

'The adventitious production of iron in the smelting of copper' revisited: metallographic evidence against a tempting model

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ABSTRACT: *The concentrations and distributions of copper in corroded iron artefacts are critical for interpreting the origin of the iron metal. Microanalysis and copper element distribution maps of twelve iron artefacts from the Egyptian New Kingdom (14th-12th century BC) Temple at Timna/Wadi Mene 'iyeh near Elat-Aqaba show gradients indicating contamination by copper from the burial environment. While metallic iron can be a by-product from copper smelting, it seems unlikely it was collected and forged into iron objects. These twelve iron artefacts are the products from iron smelting, not by-products from copper smelting.*

Introduction

In an influential paper, Gale *et al* (1990) proposed a technological link between the smelting of copper and the emergence of iron smelting, built upon the identification of cupriferous iron objects from the Egyptian New Kingdom Temple at Timna/Wadi Mene 'iyeh (Fig 1), located 20-30km north of the Gulf of Elat-Aqaba. This supposition of a development of iron smelting as a by-product of copper smelting was widely accepted in the archaeological literature (see *eg* Moorey 1994: 279-80; Waldbaum 1999: 30). Some doubts, however, have been raised on theoretical grounds. Craddock (1995: 255-256) speculated '... it seems unlikely that any serviceable bloomery iron ever contained more than small traces of copper, and the copper that has been found in the totally corroded iron at Timna at least is likely to have entered the totally corroded metal from the surrounding copper-rich environment during burial.' On metallurgical and historical evidence, Craddock suspected that an iron by-product from copper smelting would be unusable. Therefore, it now seems worthwhile to re-examine the iron artefacts and to test these hypotheses against new data. We have recently studied some of the original iron samples from Timna (Gale *et al* 1990) as well as several new iron samples from the same contexts by means of microprobe analysis and element mapping to clarify the spatial distribution and likely origin of the copper within these iron samples. These iron objects from Timna have

gained remarkable importance in the archaeo-metallurgical literature due to their relatively large number and excavated context as well as potential impact on understanding the early development of iron production and use.

Iron artefacts from Timna

Twelve iron artefacts were investigated (Table 1) principally from Strata II, III and IV in order to concentrate on the Egyptian New Kingdom 19th and 20th Dynasties and Midianite phase of the Temple (Fig 1). Stratum assignments are taken from the excavation report by Rothenberg (1988: 287-90). Stratum II is Midianite, dated 12th century BC. Strata III and IV are Egyptian New Kingdom (19th-20th Dynasties), dated 14th-12th century BC. The Temple was associated with the Egyptian goddess Hathor. Stratum V is attributed to the Sinai-Arabah Copper Age Early Phase (Chalcolithic-Early Bronze I), dated 4th millennium BC. One iron ring (322/89) was attributed to Stratum V in the publication, but it was certainly intrusive from later strata. As additional samples were taken from archived Find Boxes, the sample designation number of the Find Box Number is preferred followed by the object number (such as Find Box 322/Object 89). To allow cross-referencing, the column marked Rothenberg (1988) in Table 1 gives the metallurgical catalogue numbers and figure illustration numbers in that report. Figure 2 illustrates eight of the iron artefacts.



Figure 1: The Hathor Temple at Timna at the end of excavation. The walls and standing stones represented are attributed to Strata II to IV (Rothenberg 1988: 86). The photograph shows features from different layers.

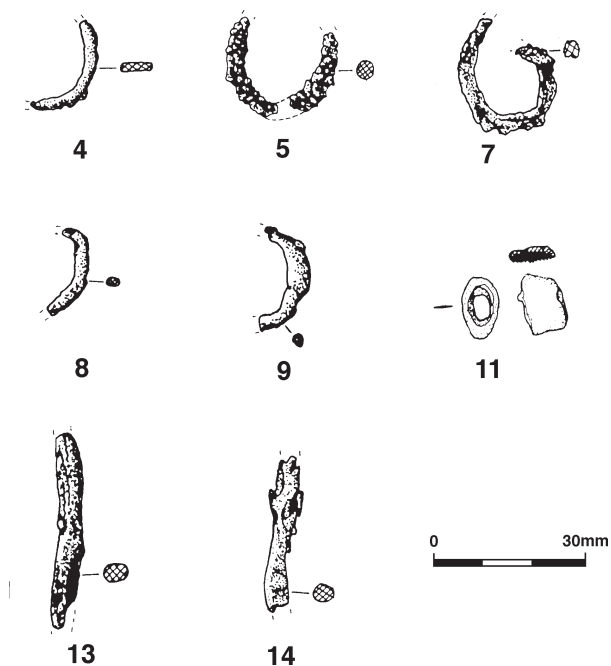


Figure 2: Illustrations of eight of the iron artefacts included in this study. The numbers are those in the column marked Rothenberg (1988) in Table 1.

