

## Preliminary study of two antique Illyrian helmets (V-IV B.C.) excavated in northwest and northeast of Albania

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**Abstract:** This study combines several analytical techniques to preliminary investigate two Illyrian helmets (V-IV century B.C.) excavated in Nënshat (Shkodra, northwest Albania) and in Krumë (Has, northeast Albania). They were stored in the Center of Albanian Studies, after being excavated in 1963 in damaged conditions but have never been studied until now. The aims of this paper are the investigation of the helmets' alloy and production technique; hence  $\mu$ -X ray fluorescence ( $\mu$ -XRF), scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS), optical microscopy (OM) and Vickers microhardness tester were used to study the elements content, microstructure inclusions and its characteristics due to the production process. The Illyrian helmets alloy resulted copper-tin (Cu-Sn) with less than 1% of iron (Fe) and arsenic (As). The microstructure was composed by straight and bended twins, with 160.5 and 169.6 mean Vickers microhardness values, suggesting that these objects' alloys were casted, annealed and then cold worked to obtain the final distinctive shape. Elements such as sulfur, silicon, iron, arsenic (S, Si, Fe, As) can originate from the copper mineral used for the alloy production. This paper is part of a campaign in Albania to better understand the past with the use of physical analytical techniques.

**Keywords:** Illyrian helmet; Albania; copper-tin alloy; microstructure; production technique; copper mineral.

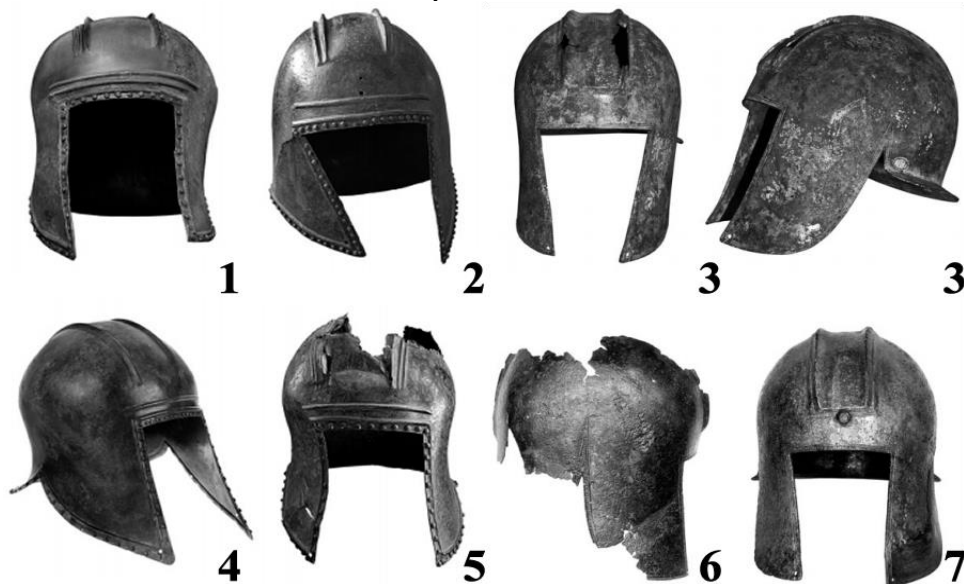
### 1. Introduction

The Illyrian helmets are widely spread across the Balkan Peninsula and beyond. Their main characteristics are: quadrilateral or trapezoidal face opening, triangular or elongated cheek / ear blinkers, neck guard, ridge along its length. Although, there is a great diversity not only in forms and shapes but also in territorial spread, three clear typologies of the Illyrian helmets can be distinguished from the VIII to the III century B.C.

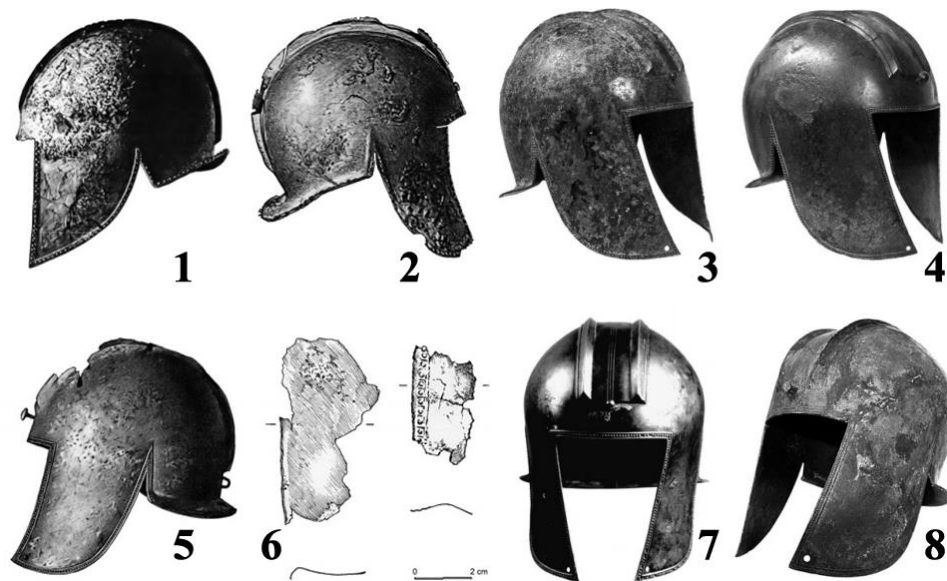
The first one is the simplest with a low crown and no neck guard. The second group has triangular cheek blinkers, elongated neck guard, horizontal ribs on the front and decorations along the borders. There was a period of experiments that preceded the third

typology with various details, some of them borrowed from Corinthian or Chalcidian helmet types.

The first typology of the Illyrian helmets (dating from the end of VIII century to the VII B.C.) were excavated mainly on the Peloponnese, with a single helmet from Borove in East Albania. The second type of helmets (from the VII to the middle of VI century B.C.) were found between Thessaloniki, Olympia and Kozani (Greece and Republic of North Macedonia) and also in the Northern Balkans which testifies the trade connections. The helmet elements during the second period of development, tended to get simplified and less solemn. The third typology (dating from the second half of the VI century to the IV B.C.) is divided into two subgroups: helmets from the first subgroup had side gussets and the ones from the second had round opening cuts for the ears. However more and more different workshops produced these helmets and considering that they did not progress simultaneously, a diversity in elements and territories is observed in today's findings. The most interesting elements of the Illyrian helmets, belonging in the first subgroup, are: juxtaposed lions on the forehead (borrowed from the Corinthian type); floral motifs; cheek-pieces in the form of a ram's head; incised arrow; artisan or workshop handwritings; boars and riders on the forehead and the cheekpieces; along with studs on the helmet edge; golden and silver embossed rectangular sheets around a square opening; high crown and stamped circles. These helmets were excavated in: Albania (Belsh, Perlat, Apollonia, Durrës, Dibër, Borovë, Dushk); Kosovo (Dardana, Shiroka, Denji, Korisha, Banja e Pejës); Greece (Sindos, Archontiko, Argolis, Kilkis, Thermi, Isthmia, Olympia); Republic of North Macedonia (Rečica, Kozani, Gornja Porta, Trebenishte); Italy (Sicily); Rumania (Timișoara); Serbia (Ražana, Trstenik); Montenegro (Budva); Bosnia and Herzegovina (Podstinja, Kiseljak). Illyrian helmets, classified in the second subgroup (dating the middle of the V century to the IV B.C.), were distinguished by the ear shaped cuttings. They were excavated in Greece, Albania and also in Italy. Various Illyrian helmets excavated in different regions of the Balkans are shown in figures 1 and 2, to better visualize their shapes and characteristics.



