

FIRST DATA ON THE NATURE AND ORIGIN OF THE METALWORK FROM TELL EL-FARKHA

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INTRODUCTION

The metalwork from Tell el-Farkha is a major assemblage of late 4th millennium utilitarian copper artefacts, presented in detail by CZARNOWICZ (2012). More than 30 objects were found during excavations in the settlement, with a further seven finds from the cemetery. Fish hooks, harpoons (Fig. 1) and knives dominate, but also several awls, pins with loops (Fig. 2) and fragments of bracelets (Fig. 3) were found as well as small unidentified pieces, and working debris such as a casting prill and a plain rod. This progress report focuses on three main aspects of the assemblage of metal finds from Tell el-Farkha, namely their microstructure, their chemical composition, and their isotopic composition. This links back to three different choices that the early metal smiths may or may not have taken: how to work the metal, whether they selected or influenced the metal for particular properties determined by its composition, and where they may have obtained the metal from.

The first choice provides some insight into how skilled the metal smiths were who produced these artefacts. There are different ways how to shape metal artefacts, and some of the properties of metal depend on how it is being worked. Objects can be cast into shape, and then further treated by hammering, either to complete the forming process, or to improve the hardness of the metal, or both. If metal is worked or hammered too much it can become brittle, and annealing, that is re-heating to about half the melting temperature would be necessary to make it soft again and facilitating further deformation through hammering. The microstructure of the metal would show whether it is in its original as-cast state, or whether it has been subsequently deformed through hammering, and/or annealed (*e.g.* SCOTT 1991). Casting and hammering would be relatively basic methods of metal working, while evidence for annealing, which also leaves very characteristic microstructures, would indicate an advanced level of knowledge and experience.

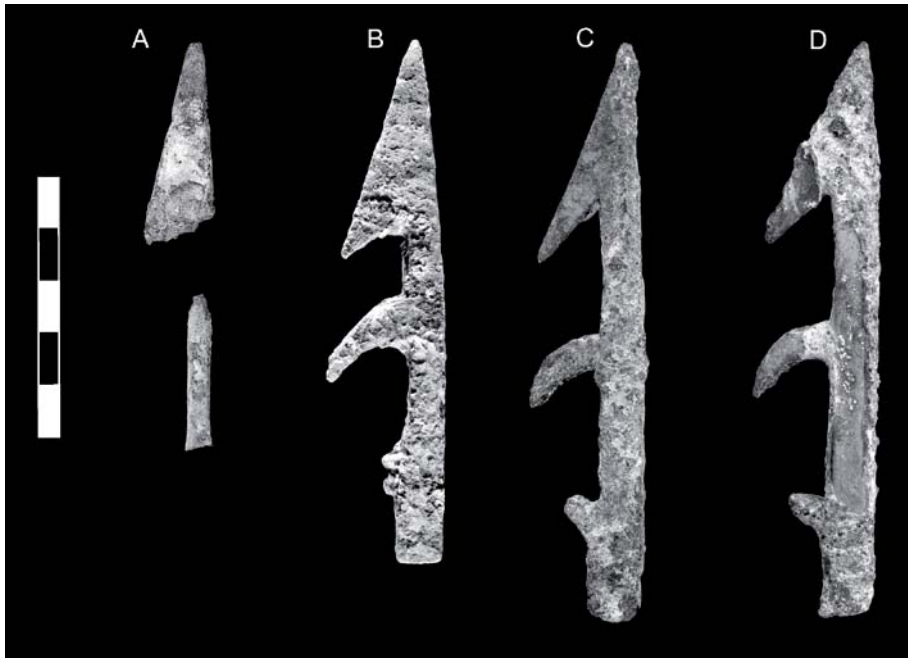


Figure 1. Four harpoons from Tell el-Farkha. Scale in cm (photo by M. CZARNOWICZ).

Secondly, it is interesting to find out whether there is a relationship between the type or function of certain objects and the metal from which they are made. In this early period, one would expect that either copper or copper-arsenic alloys were used, while further to the east more complex alloys rich in antimony and arsenic were also used (*e.g.* the famous metalwork from Nahal Mishmar, TADMOR *et al.* 1995). The alloys are typically harder than pure copper, melt at lower temperatures, and have a lighter colour. So both for functional objects such as knives and other tools, and for decorative items such as jewellery one could expect to see a preferred use of alloyed copper. For wires, in contrast, one would probably expect to see the softer and less brittle pure copper being used.

The third question concerns the geological origin of this metal. There are no copper ores in the Nile Delta and no copper slags known from Tell el-Farkha. Thus, the metal itself may well have been smelted elsewhere even though at least one casting drop was found indicating the presence of a local metal workshop. Substantial long-distance trade took place already in this early period, for instance with lapis lazuli from Afghanistan reaching Egypt. There are several large copper sources with known or suspected 4th millennium mining evidence in the wider vicinity of Tell el-Farkha that could have supplied this metal, such as Feinan or Timna in current-day Jordan and southern Israel (HAUPTMANN 2000, and references therein), or the Sinai Peninsula (ABDEL MOTELIB *et al.* 2012), or smaller deposits

in Egypt and Saudi Arabia along the Red Sea coast. Even Anatolia or Iran cannot *a priori* be excluded as potential source areas, given the evidence for lapis lazuli from Central Asia being found in Egypt.