The excavation reports and artefact descriptions were published by Rothenberg (1988, 1990). The iron artefacts were found in context with far larger quantities of copper alloy objects. Most of the iron consisted of jewellery, such as rings, beads, a small bracelet, rods, a tube and an ear-ring fragment decorated with gold foil. The estimated total weight of iron artefacts from the Temple at Timna was about 100 grams. Most iron artefacts found in the excavations at Timna were totally corroded. Several disintegrated on exposure (Rothenberg 1988: 147). Miscellaneous fragments of corroded iron artefacts were also saved in the Find Boxes, but object types could not always be identified.

Several sub-samples from the total number of corroded iron artefacts have already been investigated using X-ray fluorescence (XRF), lead isotope analysis and metallographic examination. Unfortunately, each iron artefact was not always examined with the same set of analytical techniques. Eleven iron samples were analysed by Gale *et al* (1990: 186-7) using XRF: 72/10, 178/2, 228/28, 322/90, 51/-, 282/29, 279/289, 283/228, 343/97, 8/48 and 500/69. Two SEM microanalysis distribution maps for copper in iron artefact 72/10 were

Table 1: List of iron artefacts from Timna examined for this study with EPMA

Find Box /Object No	Stratum	Locus & square	Description	Rothenberg (1988)		Gale <i>et al</i> (1990)	
				Cat	Fig	LIA	XRF
8/48	surface layer	112:BC 16-17	rod	20	54:15	+	+
51/-	II	101:EF 15-16	fragment	-	-	+	+
138/9	II (I)	110:CD 13-14	ring	14	54:8	-	-
204/8	II	110:E 13-14	ring	11	54:5	-	-
228/28	II	110:BCD 13-14	rod	19	54:13	+	+
256/1	III	109:G12	tube	17	54:11	-	-
282/29	-	110:AB 13-15	ring	15	54:9	-	+
283/28	III-II	106,107:D 7-9	ring	-	-	+	+
322/89	V	101:F 15-16	ring	10	54:4	-	-
343/95	III	109:F 12	ring	13	54:7	-	-
343/96	III	109:F 12	fragment	-	-	-	-
343/97	III	109:F 12	fragment	-	-	+	+

The column marked Rothenberg (1988) cites the metallurgical catalogue number (Cat) and figure illustration numbers (Fig) in that report. Stratum assignments are also taken from Rothenberg (*ibid*: 287-90). The Gale *et al* (1990) column lists the samples investigated using lead isotope analysis (LIA) and X-ray fluorescence (XRF).

published and it was noted that atomic absorption spectrometry gave a result of 0.3% Cu for this object. This was not exactly the same set of iron samples analysed by Gale *et al* (1990: 189) measuring lead isotope ratios. Lead isotope data are available for sixteen samples numbered 319/288, 309/2, 623/1, 250/2, 51/27, 328/2, 51/1, 51/2, 72/1, 228/28, 322/90, 51/-, 283/228, 343/97, 344/10 and 8/48. Tylecote (1988: 186-90) examined only five samples with corroded iron: 51/20, 51/36, 72/10, 154/3 and 279/26. The metal was almost completely corroded, so little information was derived on composition or regarding production techniques. At the time, no microanalysis was done on these iron samples. None of these five iron samples were discussed by Gale *et al* (1990). Furthermore, Tylecote's five metallographic sections were not available for further examination. Thus, there are some gaps in the surviving collection of iron artefacts and fragments available for further investigation (Table 1).

