Diachronic changes of ancient Egyptian and Nubian metallurgy

Case study of material from the Egyptian Museum of Leipzig University

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Figure 1 – Photo documentation of the set of analyzed artefacts arranged into chronological groups. The numbers are inventory numbers of the ÄMUL collection

INTRODUCTION

The Ägyptisches Museum – Georg- Steindorff – der Universität Leipzig (ÄMUL, Germany) holds an important collection of ancient Egyptian and Nubian artefacts. The sampled 86 artefacts represent the development of ancient Egyptian metallurgy in more than one and a half millennia, from Dynasty 1 (ca. 3100 – 2900 BC) until almost the end of the New Kingdom (ca. 1200 BC).

The most important assemblages are from the (Early Bronze Age) Dynasty 1 Abusir (Bonnet 1928), Dynasty 2 Tomb of King Khasekhemwy at Abydos (Kuhn 2011) and the Old Kingdom cemetery at Giza (Steindorff – Hölscher 1991). The largest sampled corpus is from the Nubian site Aniba, from the Middle Bronze Age Nubian C-Group Cemetery N and from the Late Bronze Age New Kingdom Cemetery S (Steindorff 1935 – 1937).

The sampled artefacts can be divided into several morphological categories: full-size tools, model tools, full-size vessels, mirrors and other metal objects (e.g. bolts). A diachronic change of the ore sources and technology as well as other issues can be studied in detail on the corpus (see Methodology and the discussion of the results).

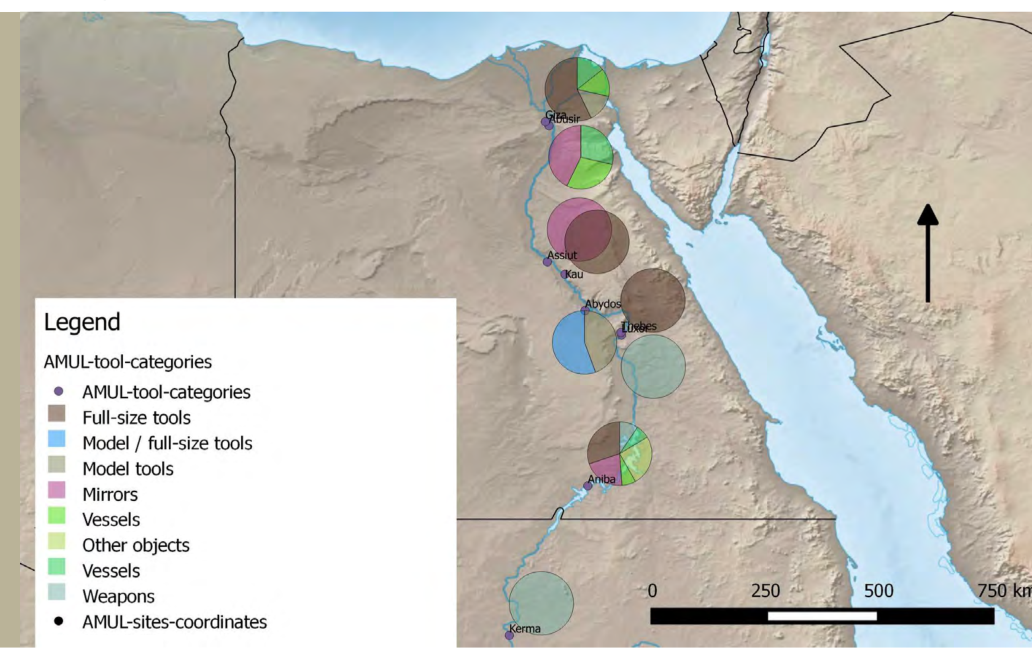

Figure 2 – Map of Egypt and Nubia with marked locations of the examined set of artefacts

METHODOLOGY

X-Ray radiography and X-Ray CT tomography – visualization of the constructions and mechanical state of the artefacts

Sampling – drilling of metallic material in the amount of 60-100 mg and sawing of 1x2x2 mm samples

X-Ray diffraction analysis (powder diffractometer Bruker AXS D8) – qualitative and semi quantitative phase analyses of powdered corrosion products and metallic phases (16 artefacts)

Energy dispersive X-Ray fluorescence spectrometry (X-Ray fluorescence spectrometer Spectro Midex) – quantitative chemical analysis of the base composition of the artefacts (92 artefacts)

Optical microscopy (metallographic microscope Olympus PME3) – identification of metallic structures (31 artefacts)

