

OLD KINGDOM COPPER TOOLS AND MODEL TOOLS

Martin Odler

with contributions by

Jiří Kmošek, Ján Dupej, Katarína Arias Kytnarová, Lucie Jirásková, Veronika Dulíková,
Tereza Jamborová, Šárka Msallamová, Kateřina Šálková and Martina Kmoníčková



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To my parents
and grandparents

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11. Case studies

11.1. Archaeometallurgical study of copper alloy tools and model tools from the Old Kingdom necropolis at Giza

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Introduction

Archaeometallurgical analyses of ancient Egyptian artefacts are needed in order to better understand the context and development of ancient Egyptian metallurgy. This archaeometallurgical study was carried out within the project *Early Copper Metallurgy in Ancient Egypt – a case study of the material from Ägyptisches Museum der Universität Leipzig*. The two-year project, funded by the Grant Agency of the Charles University and University of Chemical Technology, started in 2015 and focuses on artefacts in the collection of the Egyptian Museum – Georg Steindorff – of the University of Leipzig (ÄMUL) (Figure 225).

Arsenic copper alloys

Arsenic is a characteristic admixture of many copper ores and, therefore, we can find traces of arsenic in most ancient Egyptian objects made of copper alloys. It is generally assumed that copper objects with arsenic content over the limit of 1 weight % are products of deliberate use of arsenic rich copper ores or of intentional combining of arsenic and copper ores.⁷⁹⁵ Arsenical copper had been used from the Naqada culture until the Second Intermediate period; since the Middle Kingdom, arsenical copper

alloy was gradually replaced by tin bronzes.⁷⁹⁶ The average arsenic concentration in Egyptian artefacts made from arsenical copper was in the range of 2–3 weight % up to the Middle Kingdom; afterwards, the concentration of arsenic slightly decreased.⁷⁹⁷ The composition of arsenical copper alloy working tools and their models seems to be very similar.⁷⁹⁸ The production of arsenical copper alloys can be accomplished through several metallurgical processes, namely the smelting of secondary copper ores rich in arsenic or co-smelting under reduction condition of copper ores with oxides or sulphides containing arsenic.⁷⁹⁹ The alloys copper arsenate olivenite ($Cu_2(AsO_4)(OH)$) or copper sulpharsenide enargite (Cu_3AsS_4) could be used for the production of arsenic copper, while copper ores with sulpharsenide ore of iron – arsenopyrite ($FeAsS$) were usable for the co-smelting process.⁸⁰⁰ Sulphide ores cannot be reduced directly by carbon monoxide and need to be roasted and transformed from a sulphide into an oxide form before the reduction. The roasting of arsenic ore arsenious trioxide (As_2O_3) produces a white fume or smoke which causes serious and long-term health hazards to the human body.⁸⁰¹

Copper arsenic alloys offer some advantages in casting and mechanical properties in comparison with pure copper. With increasing amount of arsenic in a copper alloy, the melting point of the resulting alloy decreases.⁸⁰² The melting and alloying processes of copper-arsenic alloys under oxidizing conditions are accompanied with a loss of arsenic, due to its high volatile properties at higher temperatures.⁸⁰³ On the contrary, melting and alloying of this alloy under highly reducing conditions is not accompanied by the loss of arsenic and the formation of white vapours of arsenic oxide.⁸⁰⁴ The large solidification temperature interval of the copper-arsenic system results in a microsegregation tendency connected with non-equilibrium solidification.⁸⁰⁵ The consequence of this phenomenon in alloys containing less than app. 25% is the formation of γ -phase Cu_3As ; it could be also visible in non-equilibrium copper alloys containing 3% of arsenic.⁸⁰⁶

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⁷⁹⁰ Czech Institute of Egyptology, Faculty of Arts, Charles University. This study was supported by the Grant Agency of Charles University within the project GA UK No. 38715 and with the contribution of the Intern Grant Agency of the University of Chemistry and Technology in Prague within the project No. 10681501. The project is a cooperation with the Czech Institute of Egyptology, Faculty of Arts, Charles University (Martin Odler), the University of Chemistry and Technology (Jiří Kmošek), Ägyptisches Museum – Georg Steindorff – der Universität Leipzig (Dietrich Raue, Karl-Heinrich von Stülpnagel), Institut für Mineralogie, Kristallographie und Materialwissenschaft der Universität Leipzig (Gert Klöß, Andreas König) and the Institute of Nuclear Physics, the Czech Academy of Science (Marek Fikrle).

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⁷⁹⁵ Moorey (1994); Craddock (1976).

⁷⁹⁶ McKerrell (1993); Philip (2006, 212).

⁷⁹⁷ Cowell (1986, 99).

⁷⁹⁸ Cowell (1986, 99).

⁷⁹⁹ Tylecote (1992, 10); Lechtmann and Klein (1999, 498).

⁸⁰⁰ Lechtmann and Klein (1999, 498).

⁸⁰¹ Lechtmann and Klein (1999, 499).

⁸⁰² Subramanian and Laughlin (1988).

⁸⁰³ McKerrel and Tylecote (1972).

⁸⁰⁴ Budd (1990).

⁸⁰⁵ Subramanian and Laughlin (1988).

⁸⁰⁶ Northover (1989).



FIGURE 225: SET OF ANALYZED ARTEFACTS. DASHED LINES AND ARROWS INDICATE THE SAMPLED LOCATIONS (PHOTO J. KMOŠEK, © FACULTY OF ARTS, CHARLES UNIVERSITY, CZECH INSTITUTE OF EGYPTOLOGY).

Context	ID of artefact	Artefact	Site	Period	Length (mm)	Width (mm)	Height (mm)	Weight (grams)	Analysis*
G66	ÄMUL 2118	model chisel	Giza	Dynasty 5 [PM]; late Dynasty 5 [Smith], reign of Nyuserra [Ziegler]	78	6	2	5.3	XRD
G91	ÄMUL 2120	model chisel	Giza	late Dynasty 5 – early Dynasty 6 [Giza-Projekt]	64	3	2	2.1	ED-XRF, OM, XRD, SEM/EDS
G90	ÄMUL 2129	spatula/ingot?	Giza	late Dynasty 5 – early Dynasty 6 [Spiekermann]	92	16	2	6.7	ED-XRF, OM, SEM/EDS, micro HV
G81	ÄMUL 2130	razor	Giza	Dynasty 5 end or Dynasty 6 [PM]	100	45	11	47.4	OM, XRD, SEM/EDS
G85	ÄMUL 2131	razor	Giza	Dynasty 5-6	122	57	4	34.4	ED-XRF, OM, SEM/EDS, XRD, micro HV
G82	ÄMUL 2170	model chisel	Giza	late Dynasty 5 [Jirásková]	148	7	2	15.9	ED-XRF
–	ÄMUL 2600	chisel	Giza	Dynasty 5-6	71	6	5	9.3	ED-XRF
–	ÄMUL 5513	razor	unknown	Dynasty 6	143	59	3	55.8	ED-XRF, OM, SEM/EDS, micro HV

* OM: Optical Microscopy; XRD: X-Ray Diffraction analysis; ED-XRF: X-Ray Fluorescence Spectrometry; SEM/EDS: Scanning Electron Microscopy with X-Ray Energy Dispersive analyzator; micro HV - Vickers Microhardness testing

FIGURE 226: SUMMARY OF PARAMETERS OF THE ANALYZED SET OF ARTEFACTS.