METHODOLOGY

All available metal finds were analysed in April 2012 on site in Tell el-Farkha using portable equipment, including 13 registered artefacts and those more recent finds still under study in the site base (find numbers are preceded in the table by W, C, EN and ES for West, Central, East North and East South to indicate the Kom where they were found). The purpose of the study was firstly to properly identify and characterise the metal, and secondly to try and relate the metal compositionally to other analysed artefacts from the wider region, complementing the typological study based on visual examination and comparison to finds from elsewhere, published recently by CZARNOWICZ (2012).



Figure 2. Collection of pins with loop from Tell el-Farkha; object marked D is C719. Scale in cm (photo by M. CZARNOWICZ).



Figure 3. Bracelet C263. Maximum width circa 6cm (photo by R. SŁABOŃSKI).



Figure 4. Analysis of a stone tool using pXRF at Tell el-Farkha (photo by M. JÓRDECZKA).

The analytical work used first an optical microscope to inspect the corrosion state of the metal and to identify the most suitable areas for analysis by portable X-ray Fluorescence. The analytical instrument, an InnovX Delta Plus, was used in its Alloy Plus and Mining modes respectively, analysing each artefact at least twice and with both modes to obtain a range of analytical data (Fig. 4). The fundamental and unavoidable limitations of this type of analysis are by now well known and need only briefly repeated here. The calibration is set up using a large number of certified reference materials with ideal surfaces: clean, homogenous material with a flat surface at a well-defined narrow distance to the detector window of the instrument. Under such conditions, relatively good levels of analytical precision and accuracy

can be obtained for base metal concentrations above circa half of one percent, or even less, depending on the elements in question. The actual measurements of the archaeological artefacts, in contrast, were done on irregularly curved and rough surfaces of heavily corroded and heterogeneous material, often at a somewhat larger distance from the window and/or on small fragments resulting in insufficient coverage of the area irradiated by the primary beam. Even with clean and homogenous metal surfaces such morphological deviations from the ideal flat surface are known to result in strong discrepancies between the certified and the measured composition, severely limiting the quality of the data in the field. A further massive distortion of the original composition would occur through the effects of corrosion, as discussed in more detail below.

To partially mitigate the effects of surface condition and shape, the interior of several artefacts was exposed using a small diamond-coated cutting wheel (Fig. 5). The exposed fresh metal was then re-analysed using a small-spot XRF mode (Fig. 6). Only the data from these samples are reported as numerical values in the table below, while the surface analyses are only discussed qualitatively. The cut pieces were then mounted in resin and polished to a mirror-like finish using standard metallographic procedures for study by optical microscopy, and the debris from the sampling and preparation used for lead isotope analysis using the method detailed in NIEDERSCHLAG *et al.* (2003).



Figure 5. Cutting of finds at Tell el-Farkha to reveal fresh metal surfaces (photo by M. JÓRDECZKA).

MACROSCOPIC INSPECTION

Optical inspection of the artefacts confirmed the expected corrosion pattern for such archaeological finds, typically consisting of a layered structure of different mineralogical and chemical composition. Starting from the outside, this sequence begins with a mostly green surface or growth corrosion dominated by copper hydro-carbonates and possibly chlorides, and incorporating various amounts of soil particles; depending on the state of cleaning and conservation treatment applied to each artefact, this layer is not always preserved. Beneath this is a dark red layer of predominantly cuprite, which is assumed to indicate the position of the original surface of the artefact, but also to extend into its body as corrosion progresses (SCOTT 2002).

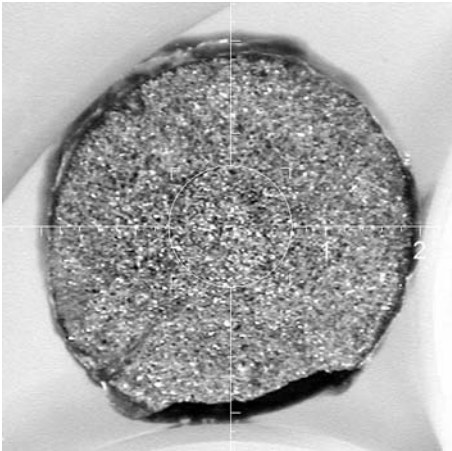


Figure 6. Bracelet C263 as seen through the camera of the XRF instrument. Diameter of cross section 4mm.