Scanning electron microscopy with X-Ray energy dispersive analyser (TESCAN VEGA 3 with EDS analyser Oxford Instruments INCA 350) – quantitative chemical microanalysis of structural phases and base composition (31 artefacts)

Vickers micro hardness testing (microhardness tester Future Tech FM 700) – analysis of mechanical properties – microhardness tests (22 artefacts)

Neutron activation analysis (in progress) – specification of ED-XRF results and identification of trace elements (Se, Te, Ag, Au, Pt, Ni, As, Sb, Bi, ...) of 80 artefacts

Lead isotopic analysis (in progress) – characterization of the geographic provenience of copper ores used in the production of 40 artefacts

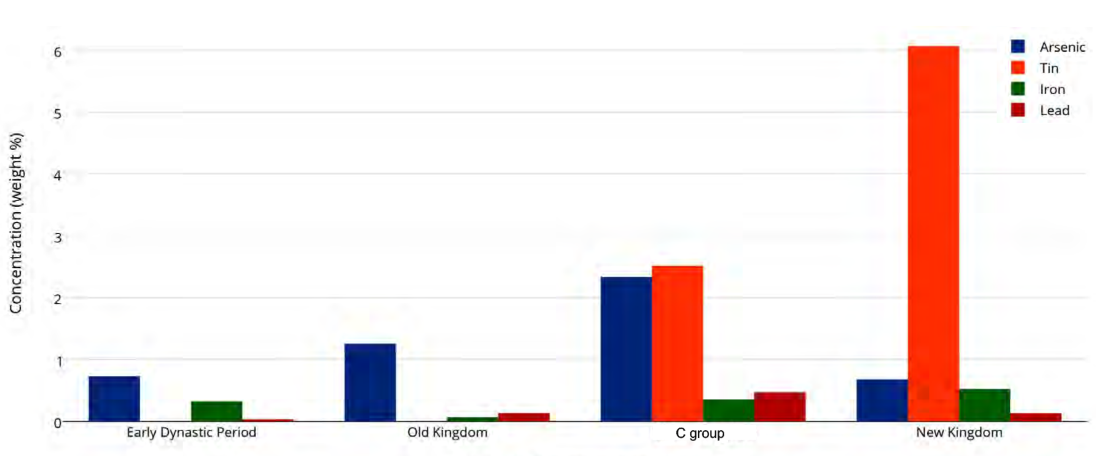

Chart 1 – Bar chart of the development of the chemical composition of copper alloy artefacts from the Early Dynastic period to the New Kingdom

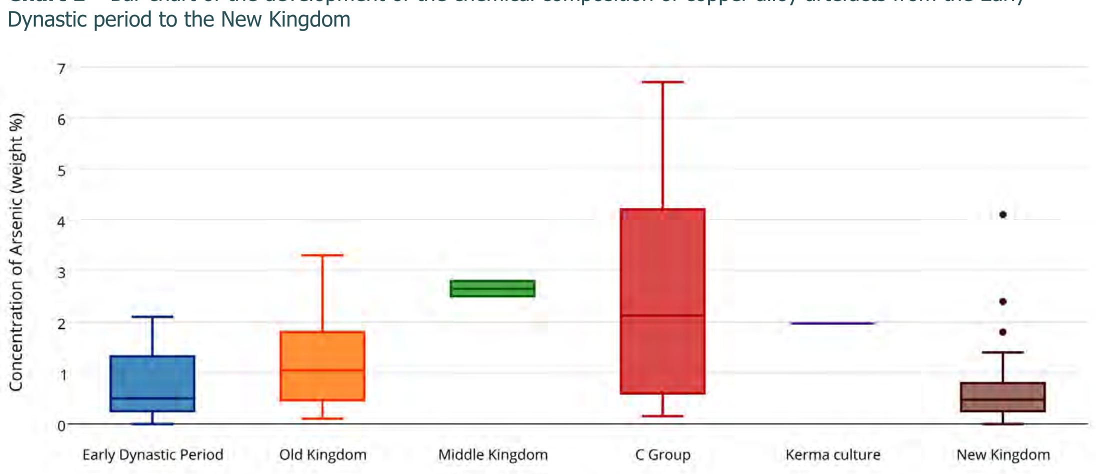

Chart 2 – Box plot chart presenting changes in the concentration of arsenic across the individual periods of ancient Egypt and Nubia