A deliberate addition of arsenic in copper is mostly associated with improved mechanical properties of the resulting alloy. The mechanical hardening processes most often consist of a final cold hammering operation after the forging and annealing cycles. The hardness of metallic objects is determined mainly by the composition of the alloy and by the degree of thermomechanical processing. The ductility level is almost constant until the solid solubility limit of arsenic in copper is reached (about 8 weight % of As in the equilibrium system); with

higher concentrations of arsenic, the metal may become brittle. No significant relation between the arsenic content and the measured hardness in non-worked state was found, as the results of recent research have shown.⁸⁰⁷ The hardness of the manufactured artefacts was achieved intentionally above all by cold hammering operations in the final step of the artefact fabrication.⁸⁰⁸

⁸⁰⁷ Lechtmann (1996, 494); Pereira *et al.* (2013, 2052).

⁸⁰⁸ Budd and Ottaway (1995).

Elimination of the hardening effect, which is necessary if the material is to be deformed to a higher degree, was performed by annealing operations. Arsenic copper recrystallizes at temperatures ranging from 300 to 400°C⁸⁰⁹, but a temperature up to 600°C is needed for complete recrystallization of the whole microstructure.⁸¹⁰

Corrosion deterioration of artefacts

The evaluation of the impact of corrosion damage on the examined archaeological artefacts is necessary for the correct interpretation of the results obtained from the chemical and phase composition analysis. Due to long deposition in a sandy environment and their extraction from the sand, archaeological objects made of copper alloys are exposed to external influences, above all of oxygen, moisture, chloride ions, etc. These influences have a negative impact on the material substance of artefacts and lead to their degradation and, in extreme cases, to the complete disappearance of the metallic core of the artefacts and loss of important information. The surface layers of buried copper alloys usually consist of a bottom layer of red cuprous oxide cuprite, which usually indicates the limit of the ‘original surface’ of the artefact.⁸¹¹ Copper corrosion products with oxidation state +I are usually covered by outer layers of green or blue copper compounds in oxidation state +II, which consist of one or more copper salts.⁸¹² In an aggressive environment, these corrosion processes continue until the whole metallic core of the artefact is completely corroded. The corrosion products usually contain particles of the sand environment, such as quartz, gypsum, rutile etc. The biggest problem associated with the material survey of archaeological metal artefacts is their significant corrosion damage, which influences the chemical composition and mechanical properties of the artefacts. Valuable information can sometimes be retrieved from corroded metallic fragments, as parts of their structure may remain uncorroded, or a pseudomorphic replacement of the phases by the corrosion products may occur. Corroded metallic artefacts are usually studied in the form of drilled powder samples or cross sections, which allows partial elimination of errors caused by the corrosion damage and the presence of corrosion products on their surface.

Methodology

The eight sampled artefacts can be dated to the Old Kingdom and with one unprovenanced exception, they were found at Giza (Figures 225 and 226). Four of them were chisels, two from each of Tombs D 37, Shaft 1 (ÄMUL 2118) and D 44 (ÄMUL 2120). The first one is datable to the reign of Nyuserra and the latter to late Dynasty 5 or early Dynasty 6. Both of them represent cross-cut chisels of Variant D1 with two bulges, the most

frequent Old Kingdom copper alloy model chisel. The chisel from Tomb D 7, Shaft 1 (ÄMUL 2170) is a model blade of Variant G4 imitating full-size picks. The fourth chisel is flat, with a single bevel and flared blade. It was found during the excavations of the mastabas at Giza by Georg Steindorff (ÄMUL 2600). The burr indicates that the chisel was actually used as a chisel, rather than as a model. Its Old Kingdom dating is, however, uncertain. Three razors were analysed as well. Two full-size razor blades were found at Giza in Tombs D 15, Shaft A (ÄMUL 2130) and D 203, Shaft 1 (ÄMUL 2131), both of them are most probably from late Dynasty 5 or early Dynasty 6. They represent Type C of razor blades, with an attached tang. The third razor blade (ÄMUL 5513) is unprovenanced but on the typological basis (wide blade), it can be dated to late Dynasty 6 and assigned to Type B7 of razor blades. It was made of a sheet and therefore most probably served as a model of razor blades. One artefact, fragmentarily preserved ÄMUL 2129 from the tomb D 208, Shaft 9, was named as a cosmetic spatula in the literature, datable to late Dynasty 5 or early Dynasty 6. It has been proposed in this monograph that these objects might have been Old Kingdom metal ingots. It has to be noted that 28 more artefacts from Giza examined for sampling did not have any metal core. These objects include above all Old Kingdom model tools (Figure 226).

In order to analyse the bulk composition of archaeological metal artefacts, it is often necessary to take samples. In the case of the archaeometallurgical survey of the set of copper alloys tools from the collections of the Ägyptisches Museum – Georg Steindorff – der Universität Leipzig, the sampling of the artefacts was performed directly in the university museum in Leipzig and the samples were evaluated in different analytical laboratories in the Czech Republic. Before the sampling, the selected artefacts were visualized by X-Ray radiography in order to characterize the actual state of the metallic core of the artefacts and the possibilities of their sampling. After the evaluation of X-Ray radiography visualizations, samples of metal and corrosion products were taken for material examination. The samples for ED-XRF analysis were taken with a HSS-spiral drill with a surface layer of TiN and a diameter 1 or 1.2mm through a handle Proxxon drilling machine. The drill was cleaned after each operation and replaced after a few cycles of sampling. The samples in the amount of 60–100mg were stored and transferred in small polyethylene containers. The samples for optical microscopy observation, SEM/EDS analysis and micro HV testing were taken using a hand saw with a 0.2mm thick steel blade; the maximum size of the sample was 1x2x2mm. The samples of corrosion products in the amount of 1g for XRD phase analysis were scratched from the surface of the selected objects. During the sampling, great attention was paid to the locations from which the samples were taken in order not to compromise the structure and the aesthetic value of the artefacts. The sampling locations were selected so

⁸⁰⁹ Northover (1989).