For most objects from Tell el-Farkha studied here the transition between the green and the red layer is irregular, resulting in a pitted surface for the cuprite layer after cleaning. Most artefacts analysed in this study have been cleaned to this surface, although several have still a prominent green surface layer, either because they are still awaiting conservation treatment, or because they were so thin as to have been completely mineralised and not preserving even the cuprite layer. A few of the larger artefacts show a fresh localised layer of powdery greenish material on top of the cleaned surfaces, indicating ongoing corrosion processes, possibly akin to bronze disease. In some cases

beneath the cuprite layer some metal is preserved. Where visible, this preserved metal appears very granular and brittle, indicating an advanced state of intergranular corrosion.

Apart from the implications for conservation treatment, an understanding of the different corrosion layers is of relevance for the interpretation of the chemical results obtained by surface analysis. Due to the non-invasive nature of the pXRF analysis and its limited penetration depth of only a few tens of microns any analysis will be specific not for the whole artefact, but for the particular layer analysed. Since the corrosion processes which formed these layers affect different elements in different ways, the various layers of a single artefact often differ fundamentally in their composition from each other, and from the original metal core.

METALWORKING TECHNIQUES

As already seen macroscopically, most of the cut artefacts were severely corroded, often to the extent that no traces of the original microstructures remained. Others, however, have sound metal surviving as well as transitional areas of partial corrosion, providing good insight into the metalworking techniques employed in the manufacture of the artefacts analysed. In all cases where sufficient material is preserved to recognise it, it is an as-cast structure. This is particularly apparent in samples that are highly alloyed, such as the metal prill EN359, the rod EN554, and the bracelet C263.

The metal prill has two metallic phases, a dendritic copper-coloured alpha phase and a whiter interstitial phase on grain boundaries and triple points (Fig. 7a, b). The only other sample with substantially preserved metal, the harpoon E/12/10, has only about 2 wt% arsenic in the copper (see below), but still enough to show an as-cast structure. An as-cast structure is also preserved in the fully corroded sample from the harpoon

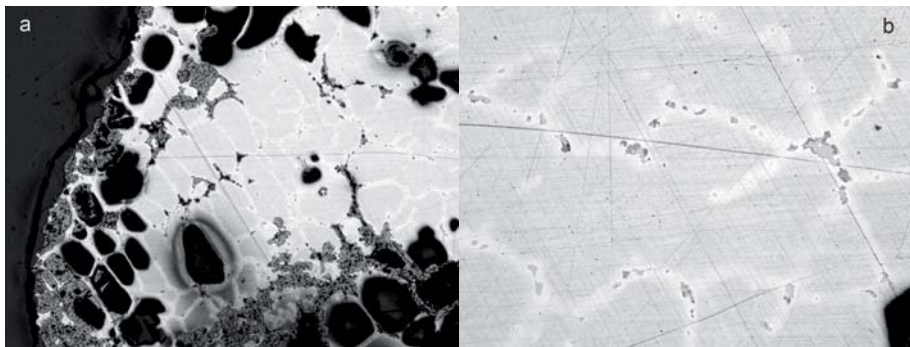


Figure 7a, b. Polished section of the metal prill EN359, showing a well-developed as-cast texture with dendritic alpha phase surrounded by a lighter network of metal enriched in arsenic, and the formation of intermetallic copper-arsenic compounds in the grain boundaries and triple points (b). Note the selective corrosion of the more copper-rich alpha phase, leaving voids (black) in their shape (a).

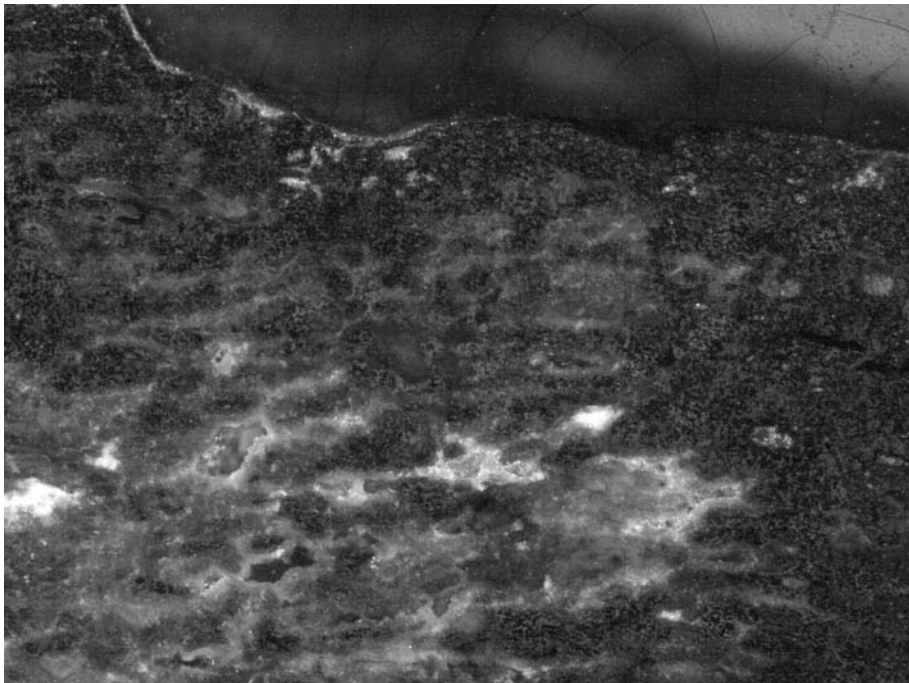


Figure 8. Polished section of a fully corroded part of the harpoon EN180, showing a slightly deformed as-cast texture with dendritic alpha phase preserved as 'ghost' structure in the corrosion products.