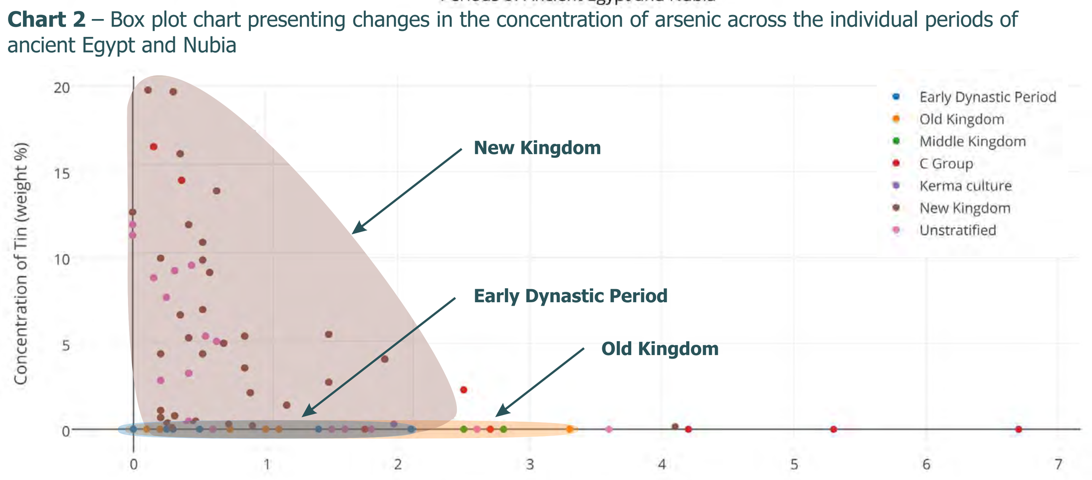

Chart 3 – Scatter plot of the ratios of arsenic and tin concentrations in artefacts from different periods and cultures

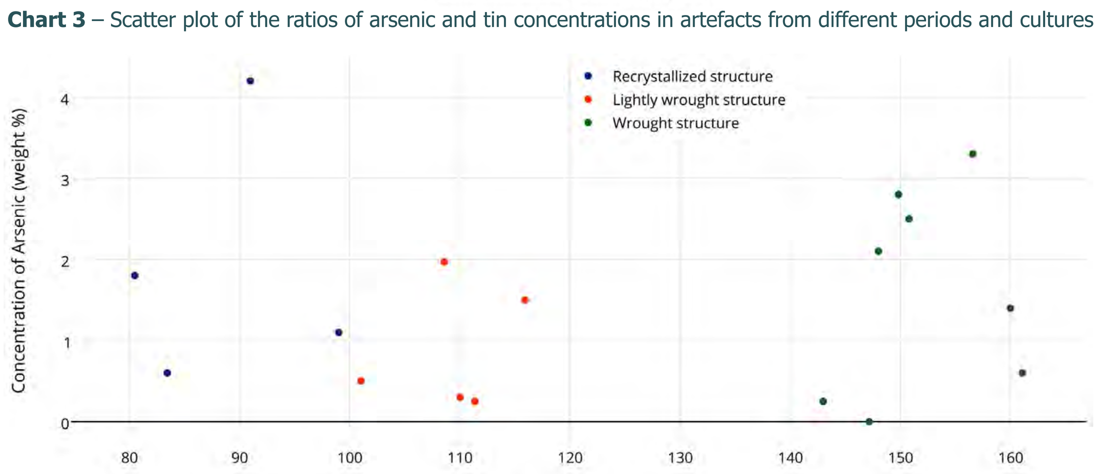

Chart 4 – Scatter plot of the ratios of arsenic concentrations and values of microhardness

Alloy	Definition	Shortcut	Amount of artefacts	In common use
copper	< 0.05 % of admixtures	Cu	1 %	from Dynasty 1
copper with admixtures	0,05 - 1 % of admixtures	Cu+	24 %	from Dynasty 1
arsenical copper	> 1 % As	CuAs	26 %	from Dynasty 1
nickel arsenical bronze	> 1 % Ni, > 1 % As	CuAsNi	1 %	from Dynasty 1
tin bronze with arsenic	> 1 % Sn, > 1 % As	CuSnAs	7 %	from Second Intermediate period
tin bronze	1-8 % Sn	CuSn	33 %	from Second Intermediate period
lead bronze	> 1 % Pb	CuPb	2 %	from Second Intermediate period
tin lead bronze	> 1 % Sn, > 1 % Pb	CuSnPb	5 %	from Dynasty 18-19
brass	> 1 % Zn	CuZn	1 %	out of context (imitation)
gun metal	> 1 % Sn, > 1 % Pb, > 1 % Zn	CuSnPbZn	1 %	out of context (imitation)
tin bronze with zinc	> 1 % Sn, > 1 % Zn	CuSnZn	1 %	out of context (imitation)