⁸¹⁰ Budd (1990).

⁸¹¹ Scott (2002, 42).

⁸¹² Selwyn (2004, 65).

Analytical techniques	Shortcut	Number of analyzed artefacts	Information expected
X-Ray diffraction analysis	XRD	4	Determination of phase composition of corrosion products.
Optical microscopy	OM	5	Identification of different phases, inclusions, the thermomechanical processes applied during artefacts production and processes of corrosion deterioration.
Scanning electron microscopy with X-Ray energy dispersive analyzator	SEM/EDS	5	Determination of main chemical phases present in metal alloy and distribution of the chemical elements in the inclusions.
Energy dispersive X-ray fluorescence spectrometry	ED-XRF	6	Identification of alloy elemental composition.
Vickers Microhardness testing	micro-HV	3	Determination of mechanical properties - hardness of the artefacts.

FIGURE 227: TECHNIQUES USED TO CHARACTERIZE COMPOSITION AND MECHANICAL PROPERTIES OF THE EXAMINED ARTEFACTS.

that they were not under the influence of the corrosion processes occurring on the surface of the artefacts and in order to achieve the best characterization of the chemical and phase composition of all the artefacts (Figure 227).

X-Ray diffraction analysis

Qualitative and semiquantitative phase analyses of powdered corrosion products taken from the artefacts were performed at the Laboratory of X-Ray Diffractometry of the University of Chemistry and Technology in Prague. The X-Ray powder diffraction data were collected at room temperature with a Bruker AXS D8 θ - θ powder diffractometer with parafocusing Bragg-Brentano geometry using CoK α radiation ($\lambda = 1.79028\text{\AA}$, U = 34kV, I = 20 or 30mA). The data were scanned with an ultrafast detector LynxEye over the angular range 16–106° (2 θ) with a step size of 0.0196° (2 θ) and the counting time of 19.2s step⁻¹. Data evaluation was performed with the software package HighScore Plus 4.0.

Optical microscopy

Five samples designated for optical microscopy observation were mounted in the Epoxy-resin Specifix-20 kit produced by STRUERS®. The mounted cross sections were grounded with SiC abrasive paper of 800 to 4000 grit size and then polished with diamond paste and detergent at a rotary polishing wheel with 3, 2 and 0.7 μm diamond size. Metallic cross-sections were etched by aqueous ferric chloride solution, and the other samples without a metallic core were observed in unetched condition. The observation was performed using the optical metallographic microscope Olympus PME3 in bright field observation mode.

Energy dispersive X-Ray fluorescence spectrometry

ED-XRF analyses were performed at the Nuclear Physics Institute of the Czech Academy of Science on the powdered metallic samples (ÄMUL 2170 and 2600) and the mounted and polished cross sections (ÄMUL 2120, 2129, 2131 and 5513). The dispersive X-Ray fluorescence spectrometer Spectro Midex with automatic motor driven XYZ precision table was used to measure the energy. The spectra from the metallic samples were acquired for 900 seconds from the distance of 2mm with the use of 0.1mm micro focus collimator. The SDD detector with FWHM <160eV was used for detection, measured at the Mn-K α line. An X-Ray tube with Mo anode with voltage 48kV was utilized for the excitation of the characteristic RTG emission. The calibration was performed using Materials and Standards CRM MBH Analytical, standards 318–322 of ČKD,⁸¹³ standard IARM 159A and fine metals standards As 3N, Bi 6N, Fe 4N, Ni 4N, Sb 5N, Sn 5N, Zn 5N.

Scanning electron microscopy with X-Ray energy dispersive analyser

The electron microprobe analysis was performed on the mounted cross sections by the scanning electron microscope TESCAN VEGA 3 equipped with secondary electrons (SE), backscattered electrons (BSE) detectors and the EDS analyser Oxford Instruments INCA 350. The analyses were performed at the Department of Metals and Corrosion Engineering at UCT Prague. The measurement was carried out under 20kV accelerating voltage from the distance of 15mm and with detection of secondary and backscattered electrons. The EDS spectra were acquired for 90s lifetime with dead time adjusted to 30–40%. The samples mounted in epoxy resin were coated with a 5nm layer of gold before measuring, in order to improve the electric conductivity of their surface.

⁸¹³ Českomoravská–Kolben–Daněk.

Vickers micro hardness testing

Hardness measurements were carried out with the Future Tech FM 700 microhardness tester. The tests were performed on the mounted and polished cross-sections of the metallic samples with the load of 100g, dwell time of 10s and under the test load of 980.7mN. At least three indentations were made for each sample in order to achieve representative results.

Results and discussion

Corrosion deterioration

An inspection of corrosion products presented on the surface of the metallic artefacts was carried out with the aim to better understand the corrosion processes that take place on archaeological alloys and the description of the environments of long-term storing of the artefacts. Most of the artefacts in the examined set were covered by a compact layer of copper corrosion products, containing in some cases impurities from the sand environment. Samples of corrosion products from selected artefacts were analysed by X-Ray diffraction analysis (XRD) and SEM/EDS analyses and the summarized results are presented in Figures 227 and 228. The razor ÄMUL 2130 with fragments of mineralized textiles was documented by a digital camera with macro lens (Figure 229).