EN180. This sample may have some indication of a small degree of deformation, indicated by the sub-parallel orientation of the dendritic structure as it appears in the corrosion products (Fig. 8).

None of the wire or pin samples contain sufficient metal or residual structures in the corrosion products to determine their manufacturing mode; they are too thoroughly corroded. Whether this is due to the small size of these objects, or due to induced stress as a result of hammering, is impossible to say at this stage of our research.

Several fragments of bracelets were analysed (C263, C264; EN01/2), all three showing a rather unusual microstructure (Fig. 9). While clearly still based on copper as the main component, they are very rich in sulphide inclusions. C263 and C264 are also rich in corroded lead metal. Only the fragment C263 has sound metal exposed in the cut surface, while C264 and EN01/2 are fully corroded, but still show the characteristic sulphide inclusions preserved in the corrosion products.

So far, we have only seen as-cast structures in all of the objects, with no indication for any working through hammering. However, the cross sections of some of the wires are angular and may have been formed by hammering a rolled-up thin sheet into a square

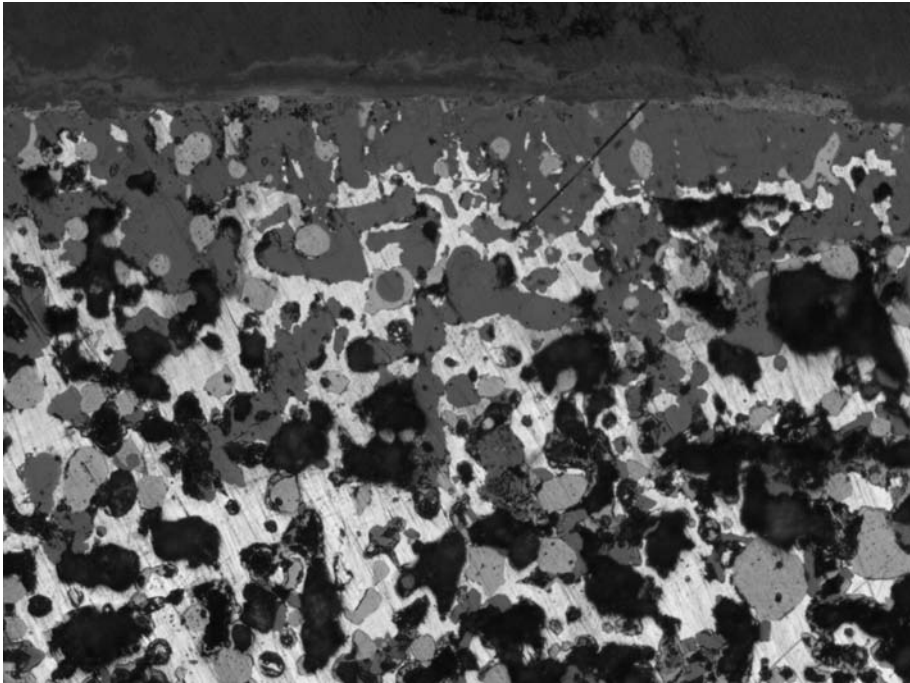


Figure 9. Polished section of the bracelet C263, showing a well-developed as-cast texture with dendritic alpha phase of copper together with numerous round sulphide inclusions and various corrosion products (different grey shades). Width of image circa 0.2mm.

Table 1. Indicative XRF analyses (in wt%) on cut metal surfaces exposing the less corroded core, using a small collimator. Measurement time 300 to 600 seconds, in air. Sulphur not sought, oxygen not measured; data normalised to 100 wt% metal. Fe, Co, Zn and Sn searched for but not found, with assumed detection limits in the order of 0.02 to 0.05 wt%. Cells with ‘-’ indicate that the element was not found. The column As* reports pXRF analyses of the surfaces of the objects; note that the pXRF values are typically only about 1/3 of the values found in the core, except for the bracelets where they are significantly higher, possibly indicating a surface enrichment in arsenic for these particular samples.