Table 1 – Parameters of copper alloys represented in the analyzed set of artefacts

CHEMICAL COMPOSITION ANALYSIS

The copper alloys used for the production of the analyzed artefacts can be divided into groups based on their chemical composition. The most common artefacts were made of a tin bronze alloy, an arsenical copper alloy or copper with admixtures of arsenic, iron and lead. Six artefacts were identified as tin bronze with arsenic; they were most probably results of recycling of older artefacts made of arsenical copper. Few artefacts from the New Kingdom were made of an alloy of tin-lead bronze and lead bronze. One object was made of non-alloyed pure copper and one of an alloy of copper nickel and arsenic.

The most frequent elements in copper alloys are arsenic, tin, lead and iron. Arsenic was intentionally used for the alloying of copper at least from the Early Dynastic period; the concentration of arsenic reached the maximum in the Middle Kingdom. Starting with the Middle Kingdom, arsenic was gradually replaced by tin. Regular concentrations of arsenic do not exceed the limit of 4 weight percent. Higher concentrations are visible only in the production of the C-group culture in Nubia. The low concentration of lead seems to correspond to the concentration of arsenic, but it was not used intentionally, and nor was iron.

METALLOGRAPHIC ANALYSIS

In most cases, the structures were formed by a single-phase solid solution of copper and arsenic or copper and tin. Only one full-size tool from the late Old Kingdom with 6 % of arsenic analysed by metallographic methods contained a two-phase structure of α copper and arsenic rich γ phase (Cu_3As). A bronze vessel with 10 % of tin from Aniba dated to Dynasty 18 contained a two-phase structure of α copper and non-equilibrium phase δ ($Cu_{41}Sn_{11}$). Three analysed structures were formed by dendrites originating from the casting operations. The most frequent structures were formed by recrystallized or wrought grains with non-metallic inclusions in different states of deformation. These structures are corresponding to different thermomechanical techniques of metal processing, especially casting, annealing and hammering.

Metallographic structures	Operational sequence	In common use
dendritic structure	casting	from Dynasty 1
recrystallized grains of α-Cu phase with non-deformed non-metallic inclusions and annealing twins	casting + annealing	from Dynasty 1
recrystallized grains of α-Cu phase with elongated non-metallic inclusions and slip bands	casting + (forging + annealing) + final annealing and fine forging	from Dynasty 1
extensively deformed grains of α-Cu phase with large amount of slip bands and elongated non-metallic inclusions	casting + (forging + annealing) + final extensive forging	from Dynasty 2

Table 2 – Metallographic structures and associated operational sequences of the analyzed artefacts

MICROHARDNESS TESTS

The microhardness of the tested arsenical copper alloys ranges between 80 and 160 Vickers hardness units. The results clearly indicate that microhardness depended more on thermomechanical processing of the artefacts than on the content of arsenic and its alloying effect. The hardness of artefacts with wrought structures and low concentration of arsenic is much higher than that of recrystallized structures with a high portion of arsenic.

