration of the vessel place it in the Hallstattian period.

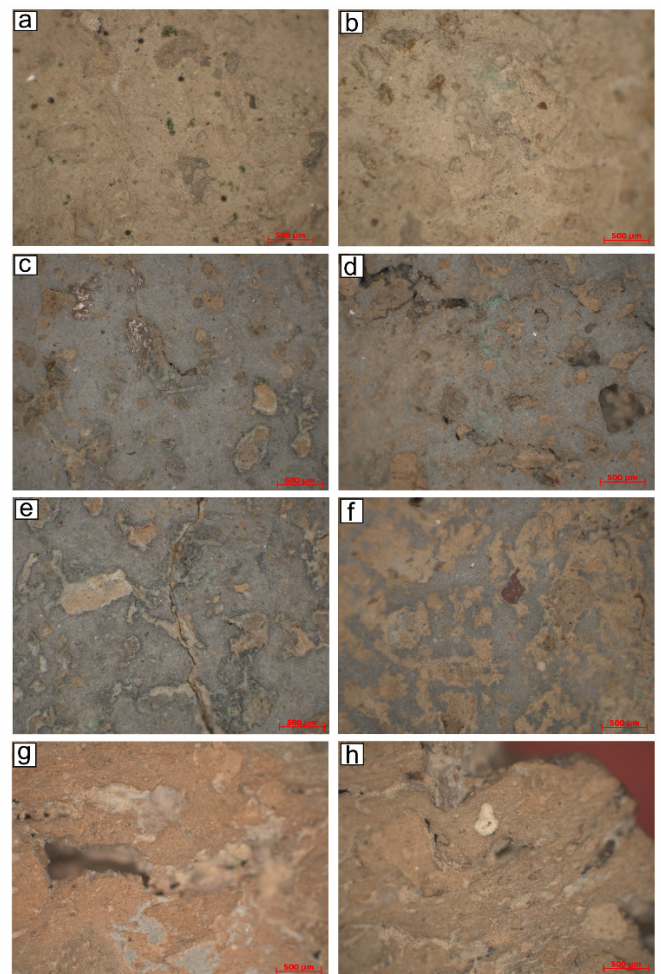
The *microscopic analysis* (Fig.12) revealed a porous paste, with a negligent clay kneading, in which are visible, in addition to grog, natural inclusions such as iron oxides, calcite and traces of organic matter. Also, on the inner surface of the sample, are visible areas with green dot marks, most likely corrosion products of copper (Fig.12/c-e), resulting from the contact between the metal object and the pottery fragment.

In the *SEM micrographs* for the core of the ceramic fragment (Fig.13) well-individualized mineral particles and grog are visible, with a low homogeneity, aspects that suggest relatively low firing temperatures.

The compositional analysis (Table 3) of the ceramic core indicated the presence of chemical elements such as Si, Al, Mg, P, Ca, K, Na, Fe, Ti, O and O, specific to the clay raw material, which is composed of aluminosilicates,

<sup>41</sup> PAPADOPOULOU/VASSILIOU 2021, 236/Table 4.

<sup>42</sup> PAPADOPOULOU/VASSILIOU 2021, 236/Table 4.

