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# News from the Institute for Archaeo-Metallurgical Studies

Thilo Rehren, Acting Director

## People and Research

The positive trend in the Institute's work has healthily continued throughout 2002. We are now involved in a number of exciting and important fieldwork projects in many regions of the world, from South America to Africa to Central Asia, and with a firm eye on China. Obviously, such an activity requires the concerted efforts of many people and support from a range of sources. This year, we are most grateful for the ongoing support from BHPBilliton plc for our studentship in archaeo-metallurgy (*see iams* 21 and 22) which has entered its second year, and for the most generous funding we receive from the Sternberg Foundation and from Lioncom, without which IAMS in its present form would not exist. In addition our work and that of our students is receiving substantial funding from an increasing number of other institutions, such as the Alexander S Onassis Public Benefit Foundation; the Dixon Research Studentship of the University of London; the Wenner Gren Foundation in the USA; the RF Tylecote Fund; the Graduate School of UCL; the ORS of Universities UK; the Art and Humanities Research Board are all helping to fund our research students. Our research has received financial support this year from the Gerda Henkel Stiftung in Düsseldorf; the British Academy; the Robert Kiln Charitable Trust; the Circle for Central and Inner Asia; the Institute of Archaeology; and the Society of Antiquaries of London.

Among the people I want to highlight this year is Dr Akin Ige, a colleague from the Natural History Museum of the Obafemi Awolowo University in Nigeria. Dr Ige has been visiting us in June last year, and we have together started a small research project on the Jaruba iron smelting practice. An initial account of this work can be found later in this issue (p. 5-20). Nigeria, as most of Africa, is a country rich in under-researched iron metallurgy, with an intriguing complexity in the detail even on the technological level. I, at least, have not seen before iron slag with more than ten percent by weight titania, neither have I been able to find reference to such slag in the literature. The ancient iron masters of the Jaruba will not have known about titania; but they will have had their reason for choosing a certain black sand to add to their ore. Early in 2003, the Royal Society has awarded us a grant to enable Dr Ige to spend another three months at the Institute and to continue this joint research.

Little do we know, and this will hardly change, about the ritual which was accompanying ancient iron smelting. One long-standing issue in African iron metallurgy has been the strict separation of living quarters and smelting sites, the latter being associated with witchcraft, or requiring special protection from uninitiated people; a separation reinforced by rituals and taboos. Current research now by Shadreck Chirikure in Zimbabwe has uncovered a much closer association of living and smelting spaces in the Nyanga area, indicating that such taboos were not universally valid. On second thought, this is hardly surprising, given the cultural diversity and geographical and temporal expanse of this continent. It needed to be demonstrated, though, and I am glad that IAMS was able last year to contribute substantially to Shadreck's time in London as a master student. I am even more pleased to report that Shadreck now has enrolled as a research student, with considerable outside funding, to continue this important work. The aim here is not only to learn about archaeo-metallurgy, but also to contribute to the educa-

tion of the future generation of archaeologists, for a time when government and economy in Zimbabwe are in a state again to enable this potentially rich country to research its own history.

Our work at Timna is also making good progress, and two important papers in this issue are covering the rock art and mining archaeology, respectively. I am particularly pleased to see that a common sense, low-cost approach can actually produce more, and more relevant, insight into ancient mining technology than the best cutting-edge instrumental analysis work: After all, it is the ancient miners' activity and behaviour that we are interested in!

## Teaching

Teaching of archaeo-metallurgy at the Institute of Archaeology is well established throughout our programme of study; at undergraduate, master and research student level. The 3<sup>rd</sup> year archaeo-metallurgy option for undergraduates is popular as ever, with close to twenty percent of all students taking it.

The MSc in Technology and Analysis of Archaeological Materials, details of which can be found on the back cover of this issue, includes two different taught elements on archaeo-metallurgy. I am always happy to discuss the content of this programme, and possible dissertation topics, with prospective students. At present, there are a number of exciting possibilities, from the study of medieval gold production in the Austrian Alps to lead smelting in Classical Antiquity, early Islamic copper metallurgy in Uzbekistan, and South American silver smelting. As always, there is a strong and direct link of these dissertation topics to ongoing research of members of staff, typically resulting in joint publications. This gives the students a unique exposure to academic work, and underlines the strong research tradition of both the Institute for Archaeo-Metallurgical Studies and the Institute of Archaeology.

Three new research students have enrolled this year with archaeo-metallurgical topics: Shadreck Chirikure is continuing his previous study of Nyanga iron smelting in Zimbabwe, Michael Charlton is applying Darwinian evolutionary theory to iron smelting, using material from Wales and Uganda, and Anna Karatzani is investigating the technology and social context of metal threads in Greek orthodox ecclesiastical textiles.

I hope to have this issue out in time for this year's two-week Summer School, beginning 16 June with Professors Tim Shaw and Beno Rothenberg on ancient mining technology, and continuing on 23 June with lectures on ancient metallurgy by Professors HG Bachmann, Beno Rothenberg, Vince Pigott and myself.

## Journal

It is with great sorrow that I report the death of Angus Mathieson, who for the last two and a half years worked as the design and copy editor for this journal. He had developed and implemented the new layout, carefully based on the initial design by Abram Games, and had become a close friend. He had just begun work on this issue, the first paper set and ready for the next, when he suffered a fatal heart attack. I want to dedicate this issue of our journal to his memory.

# Early brass in the ancient Near East

Christopher P. Thornton & Christine B. Ehlers

## Abstract

*Discussions of early brass production in the Near East have been, to date, inherently tenuous as they rely on isolated and questionable finds and analyses. It is a history lacking reliable data in support of the position that the production of pre-1<sup>st</sup> millennium BC high-zinc copper alloys was part of an informed, deliberate process employed by metalsmiths. The results of analyses conducted on samples taken from excavated materials, now housed in museum collections, provide evidence for earlier and more widespread intentional brass-making than has been available heretofore. The data indicate that high-zinc copper-base alloys were used to fashion artefacts found in mid-2<sup>nd</sup> millennium BC contexts at the sites of Tepe Yahya and Nuzi. Perhaps more significantly, comparison with contemporary materials suggests that these brasses were the result of intentional choices made on the part of early metalsmiths.*

## Introduction

The history of copper-zinc alloys such as brass (Cu-Zn), gunmetal (Cu-Sn-Zn), and latten (Cu-Zn-Sn) is shrouded by poor excavation reporting, questionable analyses, and numerous misconceptions. Early claims of intentional brass (*i.e.* above 8 wt% Zn) from sites such as Early Bronze Age Gezer (Macalister 1912: 265) in Palestine and at Neolithic Jiangzhai and Jiaoxian (Rubin & Tsun 2000) in China have been rightly criticized, if not entirely discredited (see Craddock 1980 and Zhimin 2000, respectively). The reason for such skepticism is that high-zinc copper-base alloys are extremely difficult to make due to the volatility of zinc at temperatures above 906 °C; *i.e.* 177 °C below the melting point of copper (see Pollard & Heron 1996 or Craddock 1990 for more detailed descriptions of copper-zinc production). Although zinc ores (e.g. sphalerite, ZnS) are often found in association with copper and lead ores, and could easily have found their way into a smelt accidentally, the majority of zinc present in the ores would vaporize leaving only a trace (up to 1 wt%) in the resulting copper metal unless deliberate measures were taken to prevent the loss of zinc by reintroduction of the vapor back into the smelt. In rare instances, such as under extreme reducing conditions, copper-zinc alloys with up to 8 weight percent zinc could be produced by mixing copper and zinc ores in a furnace. This has been used to explain low-zinc copper-base artefacts such as an Early Bronze Age dagger from the Cyclades (5.1 wt% Zn), two slightly later axes from Namazga-Depe in Turkmenistan (unknown) and Beth-Shan in the Levant (6.5 wt% Zn as well as Sn) (see Craddock 1978: 2-3), numerous finds from the 3<sup>rd</sup> millennium graves of Umm an-Nar in the Persian Gulf (4.1-10 wt% Zn; Frifelt 1991: 100) and possibly a few copper-zinc alloys from the MBA site of Trianda on Rhodes (S. Stos, personal communication).