Artefact	Artefact ID	Site	Region	Category	Period/culture	Dynasty	Cu	As	Sn	Fe	Pb	Ni	Zn	Ag	Sb	Se	Alloy
vessel	2160	Abusir	Memphis	full-size vessel	Early Dynastic period	Dynasty 1	100.0	0	0	0	0	0	0	0	0	0	Cu
vessel	2162	Abusir	Memphis	full-size vessel	Early Dynastic period	Dynasty 1	92.8	1.5	0	0.9	0	4.8	0	0	0	0	CuNiAs
adze	2211	Abydos	Upper Egypt	model tool	Early Dynastic period	Dynasty 2	99.6	0.3	0	0	0.1	0	0	0	0	0	Cu+
axe	2212	Abydos	Upper Egypt	model tool	Early Dynastic period	Dynasty 2	98.5	1.1	0	0.4	0	0	0	0	0	0	CuAs
harpoon	2213	Abydos	Upper Egypt	model tool	Early Dynastic period	Dynasty 2	99.5	0.25	0	0.2	0	0.05	0	0	0	0	Cu+
chisel	2216	Abydos	Upper Egypt	model tool	Early Dynastic period	Dynasty 2	98.8	0.5	0	0.3	0.26	0.14	0	0	0	0	Cu+
needle	2220	Abydos	Upper Egypt	model or full-size tool	Early Dynastic period	Dynasty 2	99.5	0	0	0.5	0	0	0	0	0	0	Cu+
needle	2217	Abydos	Upper Egypt	model or full-size tool	Early Dynastic period	Dynasty 2	99.15	0.65	0	0.2	0	0	0	0	0	0	Cu+
needle	2218	Abydos	Upper Egypt	model or full-size tool	Early Dynastic period	Dynasty 2	99.5	0.25	0	0.2	0	0.05	0	0	0	0	Cu+
needle	2219	Abydos	Upper Egypt	model or full-size tool	Early Dynastic period	Dynasty 2	97.6	2.1	0	0.3	0	0	0	0	0	0	CuAs
needle	2222	Abydos	Upper Egypt	model or full-size tool	Early Dynastic period	Dynasty 2	98.4	1.4	0	0.2	0	0	0	0	0	0	CuAs
razor	2131	Giza	Memphis	full-size tool	Old Kingdom	Dynasty 5–6	97.95	1.85	0	0.2	0	0	0	0	0	0	CuAs
adze	2600	Giza	Memphis	full-size tool	Old Kingdom	Dynasty 5–6	98.87	0.73	0	0.2	0.2	0	0	0	0	0	Cu+
chisel	2120	Giza	Memphis	model tool	Old Kingdom	Dynasty 5–6	99.9	0.1	0	0	0	0	0	0	0	0	Cu+
vessel	2169_1	Giza	Memphis	full-size vessel	Old Kingdom	Dynasty 5–6	99.25	0.2	0	0.2	0.3	0.05	0	0	0	0	Cu+
vessel	2169_2	Giza	Memphis	full-size vessel	Old Kingdom	Dynasty 5–6	98.4	1.1	0	0.2	0.3	0	0	0	0	0	CuAs
vessel	2169_3	Giza	Memphis	full-size vessel	Old Kingdom	Dynasty 5–6	98.54	1.0	0	0.2	0.26	0	0	0	0	0	CuAs
spatula?	2129	Giza	Memphis	full-size tool	Old Kingdom	Dynasty 5–6	98.0	1.8	0	0.2	0	0	0	0	0	0	CuAs
razor	5513	unknown	unknown	full-size tool	Old Kingdom	Dynasty 6	96.7	3.3	0	0	0	0	0	0	0	0	CuAs
axe	3952	Thebes	Thebes	full-size tool	Middle Kingdom	Dynasty 12–13	97.05	2.5	0	0.2	0.2	0.05	0	0	0	0	CuAs
razor	5515	Qau	Upper Egypt	full-size tool	Middle Kingdom	Dynasty 12–13	96.7	2.8	0	0.5	0	0	0	0	0	0	CuAs
dagger	5074	Luxor	Thebes	forgery	19-20 century	19–20 century	76.8	0.1	2.5	0.2	20.1	0.3	0	0	0	0	CuSnPb
dagger	2152	Aniba	Nubia	full-size weapon	C group	Second Intermediate period	94.15	5.3	0	0.5	0	0.05	0	0	0	0	CuAs
mirror	4703_1	Aniba	Nubia	full-size tool	C group	Second Intermediate period	97.65	0.6	0	0.3	1.4	0.05	0	0	0	0	CuPb
mirror	4703_2	Aniba	Nubia	full-size tool	C group	Second Intermediate period	97.0	2.75	0	0.2	0	0.05	0	0	0	0	CuAs
mirror	4701	Aniba	Nubia	full-size tool	C group	Second Intermediate period	92.75	6.7	0	0.2	0.3	0.05	0	0	0	0	CuAs
mirror	4700	Aniba	Nubia	full-size tool	C group	Second Intermediate period	95.48	4.2	0	0.3	0	0.015	0	0	0	0	CuAs
tweezers	4647	Aniba	Nubia	full-size tool	C group	Second Intermediate period	93.3	2.55	2.35	0.8	0.5	0.5	0	0	0	0	CuSnAs
tweezers	2182	Aniba	Nubia	full-size tool	C group	Second Intermediate period	82.75	0.15	16	0.2	0.9	0	0	0	0	0	CuSn
axe	4698	Aniba	Nubia	full-size weapon	C group	Second Intermediate period	97.73	1.75	0	0.2	0.2	0.015	0	0.05	0	0.05	CuAs
axe	4697_1	Aniba	Nubia	full-size weapon	C group	Second Intermediate period	82.85	0.35	14.1	0.2	2.5	0	0	0	0	0	CuSnPb
ferrule	4697_2	Aniba	Nubia	full-size object	C group	Second Intermediate period	98.9	0.6	0	0.5	0	0	0	0	0	0	Cu+
dagger	3791	Kerma	Nubia	full-size weapon	Kerma culture	Kerma culture	97.33	1.97	0.3	0.2	0.1	0.05	0	0.05	0	0	CuAs
adze	2141_1	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18, second half	90.3	0.55	8.9	0.2	0	0	0	0.05	0	0	CuSn
adze	2141_2	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18, second half	96.55	2.4	0.8	0.2	0	0	0	0.05	0	0	CuAs