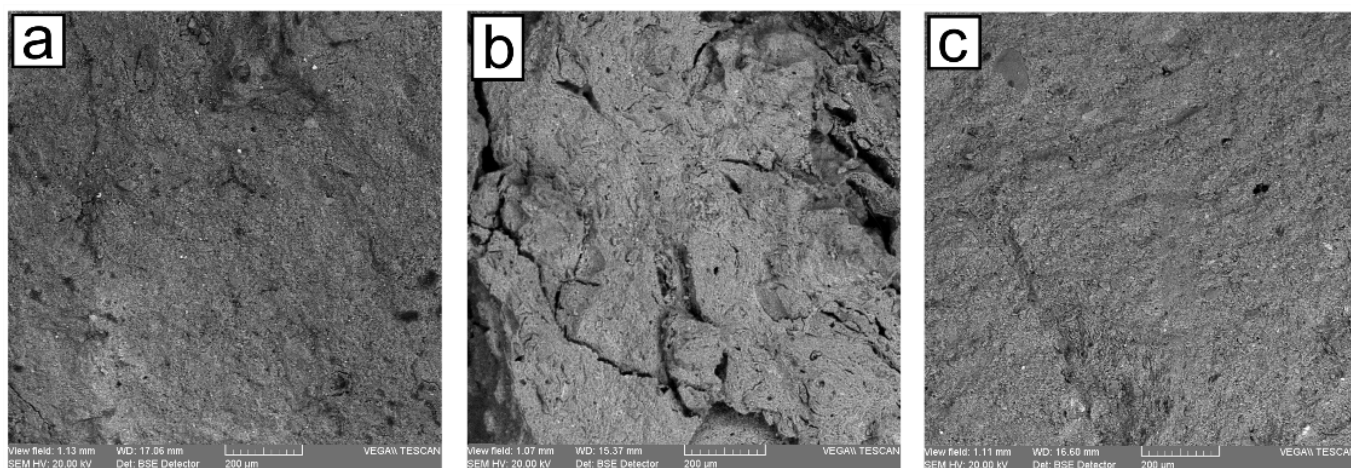


**Fig.12.** Optical microscopy of the analyzed pottery shard. a-b. exterior; c-f. interior; g-h. ceramic core (50× magnification) (Inkscape 0.92.1; AdobePhotoshop CC 2019).

feldspars and other mineral components<sup>43</sup>. The elements with archaeometric value are Fe, Ca, P and C, representing indicators of the type of clay used, firing temperatures and vessel functionality.

The presence of carbon in the ceramic paste indicates that the firing temperature of the vessel did not reach

<sup>43</sup> SANDU *et alii* 2010, 75-82; VASILACHE *et alii* 2014, 147.



**Fig.13.** SEM micrographs of the pottery sherd (a. exterior surface; b. ceramic core; c. inner surface) (Inkscape 0.92.1; AdobePhotoshop CC 2019).

**Tab. 3.** Elemental composition of the analyzed pottery shard (AdobePhotoshop CC 2019).

Elemental Composition in Weight Percent (%)													
Sample	Si	P	Al	K	Ca	Na	Mg	Fe	Ti	Cl	Cu	C	O
Ceramic core	27,08	0,39	10,33	3,39	1,64	0,39	1,83	4,20	0,53	0	0	0,97	49,24
Ceramic exterior 1	23,53	0,43	8,27	3,44	1,75	0,30	1,89	5,31	0,84	0	0	2,96	51,27
Ceramic exterior 2	21,13	0,61	7,65	4,17	2,68	0,58	1,55	3,82	0,97	0,62	2,99	5,73	47,50
Ceramic interior 1	25,72	0,18	8,98	3,96	1,95	0,36	2,28	4,79	1,04	0	0	0	50,73
Ceramic interior 2	25,00	0,29	8,66	3,98	1,97	0,32	2,41	4,81	0,94	0	1,28	0,70	49,62

700°C<sup>44</sup>, fact also supported by the macro- and microscopic presence of carbonates in the ceramic paste. Concentrations higher than 4% of iron or 6% of calcium are indicators of the type of clay used in the manufacturing the vessels<sup>45</sup>, and the obtained results indicate that the fragment analyzed in this study was made of a ferruginous clay, with a content of Fe of 4.20%. Phosphorus is a common element in nature, but also an indicator of human activities, being present in higher concentrations in settlements<sup>46</sup>, but not exceeding 0.5-1%. In the study of ceramics, it was established that values higher than 2% result from the use of the vessel for boiling or for storage phosphorus-rich liquids<sup>47</sup>, which indicates that the studied ceramic fragment does not belong to a vessel used for this purpose.