These isolated finds are generally agreed to be accidentally produced copper-zinc alloys, the implication being that early metalworkers were not aware of the zinc content until much later in history or were not in possession of the technical knowledge to control it. Dating to the 1<sup>st</sup> millennium BC, three gunmetal fibulae from Gordion in Anatolia with roughly 15 weight percent tin and above 10 weight percent zinc (see Young 1981: 287-290), as well as scattered examples with 10 to 20 weight percent zinc found throughout the eastern Mediterranean world are widely accepted as being the first deliberately-produced copper-zinc

alloys (Craddock 1988: 320; Pollard & Heron 1996: 201). The method presumed to have been used to produce these early brasses is known as cementation (a.k.a. the calamine process), whereby smithsonite (ZnCO<sub>3</sub>; zinc spar) is heated with copper metal in a closed crucible. Once the temperature rises above 906 °C, the zinc vaporizes and is absorbed into the solid copper solution. Experimentation has shown that the upper limit of zinc uptake by the cementation process is roughly 28 to 30 weight percent zinc, although up to 10 weight percent zinc can be lost through subsequent remelting (Pollard & Heron 1996: 199; Ponting 1999: 1315). The smithsonite used in the cementation process can either be found as a natural ore or can result from sphalerite roasting and sublimation in a special furnace to collect the zinc vapour in the form of zinc oxide (Ponting 1999). The use of these two alternative methods is usually distinguishable based on chemical analysis of the resulting metal; significant levels of manganese and iron in finished alloys suggest the utilization of natural smithsonite with the absorption of manganese and iron into the smelt, while the iron and manganese in sphalerite will not vaporize with the zinc during the roasting and sublimation process and result in finished alloys containing notable zinc levels, but lacking significant levels of manganese and iron (Ponting & Segal 1998: 117).

Although generally attributed to the first millennium BC, the invention of the cementation process is probably related to the invention of co-smelting and other mixed-ore smelting techniques in the Chalcolithic of the Near East, documented as early as the fourth millennium BC (see Rapp 1986). Indeed, the development of co- and mixed-ore smelting methods was likely responsible for the explosion of copper-base alloying in the third millennium BC to include copper-arsenic, lead, nickel, antimony, and tin. Despite the ubiquity of zinc ores that naturally occur in association with copper ores, there is little evidence of widespread copper-zinc alloying before the first millennium BC.

A few notable exceptions that have been published but not often remarked upon in the context of early brass involve the corpus of metal objects from Thermi on the Greek island of Lesbos, a site that spans most of the Aegean Early Bronze Age (circa 3000-2500 BC). This collection is most famous for having some of the earliest tin bronzes in the Aegean (a pin from Thermi I) as well as the oldest example of tin metal (a bangle from Thermi IV) (Begemann *et al.* 1992). For this reason, two major lead isotope provenance studies, Stos-Gale (1992) and Begemann *et al.* (1992), have been carried out on this material in order to address questions about the early sources of tin in the Aegean, with little attention having been paid to the alloying and production technologies used to fashion these artefacts. Given that copper-zinc alloys occur alongside the copper-tin alloys from levels I-V at Thermi, a more comprehensive compositional and metallographic examination of objects from this collection could help to place these objects in a more informed context in the development of brass and tin bronze technology. Table 1 documents the numerous copper-zinc alloys (above 8 wt% Zn) at this site in relation to other early examples.

Begemann *et al.* (1992: 219) wrote of the early copper-zinc alloys from Thermi, "We consider this to be a chance occurrence, not the beginning of intentional brass technology," a sentiment shared by Stos-Gale (1992: 160). The assertion that



TABLE 1: Early copper-zinc alloys (above 8 wt% Zn) in the Ancient Near East

Site (level)	Type	Time	Major Elements other than Cu (wt%)					Reference
			As	Sn	Zn	Pb	Fe	
Gordion	3 Fibulae 1 Bowl	8 <sup>th</sup> -7 <sup>th</sup> century BC		6-16	>10 12	2.0	0-4	Young 1981
Ugarit	Ring	c. 1400 BC			12			Schaeffer-Forrer <i>et al.</i> 1982
Nuzi (II)	Ring	c. 1400 BC		0.4	14.4	4.73		Bedore & Dixon 1998
Nuzi (II)	Ring	c. 1400 BC		6.3	12.2	3.35		Bedore & Dixon 1998
Tepe Yahya (IVA)	Bracelet	c. 1700 BC			19.4	0.86		Thornton <i>et al.</i> 2002
Tepe Yahya (IVA)	Ribbon	c. 1700 BC			17.0			Thornton <i>et al.</i> 2002
Tepe Yahya (IVA)	Fragment	c. 1700 BC		0.78	16.9	1.82		Thornton <i>et al.</i> 2002
Umm an-Nar	Dagger	late 3 <sup>rd</sup> mill. BC			10.0			Frifelt 1991
Altyn depe	Blade?	mid 3 <sup>rd</sup> mill. BC	2.2	6.6	16.0	12.0		Egor'kov 2001
Thermi (P.P.)	Ornament	mid 3 <sup>rd</sup> mill. BC		2.21	10.3			Begemann <i>et al.</i> 1992
Thermi (V)	Disc	mid 3 <sup>rd</sup> mill. BC		9.2	16.9			Begemann <i>et al.</i> 1992
Thermi (IIIb)	Pin	early 3 <sup>rd</sup> mill. BC	2.8		8.52			Begemann <i>et al.</i> 1992
Thermi (II)	Knife	early 3 <sup>rd</sup> mill. BC			12	0.5		Stos-Gale 1992

these early copper-zinc alloys were accidental is founded on an implicit assumption that can now be re-evaluated in light of recent findings. The interpretation of these alloys as accidental or unintentional implies that ancient metalworkers were somehow unaware or not technologically capable of managing the materials of their own craft. Alternatively, we suggest that these early examples of copper-zinc alloying attest to a more widespread and earlier working knowledge and use of brass production in the Near East than is commonly accepted.

In recent years, two separate archaeometallurgical projects on artefact collections from Harvard University museums were analysed at the Center for Materials Research in Archaeology and Ethnography (CMRAE), Massachusetts Institute of Technology, under the direction of Professor Heather Lechtman, both of which uncovered evidence for intentional copper-zinc alloy production in the mid-second millennium BC. Although a millennium later than the Thermi objects, the artefacts from Nuzi, in northern Mesopotamia, and Tepe Yahya, in south-eastern Iran, are synchronic with a single find from Ugarit (Ras Shamra) with 12 weight percent zinc (Schaeffer-Forrer *et al.* 1982, in Craddock 1988) and the much-contested pin from Gezer mentioned above, both from the Levant. In order to add to the discussion and further the general understanding of early copper-zinc alloys in the Ancient Near East, the results of chemical and metallographic analyses of these artefacts are presented below.

## Methodology

Artefacts analysed from the Nuzi and Yahya collections were drawn, photographed, and external features were noted before an approximately 5 mm sample was cut from each object using a hand-held jeweller's saw. Each section was then hot-mounted in polyester resin, manually ground on 200-600 CarbiMet grinding strips to reveal a metal surface, and polished on mechanical wheels using oil-based diamond paste (1-6 micron) and water-based alumina abrasives (0.05-0.3 micron). The mounted samples were etched with a variety of chemical solutions to

bring out features of the microstructure; both polished and etched samples were examined and documented using a Leitz Metalloplan metallographic microscope and a Wild M420 Macroscope.

To determine the chemical composition of the artefacts and their internal features, both the Nuzi and the Yahya artefacts mounted for metallographic analysis were subjected to wavelength-dispersive spectrometric (WDS) electron microprobe analysis (EMPA) on a JEOL Superprobe at M.I.T. In addition, a second sample was cut from each of the Yahya artefacts using a jeweller's hand-saw and dissolved in a solution of nitric and hydrochloric acid in order to be analysed on a VG Excell inductively-coupled plasma mass spectrometer (ICP-MS) at Thermo Elemental. Although measuring at different detection limits, the results from the EMPA and the ICP-MS of the Yahya artefacts were corroborative.

## Results and Discussion from Tepe Yahya

The three brass artefacts analysed from Tepe Yahya, housed in the Yahya collection of the Peabody Museum at Harvard University, were all excavated from the same archaeological context dated to the end of the Late Bronze Age period 'IVA' (circa 1700 BC; see Thornton *et al.* 2002). They were found in an area of the site that exhibits more resemblance in material culture to the Bactrian Margiana Archaeological Complex (BMAC) of Central Asia than to the local material culture of the preceding Middle Bronze Age period (Period IVB: 2400-2000 BC). This distinctive material culture, which is found throughout Iran at this time, has been discussed by Hiebert and Lamberg-Karlovsky (1992), and interpreted as an actual movement of people and not just a result of trade or cultural adoption. Within the metals collection from Tepe Yahya, there is a dramatic difference in chemical composition and form between the objects found within the 'Central Asian' area of well-built domestic houses on the southern side of the tell site in Period IVA (including the three brasses presented here) and contemporary artefacts from the northern area of the site that are a

direct continuation of the Period IVB culture (see Thornton *et al.* in press). In addition to the copper-zinc alloys, ornaments in the 'Central Asian' area of the site were made of tin bronze, leaded tin bronze, and even 'proto-pewter' (Pb-Sn), while those from the local Period IVB areas of the site were predominantly arsenical copper.