**Figure 1:** 1. *Agia Paraskevi near Kozani*, 2. *Anarahi*, 3. *Thessaloniki*, 4. *Ano Kopanos*. 5. *Kozani*, 6. *Trstenik*, 7. *Sremska Mitrovica*. [1]



**Figure 2:** 1. Gorica, 2. Putičevo, 3. Pečka banja, 4. Pečkabanja, 5. Grude, 6. Glasinac, Rusanovići and Taline, 7. Gavojdia, 8. Ražana.[1]

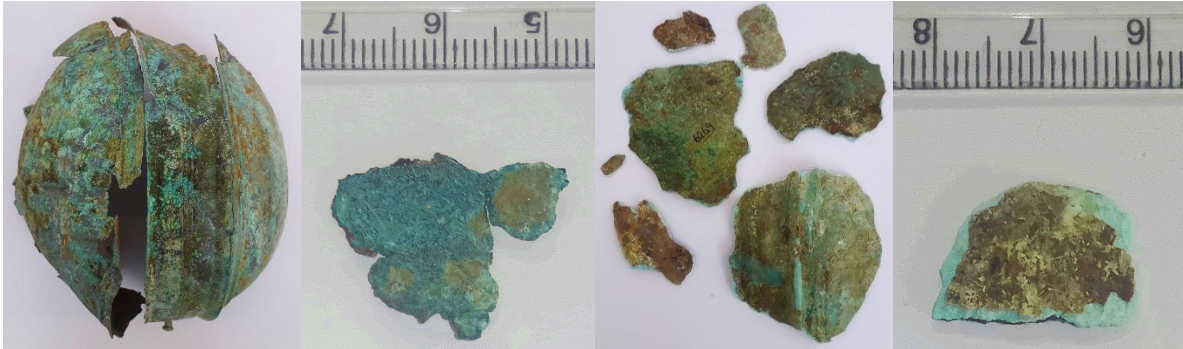
Although these helmets had various developing elements over time, they were unique and studying them helps to better understand the ancient Balkan history and the relations between its populations. [1-8]

Taking advantage of the fact that both helmets were excavated in a damaged state, gave us the possibility to take samples and to use destructive analytical methods in this study. The purposes of this paper are the investigation of the helmets' alloy and their production technique. Although Albania has a rich cultural heritage fond, its study with physical analytical methods begin only in the years 2011-2012. This article is part of a national campaign in order to better understand the past of this region's population and the relations with the neighboring countries.

Further, in the article's sessions the following nomenclatures will be used:  $\mu$ -X ray fluorescence –  $\mu$ -XRF, scanning electron microscopy-energy dispersive spectroscopy – SEM-EDS, optical microscopy – OM, copper – Cu, tin – Sn, iron – Fe, arsenic – As, sulfur – S, silicon – Si, phosphor – P, calcium – Ca.

## 2. Materials and Methods

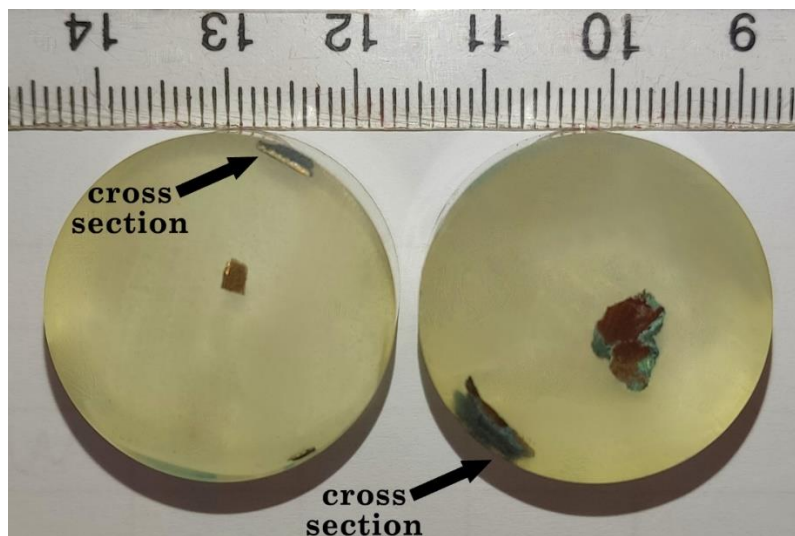
Both Illyrian helmets have the same characteristics (after the restoration process) such as: half spherical shape, ridge along its length, quadrilateral or trapezoidal face space, triangular blinkers near the ears to protect the warrior cheeks. Although there is a similarity with the helmets excavated in North Macedonia, their distinct features define them as Illyrian helmets. Figure 3 shows both helmets before restoration, along with the corresponding samples given to us from the Center of Albanian Studies in Tirana. [9-13]