		CL	Ni	CU	AS	AS*	AG	SB	AU	PB	BI
HARPOON	E/12/10	-	-	97.2	2.8	0.5	-	-	-	-	-
HARPOON	EN180	-	0.02	98.3	1.6	-	-	-	0.1	-	0.1
FISH HOOK	C814	-	-	98.3	1.7	0.5	-	-	-	-	-
WIRE	W01 24	-	0.02	97.6	2.4	0.8	-	-	-	-	-
WIRE	C734	-	0.06	92.3	-	0.1	5.1	-	2.6	-	-
WIRE?	C566	-	-	100.0	-	0.2	-	-	-	-	-
PIN W LOOP	C719 B	-	0.05	94.0	-	-	4.3	-	0.4	1.3	-
BRACELET	C264	-	-	93.9	1.2	9.9	-	0.04	-	4.8	-
BRACELET	C263	12.3	0.03	81.3	2.0	1.7	-	0.09	-	4.4	-
BRACELET	EN/01/2	-	-	99.5	0.5	2.7	-	-	-	-	-
ROD	EN554	-	0.55	65.6	0.3	0.5	13.9	-	19.7	-	-
PRILL	EN359	-	-	96.6	3.4	0.6	-	-	-	-	-

mould; unfortunately, none of the structures of these samples reveal anything diagnostic regarding their manufacturing. The corrosion of the artefacts varies from heavy to complete, and this affects particularly the smaller artefacts, such as wires and pins, but also the thin blades of knives. These are all completely corroded, and we were unable to see any positive residual textures. This may indicate that these were hammered into shape, which would have destroyed the original texture and added a lot of stress into the metal – which in turn would have made it corrode more and faster than the as-cast objects. Some shapes, such as the end of some of the pins with loops that are rolled back onto themselves, are clearly hammered into their final shape.

CHEMICAL COMPOSITION

Only about a third of all artefacts were analysed on freshly-cut inner surfaces, while all finds were analysed non-invasively on their outer preserved surfaces. Table 1 reports the analytical data obtained on metallic cores of cut samples; it has to be borne in mind though that even these freshly-cut surfaces include deep-rooted corrosion areas. This, and the field character of the analyses, prevents us from claiming that the reported data is quantitative; however, clear patterns do emerge and can be reliably interpreted.

The data indicates that most artefacts were made from arsenical copper, with arsenic concentrations in the order of half a percent to three percent by weight. Eight of the twelve analysed artefacts have more than half a percent arsenic. A similar picture emerges from the surface analysis of the entire assemblage; here, overall arsenic levels appear to be significantly lower, on average only giving one third of the readings found within the artefacts' cores, which may reflect a depletion effect of the corrosion processes. Only the three bracelets show significantly higher arsenic readings on their surface compared to the core analyses. Overall, 14 of the surface analyses show 0.2 wt% arsenic or more, a further nine show a clear arsenic peak which may represent a concentration in the order of 0.1 wt%, while only four artefacts gave no good arsenic signal.

Half of the cut analysed artefacts gave nickel signals; one in the order of one half of one percent, the others much lower, but still clearly visible. There does not seem to be a positive correlation of nickel and arsenic; the objects with the highest arsenic values have low or no nickel, while high nickel values are found in low-arsenic artefacts.

Remarkable and unexpected is the presence in three artefacts of gold and silver in significant quantities. The pin with lop C719 has about 4 wt% silver and nearly half a percent by weight gold, the small wire C734 has 5 wt% silver and two and a half percent gold, while the rod EN554 has around 14 wt% silver and nearly 20 wt% gold. These concentrations are well above normal trace element levels in copper, rarely exceeding 0.1 wt% for silver and 0.01 wt% for gold. However, they are also far too low to have a noticeable effect on the colour of the alloy, or its properties other than making it generally somewhat harder than pure copper would be. It may be significant that the pin and the wire both are among the arsenic-free finds, while the rod has only a very small amount of arsenic.

The two bracelet fragments C263 and C264, finally, both have around four to five weight percent lead, one to two percent arsenic, and are the only finds with noticeable antimony contents. From their composition, they may well come from the same object, as is also indicated by the similar microstructure with the unusual high sulphur content (not analysed). The third bracelet fragment, EN/01/2, is rather pure copper with just a small amount of arsenic and no other metals found in the analysis. The microstructure of this sample, although completely corroded, does show a high sulphur content, indicating that there is a degree of similarity among all three bracelet fragments which is not shared with any of the other analysed objects. It had been mentioned earlier that the three bracelet fragments are the only pieces which showed much higher arsenic values during pXRF analysis of their surface compared to the point-mode XRF of the interior metal exposed through a clean cut; all other artefacts showed the opposite phenomenon, much lower arsenic concentrations on the surface compared to the core. It is tempting to think that this is due to the presence of an arsenic-rich layer at the surface of these bracelets, either through inverse segregation of arsenic during the slow solidification of the cast, or through a specific surface treatment. In both cases this would have resulted in the

formation of a more silvery surface, most likely a desirable colour effect (SMITH 1973). However, one would need to do a more detailed metallographic study of the bracelet fragments to investigate this idea further.