The three brass pieces analysed from Period IVA are a small fragment of cylindrical shape, a broken 'bracelet' fragment, and a piece of 1-2 mm thick 'ribbon' wire (not analysed metallographically). The bracelet fragment was covered by an even yellow-green patina, with one end having been more heavily corroded. X-radiography of this object showed that the modern corrosion surface does not mimic the original shape of the metal, which was of uniform thickness throughout (about 4 mm). A transverse section of the bracelet revealed a large stress fracture in the centre of the object that runs longitudinally through the length of the object. The metal itself, which contains roughly 19.4 weight percent zinc and 0.86 weight percent lead, has a bright golden hue and contains a number of inclu-

ring step suggests it was decorative rather than utilitarian, and likely a bracelet, as labelled by the excavators.

The cylindrical fragment had an even yellow-green patina similar to that found on the bracelet, with one end of the piece having been flattened into a finished edge. Although not x-rayed, a small stress fracture can be seen in the bottom centre of the transverse section. The metal, which contains roughly 16.9 weight percent zinc, 1.82 weight percent lead, and 0.78 weight percent tin, again had a golden hue, but slightly less lustrous in appearance due to decreased zinc and increased lead content.

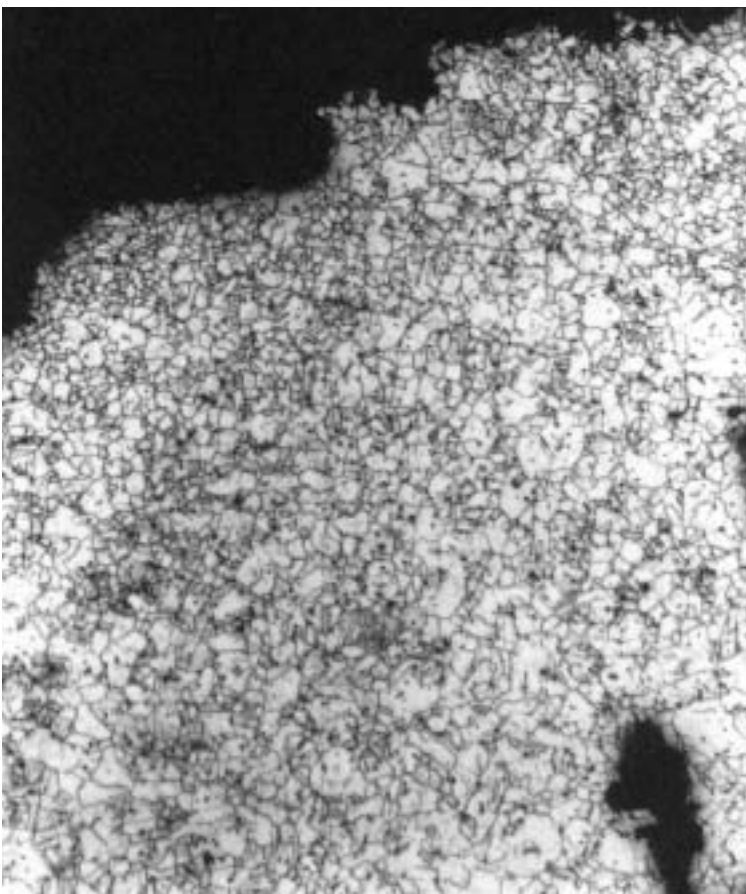


Fig. 1. Photomicrograph of bracelet fragment demonstrating grain size gradation from edge to central fracture. 50x mag. Etched; alcoholic ferric chloride.

sions determined by EMPA to be zinc sulphide and lead inclusions. Etching with alcoholic ferric chloride ( $\text{FeCl}_3$ ) and potassium dichromate ( $\text{K}_2\text{Cr}_2\text{O}_7$ ) revealed fairly equiaxed grains containing annealing twins, with smaller grains toward the outer edge and larger grains along the fracture and centre of the object (Fig. 1). This, along with the absence of strain lines, indicates the final step in production was an annealing episode. The fact that this object was not work-hardened as a final manufactu-



Fig. 2. Photomicrograph of the V-shaped fissure of cylindrical fragment. Note the flow of the deformed inclusions towards the edge. 50x mag. Etched; alcoholic ferric chloride.

Etching with ferric chloride and potassium dichromate again revealed equiaxed grains (although roughly half the size of the bracelet's grains) with annealing twins, and no strain lines. This cylindrical fragment contains a higher number and concentration of zinc-sulfur and lead inclusions than are present in the microstructure of the bracelet, and the inclusions have been plastically deformed and elongated towards the V-shaped fissure on the edge of the object (Fig. 2). The presence of this fissure suggests that this object was initially cast with a rectangular cross-section, but through episodes of working and annealing of the exterior edges in opposite directions, the metal was plastically deformed and bent around into a circular shape leaving only the V-shaped fissure where the two edges met. The presence of distinct clusters of small grains on the edge of the bracelet fragment (not shown) may suggest that both objects began with rectangular transverse sections.



There are a number of intriguing questions that can be addressed using the results of chemical and metallographic analyses of these brasses from Tepe Yahya. First, and perhaps the most obvious, is how were they made? Cementation seems to be the most likely explanation. Although iron could not be detected using the ICP-MS due to isobaric interference with the argon carrier gas, the average manganese composition of the entire collection was 7 ppm with a maximum of 28.5 ppm, suggesting an anthropogenic zinc oxide was used in the process and not a natural ore. The second question that must be asked is where did this brass come from, as there is almost no indication of metalworking in any period at Tepe Yahya. Based on the archaeological context, one possible explanation is that brass, tin bronze, and other alloys came to Yahya along with the material culture (if not the people) from the BMAC (see Thornton *et al.* in press). Finally, why were the fragment and the bracelet worked in such different ways if we presume that both were intended to have a circular cross-section? Is this indicative of two different metalworkers (perhaps a product of the 'cottage' industry suggested by Heskell (1982) for the earlier periods at Yahya) or simply the same metalworker with a different goal for each object? These questions may be far more difficult to answer.

## Results and Discussion from Nuzi, Iraq

The artefacts reported here form part of the Nuzi collection housed at the Semitic Museum at Harvard University, excavated from 1927-1931 by a joint project of Harvard University and the American School of Oriental Research, Baghdad. Two finger rings were recovered from Stratum II occupation levels in the temple complex on the main mound. Stratum II, the most extensive Bronze Age occupation at the site, is identified as Mitannian, Nuzi Ware having been identified in a number of contexts (Starr 1939; Shoemaker 1996). The Stratum II occupation was destroyed and abandoned and much of the Mitanni occupation was sealed by this destruction layer, dated to c. 1350 BC, which clearly marks the end of a Mitanni presence at Nuzi (James Armstrong, personal communication).

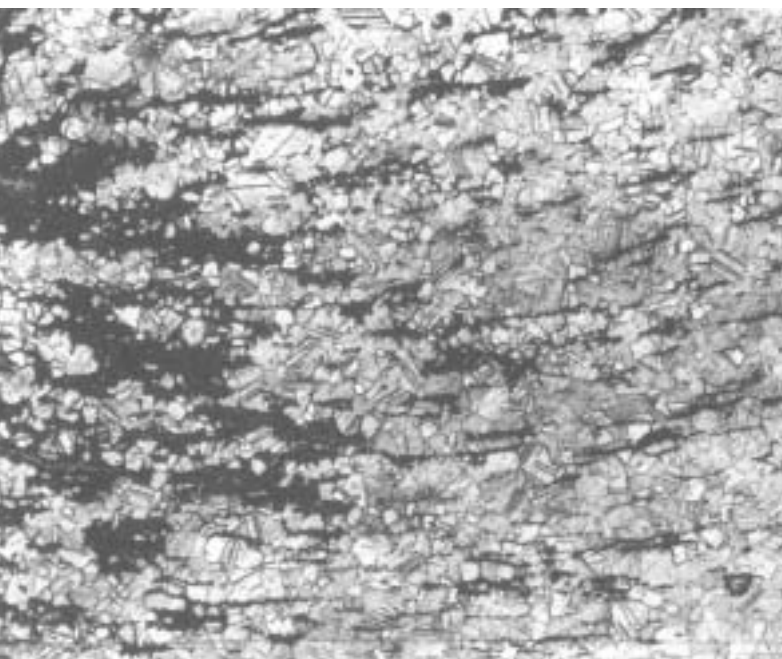


Fig. 3. Photomicrograph of band ring showing deformed inclusions and grain structure. 200x mag. Etched; alcoholic ferric chloride.