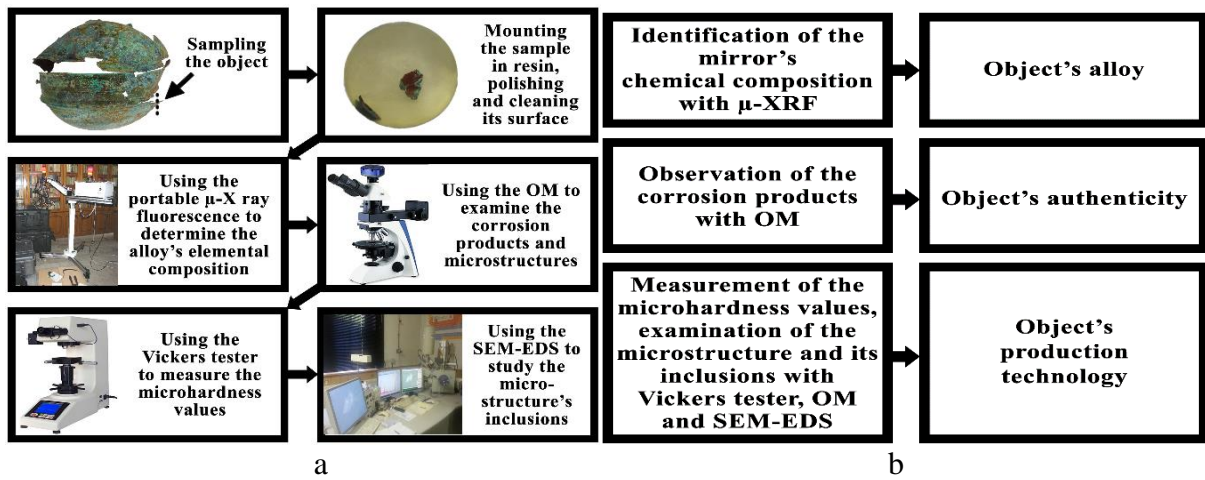
**Figure 3:** Left - damaged Illyrian helmet excavated in Nënshat (Shkodra, no 4649) with the corresponding sample; right - fragments of Illyrian helmet found in Krumë (Has, no 6269) with the corresponding sample. Center of Albanian Studies in Tirana made available these samples for this study.

To fulfill this study's objectives transportable  $\mu$ -XRF spectrometer ARTAX Bruker with Spectra ARTAX Version 7.2.5.0 and M-Quant-Calib is used to qualify and quantify the helmet's alloy (60 $\mu$ m spot diameter; Mo anode; 50kV max tension; 600 $\mu$ A max current; Si drift detector; resolution 146eV FWHM for 10kcps; Rh target). The microstructure's inclusions are studied with SEM-EDS XL30 ESEM-FEI and EDAX Genesis Spectrum software (with ZAF correction; 2000Pa (0.01974atm) column precision; 20kV tension; 10mm working distance; 120 $\mu$ A current; IR digital camera; inside column Everhart-Thornley (ETD) SE detector, BSE and CL detector). The microstructure with the corrosion products is investigated with Kozo XJP304 optical microscope (halogen light source; 12V tension; 50W power), Vickers microhardness tester (4900mN load; 30 seconds indentation time), Sony TCC-8.1 digital camera and View Version 7.3.1.7 software.[14-19]

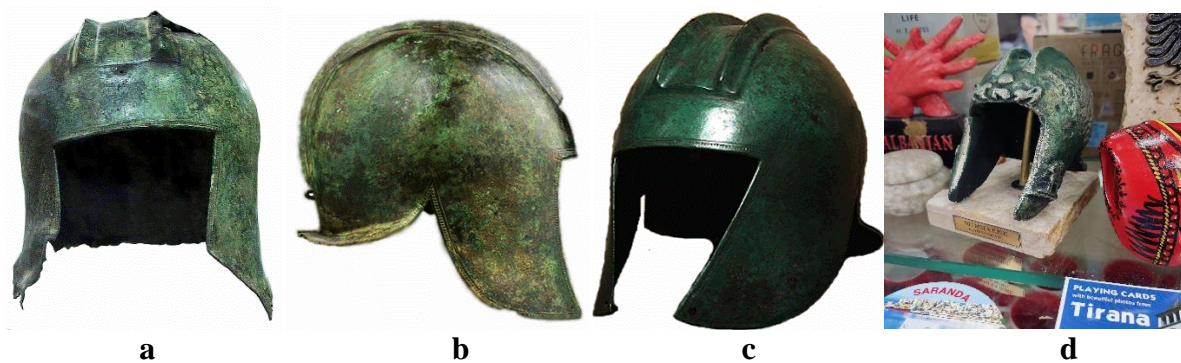
The samples of both helmets are mounted in acrylic resin (surface and cross section), polished and cleaned with ultrasounds (figure 4). After that they are studied with various physical analytical methods in order to better understand their composition and production technique. Two flowcharts, that indicate the experimental procedure in order to obtain results and the conclusions deriving from these results, are shown on figure 5. [20]



**Figure 4:** a) Samples mounted in acrylic resin (surface and cross section) of helmet excavated in Krumë (Has, no 6269, left) and the one found in Nënshat (Shkodra, no 4649, right).



**Figure 5:** a) Flowchart indicating the experimental procedure in order to obtain the results, b) flowchart showing the results converted into information and conclusions.



**Figure 6:** Illyrian helmet excavated in Albania, specifically: a) Draji-Reç (Dibër city), b) Dushk (Lushnjë city), c) Perlat (Lezhë city); d) Illyrian helmet souvenir. [3; 4]



**Figure 7:** Illyrian helmets excavated in Kosovo, specifically: Banja e Pejës (3 helmets), Dardana (1 helmet), Denji (1 helmet), Shiroka (1 helmet) and Korisha (2 helmets). [3; 4]

There are many archaeological studies about the Illyrian helmets excavated in Albania, such as in the city of Dibër, Lushnjë, Lezhë (figure 6 a), b), c)) and in Kosovo, specifically in Banja e Pejës, Dardana, Denji, Shiroka, Korisha (figure 7), but very few papers where they are examined with physical analytical methods. [3; 4] This was the main reason

for conducting this study. The popularity of the Illyrian helmets as a cultural item is shown also in the souvenirs production (figure 6 d)).

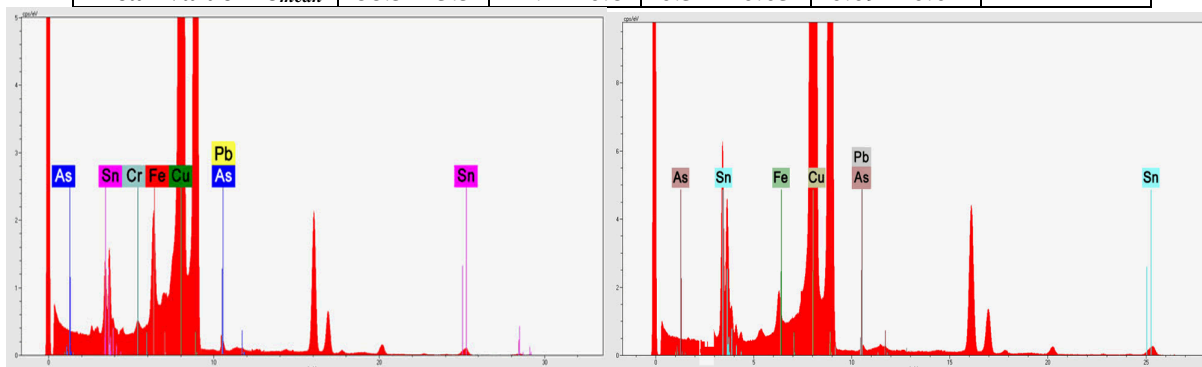
### 3. Results and Discussions

The analytical procedure starts with the  $\mu$ -XRF on 5-10 points on each sample (60 $\mu$ m spot diameter). The mean values along with the standard deviations are shown in the table below (table 1) while one XRF spectra for each sample are presented in figure 8. In both cases we have binary Cu-Sn alloys (92% Cu - 7.1% Sn and 88.5% Cu - 11.1% Sn), with minor elements such as Fe and As (less than 1%); Cr is detected in the first sample with a content of 0.06% which is a possible contamination from soil. Tin is added in copper in order to reduce its melting temperature, increase the hardness and resistance to the corrosion process. The maximum percentage of Sn in Cu, for a complete solubility, is 14% forming an alloy with high hardness and low brittleness. [16; 17; 21-25]