dagger	2153	Aniba	Nubia	full-size weapon	New Kingdom	Dynasty 18, early	80.4	0.1	19.2	0.2	0	0	0	0.1	0	0	CuSn
vessel	2167_1	Aniba	Nubia	full-size vessel	New Kingdom	Dynasty 18	96.2	0.1	3.45	0.2	0	0	0	0.05	0	0	CuSn
vessel	2167_2	Aniba	Nubia	full-size vessel	New Kingdom	Dynasty 18	89.8	0.2	9.7	0.3	0	0	0	0	0	0	CuSn
hooked knife	2138	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 19	80.36	0.29	19.1	0.2	0	0	0	0.05	0	0	CuSn
hooked knife	2139	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 19–20	93.6	0.65	4.9	0.4	0.4	0	0	0.05	0	0	CuSn
bolt	2191_1	Aniba	Nubia	full-size object	New Kingdom	Dynasty 18, early	99.5	0.2	0.05	0.2	0	0	0	0.05	0	0	Cu+
bolt	2191_2	Aniba	Nubia	full-size object	New Kingdom	Dynasty 18, early	98.15	0.3	0.8	0.2	0.2	0	0.3	0.05	0	0	Cu+
hook	2142	Aniba	Nubia	full-size object	New Kingdom	Dynasty 18, early	94.0	0.4	5.2	0.3	0	0.1	0	0	0	0	CuSn
hook	2146	Aniba	Nubia	full-size object	New Kingdom	Dynasty 18	94.95	0.8	3.5	0.7	0	0	0	0.05	0	0	CuSn
bolt	2192	Aniba	Nubia	full-size object	New Kingdom	Dynasty 18. early	98.44	0.9	0.21	0.4	0	0	0	0.05	0	0	Cu+
mirror	2171	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18, first half	99.4	0.3	0.05	0.2	0	0	0	0.05	0	0	Cu+
mirror	2173	Aniba	Nubia	full-size tool	New Kingdom	New Kingdom	95.5	4.1	0.15	0.2	0	0	0	0.05	0	0	CuAs
mirror	8439	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 19	79.6	1.0	17.1	0.2	2.1	0	0	0	0	0	CuSnAs
tweezers	2185	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18, first half	95.1	1.4	2.15	1.1	0.2	0	0	0.05	0	0	CuSnAs
tweezers	2186	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 19, end	92.85	1.8	4.0	1.1	0.2	0	0	0.05	0	0	CuSnAs
tweezers	2187	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18, early	95.0	0.5	4.3	0.2	0	0	0	0	0	0	CuSn
tweezers	2188	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18, first half	93.05	0.8	5.3	0.6	0.2	0	0	0.05	0	0	CuSn
hair curler	2226	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18, first half	92.1	1.4	5.4	0.7	0.2	0.1	0	0.1	0	0	CuSnAs
razor	2180_1	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18	99.36	0.29	0.1	0.2	0	0	0	0.05	0	0	Cu+
razor	2180_2	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18	99.47	0.23	0.05	0.2	0	0	0	0.05	0	0	Cu+
kohlstick	2189	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 19–20	98.5	0.75	0.3	0.4	0	0	0	0.05	0	0	Cu+
mirror	2178_1	Abusir	Memphis	full-size tool	New Kingdom	Dynasty 18	95.25	0.2	4.3	0.2	0	0	0	0.05	0	0	CuSn
mirror	2178_2	Abusir	Memphis	full-size tool	New Kingdom	Dynasty 18	92.8	0.35	6.5	0.2	0.1	0	0	0.05	0	0	CuSn
mirror	2172_1	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18	99.2	0.23	0.05	0.2	0	0.12	0	0.2	0	0	Cu+
mirror	2172_2	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18	84.74	0.6	13.5	0.2	0.4	0.1	0	0.46	0	0	CuSn
nail	2143_1	Aniba	Nubia	full-size object	New Kingdom	Dynasty 18	98.7	0.45	0.5	0.2	0.1	0	0	0.05	0	0	Cu+
nail	2143_2	Aniba	Nubia	full-size object	New Kingdom	Dynasty 18	98.35	0.2	1.1	0.2	0.1	0	0	0.05	0	0	CuSn
bolt	2144	Aniba	Nubia	full-size object	New Kingdom	Dynasty 18	98.8	0.25	0.4	0.4	0.1	0	0	0.05	0	0	Cu+
mirror	2176_1	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18, second half	83.6	0.35	15.6	0.3	0.1	0	0	0.05	0	0	CuSn
mirror	2176_2	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18, second half	87.65	0.5	10.6	0.5	0	0	0.7	0.05	0	0	CuSn
mirror	2176_3	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18, second half	96.8	0.85	2.1	0.2	0	0	0	0.05	0	0	CuSn
bolt	2145	Aniba	Nubia	full-size object	New Kingdom	Dynasty 18–19	89.2	0.5	9.55	0.2	0.5	0	0	0.05	0	0	CuSn
razor	2179_1	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18, second half	87.75	0.4	11.6	0.2	0	0	0	0.05	0	0	CuSn
razor	2179_2	Aniba	Nubia	full-size tool	New Kingdom	Dynasty 18, second half	97.25	1.1	1.4	0.2	0	0	0	0.05	0	0	CuSnAs
vessel	4417	Aniba	Nubia	full-size vessel	New Kingdom	Dynasty 18–19	90.7	0.5	6.8	0.2	0	0.1	1.7	0	0	0	CuSnPb