A small ring in the shape of a flat band was recovered from the main courtyard of the Stratum II temple (Temple A). Identified by excavators as having been part of the store of temple offerings and furnishings, this piece survived the sack of Nuzi and was found in the occupation layer just below the destruction level. A second ring with ridged surface decoration was recovered from an open area just outside the northern entrance to Temple A, sealed in a context below the destruction layer. The small finds excavated from this area are thought to have been part of the Temple A furnishings. The objects appear to have been scattered as a result of the looting and destruction of the Temple during the sack of Nuzi.

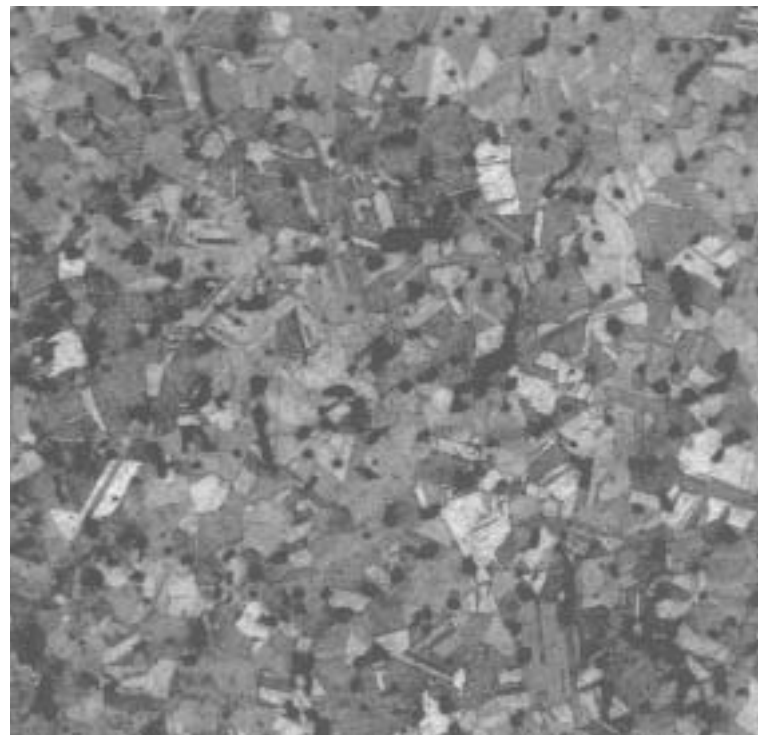


Fig. 4. Photomicrograph of brass microstructure of the decorated ring. 200x mag. Etched; alcoholic ferric chloride.

Chemical analysis identified the band as a copper (77.6 wt%), zinc (12.2 wt%), tin (6.3 wt%), lead (3.35 wt%) alloy. Metallographic analysis revealed a solid matrix of copper, zinc, and tin metal alloy with inclusions of lead that precipitated out of the solid metal solution along the grain-boundaries during the cooling of the original cast. Features of the microstructure reveal that the original cast blank, a rod or bar, was worked along both the interior and exterior surfaces, compressing and bending the metal into shape. The deformed and aligned lead inclusions along with the presence of equiaxed grains and annealing twins indicate that a number of working and annealing episodes occurred during the production of the ring (Fig. 3).

The results of chemical analysis reveal that the decorated ring was made from a leaded-brass alloy, 79.8 weight percent copper, 14.4 weight percent zinc, and 4.73 weight percent lead. The bright yellow-orange solid microstructure of the copper-zinc (brass) alloy also contains lead inclusions that precipitated out of the molten metal along the solidifying copper-zinc grain boundaries during the initial cast and cooling of the metal (Fig. 4). Metallographic examination of this piece reveals that it was also shaped from a cast blank; the small size and equiaxed

shape of grains along with the presence of annealing twins indicate that a number of alternating working and heating episodes were used to shape the original cast rod into a ring.

The production technique of shaping a cast blank into an object through sequences of hammering and annealing is well-attested in the Near East by the time of Nuzi. The striking and unexpected results of these analyses are the chemical compositions of the rings. The zinc content in these pieces, 12.2 weight percent and 14.4 weight percent, would have been sufficient to alter the appearance of the copper alloys. Even at low levels (relative to later 'true' brasses of 30 to 32 wt% Zn), the presence of zinc in copper alloys renders the metal a noticeably more golden colour than copper-tin or copper-arsenic bronzes (Craddock 1995: 293). The golden colour of the brasses used to fashion these rings would have made them distinct from contemporary bronze alloys.

The link between the selection of materials, the choices made during production, and the appearance of a finished product is documented elsewhere in the archaeological record (e.g. Hosler 1994; Lechtman 1996). The occurrence at Nuzi of two pieces of jewellery made from an alloy that would have resulted in a finished product with a distinctive colour and appearance is significant. In addition to these brass rings, six other artefacts from the Nuzi Collection were analyzed, all recovered from contemporary Nuzi contexts. The results of analysis of three arrowheads (C. Dixon, analyst) and three stick-pins/fasteners from the site reveal that metalsmiths were using different alloys for different kinds of objects. The arrowheads were made from copper containing small amounts of arsenic or tin along with trace amounts of other components. Two of the stick pins/fasteners were also made from copper with low levels of arsenic (less than 1 wt%); the third fashioned from a copper-tin bronze (Sn 6.37 wt%) distinct in composition from the brass rings. The materials used to fashion the arrowheads and two of the stick pins have been termed 'dirty' copper alloys, and interpreted as having been the result of smiths using polymetallic ores or recycled metals rather than having been deliberate combination of metals to produce intentional alloys (Bedore Ehlers & Dixon 1998; Heather Lechtman, personal communication). It is possible that the production methods used to retain or introduce zinc in certain alloys were employed discriminately, with specific results in mind; it is intriguing that objects of personal adornment were made from golden-coloured brass while objects made for more functional purposes were fashioned from copper and copper alloys, notably lacking zinc.

## Conclusions

The characteristics of zinc are such that its extraction and retention in an alloy require particular, refined processes during production. It seems unlikely that sites such as Thermi, Tepe Yahya, or Nuzi were centers of great metallurgical innovation, yet all three managed to acquire one of the most elusive alloys of the Bronze Age. Does this suggest that copper-zinc alloys were not as rare as previously thought, or could the lack of examples from the Bronze Age be a result of non-random sampling of metal collections - e.g., analysing large tools and weapons over small jewellery and personal ornaments? Future research has the potential to add much to this discussion, particularly on the relationship between zinc and tin, which are found both alloyed together and in association with one another at all three sites.

Requiring specific processing techniques and a deliberate investment of time and labour, the production of copper-zinc

alloys indicates a high level of working knowledge of materials and material processing on the part of metalsmiths. Perhaps, as in the Medieval period of Europe, zinc was added to copper-tin alloys as a form of cheap bronze. Alternatively, copper-zinc alloys may have been highly valued due to their golden colour, distinct from bronze alloys, and thus their resemblance to objects fashioned from gold. Ultimately, evidence of brass production and the reconstruction of the components and production processes used in the manufacture of copper-zinc alloys indicates something about the methods used and the decisions and choices metalsmiths were making to result in the desired finished product.

The results of these recent studies provide evidence that copper-zinc alloys existed almost two thousand years before the date generally accepted for the development of the cementation process, suggesting that the history of brass is longer and more complex than is generally believed. Our role as archaeologists interested in metallurgy is to figure out how and why. Answers regarding the origins and development of early copper-zinc alloys in the Ancient Near East may be as elusive today as they were twenty years ago, but at least now we know to look for them.