Iron and arsenic are two usual elements that are associated with copper sulphite minerals. Copper minerals are divided into: 1) sulphites with Cu-S bonds, 2) carbonates with Cu-C-O elements, and 3) silicates with Cu-Si-O elements. Copper is extracted easily from sulphites minerals which in the Balkans are present in Albania, Cyprus and in some areas of Turkey. (Economou-Eliopoulos et al, 2008) Hence, the northern and eastern territories of Albania are rich in copper sulphite minerals such as: pyrite FeS<sub>2</sub>, chalcopyrite CuFeS<sub>2</sub>, bornite Cu<sub>5</sub>FeS<sub>4</sub>, sphalerite [(Zn,Fe)S], covellite CuS, tennantite (Cu<sub>12</sub>As<sub>4</sub>S<sub>13</sub>), tetrahedrite [(Cu,Fe)<sub>12</sub>Sb<sub>4</sub>S<sub>13</sub>] and Cu-Zn-Au-Ag-Si compounds. [26-32]

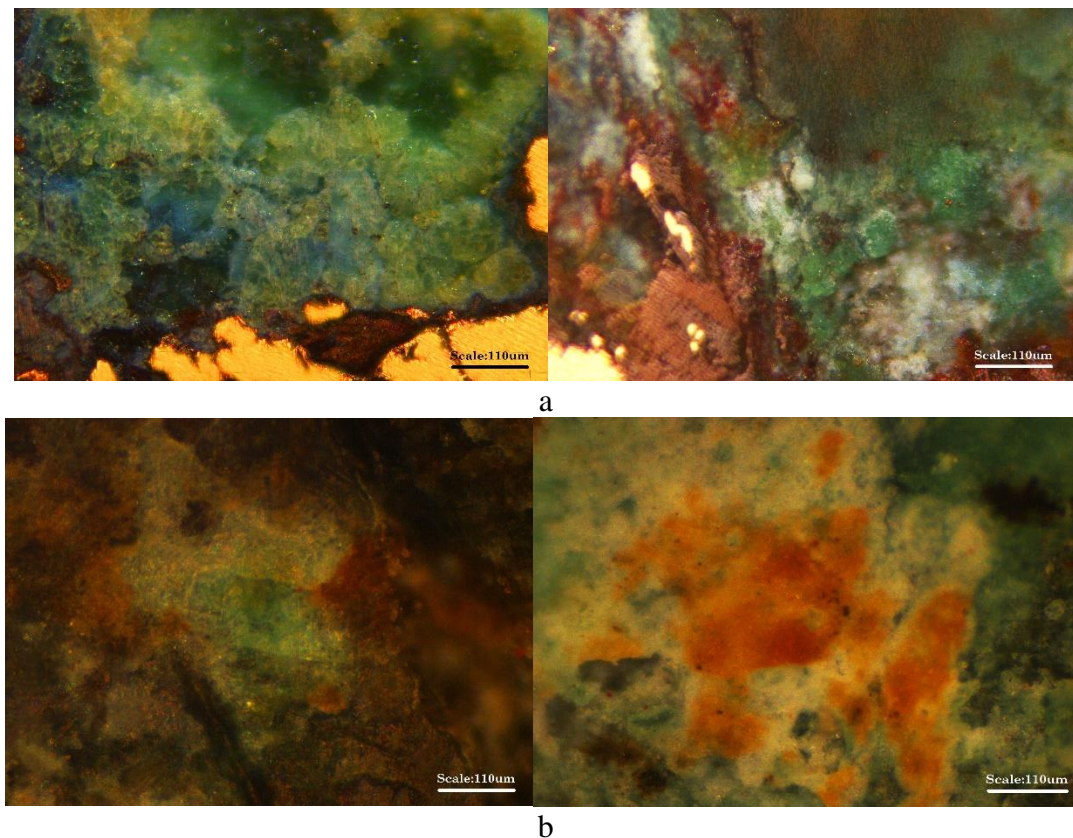
**Table 1:**  $\mu$ -XRF results of the Nënshat (Shkodra, no 4649, above) and Krumë (Has, no 6269, below) helmets' samples.

	Cu (%)	Sn (%)	Fe (%)	As (%)	Cr (%)
<b>No 4649</b>					
<b>Mean value <math>\pm \sigma_{mean}</math></b>	92 $\pm$ 4	7.1 $\pm$ 0.5	0.71 $\pm$ 0.04	0.13 $\pm$ 0.02	0.06 $\pm$ 0.01
<b>No 6269</b>					
<b>Mean value <math>\pm \sigma_{mean}</math></b>	88.5 $\pm$ 3.5	11.1 $\pm$ 0.8	0.31 $\pm$ 0.03	0.09 $\pm$ 0.02	-



**Figure 8:**  $\mu$ -XRF spectra of the samples from helmet excavated in Nënshat (Shkodra, no 4649, left) and the one found in Krumë (Has, no 6269, right).

The second step of examination includes the observation of corrosion products with optical microscope with polarized light (figure 9).



**Figure 9:** Corrosion products observed with optical microscope with polarized light of the samples from a) helmet excavated in Nënshat (Shkodra, no 4649) and b) the one found in Krumë (Has, no 6269).