The corrosion products on the surface of the copper alloy model chisel ÄMUL 2118 consist of a mixture of the copper oxide minerals cuprite (Cu_2O) and tenorite (CuO), quartz (SiO_2) and small amounts of the copper chloride mineral clinoatacamite ($\text{Cu}_2\text{Cl}(\text{OH})_3$) and calcium sulfate dihydrate ($\text{Ca}(\text{SO}_4)\cdot 2\text{H}_2\text{O}$). The corrosion products present on the surface of the copper alloy chisel ÄMUL 2120 consist predominantly of mineral cuprite; about 20% of the occurring mineralogical phases were impossible to identify from the obtained XRD spectra. The secondary electron image of a cross section of the corrosion product sample (see Figure 232a) shows a multiphase structure of different minerals, which were analysed by SEM/EDS (see Figure 231). The matrix of the sample is formed by copper oxides with the presence of areas enriched with arsenic and calcium, and particles of quartz are also present. The surface corrosion products of the copper alloy razor ÄMUL 2130 were identified by XRD as a mixture of the copper chloride minerals atacamite ($\text{Cu}_2\text{Cl}(\text{OH})_3$), clinoatacamite ($\text{Cu}_2\text{Cl}(\text{OH})_3$) and paratacamite ($\text{Cu}_2\text{Cl}(\text{OH})_3$), accompanied by quartz and small amounts of mineral rutile (TiO_2) and calcium sulfate dihydrate. The cross section of corrosion products from this artefact was analysed by SEM/EDS (see Figures 232 and 232d), and a map of chemical element distribution inside the corrosion layers was created. The core of the sample is formed by a mixture of copper chloride minerals, while minerals enriched with chlorides, arsenic and calcium are concentrated near the level of the original surface of the artefact. Visual observation identified fragments of textiles on the outer surface, which presently occurred in a fully mineralized form (see Figure 229) and were not analysed in more detail. They can be interpreted as the remains of the original textile cover in which the artefact was stored at the time of the burial. The last analysed sample of corrosion products with the remaining metallic core comes from the copper alloy razor ÄMUL 2131 and was analysed in the form of cross section. XRD analysis identified copper as the base material, cuprite as the main corrosion product and copper sulfide ($\text{Cu}_{1.8}\text{S}$). About 10% of the mineralogical phases were not identified, and this group may also include non-equilibrium phases of copper and arsenic. A better view into the corrosion of metallic matrix of this artefact could be obtained from the results of the SEM/EDS analysis (see Figures 231 and 232e). Non-equilibrium phases of copper and arsenic, copper-sulphide inclusions and copper oxide corrosion products are present inside the metallic matrix.

ID of artefact	Copper	Cuprite	Tenorite	Copper Sulfide	Atacamite	Clinoatacamite	Paratacamite	Rutile	Calcium sulfate dihydrate	$\text{Ca}(\text{SO}_4)\cdot(\text{H}_2\text{O})_2$	SiO_2	Quartz	Non-identified
ÄMUL 2118	0	Cu_2O	29	$\text{Cu}_{1.8}\text{S}$	31	0	$\text{Cu}_2\text{Cl}(\text{OH})_3$	0	0	8	28	0	
ÄMUL 2120	0		80		0	0		0	0	0	0	20	
ÄMUL 2130	0		0		0	31		16	2	5	27	<5	
ÄMUL 2131	50		35	0	5	0		0	0	0	0	10	

FIGURE 228: RESULTS OF X-RAY DIFFRACTION ANALYSIS OF CORROSION PRODUCTS PRESENTED ON SURFACE OF THE SELECTED ARTEFACTS (WEIGHT %).

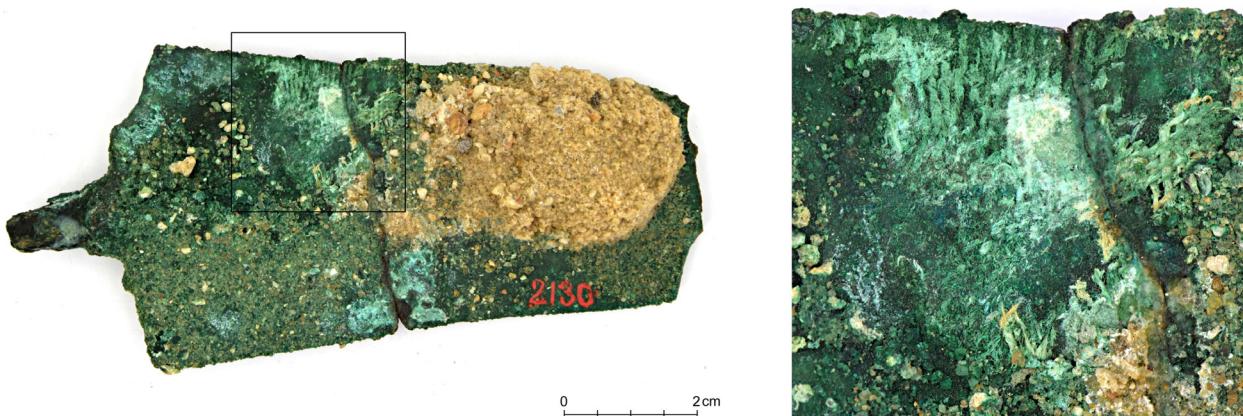


FIGURE 229: RAZOR ÄMUL 2130 WITH DETAIL OF PRESERVED FRAGMENTS OF MINERALISED TEXTILE ON THE SURFACE (PHOTO J. KMOŠEK).

Artefact	Cu	As	Sn	Fe	Ag	Sb	Ni	Zn	Bi	Pb
ÄMUL 2120	95.45	3.3	<0.05	0.2	<0.05	n.d.	<0.05	n.d.	n.d.	0.9
ÄMUL 2129	94.7	2.9	<0.05	0.3	1.9	n.d.	<0.05	n.d.	n.d.	<0.10
ÄMUL 2131	92.45	6.7	0.4	<0.20	0.2	n.d.	<0.05	n.d.	n.d.	n.d.
ÄMUL 2170	98.85	0.7	<0.05	<0.20	<0.05	n.d.	<0.05	n.d.	n.d.	<0.10
ÄMUL 2600	98.25	1.2	<0.05	0.3	<0.05	n.d.	<0.05	n.d.	n.d.	<0.10
ÄMUL 5513	95.65	3.6	<0.05	0.5	<0.05	n.d.	<0.05	n.d.	n.d.	<0.10

FIGURE 230: RESULTS OF ENERGY DISPERITIVE X-RAY FLUORESCENCE ANALYSIS OF METALLIC MATERIAL OF THE ARTEFACTS (WEIGHT %).