## Acknowledgements

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# Egyptian chariots, Midianites from Hijaz/Midian (Northwest Arabia) and Amalekites from the Negev in the Timna Mines

## Rock drawings in the Ancient Copper Mines of the Arabah – new aspects of the region's history II

Beno Rothenberg

### Introduction

Two panels of rock engravings were discovered on the walls of Mine 25 (Fig. 1) by our Arabah Expedition in 1966. Mine 25 is situated along the upper reaches of Wadi Timna, where the Timna Cliffs retreat and form a side valley, flanked on both sides by huge formations of cupriferous Nubian Sandstone and slopes with numerous mining relics, sand-filled shafts ("plates") and gallery openings. First published many years ago (Rothenberg 1972: 119-124, cf. also Anati 1981: 49-61), the rock engravings were recently re-investigated in the light of our new, much revised, concepts of the history of the Timna copper industries and related archaeological finds (Rothenberg 1988; 1999), resulting in a better understanding of this rock art. This has also substantially contributed to the understanding of the ethnic aspects of the history of Timna, the collaboration of Amalekite workers from the Negev and the 'Midianites' from North-

west Arabia, with the Egyptians of the 19<sup>th</sup> and 20<sup>th</sup> Dynasties of the New Kingdom, from the end of the 14<sup>th</sup> to the middle of the 12<sup>th</sup> centuries BC.<sup>1</sup>

Besides these two large rock engravings, there are surprisingly only very few other rock drawings inside the Timna Valley. Several small iconic images, accompanied by memorial inscriptions, were found near tombs of Roman date and were intrusive in style. This is in contrast to the Negev, where there are many thousands of rock drawings related to the indigenous population settlements and camping sites, dating from the Chalcolithic Period to Islamic times. The same is the case in the Sinai, as well as along major pathways and campsites on the way to the mining regions, where many thousands of rock drawings were reported, most of which of intrusive character related to turquoise mines and to copper mining and smelting (Rothenberg 1987; Petrie 1906; Anati 1981). The two rock engravings in the Timna mines are of special significance since they are witnesses for major activities in the Timna mines by groups of workers from afar of different ethnic origin and culture.

### Amalekite rock art - Engraving 1

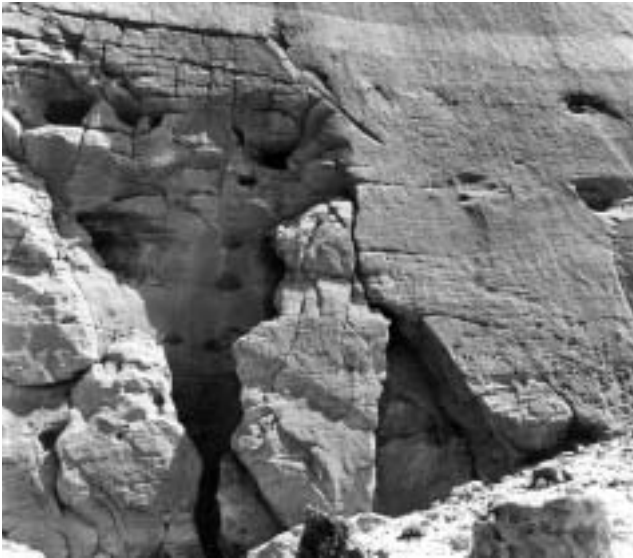
About 50 m in front of the southern tip of Mine 25, a circa 10 m high pile of rubble and large boulders lies against the hillside. Some 5 m above this pile, a large panel of 'drawings' - Engraving 1 - 5 m wide and 2 m high, was engraved on a particularly smooth area of the rock wall (Fig. 2). The rather unusual location of this rock engraving, high up on this steep and flat rock face where there is nothing to stand on for the drawing 'artist', can only be explained by the fact that we are dealing with a hillside which collapsed sometime after intensive underground mine workings were carried out inside this part of the hill. Adjacent to the left of the rubble pile, a mine shaft, now



Fig. 1. Northern half of the Arabah Survey map of the Timna Valley. Site 25 is the location of the rock engravings.



Fig. 2. Engraving 1.



*Fig. 3. Mine shaft with typical New Kingdom footholds next to the rubble pile. Engraving 1 is located to its right on the flat rock surface.*

partly damaged, was dug into the rock wall (Fig. 3), datable by its technology, shape, and footholds to the Egyptian New Kingdom, contemporary with the major mining activities in the Arabah and especially in the Timna Valley (Conrad & Rothenberg 1980). We assume that gallery workings branching out from this shaft, and from a second shaft further to the left, contributed to the collapse of the hillside. This collapse was perhaps caused by one of the many minor earthquakes of the region, the latter witnessed by collapsed installations in the excavated smelting camps, like at Site 2 and the Timna Temple (Rothenberg 1972: 149), and deformed mine workings, like that of the shaft at Site 7.

The second shaft began at the same height as the shaft close to Engraving 1, and was dug into a narrow ridge still partly preserved directly in front of a small cave, where we found habitation remains and some pottery sherds (Fig. 4). By the lighter patina we could tell that this ridge originally continued all along the hillside and the 'artist' must have stood on it whilst working



*Fig. 4. At the left, the cave and mine shaft belong to the same mine as the shaft next to Engraving 1 (at the left of the figure).*

on Engraving 1 - right next to the collar of the mine shaft. Since the collapse of the hillside also caused much damage to the shaft next to the cave, and bearing in mind the typical tool-marks and footholds still preserved on the walls of the shafts, we may assume that both shafts belonged to one and the same Egyptian New Kingdom mine. From these shafts underground galleries branched out, leaving behind a hollow hillside, which collapsed at some later stage as part of the widespread destructive erosional geomorphologic processes in the Timna Valley (Conrad & Rothenberg 1980: 57-68).

The figures of this 'picture' are deeply incised with a sharp-pointed implement in a purely linear style, typical for the indigenous inhabitants of the Negev mountains and common, in fact, all over the Middle East from the 2<sup>nd</sup> millennium BC to almost recent times (Anati 1981: 49). There is one horizontal line for the animal body, short lines for its legs and one vertical line for the human body, with bent lines for arms and legs, in contrast to the much more realistic figures of Engraving 2, which show full bodies and even details of clothing (see Fig. 5 below).

It seems that this panel was purposely made as one pictorial unit consisting of three long rows of juxtaposed figures of ostriches, ibexes, and gazelles, with several hunters and their hunting gear, perhaps an afterthought, fitted into the picture. There are two exceptions to this assembly. On the right side of the panel appears a crude representation of a chariot with two four-spoke wheels, drawn by what seems to be two ibexes with long, drawn-back horns. These are harnessed together at their heads by a heavy looking cross-beam.<sup>2</sup> It seems obvious that the 'artist' had never seen a horse-drawn chariot and copied its image by memory from Engraving 2, completely missing the horses on the original.

The second exception on the panel is the solitary iconic image of a 'human' figure, its raised large hands showing widely spread four fingers, standing above the left end of the upper row of animals. A strange object, drawn as a straight line ending in an oval sling or handle, projects from the hip of the figure and could represent some kind of weapon. This seems to be a 'magic' icon or the image of a 'higher spirit' of 'Midianite' origin and was added onto the panel by a different 'artist', perhaps the same one who created the 'Midianite' centrepiece of nearby Engraving 2 (see below).