Table 3 – Parameters of the analyzed artefacts and results of ED-XRF and SEM/EDS chemical composition analysis

Structural phases	Description	Occurrence
α-Cu	α solid solution of copper and other elements	from Dynasty 1
Cu_3As	ɣ intermetallic non-equilibrium phase of copper and arsenic	from Dynasty 5–6
$Cu_{41}Sn_{11}$ ($Cu_{39}Sn_{11}$)	δ intermetallic non-equilibrium phase of copper and tin	from Second Intermediate period
Pb+Fe, S, Se, Te, Ag	metallic particles of lead with admixtures of Fe, S, Se, Te, Ag	from Dynasty 2
Cu+S, As, Fe, Se, Te	non-metallic inclusions of copper sulphide with admixtures of As, Fe, Se, Te	from Dynasty 1
Cu+O, Fe+O	non-metallic inclusions of copper and iron oxides	from Dynasty 2
Cu+Si, O	non-metallic inclusions of copper-silicon compounds	from Dynasty 2
corrosion products of copper	cuprite (Cu_2O), tenorite (CuO), atacamite ($Cu_2Cl(OH)_3$), malachite ($Cu_2(OH)_2CO_3$)	–

Table 4 – Identified structural phases in the analyzed set of artefacts

ANALYSIS OF MICROSTRUCTURAL PHASES

Different types of inclusions have been revealed using SEM/EDS and XRD analyses of metallic cross sections. The first main group consists of oxide inclusions containing copper and iron, and the second of sulphide inclusions containing a variable amount of copper, iron, lead, arsenic, selenium and tellurium. These sulphide inclusions are usually formed by various copper sulphide minerals (chalcocite, digenite). Selenium and tellurium incorporated into these non-metallic and non-equilibrium metallic microstructural phases were found in concentrations below 5 %. The mutual presence of selenium and tellurium in sulphide inclusions is most probably associated with the copper sulphide ore deposits. Non-metallic inclusions composed of copper-silicon compounds and metallic particles of lead with admixtures of iron, sulphur, selenium, tellurium and silver were also identified in some cases. The other determined elements such as silicon, chlorine and calcium are most likely related to the corrosion processes.

CONCLUSIONS AND FURTHER RESEARCH

- Arsenical copper was known in Egypt already in the Naqada culture. It is present in our corpus from the earliest artefacts of Dynasty 1. Arsenic was used as the main alloying element until the Middle Kingdom, when it was gradually replaced with tin.
- The hardness of artefacts was intentionally achieved by mechanical hardening rather than using the alloying effect of arsenic.
- The production techniques of casting, alloying, annealing, hot or cold hammering and surface finishing were commonly used from the Early Dynastic Period.
- The largest part of the analysed artefacts was made of rich sulphide copper ores, which is indicated by the presence of selenium and tellurium in non-metallic inclusions.