Regarding the analysis of the surfaces of the ceramic fragment, in addition to the specific elements of the raw material, traces of Cu and Cl were identified, resulting from the contact with the metal pieces. In this regard, the precise information on the context of the discovery or subsequent

<sup>44</sup> PAPACHRISTODOULOU *et alii* 2006, 352; NODARI *et alii* 2007, 4669-4670; RAVISANKAR *et alii* 2010, 189; VASILACHE/KAVRUK/TENCARIU 2020, 7.

<sup>45</sup> MANIATIS/TITE 1981, 61; NADEAU/TITE 1987, 242-243; VASILACHE *et alii* 2014, 144.

<sup>46</sup> GOLYEVA *et alii* 2018, 313; SALISBURY 2020, 199-211.

<sup>47</sup> SANTOS RODRIGUES/LIMA DA COSTA 2016, 298.

storage are not known, which makes it difficult to correctly interpret the traces of Cu on the surface of the ceramic fragment (this can be the result of contamination either in the depositional environment, either due to the incorrect storage).

The  $\mu$ FT-IR analyzes were performed on the ceramic core and on the surfaces (Fig.14) and based, on the characteristic group vibrations, we could establish the nature of the chemical compounds.

The 4000-3000 cm<sup>-1</sup> domain is attributed to the waters, and the water absorbed<sup>48</sup> in the ceramic fragment was also detected through the peaks at 3384 cm<sup>-1</sup>, 3235 cm<sup>-1</sup> and 1627 cm<sup>-1</sup>, corresponding to the -OH deformations. This can result from depositional processes in the soil or from cleaning the ceramic fragment.

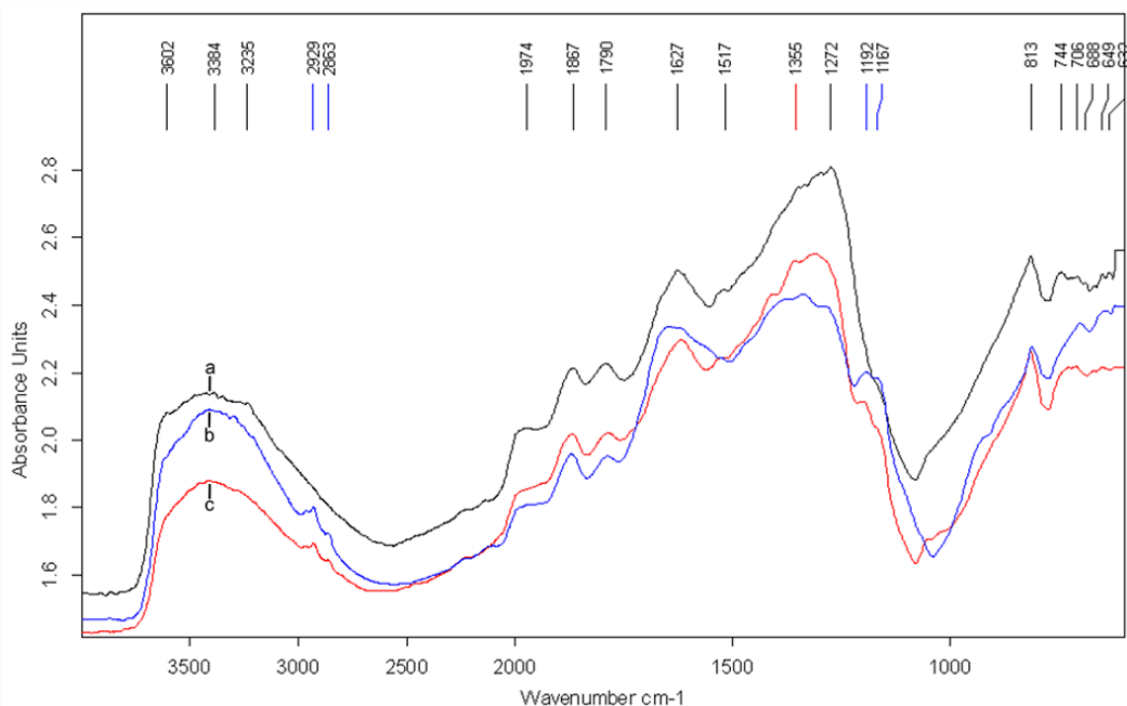
The peak located in the region 3500-3750 cm<sup>-1</sup> and the one at 688 cm<sup>-1</sup> are attributed to kaolinite<sup>49</sup> (Al<sub>2</sub>Si<sub>2</sub>O<sub>5</sub> (OH)<sub>4</sub>), and the missing doublet from 915 cm<sup>-1</sup> indicates that the ceramic fragment was burned at temperatures higher than 500°C.