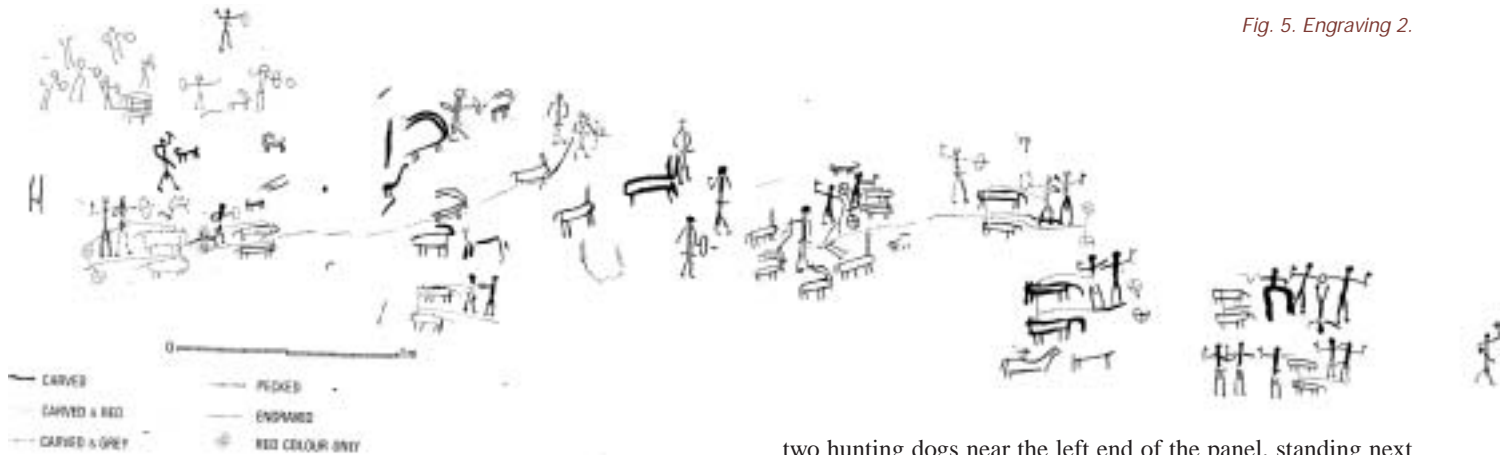
Although the indigenous style of Engraving 1 was common in the region for several thousand years, its location next to the typical Egyptian New Kingdom mine shaft of Mine 25 seems convincing evidence for its New Kingdom date. Engraving 1 must have been made by workers from the Negev working with the Egyptians and 'Midianites' in the Timna mines (Rothenberg 1998).<sup>3</sup> Furthermore, the image of the chariot on Engraving 1 was evidently a copy of the Egyptian New Kingdom chariots of Engraving 2, and the 'Midianite' icon of the 'human' figure with large, widely spread hands, is well-dated to this period (see below). The inhabitants of the Negev in the Late Bronze - Early Iron Age have been identified with the indigenous seminomadic tribe mentioned in the Bible as "Amalekites dwell in the Negev region" (Numbers 13:29), and Engraving 1 is an impressive example of indigenous Amalekite rock art.

### **Egyptian and 'Midianite' Petroglyphs - Engraving 2**

About 100 m along the cliffs, a narrow canyon opens into the mountainside. It is about 40 m deep, 20 m high, and about 5 m



Fig. 5. Engraving 2.



wide. On its right side, a 9 m long and 1.5 m high panel of rock engravings, Engraving 2 (Fig. 5) was cut into a smooth stone frieze sheltered from the occasional torrential rain of the area by overhanging rocks. On the floor, below the engravings, we found a group of large bowls made of very soft sandstone (Fig. 6), the like of which we have only found in the Hathor Temple of Timna, used in a ritual context. Some sherds of the Egyptian New Kingdom were found next to the stone bowls. This set-up suggest that the canyon of Engraving 2, and perhaps also



Fig. 6. Stone bowls on the floor of the canyon of Engraving 2.

Engraving 1, had a ritual function in the very heart of the New Kingdom mining region of Timna, one of the many cult sites related to the mining as well as smelting sites of the Timna Valley (Rothenberg 1972).

Contrary to the style of Engraving 1, which was purely linear, the figures of Engraving 2 have 'volume' and also show details of clothes, the latter presumably in order to emphasize ethnic characteristics of the different groups of people represented here. It seems that Engraving 2 was begun in the centre of the panel with the group of tall hunters with emphasized large heads, carrying long hilted straight-bladed swords and, of especial significance, all wearing tasselled kilts (Fig. 7). Some of the hunters brandish a battleaxe, others carry bow and arrow ready for action. We propose to identify these hunters with the 'Midianites' from Northwest Arabia, the third partner of the Egyptian New Kingdom copper industry of Timna (see below).

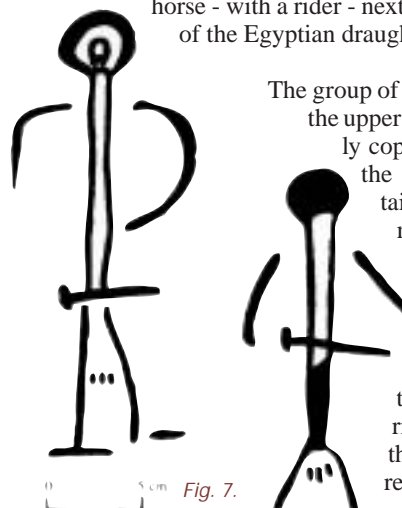
All around the hunters appear different animals, representing the game animals common at the time in the region: ibexes, oryxes, one ostrich, two leopards and one cheetah. There are also

two hunting dogs near the left end of the panel, standing next to a hunter which does not belong to the two main groups of engravings on the panel. This appears to be a somewhat later addition.

The dominant theme of Engraving 2 is the arrangement of four-spoked manned chariots. The chariots have rear-positioned wheels, but no sidescreen. In the first publication of Engraving 2 (Rothenberg 1972: 122), we assumed that the plump bodies of the draught animals represented oxen, but we now accept the identification of these engravings as horses<sup>2</sup> harnessed to the front of the pole. One or two armed men are standing on each chariot. Some carry a shield or a bow and all hold a typical Egyptian New Kingdom battleaxe in a raised hand. To free the hands of the charioteer, the reins are tied around his waist. This is a feature well known from New Kingdom wall-paintings. All occupants of the chariots wear loincloths folded into a pointed apron in front; this is a common garment of the Egyptians of this period, as can be seen, for example, on the reliefs of Queen Hatshepsut's temple at Deir el-Bahri (Breasted 1906/7, IV: 204).

Many of the lines of the chariot engravings are filled with red ochre-like colour, and here and there also a grey-white fill can be seen. Some of the chariot wheels were actually painted with red colour straight onto the rock without a previously engraved image. This technique has not been seen on any of the petroglyphs of the region, but is well known from Egyptian wall-painting techniques.

It is somewhat difficult to explain the beautifully-engraved horse on the low edge of the panel, close to the manned chariots on its right side.<sup>4</sup> According to the patina, this horse is an ancient drawing though probably later than the chariots next to it. Perhaps a later visitor to the site had the same problem identifying the plump draught animals and expressed his interpretation by this almost perfect, well-proportioned image of a horse - with a rider - next to the 'problematic' images of the Egyptian draught animals.



Detail of Engraving 2.

The group of barely discernible images in the upper left corner of the panel, badly copying some of the figures of the original engraving, is certainly a much later addition made by rubbing with a blunt implement. The scene on the right end of Engraving 2 is rather enigmatic. The whole group, including the draught animals harnessed to a centre pole with no chariot wheels behind them, and the two groups of three figures, drawn exactly like the cha-



rioteers with battleaxes in raised hands, apparently represent some kind of ceremonies. This is particularly true for the group with one figure standing on its head and the two figures behind holding together a strange round object - it seems to express an iconic idea - enigmatic for us<sup>5</sup> - in contrast to the narrative character of both of the main groups of Engraving 2.



Fig. 8. 'Midianite' iconic image on a sherd from the Hathor Temple of Timna.

A solitary figure with a raised hand, widely spread fingers and wearing a long sword is engraved on a separate plane of the rock at the extreme right of Engraving 2. This is obviously an iconic image. Besides the widely spread fingers of its raised hand, however, it does not show any of the characteristics of the solitary figure on top of Engraving 1 which we consider to be a 'Midianite' icon; it seems to represent a similar iconic image. In connection, we should mention that among the characteristic motifs of the decorations on 'Midianite' pottery found in the mining and smelting camps and the Hathor Temple of Timna (Rothenberg & Glass 1983: 92), as well as at Barqa el-Hetiye (Fritz 1994: Abb. 12:13) in the mining region of Feinan (Jordan), there appears a human figure with emphasized large headgear and widely spread raised hands - no doubt a characteristic 'Midianite' iconic motif (Fig. 8).

## The ethnohistoric message of Engraving 2

During our first surveys and excavations in the smelting camps 1959 to 1969, we had serious problems identifying and dating the pottery finds. Everywhere, first on the surface of the sites of our survey (Rothenberg 1962; Aharoni 1962: App. 1) and later on in all the strata of our excavations in the smelting camps, we found three totally different kinds of pottery, the dating of which was at the time extremely difficult (Rothenberg 1988: 3-11):

1. Rough handmade pottery of a type to be found in the Negev settlements, called 'Negebite Ware', and generally dated Iron Age II;<sup>6</sup>
2. Local wheel-made 'kitchenware', which appeared to have some comparisons at sites in the Levant of an earlier, Late Bronze Age, date; and
3. Hand-made vessels with polychrome decorations, the latter reminiscent of motifs of decorations of Aegean pottery. When first found in our surveys, we related this decorated pottery to the Edomite pottery of Jordan (Aharoni 1962: 66). However, when our subsequent excavations showed that the copper industry of Timna, and the wheel-made pottery found in its layers, was apparently of a much earlier date, the unique polychrome pottery with Aegean decorations, until that time unknown to archaeologists of the region, presented a

complex problem of dating and provenance and of the ethnohistoric aspects of the Timna copper industry (Fig. 9).