Artefact	Localization	Spectrum	Cu	As	Fe	Ag	Se	Te	Pb	Si	S	O	Cl	Ca
ÄMUL 2120	Corrosion products	2120/1	96.6	0	0	0	0	0	0	0	0	3.2	0.2	0
	Corrosion products	2120_2	35.4	28	0	0	0	0	0	0.3	0.9	13.5	0	21.9
	Corrosion products	2120_3	2	0	0	0	0	0	0	69.1	0	28.9	0	0
ÄMUL 2129	Matrix	2129_1	98	1.8	0.2	0	0	0	0	0	0	0	0	0
	Inclusions	2129_2	49.3	0	1.5	0	5	5.1	32.2	0	6.9	0	0	0
	Inclusions	2129_3	8.1	0	71.5	0	0	0	0	0	0.9	19.5	0	0
	Corrosion products	2129_4	65.6	1.2	0.4	9.1	0	0	0	0	9.3	0	14.4	0
ÄMUL 2130	Corrosion products	2130/1	75.7	1.6	0.7	0	0	0	0	0	0	13.2	8.4	0.4
	Corrosion products	2130/2	45.3	17.7	0	0	0	0	0	0	0	21	9.1	6.9
ÄMUL 2131	Matrix	2131/1	92.9	6.6	0.2	0	0	0	0	0	0.3	0	0	0
	Intermetallic phase	2131/2	71.8	28.2	0	0	0	0	0	0	0	0	0	0
	Inclusions	2131/3	74.1	0	3.3	0	1.5	0.7	0	0	20.4	0	0	0
	Corrosion products	2131/4	87.3	2.4	0	0	0	0	0	0	0	9.6	0.7	0
ÄMUL 5513	Matrix	5513/1	96.7	3.3	0	0	0	0	0	0	0	0	0	0
	Inclusions	5513/2	79.4	0	6.1	0	1	0	0	0	13.5	0	0	0
	Inclusions	5513/3	53.4	0	31.8	0	0	0	0	0	0.8	14	0	0

FIGURE 231: RESULTS OF THE SEM/EDS ANALYSIS OF THE MAIN CHEMICAL PHASES OCCURRING IN THE ANALYZED ARTEFACTS (WEIGHT %).

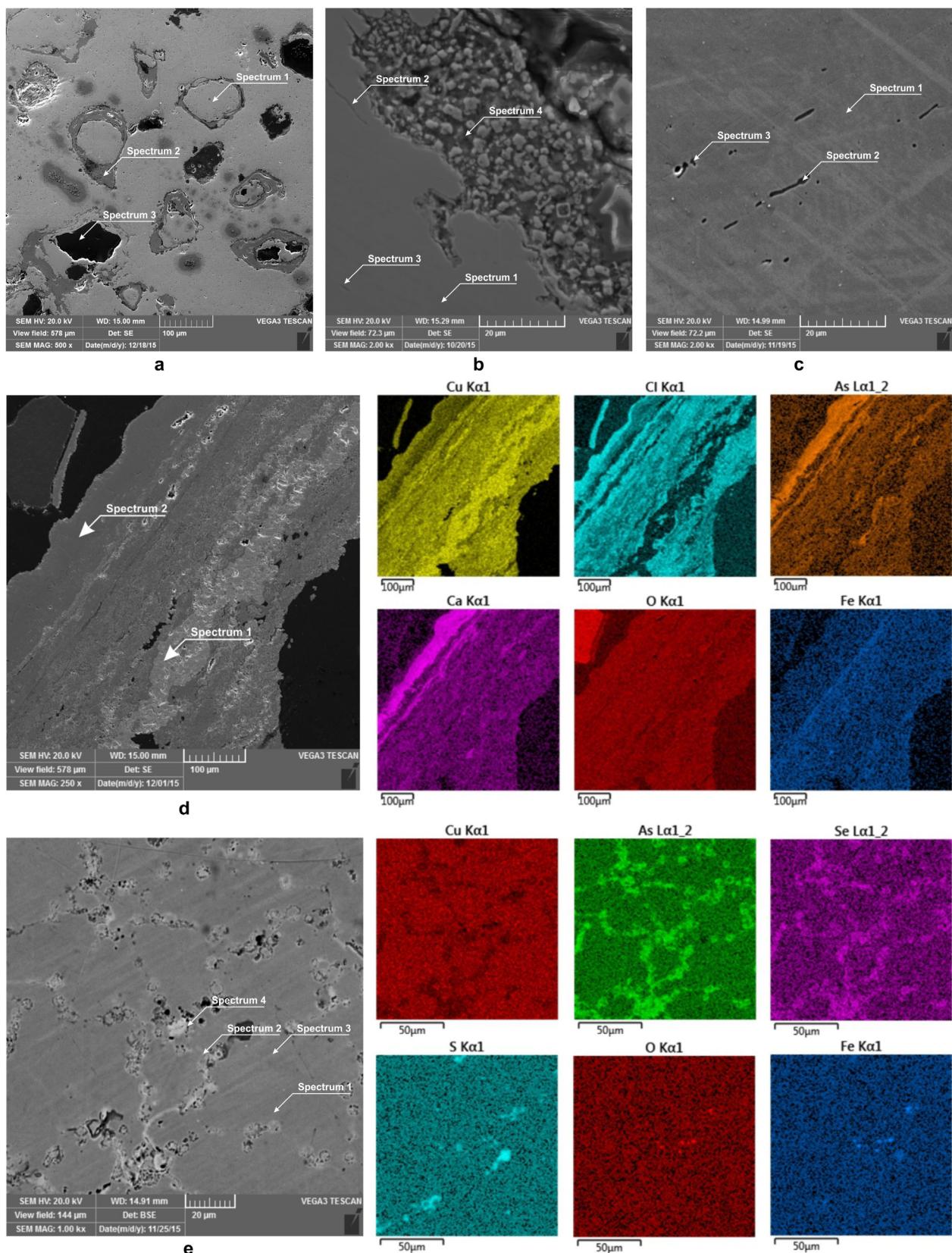


FIGURE 232: SEM IMAGES OF CROSS SECTIONS OF ANALYZED SAMPLES. A - CORRODED STRUCTURE OF SAMPLE ÄMUL 2120 (SE IMAGE, PHOTO T. JAMBOROVÁ), B - PARTLY CORRODED STRUCTURE OF SAMPLE ÄMUL 2129 (SE IMAGE, PHOTO T. JAMBOROVÁ), C - METALLIC STRUCTURE OF SAMPLE ÄMUL 5513 (SE IMAGE, PHOTO T. JAMBOROVÁ), D - CORRODED STRUCTURE OF SAMPLE ÄMUL 2130 AND MAP OF ELEMENTAL DISTRIBUTION (SE IMAGE, PHOTO T. JAMBOROVÁ), E - METALLIC STRUCTURE OF SAMPLE ÄMUL 2131 AND MAP OF ELEMENTAL DISTRIBUTION (BSE IMAGE, PHOTO T. JAMBOROVÁ).