Iron was used originally for jewellery, but not for practical purposes such as tools (see Snodgrass 1980, 1982). Rothenberg's (1972: 174) initial interpretation assumed that the few decorative iron artefacts from the Timna Temple were representative of the earliest stage of the production of iron in its own right, separate from copper smelting. Lead isotope, compositional analysis

and experimental evidence, however, suggested an alternative interpretation. Gale *et al* (1990: 189-90) concluded:

'Iron artefacts dating between 1318 and 1156 BC, found both at Site 2 and at the site of the Hathor Temple contain noticeable amounts of copper. There is a strong indication that the iron used to make these artefacts was produced locally as an adventitious by-product of copper smelting. Isotope ratios of traces of lead present in Timna ores and fluxes, as well as in the iron artefacts, correlate within the margins of error, thereby proving both the provenance of the artefacts and the origin of iron from the mines and smelting centres of the Timna Valley. It seems that in Timna metallic iron was truly born in a copper smelting furnace.'

Semiquantitative XRF was used by Gale *et al* (1990) to detect major and minor element concentrations on the corroded surfaces of the eleven iron samples. The XRF analyses of the corroded iron artefacts show copper and occasionally manganese and zinc to be present. No nickel was detected, so the iron artefacts were not made from meteoric iron (*ibid*). The identification of copper (with estimated concentrations between 0.1-0.5%), however, on the surface of some iron artefacts was interpreted further as representing the

Table 2: Elemental compositions of remaining metallic iron measured by EPMA

Sample	Composition (wt%)										
	Pb	Zn	Ni	Mn	Co	P	Cu	S	Cl	Ca	Fe
138/9	0.100	0.000	0.000	0.037	0.144	0.004	0.118	0.000	0.015	0.015	92.832
282/29a	0.172	0.011	0.004	0.000	0.125	0.000	0.032	0.013	0.008	0.000	93.595
282/29b	0.101	0.000	0.000	0.000	0.061	0.000	0.033	0.023	0.012	0.009	93.597
283/228	0.010	0.016	0.000	-	-	0.000	0.177	0.013	-	0.018	95.478
322/89 1	0.005	0.007	0.007	0.000	0.109	0.009	0.000	0.014	0.054	0.000	93.003
322/89 2	0.067	0.000	0.004	0.015	0.096	0.013	0.000	0.014	0.000	0.000	92.496
343/96	0.000	0.004	0.000	0.000	0.198	0.026	0.140	0.000	0.016	0.074	97.096
343/97	0.102	0.000	0.015	0.000	0.108	0.013	0.021	0.000	0.011	0.014	95.201

The low totals and detectable chlorine (Cl) suggest that there is some corrosion of the remaining metallic iron. The elements Si, Mg, Ag and As were also sought, but not detected. The data are not normalized. Only those samples with surviving metallic iron are included.

link between copper smelting and iron smelting. Although copper was not detected on three of the eleven samples, no argument was made for independent smelting of iron. The two copper element distribution maps for artefact 72/10 were interpreted by Gale *et al* (1990: 188) as showing ‘... the copper is homogeneously distributed over the total exposed area. There is no indication of depletion, nor of copper agglomeration in the core of the sample. This evidence suggests that object 72/10 was made from wrought iron obtained as a by-product of copper smelting.’ Regrettably, the two published copper element distribution images are very poor quality. It is critical to note now that their SEM investigation of 72/10 was actually made on a ‘freshly broken cross section’ (*ibid*, 187) and not on a polished metallographic cross section.

This conclusion would make the iron artefacts from the Timna Temple fundamentally different from other iron artefacts from other sites of comparable date. Other early finds of iron artefacts in Late Bronze and Early Iron Age archaeological contexts have been listed by Waldbaum (1978, 1980, 1999). Metallographic investigations of iron jewellery from the Umm ad-Dananir region of the Baq’ah Valley in Jordan (c1200-1050 BC) documented steel with very low concentrations of copper, usually under 0.02% (Pigott *et al* 1982, Notis *et al* 1986). Nevertheless, too few technical studies have been completed on early iron and/

or steel artefacts from the region. The initial appearance of smelted iron and steel objects and the cultural attribution for its development still remain some of the most important research questions in archaeometallurgy for the Eastern Mediterranean (Waldbaum 1999).

Metallography and microanalysis

The present study now extends aspects of the earlier metallographic (Tylecote 1988) and compositional programme of analysis (Gale *et al* 1990) with the use of electron probe microanalysis (EPMA) of metallographic sections on twelve samples from iron objects and fragments selected from Temple Strata II, III and IV. Unfortunately, sample 72/10 (Gale *et al* 1988: 186-8) was not available for study. The objectives were to document the distribution of copper within the iron corrosion products as well as the original matrix of the objects using elemental dot maps and to analyse any remaining, uncorroded iron metal.