On the basis of the existing data there does not seem to be a clear pattern of correlation between function and composition, apart from the three bracelet fragments. The amount of alloying components lies in the low percentage range for most artefacts, with emphasis on arsenic as the main alloying element. However, the harpoons are not more alloyed than the wires, which could suggest that there was no selection yet for hardness (harpoons) or ductility (wires) when picking a piece of metal in order to produce a specific object. The largest effect from alloying component would have come from the four to five percent of lead in the two bracelets C263 and C264, and the high sulphur content in all three bracelet fragments. These would have given the metal a much higher fluidity and longer cooling range than the normal low-alloy arsenical copper would have had. This might have been a noticeable property which the metal founders exploited when casting the bracelets with their fine surface detail. Interesting in this respect is also that at this early period, copper smelting is supposed to be based almost entirely on the exploitation of oxidic ores such as malachite; the smelting of sulphidic ores was not yet common practice. Much later, from Hellenistic times in the eastern Mediterranean and in Zhou period and later China, people would add lead to bronze specifically for complex castings to improve the metal's fluidity. It seems that this effect of lead was already empirically known to the metal smiths of the late fourth millennium BC, even though this is most likely the selection of a naturally fluid alloy rather than the conscious addition of lead and sulphur to copper.

GEOLOGICAL ORIGIN – LEAD ISOTOPE ANALYSES

Lead is a relatively common trace element in most natural rocks. It occurs in nature in four stable isotopes, that is in four different varieties which all have the same chemical behaviour, but slightly different mass. Three of these isotopes form continuously through the slow but persistent radioactive decay of uranium and thorium, two other trace elements present in most rocks. Through this process the relative proportion of the four lead isotopes changes over time as the three radiogenic isotopes increase in quantity, at different speeds, while the fourth one remains unchanged, therefore over time relatively decreasing in abundance. This process happens in a predictable manner, depending in detail on the relative proportions of the three elements lead, uranium and thorium in the rock. During ore formation, the lead is removed from the parental rock and concentrated together with other metals such as copper or silver, but typically separated from uranium and thorium; at this stage, the lead isotope abundance ratio in the ore is therefore effectively fixed, while the lead isotope ratio in the surrounding rocks continues to evolve due to their remaining content in uranium and thorium. Measuring the lead isotope abundance ratios in ore deposits therefore provides a measure for the

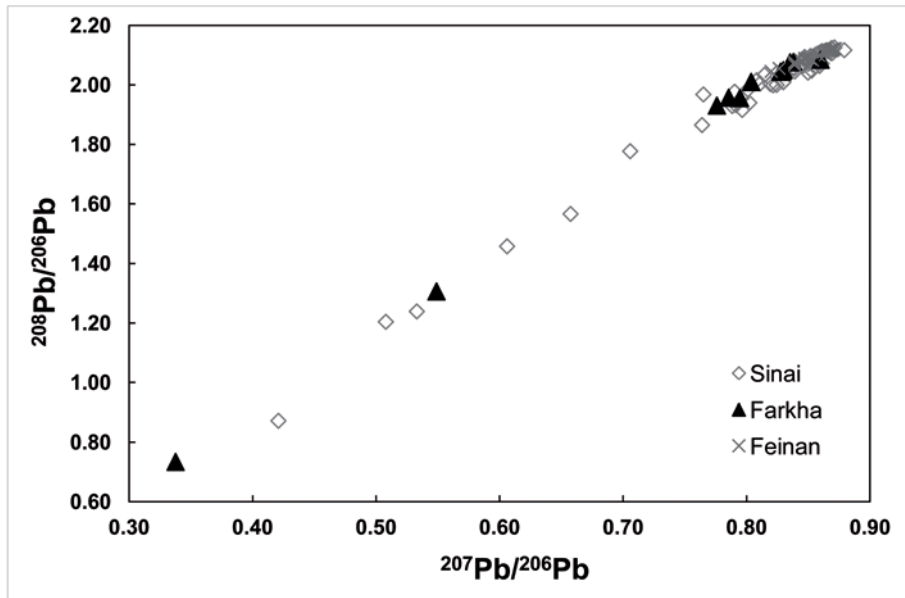


Figure 10. Lead isotope abundance ratios for 13 Tell el-Farkha objects (triangles) compared to data for copper and copper ore from Feinan in Jordan (x, data from HAUPTMANN *et al.* 1992) and the Sinai (chevrons, data from ABDEL-MOTELIB *et al.* 2012). Note the long tail of LIA data to the lower left-hand corner of the diagram, indicating a very radiogenic signature of the metal.

geological age of the formation of the ore deposit. During smelting, this isotope ratio is transferred without change into the metal, where lead occurs again as a trace or minor element in the copper, so that the lead isotope ratio of a copper artefact represents the lead isotope ratio of the ore deposit from which it was smelted. Different ore deposits form at different geological times and in different geological environments which enables one to distinguish between metal that was made from ores from different deposits, through the analysis of the lead isotope ratios in these metals. However, ore formation is a ubiquitous process, and can happen at the same time in different parts of the world, leading to geographically unrelated ore deposits which have the same lead isotope signature. Depending on the specific formation processes long drawn-out ranges of isotope ratios within single deposits can occur, which are then not very specific. Finally, there are copper ore deposits which are relatively poor in lead but contain relatively high concentrations of uranium and thorium which can lead to a highly variable lead isotope signature. Such lead is usually called radiogenic.