The intense peaks from 2929 cm<sup>-1</sup> and 2863 cm<sup>-1</sup> are attributed to organic carbon<sup>50</sup>, present only on the surfaces

<sup>48</sup> DAMJANOVIĆ *et alii* 2011, 826.

<sup>49</sup> CHEN *et alii* 2015, table 1; OANCEA *et alii* 2017, 5085.

<sup>50</sup> COLUMBINI *et alii* 2005, 85-36; MARITAN *et alii* 2005, 42; DAMJANOVIĆ



**Fig.14.** FT-IR spectra of the pottery shard (a. ceramic core; b. exterior surface; c. inner surface) (Inkscape 0.92.1; AdobePhotoshop CC 2019).

of the ceramic fragment, with a higher intensity on the outside. The identified organic carbon, as in the case of metal parts, most likely comes from soil contamination during depositional processes.

Also, in all samples were identified carbonates<sup>51</sup> visible in the 1300-1500  $\text{cm}^{-1}$  domain, indicating that the firing temperatures of the vessel did not exceed 700-750°C. At 706  $\text{cm}^{-1}$  was detected the aragonite<sup>52</sup>, the anhydrous version of calcium carbonate, which may come from recrystallization during depositional processes.

The class of silicates is represented by the Si-O stretches of quartz<sup>53</sup> in the region 1900-1870  $\text{cm}^{-1}$ , the representative peaks being visible at 1974  $\text{cm}^{-1}$  and 1867  $\text{cm}^{-1}$ . In addition, quartz<sup>54</sup> was also identified by the peaks at 1192  $\text{cm}^{-1}$ , 1167  $\text{cm}^{-1}$ , 744  $\text{cm}^{-1}$  and 649  $\text{cm}^{-1}$ . Muscovite<sup>55</sup> ( $\text{KAl}_3\text{Si}_2\text{O}_{10}(\text{OH})_2$ ) is also present through the peaks at 1272  $\text{cm}^{-1}$  and 813  $\text{cm}^{-1}$ .

The presence of feldspars<sup>56</sup> was noticed through the peak from 1790  $\text{cm}^{-1}$ , while the iron oxides<sup>57</sup> were detected at 632  $\text{cm}^{-1}$ , belonging, like the other minerals, to the raw material.

Thus, the EDX and  $\mu\text{FT-IR}$  analyzes illustrate the use of a local kaolinitic clay, with a high iron content. The presence of feldspars is related to the aluminosilicates from the clays, as well as to the potassium and magnesium in

the EDX analysis. The presence of iron oxides identified by macro- and microscopy is also found in the FT-IR spectra, which supports the ferruginous nature of clay established by EDX. The correlation of the EDX with the  $\mu\text{FT-IR}$  results indicated the presence of carbonates and calcite in the paste of the analyzed shard, so that the firing temperatures did not exceed 700-750°C.