In metallographic sections of the corroded iron artefacts, a few remnants of iron metal were found in the interiors or core (*eg* Fig 3). EPMA revealed that the copper concentrations of the remaining metallic iron were usually under 0.1% (Table 2). The detection limit is approximately 0.01% for copper in iron. EPMA dot distribution maps of the copper in the iron corrosion for sample 322/89 reveal higher concentrations on the

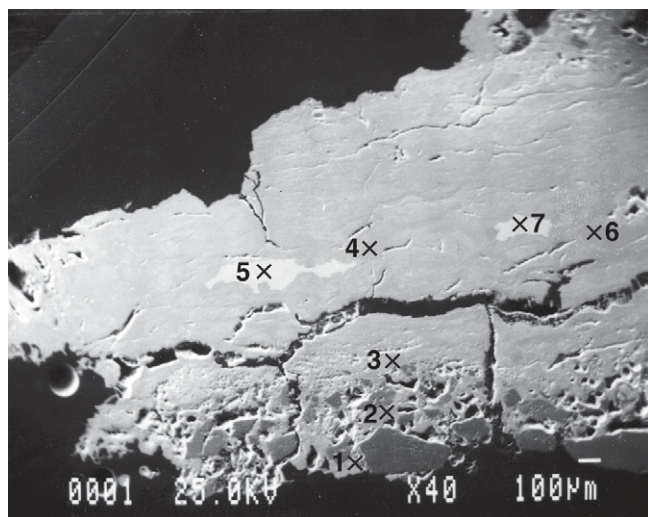


Figure 3: Backscattered electron image of sample 322/89. The point analyses 1, 2 and 3 are in the outer corrosion products around grains of quartz sand. Points 4 and 6 are in the corrosion at the centre. Points 5 and 7 are in the remaining metallic iron.

surface around entrapped grains of quartz sand than in the metal (Fig 4). The copper concentrations are observed to decrease toward the interior of the corroded iron objects (Table 3). Copper is also observed to be concentrated along fractures in the corroded iron. Similar distributions of copper were also observed in the other samples in this study. The EPMA copper distribution map for sample 282/29 is presented in Figure 5. The distribution was not homogeneous throughout the metallographic section of the corroded

Table 3: Point analyses in the metal and corrosion products of sample 322/89, measured by EPMA

Point No	Composition (wt%)										
	Pb	Zn	Ni	Mn	Co	P	Cu	S	Cl	Ca	Fe
1	0.058	0.000	0.000	0.045	0.039	0.007	0.075	0.013	0.212	0.157	51.709
2	0.063	0.014	0.009	0.099	0.052	0.108	0.049	0.115	0.181	0.189	49.572
3	0.060	0.002	0.000	0.034	0.063	0.105	0.069	0.048	0.222	0.252	54.667
4	0.035	0.000	0.003	0.000	0.038	0.029	0.013	0.035	0.673	0.151	52.772
6	0.000	0.000	0.015	0.003	0.033	0.021	0.027	0.067	0.332	0.187	56.130
5	0.005	0.007	0.007	0.000	0.109	0.009	0.000	0.014	0.054	0.000	93.003
7	0.067	0.000	0.004	0.015	0.096	0.013	0.000	0.014	0.000	0.000	92.496

The points labelled 1-7 are marked on Figure 3. Points 5 and 7 are in remaining metallic iron. The data are not normalized. Estimating oxygen by difference from the totals, the corrosion product analyses with approximately 49-56% Fe are iron oxides such as limonite (iron hydroxides) not magnetite.

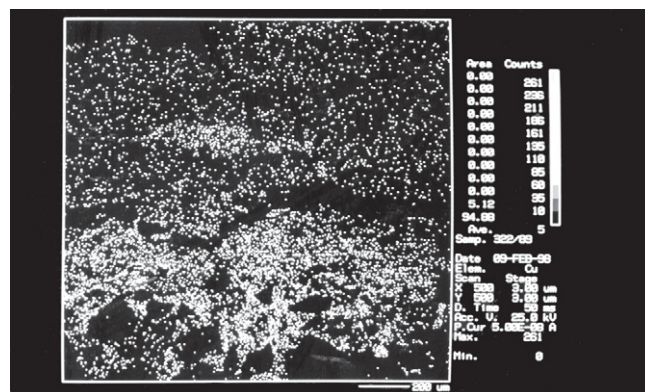


Figure 4: EPMA elemental map for copper in sample 322/89. Note the very low copper count rates. The intensity of points near the surface (bottom of image) and (dark) quartz sand reveals the relatively higher concentrations there. Compare point analysis compositions in Table 3 and Figure 3.