From the OM pictures with polarized light and based on the literature cuprite  $\text{Cu}_2\text{O}$  (red), cassiterite  $\text{SnO}_2$  (black) were distinctive. These oxides form the first layer of the corrosion products, which is created during the object usage. From the second one, malachite was visible  $\text{Cu}_2\text{CO}_3(\text{OH})_2$  (green) and in few cases also azurite  $\text{Cu}_3(\text{CO}_3)_2(\text{OH})_2$  (blue). Usually these components are created during the first period of conservation in soil (inland or near the sea). The above results are qualitative analysis based on OM observations and on literature, in the cases where XRD is not an option due to insufficient sample's amount. These OM pictures with polarized light are proof that the helmets are authentic and not replicas because it is impossible to create the first and second corrosion layer artificially. [14; 15; 33]

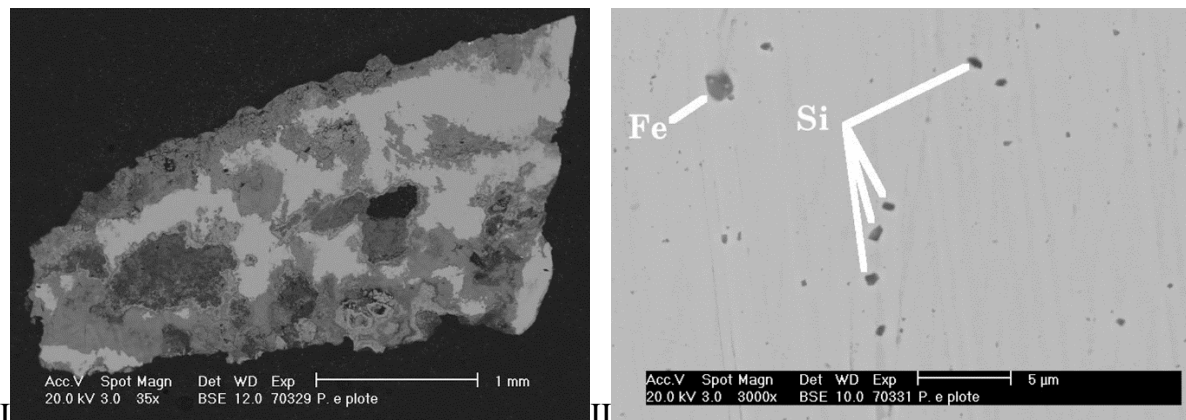
Table 2 shows the Vickers microhardness values of both helmets' samples, along with the mean values and relative error. The results of sample from helmet no 4649 (Nënshat, Shkodra) falls in the interval from 146.2 to 176.8 HV, while the mean value is 160.5HV. The values obtained from the second sample of helmet no 6269 (Krumë, Has) are similar, from 163.9 to 175.8 HV with a mean value of 169.6HV. This is expected considering that the percentage of copper and tin are close in both samples as well.

The Vickers microhardness value of copper can increase either from mechanical processing or from alloying. Pure cast Cu has a Vickers microhardness value around 40-50 HV and it can increase to 100-120 HV if it is cold worked. When Cu forms an alloy with Sn, up to 14%, the Vickers microhardness value can get up to 220 HV. [19; 20]

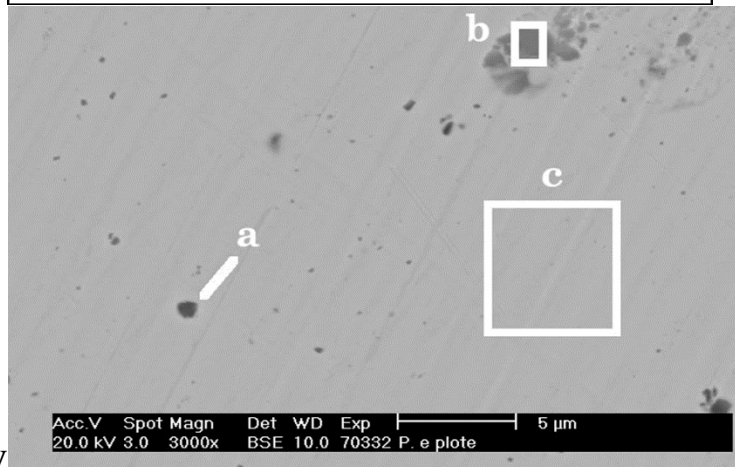
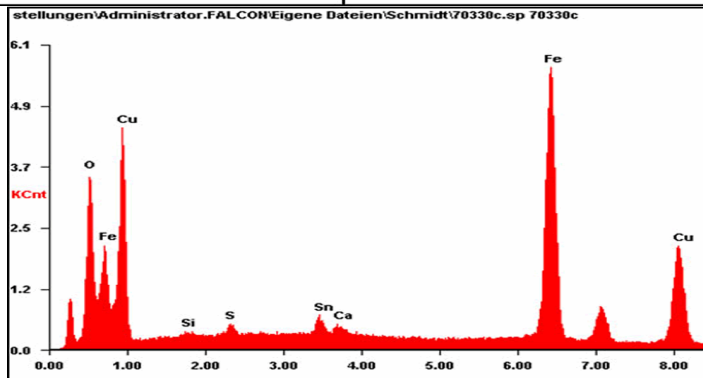
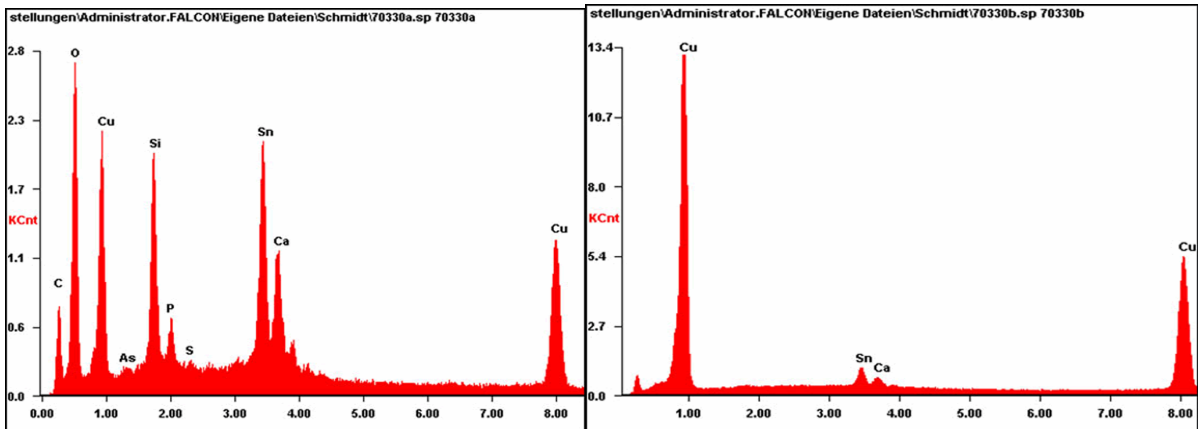
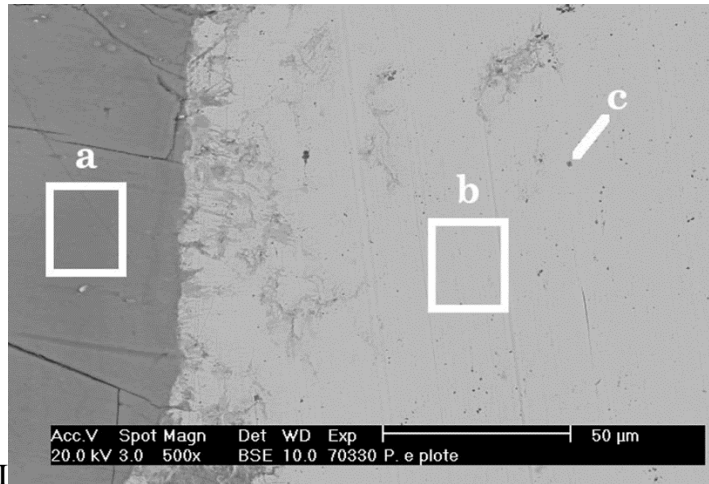
**Table 2:** Vickers microhardness values of the Nënshat (Shkodra, no 4649) and Krumë (Has, no 6269) helmets' samples.