The discovery in the Timna Mining Temple (Rothenberg 1988) of numerous votive gifts to the goddess Hathor, carrying the names of the Egyptian pharaohs of the New Kingdom (from Seti I to Ramesses V), found together with the same three kinds<sup>7</sup> of pottery in all the layers of the Temple, provided the first solid date for the major copper industry at Timna. The date of the three kinds of pottery was those dated to the period from the end of the 14<sup>th</sup> century to the middle of the 12<sup>th</sup> century BC, a date also confirmed by <sup>14</sup>C (Rothenberg 1990: notes 21- 23). Subsequent petrographic comparison (Glass 1988; Rothenberg & Glass 1983) of the polychrome 'Aegean' pottery of Timna with the same kind of pottery found by Peter Parr in his survey in Northwest Arabia in 1968 (Parr *et al.* 1970) established that this pottery was in fact manufactured at the ancient town of Qurayyah in Hijaz/'Midian', and probably also at other towns of Northwest Arabia. We therefore suggested to name it 'Midianite pottery' (Rothenberg 1969; 1972: 154-162).

However, we do not have any evidence for the date of the actual beginning of the appearance of 'Midianite' pottery, or the end of its use, in its 'home country' Northwest Arabia. Moreover there is no indication for the existence of a potting tradition in the region out of which the 'Midianite' pottery could have developed. On the contrary, according to the available evidence from Qurayyah and other sites in the Hijaz, the 'Midianite' pottery appeared rather suddenly - seemingly together with the very imposing 'oasis urbanism' - and, according to the evidence from Timna, remained almost unaltered for at least 150 years, after which it equally suddenly disappeared. It is important to emphasize here, that at all sites in the Levant where 'Midianite' sherds were found (Rothenberg & Glass 1983; Parr 1988; Knauf 1988: 21-23), their dating was within the range of the New Kingdom date of Timna.

The finds in the Mining Temple established the identity of the three ethnic elements working together in the mines and smelters of Timna: Egyptians of the Ramesside New Kingdom, the 'Amalekites' from the Negev, and the 'Midianites' from the

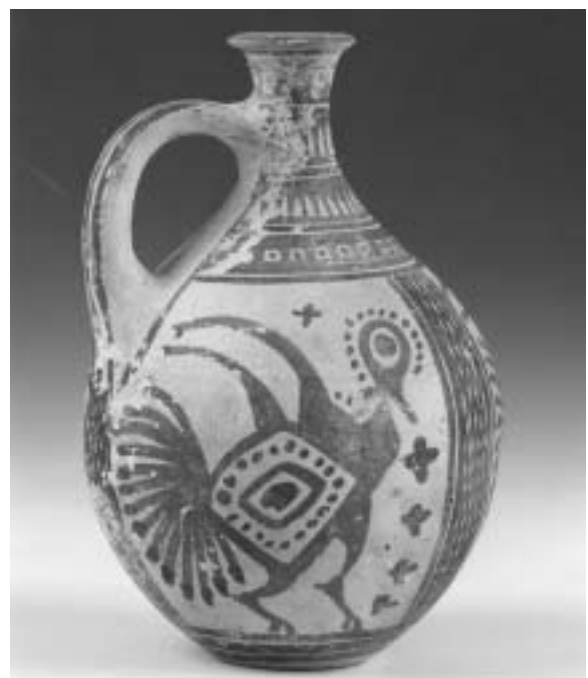


Fig. 9. A typical 'Midianite' juglet (Bible Lands Museum Jerusalem).



Fig. 10. 'Philistine' warriors with typical gear and dress on the battle scene at Medinet Habu.

Hijaz. However, the appearance in the Timna mines and smelters of 'Midianite' workers from Northwest Arabia, who according to our excavations at Timna brought with them on camel back large quantities of their pottery, evidently mass-produced in their Hijaz 'urban oases' (Parr 1988), is the core of what we called the 'Midianite enigma' (Rothenberg 1998): Who are the 'Midianite' miners/potters and how and why did they suddenly appear in this rather inhospitable and isolated region of the Near East - and what kept them going for several generations, only to 'disappear' again in the middle of the 12<sup>th</sup> century BC?

At the time, when we identified the two different types of peoples, represented in the two major 'scenes' of Engraving 2, as Egyptians on their chariots and 'Midianites' hunting the game animals of the region, the 'strange desert people from Midian' seemed rather enigmatic. I did not realize then that the solution of the 'Midianite enigma' was right there in the details of the engraving, which clearly emphasizes the typical ethnic characteristics of the people. The tall men with the large headgear, long sword and tasselled kilt represent 'Sea People', *i.e.* the 'Midianites' are 'Philistines' of ultimate Aegean origin, who had already immigrated into Northwest Arabia already as early as the time of Seti I (1318-1304 BC), the earliest date of the 'Midianite' pottery according to the Hathor Temple. Comparative documentary evidence can be found at the great Mortuary Temple of Ramesses III at Medinet Habu (Nelson 1930). Here, in the great sea and land battles of Ramesses III against various groups of invading 'Sea Peoples' (Fig. 10), the different groups of 'Philistines' are depicted in their typical wear. Amongst them are several groups, named on the hieroglyphic inscriptions as Peleset, Tjekker and Denyon, which all wear the same 'outfit': the typical large headgear, a long sword, and a kilt with three tassels - the same as the group of 'hunters' on Engraving 2 of Timna. We therefore propose now to understand the 'Midianite' hunters as representing an early wave of migrating 'Philistines', a dominant component of the 'Midianite/Philistine' community in the Northwest Arabian oasis settlements, collaborators of the Egyptians in the New Kingdom copper industry of the Arabah.

But why would 'Sea People' immigrate to the deserts of Arabia? The lack of systematic archaeological excavations in the urban oases of the Hijaz, and particularly the lack of proper archaeological explorations of the extensive mineralised zones

of Northwest Arabia, make it difficult to reach comprehensive conclusions. However, we may assume that migrating metallurgists of ultimate Aegean/Anatolian origin (Parr 1996: 216-7; Mendenhall 1984: 144; 1992: 817) were drawn to Arabia - seemingly together with Egyptians of the New Kingdom (see below) - because of its widespread gold, silver and base-metal deposits. It is important to mention that in this mineralized zones of Hijaz traces of ancient mine workings have been discerned (Roberts *et al.* 1977; Rothenberg 1998).

The Egyptian-'Midianite' partnership for about 150 years in the Timna mines strongly suggests a similar partnership - with the Egyptians perhaps even as initiators - in the mineral exploitation in Northwest Arabia. Although the mineralised zones of North Arabia have not yet been sufficiently explored by archaeologists,<sup>8</sup> there is some new evidence for Egyptian activities in Northwest Arabia which is relevant for our present considerations. During recent excavations at Tayma (Abu-Duruk 1989: 17, Pl. 9; 1990: 15), a large ancient town of great importance for the history of Arabia, 'Midianite' pottery was found in several parts of the town and in burials. Inside some of the burials, Egyptian scarabs and faience amulets of the New Kingdom were found together with contemporary 'Midianite' pottery. A.H. Masry (1990: 5) summed up the significance of the Egyptian finds at Tayma: "A hieroglyphic-inscribed scarab of the New Kingdom was discovered (in the second season). This confirms the abundance of evidence for the early contacts with the Nile valley. .... Definite type similarities were observed, chiefly relating to Philistine (!) ware... particularly to the site of Timna in Sinai."

Whatever the relationship between the 'Midianites' and the New Kingdom Egyptians, it would not at all be surprising if systematic fieldwork in the mining regions of Northwest Arabia would ascertain that the Egyptians were also partners in mining activities in this region. The Egyptians, since prehistoric times and especially during the New Kingdom, intensively worked very similar gold and copper deposits on the opposite side of the Red Sea in the Eastern Desert (Rothenberg *et al.* 1998), further south in Nubia, and, for generations (from the late 14<sup>th</sup> to the middle of the 12<sup>th</sup> centuries BC), together with the 'Midianites' from the Hijaz/Midian, in the Timna mines. It would be actually quite difficult to understand why they did not extend the wide orbit of their mining activities to the Hijaz, especially as their 'Midianite' partners were actually 'at home' in this region at that time.