## DISCUSSIONS

The results obtained with the help of the three analyzes performed, allowed us to highlight two special situations: that of the bronze socketed axe with a significant content of lead and that of the sickle made entirely out of copper. The elemental composition of the socketed axe has analogies in several discoveries, recently introduced in the scientific circuit, from the settlements of Taraclia-Gaidabul and Odaia Miciurin (Republic of Moldova)<sup>58</sup>. The objects (a knife, a needle and three points attributed to the Noua-Sabatinovka culture) have concentrations of 2-3% Pb, and trace elements such as Ag, As, Sb, Ni, Co and Mn. Although the chemical compositions show minor differences, the amount of Pb used suggests the intentionality of alloying this element with bronze, which may provide evidence of this practice for a cultural entity found in a vast territory of Romania, especially in the originating area of the analyzed axe. Other pieces with a high lead content (between 1.55-2.95%) were reported also on the Romanian territory: an Oinac type socketed axe from Giurcani (Vaslui County)<sup>59</sup>, a knife<sup>60</sup> and a spearhead of Dremajlovka type<sup>61</sup> from Oțeleni (Vaslui County), objects which, through the context of the

*et alii* 2011, 862; COSTA *et alii* 2017, 573.

<sup>51</sup> NODARI *et alii* 2007, 4669; RAVISANKAR *et alii* 2010, 187;

<sup>52</sup> GASAWAY *et alii* 2017.

<sup>53</sup> AROKE/ABDULKARIM/OGUBUNKA 2013, 49.

<sup>54</sup> CHEN *et alii* 2015, Table 1; OANCEA *et alii* 2017, 5085.

<sup>55</sup> BARILARO *et alii* 2008, 273, Table 1; VASILACHE/KAVRUK/TENCARIU 2020, 12.

<sup>56</sup> SENTHIL KUMAR/RAJKUMAR 2014, 36.

<sup>57</sup> RAVISANKAR *et alii* 2010, 188; NICULAE 2011, 38, Table 5.1.

<sup>58</sup> SÎRBU *et alii* 2020, 98-100; SÎRBU *et alii* 2021, 54-57.

<sup>59</sup> LAZANU 2016, 79.

<sup>60</sup> LAZANU 2016, 83.

<sup>61</sup> LAZANU 2016, 89.

discovery or based on the analogies, were attributed to Noua culture.

Regarding the analyzed copper sickle, a similar situation was identified among the hoard from Bozia Nouă (Vaslui County), composed of three 'ingots' of copper, a socketed axe and three sickles, also attributed to Noua culture<sup>62</sup>. One of the sickles, attributed to Ghermănești type, Ruginoasa variant, was made out of copper and the elemental analysis highlighted also the presence of trace elements such as S, V, Fe, Co, Ni and As<sup>63</sup>. The use of copper in the manufacture of the Bozia Noua sickle is an important clue for the present study, suggesting, once more, a metallurgical practice that can be attributed to the Late Bronze Age cultural entities, located East of the Carpathians. The fact that experimental studies stated that the copper axes were not much more efficient than their stone equivalent, persuaded C. Renfrew to assume that copper tools, in general, were mainly prestige objects or status symbols with a social importance rather than a technological one<sup>64</sup>. Meanwhile, J. Chapman considers that there should be a small number of broken cast copper objects, due to the fact that the mastery of copper smelting and casting would stimulate new and different types of material relationships between individuals<sup>65</sup>. In respect of the symbolic value of metal artefacts, A. Vulpe is of the opinion that most of the hoards, as well as isolated discoveries, have a cultic/sacred specificity and that all the bronze hoards dating from Late Bronze Age, even fragmentary, should be considered to have a valoric function<sup>66</sup>.

The analyzes evidenced that the metal parts do not show the chemical signature of the mines from the Balkans or Central Europe<sup>67</sup>. Based on the analogies established for the investigated objects and their cultural attribution, the area of origin of the ores should be sought to the East, but the lack of chemical analysis for artifacts from this region makes it difficult to establish links with a specific area of exploitation. In this regard, the nearest tetrahedrite mines are located in Ukraine, in Savran-Sinistovska<sup>68</sup>, Dniester-Bug area, Odessa (Maiscoe/Mayske deposit), Luhansk<sup>69</sup> (Bobrikovo deposit) or Donetsk<sup>70</sup> (Nikitovka deposit) regions. Unfortunately, it is difficult to pinpoint these areas as possible sources of ore for the studied artefacts, as our arguments are based primarily on archaeological data regarding the area of distribution and transportation of goods in Late Bronze Age, performed by Noua communities.