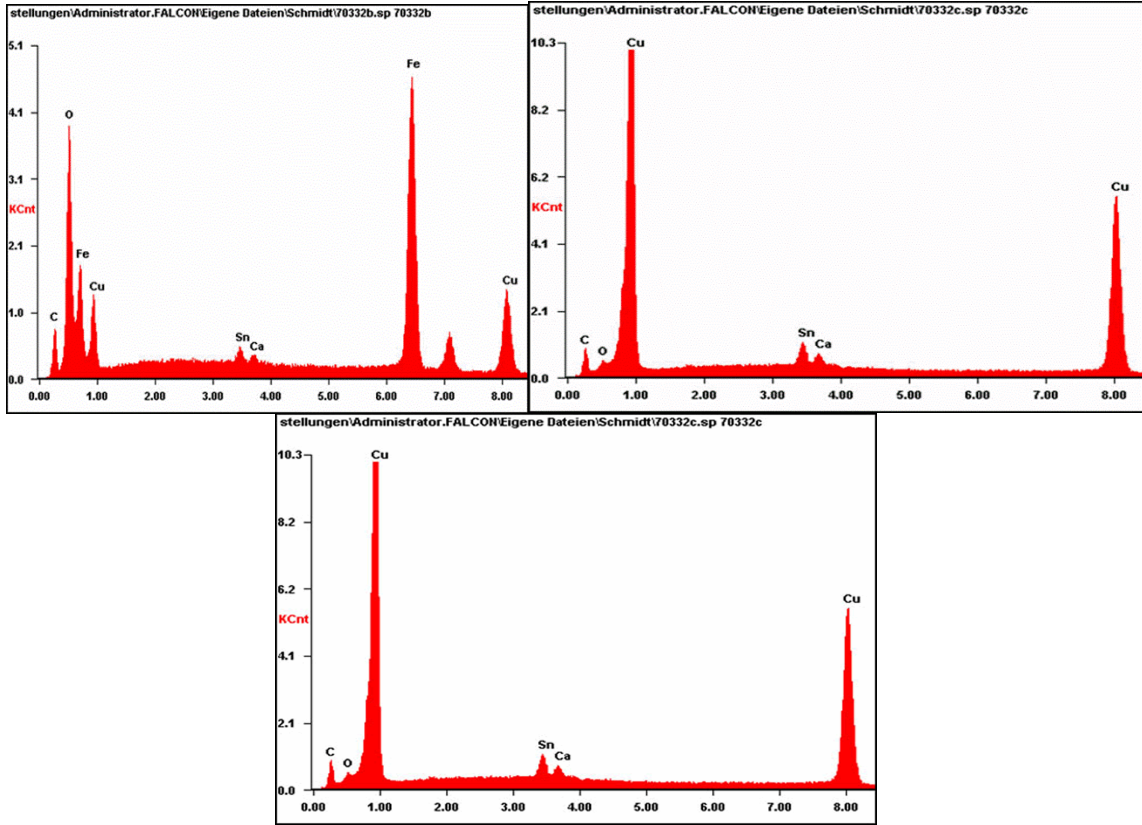
Nënshat (Shkodra) no 4649		Krumë (Has) no 6269	
$d_{\text{mean}}$ ( $\mu\text{m}$ )	HV	$d_{\text{mean}}$ ( $\mu\text{m}$ )	HV
72.4	176.8	72.9	174.4
77.3	155.1	73.7	170.6
79.6	146.2	73.5	171.5
74.9	165.2	75.2	163.9
76.8	157.1	74.2	168.3
78.2	151.5	75.1	164.3
73.7	170.6	72.6	175.8
76.8	157.1	73.7	170.6
75.1	164.3	73.9	169.7
75.8	161.3	74.4	167.4
Mean values			
76.06	160.5	73.92	169.6
Absolute and relative error			
$\Delta d = 1 \mu\text{m}$	$\Delta HV/HV = 2.6\%$	$\Delta d = 1 \mu\text{m}$	$\Delta HV/HV = 2.7\%$

Continuing with the analytical procedure with the SEM photos and EDS spectra of various points and areas examined on both helmets' samples (figures 10 and 11) while tables 3 and 4 present the elements content in weight percentage from the EDS results (mean value of the standard deviation is  $\pm 0.3\%$  for major content elements). [18]