An initial assessment of the 13 Tell el-Farkha samples shows that they extend over a very drawn-out range, as is typical for highly radiogenic lead (Fig. 10). Comparing this to the possible copper ore sources briefly mentioned above shows that the Tell el-Farkha metal has the same lead isotope signature as the Sinai ores; that it extends further down is not really a problem. This does not prove that the metal is from the Sinai, but it is

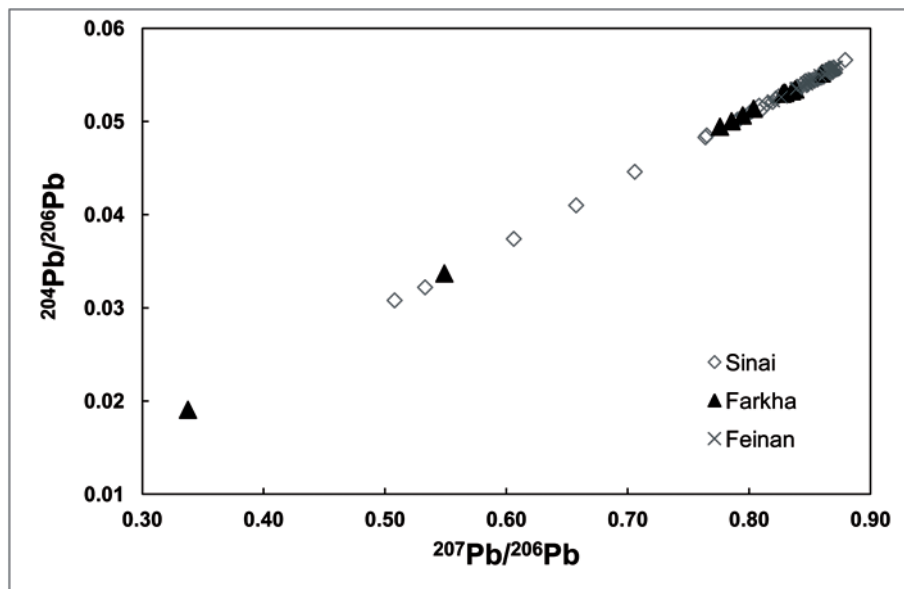


Figure 11. Alternative representation of the lead isotope abundance ratios for 13 Tell el-Farkha objects (triangles) compared to data for copper and copper ore from Jordan (x) and the Sinai (chevrons). There is less scatter along the regression line, because only the U/Pb ratios in the ore deposits are involved while in Figure 10 the values also depend on the Th/Pb ratios.

a distinct possibility. However, it is virtually impossible to distinguish between copper ores from the Sinai and from Feinan in the Arabah valley in the upper right corner of the diagram. Also the alternative isotope plot including ^{204}Pb (Fig. 11) does not provide a clear discrimination.

A closer look at the group of lead isotope data near the top right hand end of the graph confirms that the match to the Sinai ores is still clear and the problem of discrimination with copper ores from Feinan persists (Figs. 12 and 13). However, copper ores with relatively high concentrations of arsenic and lead do exist in Sinai while they are absent in Feinan (HAUPTMANN *et al.* 1992). If one disregards the gold and silver contents then it would again appear that on balance it is more likely that also these samples are from the Sinai. It is as yet unknown whether the copper ores from Sinai contain also gold, but if the silver content of EN554 is taken at face value it rather seems to be an addition than an impurity from the ore. Indeed, copper-silver alloys are known from the fourth millennium BC, *e.g.* at Ur (MÜLLER-KARPE 1990), Arslantepe in eastern Anatolia (PALMIERI *et al.* 1998) and recently also at Çukuriçi Höyük near Ephesos at the Aegean coast (HOREJS *et al.* 2010). Similarly, gold-silver alloys with varying copper contents are also known from Predynastic Egypt (GALE & STOS-GALE 1981) and the Levant (REHREN *et al.* 1996, and references therein). The origins of these less common alloys, whether they are natural or intentional alloys, remain mostly unknown.