The two metal pieces are, as stated before, fragmentary and appear to have been so since ancient times. This procedure, of deliberately breaking the metals deposited, was long disputed amongst the specialists<sup>71</sup> and

the main reasons accepted for the phenomenon are linked to: recasting; pre-monetary functionality; ritualic implications or the enchainment theory. Unfortunately, for the territory of Romania, the studies dedicated exclusively to this matter<sup>72</sup> are very few (usually the fragmentary state of the pieces is only mentioned). Still, the analysis conducted for the BzD-HaA1 hoards of Central Transylvania<sup>73</sup> highlighted that the most fragmentary objects at the end of the Bronze Age belong to the tool category, especially sickles and socketed axes, as is the case of our study. This association appears to be representative for the Late Bronze Age<sup>74</sup> (metal pieces belonging to same types as in our case were found together in many hoards, such as the ones from Ghermănești<sup>75</sup>, Heleşteni<sup>76</sup>, Bozia Nouă<sup>77</sup>, Tomești<sup>78</sup>, etc.) and is thought to have not only an 'industrial' significance (some specialists believe that these types could be the most representative for metal production at the end of the Bronze Age), but also a symbolic one, suggesting a male-female symbiosis (the socketed axe could represent the man and the sickle the woman)<sup>79</sup>. The intentional damaging of metal artefacts is also thought to be specific for Late Bronze Age, although it may have earlier origins<sup>80</sup>.

## CONCLUSIONS

Based on the existing chemical compositions and analogies, the two metal pieces could be attributed to Noua culture, specific to Late Bronze Age. Although the ceramic fragment indicates a possible dating in the Hallstattian period, the place of its discovery is uncertain, which is why, given the above, we believe that the two metal artifacts were not discovered in the same context as the shard.

The study carried out highlighted the existence of specific metallurgical practices within these communities, such as lead alloying or the use of tetrahedrite ores. Also, the study of fragmentary objects illustrated the possible duality of function that they had (practical-ritual), providing more data than a simple mention of broken objects without archaeological context. Last but not least, our study contributes to the existing elemental database, for prehistoric Romania.

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<sup>62</sup> LAZANU 2016, 133.

<sup>63</sup> LAZANU 2016, 150.

<sup>64</sup> RENFREW 1978.

<sup>65</sup> CHAPMAN 2000, 99.

<sup>66</sup> VULPE 2001.

<sup>67</sup> MARCOUX *et alii* 2002; LING *et alii* 2014; PERNICKA/LUTZ/STÖLLNER 2016; MÖDLINGERA/ TREBSCHKE 2020; MÖDLINGER/TREBSCHKE/SABATINI 2021.

<sup>68</sup> <https://www.mindat.org/loc-17830.html>.

<sup>69</sup> <https://www.mindat.org/loc-246264.html>.

<sup>70</sup> <https://www.mindat.org/loc-246264.html>.

<sup>71</sup> HOFFMANN 1999; CHAPMAN 2000; HARDING 2000, 352-368; NEBELSICK 2000; SCHIFFER 2010, 19-30; BUTTERFIELD 2017; BRANDHERM 2018; VILAÇA/BOTTAINI 2019.

<sup>72</sup> CIUGUDEAN *et alii* 2006; CIUGUDEAN 2010; REZI 2011.

<sup>73</sup> REZI 2011.

<sup>74</sup> KACSÓ 2007, 60; WITTENBERGER 2008, 17; MĂȚĂU 2010, 249-250.

<sup>75</sup> MELINTE 1975.

<sup>76</sup> MITREA 1971.

<sup>77</sup> DINU/COMAN 1964.

<sup>78</sup> PETRESCU-DÎMBOVIȚA 1977, 77.

<sup>79</sup> DIACONU 2014, 230.

<sup>80</sup> NEBELSICK 2000, 132.

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