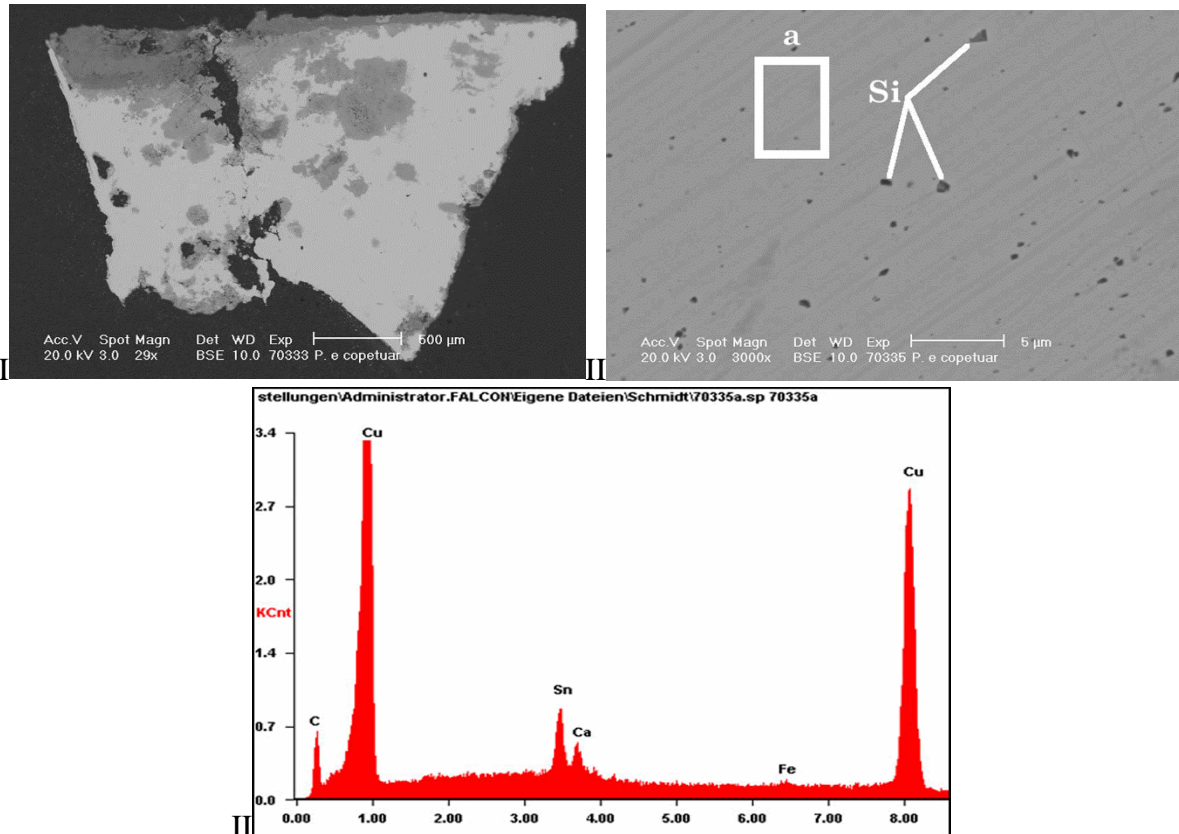


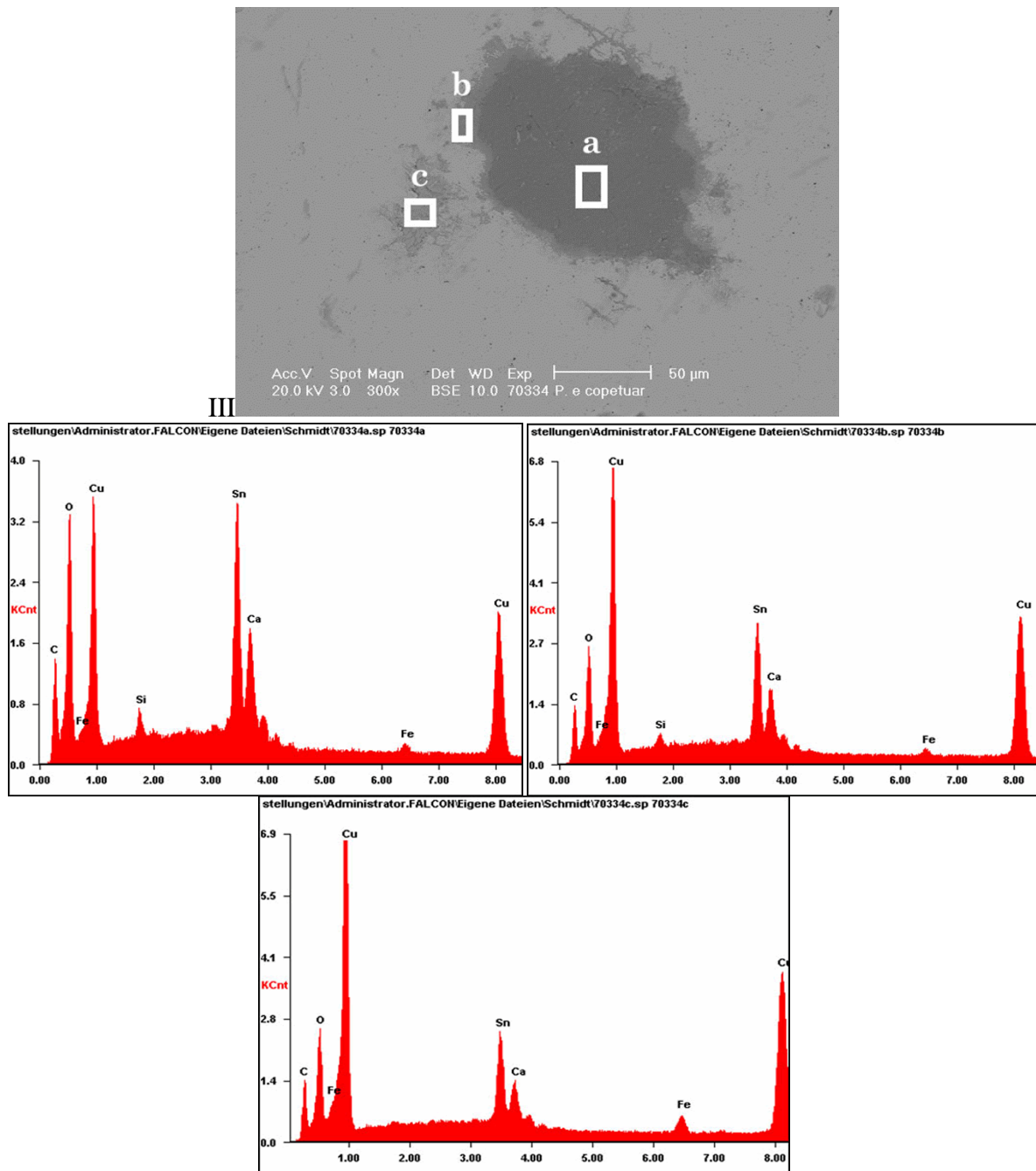






**Figure 10:** Nënshat (Shkodra, no 4649) helmet's sample: I. SEM image of the sample (magnification 35X); II. SEM image showing Fe and Si content (from EDS) in the alloy (magnification 3000X), III. SEM image (magnification 500X) showing areas a, b and point c examined with EDS, below the corresponding spectra; IV. SEM image (magnification 3000X) showing point a and areas b, c examined with EDS, below the corresponding spectra.