The project is continuing by neutron activation analysis of all samples used for the identification of trace elements, and by lead isotope analysis of selected artefacts used for the characterization of the geographic provenance of the copper ores used for the production of copper. The results of neutron activation analysis and lead isotope analysis will enable detailed determination of the alloys used for the production of the sampled artefacts.

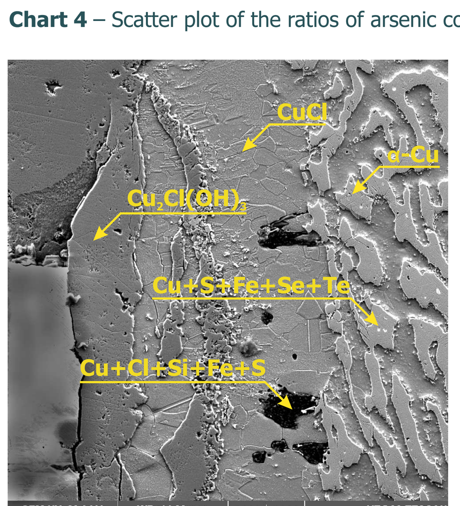

Figure 3 – Secondary electron image of the microstructure of sample 2169

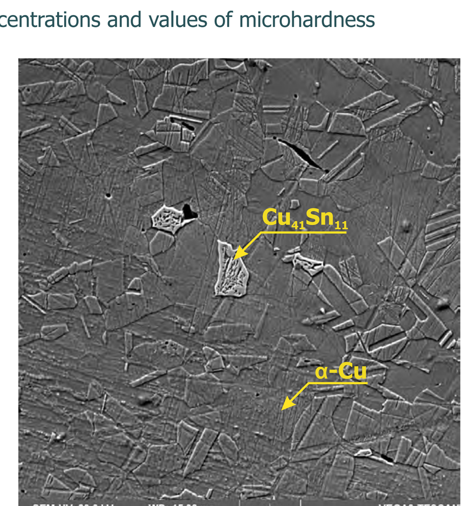

Figure 4 – Secondary electron image of the microstructure of sample 2167

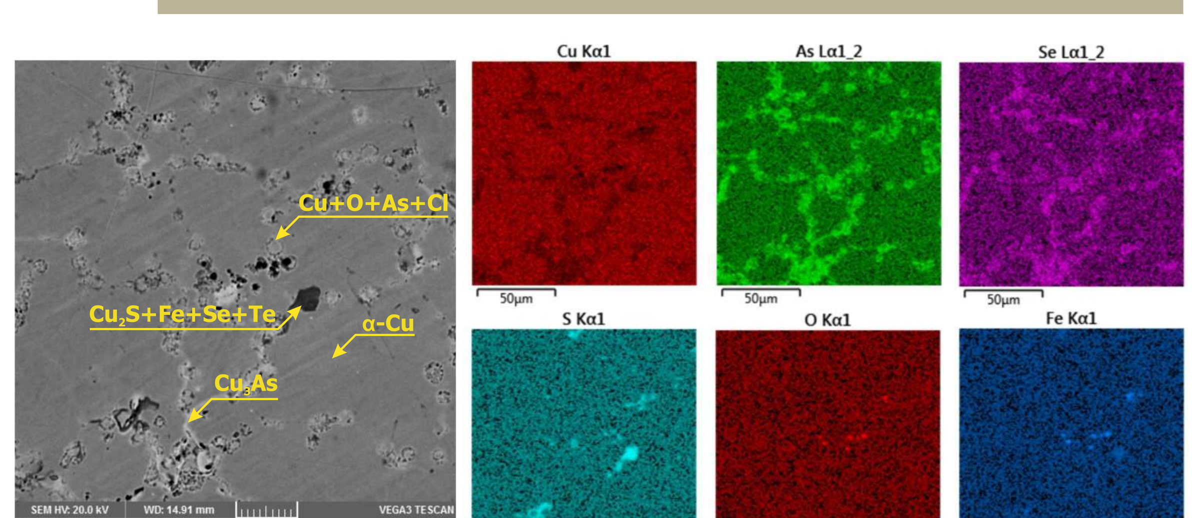

Figure 5 – Back scattered electron image of the microstructure of sample 2131 with maps of the distribution of Cu, As, Se, S, O, Fe

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