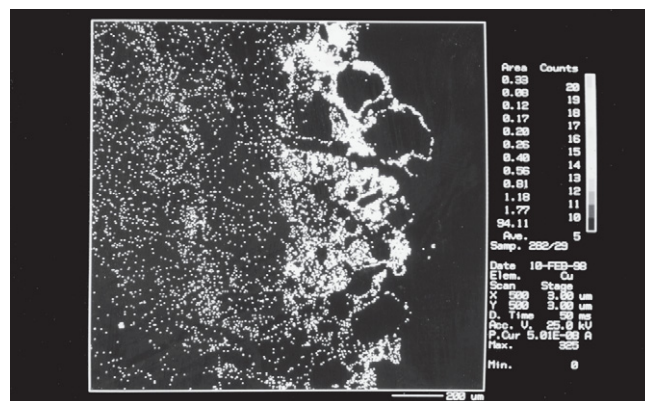


Figure 5: EPMA elemental map for copper in sample 282/29. The low copper counts document relatively higher concentrations near the surface and around quartz sand grains. Again there is a decrease in concentration toward the centre of the section, in this case to the left of the image.

iron artefacts. There is no agglomeration of copper in the core of the samples.

Previous XRF analyses by Gale *et al* (1990: 186) apparently analysed copper concentrations on the surface of the corroded iron artefacts. XRF analysis of the surface, however, did not reveal the decrease in copper concentrations toward the interior of the iron artefacts. This deficiency of surface XRF, as it was used, directed their interpretation to a misleading conclusion.

Our new study of twelve samples now documents that the observed copper distributions are uneven and decrease in concentration toward the centre of the corroded iron artefacts from Timna. The remaining metallic iron has typically less than 0.1% Cu. Sample 322/89 had no detectable copper in the iron at all. Based upon the Fe-Cu equilibrium phase diagram, these compositions would be single phase. Upon very slow cooling, copper over 0.35% is precipitated from saturated alpha iron solid solution (Lorig and Adams 1948: 6). If iron had formed together with copper in the same furnace charge, one would expect to find significantly higher non-equilibrium copper concentrations, just below the eutectoid at 8% Cu, in the iron metal (*ibid*). Copper smelting experiments by Tylecote and Boyde (1978: 45-6) produced iron-copper metal with non-equilibrium compositions.

Etching such small remnants of metallic iron was not very successful. Microhardness measurements gave most values around 100 HV₂₀. A few measurements were higher at 170 HV₂₀. Iron oxide corrosion products may preserve some relict metallographic structures for steel (Knox 1963, Scott 1989), but none were observed in these samples. The surviving metal in the centre of the artefacts is thus identified as wrought iron.

Based upon the microanalytical data as well as element mapping of the metallographic sections, the distribution of copper in the corroded iron is interpreted as contamination from burial alongside larger quantities of copper and copper alloy artefacts.

Corrosion of archaeological iron and copper

Metallic iron corrodes with characteristic and somewhat predictable results on archaeological sites. The iron rings, beads and bracelet from the Timna Temple are almost completely corroded, consisting of iron oxides or oxyhydroxides. The inner core is dense black iron oxide, usually magnetite, with few remnants of metallic iron. Insoluble iron oxides, once formed, can passivate

and somewhat protect the remaining metal in archaeological iron objects (see Turgoose 1982 and 1985, Cronyn 1990). However, this has not prevented almost complete corrosion of the iron artefacts at Timna.

When two dissimilar metals, such as iron and copper, are in direct contact or buried in close proximity, the electrochemical attack (galvanic corrosion) on the less noble metal, in this case iron, is increased, especially if the surface area of the second more noble metal (copper) is significantly larger. The corrosion rate and ultimate oxidation of the relatively small iron artefacts from the Temple seem to have been accelerated with large quantities of copper and copper alloy artefacts being present. On this basis, the iron corroded rapidly, soon followed by surface corrosion of the copper and copper alloy artefacts. The corrosion rates and depth of corrosion penetration are clearly different for iron objects and copper objects from Timna.