**Figure 11:** *Krumë (Has, no 6269) helmet's sample: I. SEM image of the sample (magnification 29X); II. SEM image (magnification 3000X) showing Si content (from EDS) in the alloy and area a examined with EDS, below the corresponding spectra; III. SEM image (magnification 300X) showing areas a, b, c examined with EDS, below the corresponding spectra.*

From the EDS results the alloy's elements (Cu and Sn) are detected with percentages similar to the ones from the  $\mu$ -XRF analysis (figure 10 III b), IV c) of helmet no 4649 and figure 11 II a) of helmet no 6269). The corrosion areas are easily distinguishable from the high percentage of oxygen, such as figure 10 III a) of helmet's no 4649 sample, also figure 11 III a), III b) and partially III c) of helmet's no 6269 sample. The microstructure of the first sample contains Fe and Si inclusions (figure 10 III c), IV a), IV b)) unlike the second sample where an iron inclusion might have been detected on figure 11 III c). Elements such as P and Ca can be contaminations from the soil while S, Si, Fe and As can originate from the copper sulphite minerals used to produce these helmets. Furthermore, the copper sulphite mineral's

origin (if its local or imported) needs following analysis, however these elements' content are an indicator. Carbon was neglected in the calculations because of the coating layer applied during EDS sample preparation. [18; 26-29]

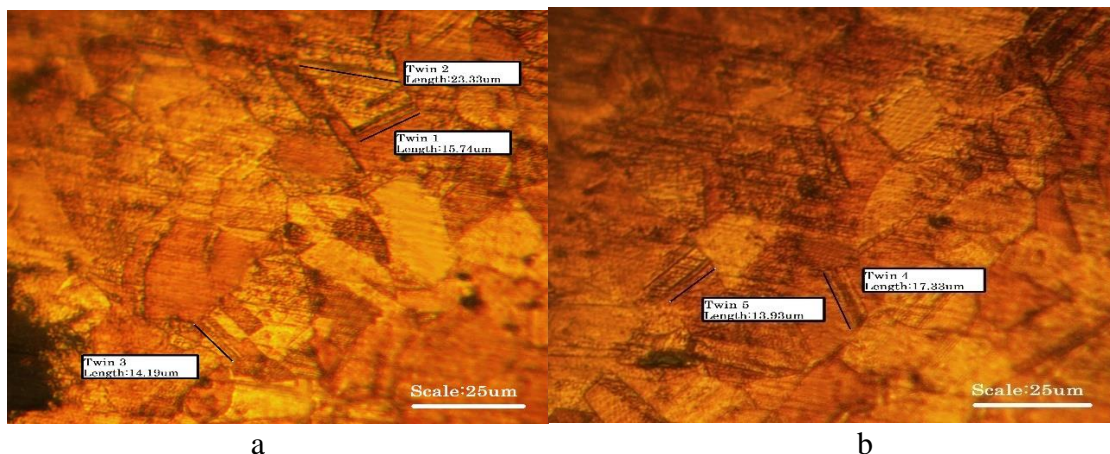
**Table 3:** EDS results (in weight percentage) of the Nënshat (Shkodra, no 4649, III and IV) helmets' sample.

Wt%	III area a	III area b	III point c	IV point a	IV area b	IV area c
O	34.4	-	13	0.6	17.5	-
As	0.7	-	-	-	-	-
Si	8	-	0.3	24.4	-	-
P	1.6	-	-	-	-	-
S	0.3	-	0.5	-	-	-
Sn	25	7.5	2.2	5.5	2	7.4
Ca	0.9	0.2	0.1	0.2	0.1	0.2
Cu	29.1	92.3	23.3	69.3	18.2	92.4
Fe	-	-	60.6	-	62.2	-

**Table 4:** EDS results (in weight percentage) of the Krumë (Has, no 6269, II and III) helmets' sample.

Wt%	II area a	III area a	III area b	III area c
O	-	31.5	20.9	17.9
Si	-	1.3	1.1	0.2
Sn	12	30.2	24.6	18.1
Ca	0.4	0.6	0.5	0.3
Fe	0.9	2.4	2.2	5.5
Cu	86.7	34	50.7	58

The last step of examination is shown on figure 12, where the samples' microstructures are etched for 8-10 seconds with  $K_2Cr_2O_7 : NaCl : H_2SO_4 : H_2O : HCl = 2 \text{ gr} : 5\text{ml} : 8\text{ml} : 100\text{ml} : 3\text{drops}$ , in order to observe the microstructure's characteristics. The presence of straight and bended twins (length less than  $25\mu\text{m}$ ) suggests that both alloys might have been casted in half spherical molds, annealed or hot worked (twin formation) and then cold worked (twin bending), for example by hammering, in order to obtain the final distinctive shape. [14; 15; 21-25]



**Figure 12:** Microstructure of helmet's sample from a) Nënshat (Shkodra, no 4649) and b) Krumë (Has, no 6269), after being etched for 8-10 seconds with  $K_2Cr_2O_7 : NaCl : H_2SO_4 : H_2O : HCl = 2 \text{ gr} : 5\text{ml} : 8\text{ml} : 100\text{ml} : 3\text{drops}$ .

Although we had the opportunity to examine these two Illyrian helmets with physical analysis methods, the results have limitations because they come from just one sample from each helmet, because taking more random samples from them was not permitted.

#### **4. Conclusions**

- The Illyrian helmets excavated in the northwest and northeast of Albania resulted copper-tin alloys with less than 1% of iron and arsenic content.
- The microstructure was composed by straight and bended twins, with mean Vickers microhardness values of 160.5 and 169.6 HV, which suggests that these objects' alloys were casted, annealed and then cold worked in order to obtain the final distinct shape.
- Elements such as S, Si, Fe and As can originate from the copper sulphite mineral used to produce these helmets. To define if the copper sulphite mineral is extracted locally or imported needs further detailed analysis.
- Although structure elements and materials could have been local, the general influence and inspiration in metal working has passed beyond borders among the Balkan regions.
- A future study goal would be the examination of other Illyrian helmets excavated in the Albanian territories, along with the copper minerals of the areas, in order to scientifically determine if the production was completely local, from the mineral extraction to the object final form.

#### **Authors Contributions**

- The conceptualization of the article "Preliminary study of two antique Illyrian helmets (V-IV B.C.) excavated in northwest and northeast of Albania" was done by O. Ç.;
- All of the data curation and visualization was done by O. Ç.;
- The  $\mu$ -XRF formal analysis and investigation were performed by O. Ç. and N. C.;
- All the OM and Vickers formal analysis and investigation were done by O. Ç., along with the samples' preparation (mounting in resin, polishing, cleaning, etching);
- The SEM-EDS formal analysis and investigation were performed by G. S.;
- Resources - the  $\mu$ -XRF, OM, Vickers tester, all the mounting materials and reagents are part of the University of Tirana laboratories in Albania while the SEM-EDS belong to the Institut für Keramik, Glass- und Baustofftechnik, TU Bergakademie Freiberg, Germany;
- O. Ç. did the writing of the original draft-article, then the correcting of the referee points and editing in order to prepare the revised-article;
- The funding was assured by DAAD (projekt-ID 57068639 & ID 57175715);
- The methodology of sample preparation and analysis procedure were consistent with the literature, provided in the article Literature;
- Project administration, its supervision and validation of the output data were performed by O. Ç. and N. C.;
- The analytical equipment's software was used by the corresponding authors that performed each formal analysis. There is no programming, coding or software developing in this article.

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equipments; and last but not least to the Institut für Keramik, Glass- und Baustofftechnik, TU Bergakademie Freiberg and DAAD (Projekt-ID 57068639 & ID 57175715) for making possible the samples' examinations with SEM-EDS in Germany.

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