The copper oxide minerals cuprite of dark red colour and tenorite of grey-black colour are the principal corrosion products of copper; they usually occur directly connected with the metallic surface.⁸¹⁴ The copper trihydroxychlorides atacamite, clinoatacamite and paratacamite, minerals of pale green colour, are often associated with specific corrosion deterioration of archaeological metals called ‘bronze disease’.⁸¹⁵ These unstable compounds are formed by a reaction of mineral nantokite (CuCl) with moisture and oxygen. This reaction takes place after the removal of the artefact from the sand environment due to chlorides incorporated in the corrosion layers. This type of corrosion damage can lead to very serious chemical and mechanical deterioration of archaeological artefacts resulting in cracking or fragmentation. Chloride ions necessary for the course of the reaction originate mainly from the degradation process of organic materials that occur in the immediate vicinity of the artefact. This fact is illustrated by the presence of mineralised fragments of textile on the surface of the corroded artefact.⁸¹⁶ The identified minerals such as calcium sulfate dihydrate also called gypsum, quartz and rutile are essential components of the soil in which the objects were originally situated after the burial. There, the minerals were under the influence of ongoing corrosion processes absorbed into the forming corrosion layers. Copper sulfides (digenite) occurring in small amount in metallic structures could be interpreted as an impurity originating from the pyrometallurgical process of the artefact production.⁸¹⁷ Corrosion layers enriched with arsenic and calcium (Figure 232d) are present in the cross sections of the artefacts ÄMUL 2120 and 2130. These enriched zones, which appear on the surface of the artefacts, play an important role in the evaluation of the elemental composition of alloys and can lead to very misleading results. One of the possible reasons for the presence of these layers could be the process of inverse segregation in low arsenic copper alloys during solidification after casting.⁸¹⁸ In this case, however, the presence of these layers is rather the result of the corrosion process in the sand, where migration of calcium and arsenic ions to the metal surface took place, resulting in the formation of corrosion products with a higher concentration of these elements.

Alloy composition

The results of the ED-XRF analysis of the chemical composition of the metallic material make it possible to characterize the material used for the production of the artefacts as an arsenical copper alloy with admixtures of other elements. The ED-XRF analysis results of the chemical composition of the artefacts are summarized in Figure 230 and the results of the SEM/EDS analysis of the

individual structural phases in Figure 231. All chemical composition results are listed in weight %. Arsenic, which was used as the main alloying element for copper, was found in concentrations between 0.7 and 6.7 weight percent. Tin is present in all the artefacts in concentrations <0.05%, with the exception of the razor ÄMUL 2131, which contains 0.4% Sn. Iron was detected in all the artefacts in concentrations ranging from <0.2 to 0.5%. The analysis of the examined artefacts revealed silver concentrations <0.05%, only the razor ÄMUL 2131 contains 0.2% Ag and the spatula ÄMUL 2129, surprisingly, as much as of 1.9% Ag. Such a high concentration of silver is most probably cumulated in the layers of corrosion products, as shown by the results of the SEM/EDS analysis (see Figure 231). Lead was analysed in the whole set in concentrations <0.1% except for the artefact ÄMUL 2131, where it was not detected. From the analytical point of view, it is very difficult to determine relatively low concentrations of arsenic and lead, and the whole situation is even more complicated if both elements occur together. This phenomenon is caused by an overlap of the peaks of arsenic and lead in the measured spectra. The whole set of the analysed artefacts contains nickel in concentrations <0.05%, while the content of antimony, zinc and bismuth is below the detection limit of the method used for the analysis.

The method of electron scanning microscopy with energy dispersive analyser enabled a detailed analysis of the individual microstructure phases, which helped better understand the exact description of the elemental distribution in the metallic alloy structures. The survey of the metallic matrix of cross sections revealed two different types of inclusions. The first group consists of oxide inclusions containing copper and iron – they were identified in two out of the three analysed samples. Iron and sulphur occurred above all in these oxide inclusions, with concentrations below 1%. The second group consists of sulphide inclusions containing a variable amount of copper, iron, lead, selenium and tellurium (see Figure 231). These sulphide inclusions are probably formed by the copper sulphide digenite, as was confirmed by the XRD analysis. Selenium and tellurium incorporated into these microstructural phases are found in concentrations lower than 5%. The chemical composition analysis of copper and arsenic non-equilibrium phases carried out for the structure of the razor ÄMUL 2131 provided highly variable concentrations of arsenic, which reached up to 28.2% (see Figure 232e). The other determined elements such as silicon, chlorine and calcium are most likely related with the corrosion processes, rather than with the production technologies of the artefacts or with their provenance.

Arsenic occurring in copper alloys in concentrations under 1% is generally assumed to be an accidental impurity, whereas amounts exceeding 1% are regarded as a deliberate addition.⁸¹⁹ In this case, the whole set with

⁸¹⁴ Scott (2002, 82).

⁸¹⁵ Scott (2002, 125); Selwyn (2004, 66).

⁸¹⁶ Selwyn (2004, 66).

⁸¹⁷ Tylecote (1990); Ottaway (1994).

⁸¹⁸ Meeks (1993, 268).

⁸¹⁹ Craddock (1976); Bray and Pollard (2012).

Artefact	Phases	Inclusions	Structures	Operational sequence	HV0.1	
					Average	Standard deviation
ÄMUL 2129	α	Cu-Fe-Se-Te-Pb -S and Cu-Fe-O	Recrystallized grains of α-Cu phase with elongated non-metallic inclusions and slip bands	Casting + (forging + annealing) + final annealing and fine forging	81	4.6
ÄMUL 2131	α + As-rich γ	Cu-Fe-Se-Te-S	Partly corroded, recrystallized grains of α-Cu phase with As-rich γ phases at the grain boundaries with annealing twins, slip bands at the outer surface and non-metallic inclusions	Casting + annealing and fine forging	91	5.2
ÄMUL 5513	α	Cu-Fe-Se-S and Cu-Fe-O	Extensively worked grains of α-Cu phase with large amount of slip bands and elongated non-metallic inclusions	Casting + (forging + annealing) + final extensive forging	157	8.3

FIGURE 233: RESULTS OF THE SEM/EDS ANALYSIS OF THE MAIN CHEMICAL PHASES PRESENT IN THE ANALYZED ARTEFACTS.