Moisture and salts are key factors for corrosion as well as the mobility of soluble chemical species. In a chloride-rich environment, such as at Timna, copper as well as iron can form soluble species in groundwater (Rose *et al* 1979: 549). However, copper species are more mobile and stay in solution better than iron species under oxidizing burial conditions. The corrosion and mobility of soluble copper species is the key to understanding the presence and distribution of copper on the surfaces of the iron artefacts from the Timna Temple. Corrosion from the numerous copper artefacts 'stains' layers in the soil of the Temple. Rothenberg (1988: 29) states, '... wherever we met the olive green "layer", there was a revealingly large number of copper objects, copper prills and many copper ore fragments.' Copper mobility in solution is considered to be intermediate, and readily controlled by adsorption to iron oxides (Rose *et al* 1979: 556).

In fact, copper in solution is attracted electrochemically to precipitate onto metallic iron. The precipitation of copper from solution onto metallic iron has been a mechanism used for cementation recovery of metallic copper from acidic mine water or leached copper sulphides, for example at Rio Tinto, Spain (Avery 1974: 175). Thus, some interaction should be expected on electrochemical principles between corroding copper/copper alloy artefacts and corroding iron artefacts in salt-rich arid burial environments, such as at Timna. Therefore, based upon burial environment and corrosion principles, we argue that the presence and distribution of copper within the surface corrosion products of the iron artefacts is simply the result of 'contamination' in burial.

Conclusions

The corrosion of iron artefacts from the Hathor Temple at Timna has been almost complete. Only a few remnants of metallic iron remained in the corrosion products. Less than 0.2% copper was detected in the uncorroded iron metal. The element distribution maps for copper indicate its distribution primarily on the surface and along cracks in the iron corrosion products. The copper concentrations and distributions observed in these corroded iron artefacts, thus, derive from the corrosion and mobility of copper from nearby copper and copper alloy artefacts. These observations confirm the hypothesis of Craddock (1995: 255-6) that the copper in the corroded iron artefacts from Timna resulted from burial conditions.

It should be made clear that some metallic iron was indeed produced during copper smelting at Timna and is present in copper ingots (Roman 1990). However, the central question is whether this metallic iron was actually separated from the copper *and* utilized for the forging of any iron objects (see Waldbaum 1980: 80). This new investigation of the iron artefacts from the Timna Temple provides no evidence that the iron by-product of copper smelting was used to make objects. The iron artefacts from Timna studied here are evidence for early iron production within the region as a separate and distinct technique from copper smelting.

The archaeological evidence from the Timna Temple represents the cultural interactions between at least three distinct groups: Egyptians, Midianites and a local population (Rothenberg 1972). Thus, iron production or control over the technique cannot be linked directly with only one of these groups at Timna. There is a lack of any well-dated evidence for Egyptian production of iron from other sites at this time, which would make an Egyptian origin seem unlikely. Other influences and possible knowledge of iron smelting should be considered too. For example, in the Hijaz (NW Arabia) Midianite pottery has been found on the surface at Survey Site 200-41 in Wadi Sharmah along with iron slag as reported by Ingraham *et al* (1981). Nevertheless, Egyptian control over copper production at Timna would at least substantiate a growing Egyptian exposure to other cultural traditions (not only Midianites) more familiar with iron smelting and working. These iron objects are representative of the very beginning of iron smelting in the region along the Egyptian frontier with the Midianites. The iron objects from the Temple are interpreted as 'offerings' so the ultimate sources of the

iron metal by trade at this date could be more widely dispersed.

Due to the contamination of the corroded iron with copper from nearby artefacts, the lead isotope data should also be re-examined for Timna, as suggested further by Craddock (1993: 592). Although lead is not as mobile as copper in burial conditions, such as those at Timna, the possibility does exist that the original lead isotope ratios for the corroded iron objects were also contaminated. Thus, a local origin of the smelted iron metal may not necessarily be correct, even if the lead isotope data coincide with those for the local geology at Timna.

Iron smelting was certainly a very minor component of the metallurgical activity within the region during the Egyptian New Kingdom. It was probably done on a very small scale leaving very little evidence to be recognized as iron smelting slag. The initial assumption (Rothenberg 1972: 174) that the iron objects in the Temple were the products of iron smelting was correct. These decorative iron objects are clearly the products of iron smelting, not by-products from copper smelting. These iron objects, thus, are technologically similar to iron objects from other sites in the region with comparable archaeological contexts. Their archaeological significance, however, as some of the earliest smelted iron artefacts known from the region remains unchanged.

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