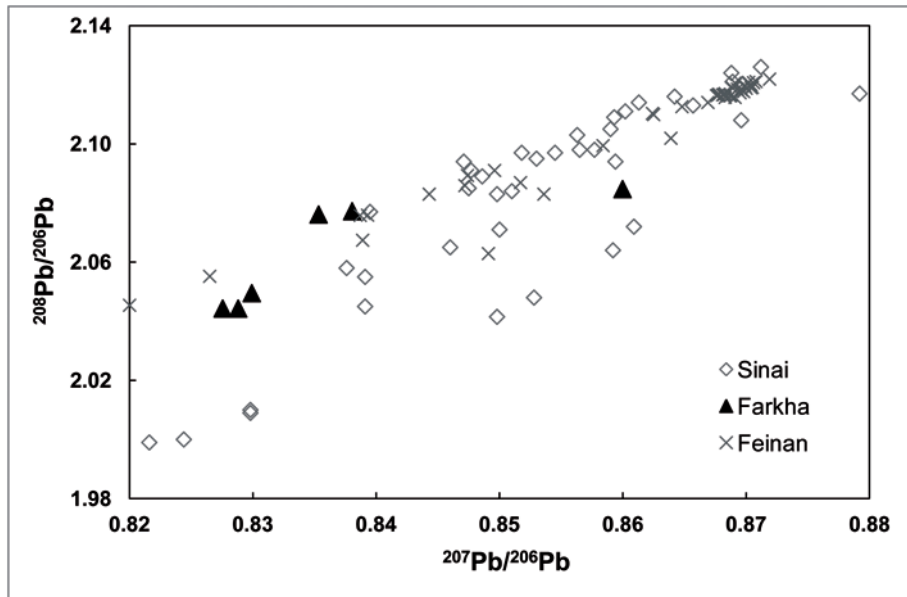


Figure 12. Detail of Figure 10, showing lead isotope abundance ratios for 6 of the 13 Tell el-Farkha objects (triangles) compared to data for copper and copper ore from Jordan (x) and the Sinai (chevrons). There is no clear discrimination between the two ore regions.

On balance, it seems safe to assume that the majority, if not all of the metal from Tell el-Farkha comes from the Sinai where there is archaeological evidence for early metal production, including for the production of arsenical copper.

CONCLUSIONS

The metal from Tell el-Farkha is predominantly arsenical copper, as one would expect at that period in time. There is no indication among the tools that the metal smiths selected more arsenic-rich alloys for artefacts with a particular function. However, there is a clear link between the bracelets and a very intriguing lead- and sulphur-rich alloy which would have been highly fluid when molten, and therefore ideal to cast, and possibly therefore selected for producing jewellery. This alloy is also likely to have been somewhat more silvery in colour than the normal copper, and this may have contributed to the appeal and selection of this metal; the surface colour effect would have been much more pronounced if there would have been a surface-enrichment in arsenic, as indicated by the pXRF analyses reported above (As* in Tab. 1).

It is reasonable to assume that the Tell el-Farkha metal comes most likely from the Sinai. That is true for all the arsenical copper artefacts, and the sulphur- and lead-rich bracelets, and the gold-silver rich copper. Feinan is possible as an additional source of copper, but it does not seem very likely based on their trace element contents.

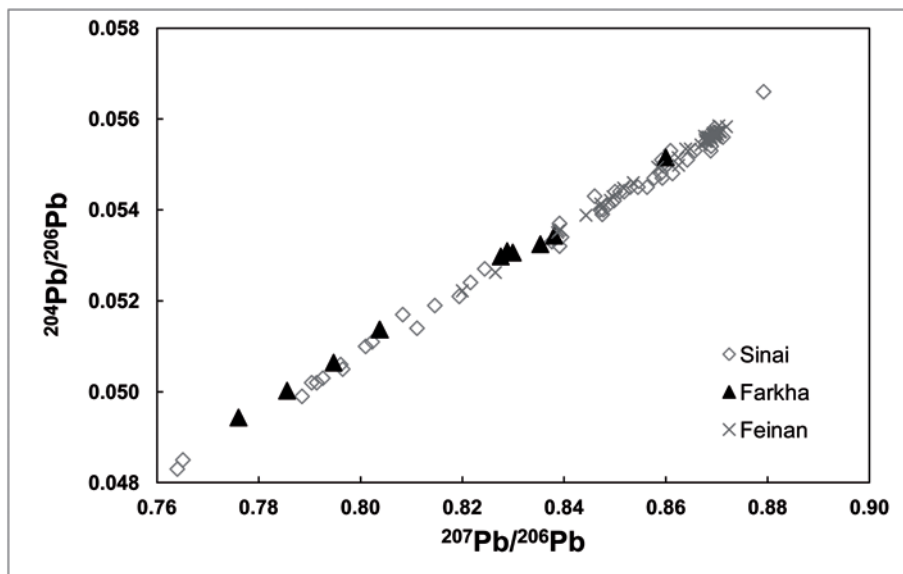


Figure 13. Detail of Figure 11, showing lead isotope abundance ratios for 10 of the 13 Tell el-Farkha objects (triangles) compared to data for copper and copper ore from Jordan (x) and the Sinai (chevrons). There is no clear discrimination between the two ore regions, except for the shorter range of data from Jordan which do not have $^{207}\text{Pb}/^{206}\text{Pb}$ values lower than 0.82, while both Sinai ores and the Farkha metal extend to much lower values.

Finally, all artefacts where the texture is preserved are in their as-cast state; there is no evidence for hammering and annealing to modify the metal properties. However, for thin objects such as the blades of knives or thin wires, and clearly the pins with loop, we may reasonably expect that they were hammered – and this may well have contributed to their bad corrosion state, particularly if they were not annealed after hammering, but left in a work-hardened state. This would have made sense for both the knives and the pins, so one can assume it – but not prove it.

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