the exception of the artefact ÄMUL 2170 is intentionally created from an alloy of copper and arsenic. The obtained results are in a good correlation with the chronology of alloy type compositions created by Cowell, which provides an average content of 3.3% As for the Early Dynastic period and the Old Kingdom.⁸²⁰ In view of the very low concentrations of bismuth and antimony, arsenic is not expected to be a Fahlore ore product. Arsenic is supposed to have been made from a relatively pure source of its ore by alloying or co-smelting with copper. Iron oxides could be used as slag-forming additives during the smelting of slags with a high melting point, but it is more probable that the integral part of copper ores was used for the production. This hypothesis is confirmed by the research by Craddock and Meeks, who came to the conclusion that low iron content in the analysed set of artefacts implies simple smelting processes without slag formation.⁸²¹ The content of iron, arsenic and antimony could be reduced using an adequate refining process or repeated re-melting of copper under certain conditions.⁸²² The presence of silver, nickel and lead in copper alloys is most likely related to the copper ore from which the artefacts were produced. The absence of the ‘Fahlore’ element group represented by antimony and bismuth makes these alloys different from European Early Bronze Age copper alloy production. The mutual presence of selenium and tellurium in the sulphide inclusions is associated with the copper sulphide ore deposits. The contents of Se and Te and their abundance ratio seem to be characteristic of the different ore deposits, and it is generally assumed that higher concentrations of both elements occur in igneous rather than sedimentary rocks.⁸²³ A major part of selenium and tellurium remains in the sulphide phase, but under oxidizing roasting conditions of sulphide copper ores, these elements are very unstable and volatile substances.⁸²⁴

No correlation between the function and chemical composition of the analysed artefacts is visible from the viewpoint of the chemical analysis. The content of the main alloying element – arsenic – in working tools (ÄMUL 2129, 2131, 2600 and 5513) varies within the range 1.2–6.7%. Working tool models (ÄMUL 2120, 2170) contain 0.7–3.3% arsenic. From this, we can conclude that working tools and their models were made from the same alloy and the concentration of the main alloying elements such as arsenic was not evidently dependent on the function of the artefacts. The analysis of a larger set of artefacts would be necessary to refine these results.

The origin of the individual chemical elements contained in the alloys can be traced back to the original ore material, partly influenced by the pyrometallurgical technologies used in the production of the alloys. According to the classification system by Pernicka, the individual elements in copper alloys can be divided into groups concerning their bearing on provenance and/or smelting technology.⁸²⁵ The elements related to the production technology group are, in our case, iron, lead and tin. The second transitional group of provenance and production technology is represented in our case by arsenic, lead, selenium and tellurium, and the third provenance group consists of silver and nickel. More detailed evaluation of the trace element composition of the artefacts in the context of the production technology and provenance will require a more accurate analysis such as the neutron activation analysis or the proton induced X-Ray analysis.

Microstructural characterization

The results of a microstructural survey of three tools are summarized in Figure 233, and the photos of the metallic structures are presented in Figure 234. The microstructures of the spatula ÄMUL 2129 and the razor ÄMUL 5513 consist of one-phase α-Cu solid

⁸²⁰ Cowell (1987, 99).

⁸²¹ Craddock and Meeks (1987, 193).

⁸²² Tylecote *et al.* (1977).

⁸²³ Rehren and Northover (1990, 222).

⁸²⁴ Tylecote *et al.* (1977).

⁸²⁵ Pernicka (2015, 253).

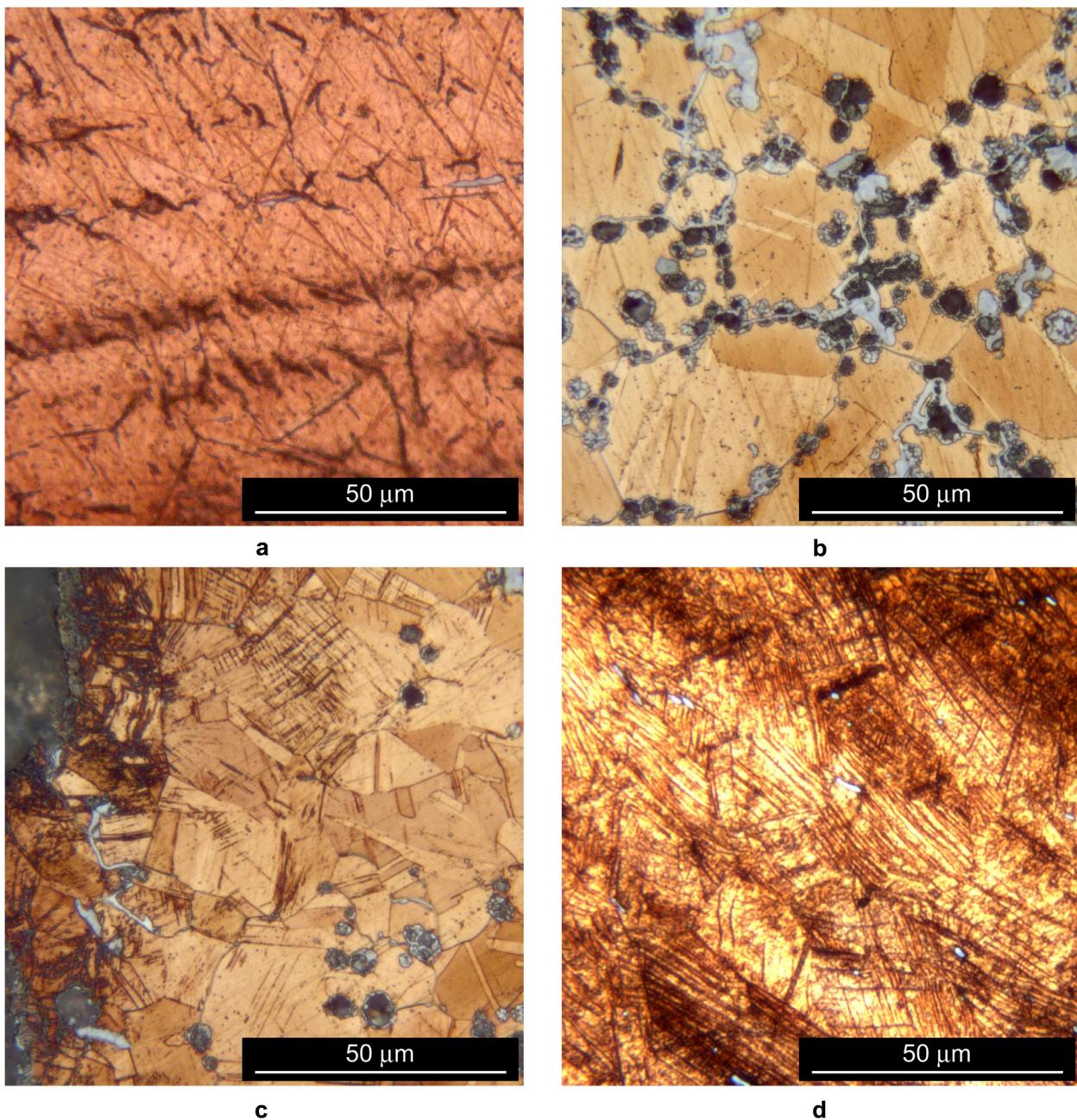


FIGURE 234: OPTICAL MICROSCOPY IMAGES OF THE METALLIC STRUCTURES AFTER ETCHING. A - RECRYSTALLIZED GRAINS OF α -Cu PHASE AND SLIP LINES (ÄMUL 2129, PHOTO M. KMONÍČKOVÁ), B - PARTLY CORRODED, FULLY RECRYSTALLIZED GRAINS OF α -Cu PHASE WITH AS-RICH γ PHASES AT THE GRAIN BOUNDARIES (ÄMUL 2131, PHOTO K. ŠÁLKOVÁ), C - PARTLY CORRODED, RECRYSTALLIZED GRAINS OF α -Cu PHASE WITH AS-RICH γ PHASES AT THE GRAIN BOUNDARIES AND SLIP LINES (OUTER SURFACE OF SAMPLE ÄMUL 2131, PHOTO K. ŠÁLKOVÁ), D - EXTENSIVELY WORKED GRAINS OF α -Cu PHASE WITH LARGE AMOUNT OF SLIP LINES (ÄMUL 5513, PHOTO M. KMONÍČKOVÁ).

solution of copper and arsenic (Figure 234 a, d) , while the razor ÄMUL 2131 consists of two-phase structure of α -Cu and As-rich γ phase, situated unequally on the grain boundaries (Figures 232e and 234b). The spatula 2129 shows recrystallized grains with slip lines and elongated non-metallic inclusions. The structure of the razor ÄMUL 2131 contains fully recrystallized regular grains with annealing twins, slip lines on the outer surface of the sample and non-deformed non-metallic inclusions, accumulated with As-rich γ phases at the grain boundaries. These grain boundaries are partly

corroded and filled with oxide corrosion products. The razor ÄMUL 5513 shows extensively worked grains with a large amount of slip lines and elongated non-metallic inclusions (Figure 234d).

The microhardness values measured on the mounted cross-sections are presented in Figure 233. Microhardness of the fine worked structure of the spatula ÄMUL 2129 with the average content of 2.9% As is 81 HV0.1, and in case of the annealed razor ÄMUL 2131 with the average content of 6.7% As, it

is 91 HV0.1. Microhardness of the intensively worked razor ÄMUL 5513 with the average content of 3.6% As has a surprisingly high value of 157 HV0.1. The standard deviation of the measured microhardness values is caused by the inhomogeneous structures of the artefacts, from the point of view of inclusion distribution and non-uniform thermo-mechanical processing. The results clearly indicate that microhardness depends more on the thermomechanical processing of the artefacts than on the content of arsenic, in accordance with the results of other studies.⁸²⁶ The production technology of the selected artefacts was probably divided into several individual steps consisting of alloy preparation, casting, forging and recrystallization annealing operations. Various combinations of these operations could have resulted in very different mechanical properties of the final products, depending on the different functions of the artefacts. We have not enough data to compare the thermomechanical processing technologies of working tools and their models, because all microstructural analyses involved working tools.

Conclusion

The results of the analysed collection of artefacts from ÄMUL provided insight into the fields of corrosion deterioration, material composition, microstructure features and mechanical properties of Egyptian working tools and their models dated to Dynasty 5 and 6. Out of the set of more than thirty artefacts from the Giza necropolis, only eight artefacts could be sampled for the purposes of the analysis, due to the considerable degree of corrosion deterioration. The metallic cores of the other artefacts were completely transformed into corrosion products. The corrosion products of the analysed artefacts consist on the one hand of a mixture of copper oxide minerals (cuprite and tenorite) with higher amount of arsenic, and on the other hand of a mixture of copper chloride minerals (atacamite, clinoatacamite and paratacamite). The corrosion products contain a certain portion of sand particles, represented by quartz, gypsum and rutile. The chemical composition of the alloys in the analysed set indicates a fairly consistent composition, corresponding to arsenic copper alloys containing arsenic up to 3.6%, iron up to 0.5% and admixtures of tin, silver, nickel and lead. There is only one exception – the razor ÄMUL 2131, which contains higher portion of arsenic, tin and silver and surprisingly does not contain lead. The chemical composition of the working tools and their models seems to be unstable in the proportion of arsenic and there is no visible correlation between the chemical composition and the function of the artefacts. Two out of the three examined microstructures were annealed and contain slip lines on the surface. The third microstructure was fully worked and contains a large amount of slip lines. An As-rich γ phase in the intergranular regions

was documented in one case, related to the presence of inverse segregation during solidification after casting. Two types of inclusions were identified in the structure of metallic cross sections. The first type is represented by mixed oxides of copper and iron, and the second type by copper-iron sulphide inclusions with a portion of selenium, tellurium and lead in some cases. The results of Vickers microhardness tests of three artefacts are comparable with the results obtained earlier by other authors and confirm that the hardness of the artefacts was intentionally achieved by mechanical hardening rather than by the alloying effect of alloys with a higher portion of arsenic. The techniques of casting, alloying, hot or cold working, annealing, final cold working and surface finishing were used in the production of the artefacts. From the obtained results it is not possible to distinguish which technology was used for the production of the arsenic copper alloys, but at least the artefacts ÄMUL 2129, 2131 and 5513 were made from rich sulphide ores, which is indicated by the presence of selenium and tellurium.

11.2. Morphometrical and statistical case study of Old Kingdom adze blades

Martin Odler⁸²⁷ and Ján Dupej⁸²⁸

Introduction

The tools of geometric morphometry have been used in anthropology and biology for decades now. On the other hand, their application in other fields, such as archaeology, has been somewhat slower. Geometric morphometry is the quantitative study of shape with the application of multivariate statistical and geometric approaches in the evaluation of the data. The historically earlier traditional morphometry used distances, lengths and angles to describe a particular specimen in the set. In contrast, geometric morphometry (GM) describes shapes using landmarks. The term originates in geography and refers to a well recognizable feature used for navigation. In GM, this term is used for an anatomically significant locus, present and repeatable in all the studied specimens.⁸²⁹ Each specimen in the set must be described by an equal number of landmarks, placed in these anatomically equivalent loci. This property, referred to as homology, is crucial for a successful application of statistics on the data.

The configurations of landmarks describing a specimen are generally in random locations and orientations

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⁸²⁹ Bookstein (1997b).

⁸²⁶ Pereira *et al.* (2013); Lechtman (1996); Junk (2003).

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