## When did it all end?

The 'Midianite' sites in the Hijaz remain so far unexcavated and their detailed history is unknown. We therefore have no archaeological information to help us to understand the processes that led to the disappearance, seemingly also quite suddenly, of the 'Midianites' and their pottery from Northwest Arabia. We can only suggest that this process is connected with the overall withdrawal of New Kingdom Egypt from their 'possessions' in the Levant sometime in the middle of the 12<sup>th</sup> century BC, when the Timna copper mines were also abandoned. Parr (1988: 81-86), in his study of the "Midianite urbanism", reaches the conclusion that the apparent withdrawal of the Egyptians also from Northwest Arabia in the middle of the 12<sup>th</sup> century BC, accompanied by the interruption of the strong commercial links between Egypt and the 'Midianite'/Sea People urban oases, "led to the collapse of a precocious and precarious 'urbanism' there" (Parr 1988: 86), *i.e.* the 'Midianites/Philistines', and their pottery, vanished from the scene of Northwest Arabia.

Parr (1988: 86) discussed the appearance of "camel nomadism" as one of the results of the disappearance in the region of the 'Midianites': "It is reasonable to suppose that the two developments are related, and that camel nomadism in northern Arabia was at least partly the result of the abandonment of sites such as Qurayya." Parr is unaware of the fact (Grigson, *in press*), that the 'Midianite' miners at Timna already used many camels, and that supplies were actually brought over from the Hijaz on camel-back. Camels were also widely used as pack animals in the mines and smelters of Timna. Since people do not disappear in mid-air, we must assume that the 'missing' Midianites must have emigrated to another region. At several Philistine towns of Philistia, as for instance at Tel Masos (Kempinski 1993: 988), 'Midianite' sherds were found in Philistine layers of the 12<sup>th</sup> century BC and further research may show that these sherds are not evidence of trade with 'Midianite' Hijaz, but a trace, even if at present rather faint, of the integration of 'Midianites/Philistines' from the Hijaz into the Philistine society of the 12<sup>th</sup> century BC in Philistia.

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## Notes

- As we shall explain below, these ethnic identifications are based on the archaeological finds from the Timna excavations and their provenance, established by petrographic and typological research.
- The revised identification of the animals as horses and not oxen is based on the expert view of Dr B. Shalmon, regional biologist, Eilat district, Nature and Parks Authority. His detailed paper will be published separately. Shalmon identified the two animals drawing the chariot on Engraving 1 as horses, the same as the animals which draw the Egyptian chariots on Engraving 2.
- During our survey in the 70s of the southernmost Negev, we located a campsite with typical Negev pottery and copper smelting remains on the route between Timna and the Negev. It appears that the workers from the Negev took some ore from the mines of Timna to produce some copper for their own use on the way home.
- The edge of the rock frieze is damaged by erosion and the legs of the horse are only partly preserved.
- Anati 1984, 58, suggested that the three man standing in front are dancing, whilst the other three, with the man upside down being dead, could be understood as a funerary procession.
- According to the petrographic study by Glass (Rothenberg 1988; Glass 1988), most of this pottery, now called 'Local, rough handmade pottery', is actually of local manufacture, believed to have been made on site by the workers from the Negev, but there is also proper Negebite Ware, originating from the Negev settlements and dated by our Timna finds to the middle of the 12<sup>th</sup> century BC at the latest. This should present a difficult problem for the archaeologists who still date the Negev settlements exclusively to Iron Age II (cf. Mazar 1990).
- Glass actually identified in his petrographic study five different groups of pottery at the Temple (Glass 1988), including local hand-made pottery (which is not 'Negebite Ware'), as well as a few sherds imported from Egypt, but this does not change the general picture of the pottery finds at Timna.
- Parr (1988) discusses this issue and comes to the conclusion that we do not have any evidence for the involvement of the 'Midianites' in the exploitation of the mineralization of the coastal zone of the Hijaz and that it may be wrong to assume that the 'Midianites' exploited the mines of the region. In my view, no proper survey of the mines as such has yet been undertaken and the issue has to be considered as still archaeologically undecided. In the light of the intensive mining activities by the 'Midianites' from the Hijaz in Timna, it would be rather odd if the local 'Midianites' would have ignored the rich mineral sources of their own neighbourhood.



# Black sand and iron stone: iron smelting in Modakeke, Ife, south western Nigeria

Akin Ige & Thilo Rehren

## Abstract

Fourteen fayalitic bloomery slags, possible ore and furnace wall samples from an ancient smelting site in southwestern Nigeria were analysed by chemical and mineralogical techniques. They derive from a skillful bloomery smelting operation extracting the maximum possible amount of iron from the ore. For most of the slags the analyses show an exceptionally rich level of titanium oxide (up to around ten wt%), indicating the use of a mixed ore comprising limonite iron stone and ilmenite-rich black sand. Some of the slags were tapped, while others may have solidified within the cooling furnace, as indicated by their morphology and mineralogical texture.



**Fig 1**  
Map of Nigeria, with Ife in the south-western part of the country, in the Yoruba region.

## Introduction

The Yoruba, whose homeland covers the eastern part of the Republic of Benin and a large portion of south western Nigeria (Fig. 1), have a rich cultural heritage, which has been appreciated by many scholars. Their forefathers were basically farmers and hunters. Therefore they needed to manufacture implements for their farming and hunting as well as weapons for defensive and offensive purposes. Most of the insignia of their office and symbols of their gods were made from iron.

Generations of the Yorubas have smelted iron and the skill has been taught from one generation to another. Iron-working in Yoruba has been the pre-eminent transformative process, a technology greedily sought and jealously guarded, for its control could promote a king's ambition and a soldier's fortune. Iron-smelting technology has often been considered divine inspiration brought to humans by culture heroes. Sacred Yoruba kings were sometimes renowned as smelters and blacksmiths. In other circumstances, the transformative powers of iron-workers are deemed so great that smelters and blacksmiths are thought dangerous and avoided by ordinary people (Adepegba 1991).

The widespread occurrence of remains of the bloomery process - mining pits and tunnels, small parts of technical ceramic including furnace shafts and tuyeres, traces of charcoal, as well as copious amounts of smelting slags - provide a rich materials base for reconstructing the technology used by the ancient Yoruba smelters. Excavations at several locations in the Ife area have revealed evidence of Late Iron Age iron smelting in the Yoruba area of southwestern Nigeria. In many areas, layers of smelting debris, such as ash, charcoal, slag, and pieces of possible tuyères was found together with fragments of mud-brick walls associated with what may be the collapsed remains of furnaces.

Recent initial excavation work by one of us at Modakeke identified three furnace structures, one of which has been excavated. It was round, built of large mud bricks, and contained ash, slag, and burnt brick. The excavation revealed a sequence of three cultural layers that contained slags, furnace walls, charcoal, roots and rootlets. The top layer was grey lateritic compact soil full of slags and furnace walls (the samples of the NG-MO and NG-IP series belong to this group). The second layer was composed of hard dark brownish soil that contained significantly less and smaller slags and furnace fragment (the NG-IS series). The third layer consists of loose and reddish brown soil and contained a furnace base of 65 cm in diameter and a wall about 7 cm thick. The two ore samples (MO7 and MO6) were collected from this layer.

The objective of this study is to determine the chemical, mineralogical and microstructural characteristics of the ore and the slags, in order to reconstruct the technology involved in the smelting process and the temperature attained during the operation. This will unravel the technical skills of the early Yoruba settlers.

This study is the beginning of a large scale scientific investigation of several smelting sites in Yoruba land to determine the origin of the technology involved in iron smelting and the relationship of this technology to the trade and migration patterns.

## Historical background

Ile-Ife is regarded by all Yoruba as their immediate origin. From there, their ancestors dispersed to establish towns in their present homeland in West Africa. Ife was a centre of the iron manufacture, though chiefly of small wares, such as nails, horse-shoes, keys, locks, and common agricultural tools; and it was estimated that there were about 500 iron smelters, smiths and other workers in iron of various kinds living within a radius of about twenty kilometers (Adeniji 1977).

From the history and art of the Yorubas it is certain that the knowledge of mining and metallurgy was prevalent among the royalty. As they spread across the land they founded kingdoms and empires, and iron mining and smelting continued because of the need to acquire weapons to fight and conquer more lands. The oldest and the most powerful of these kingdoms is the Old Oyo. However the northward growth of the Yoruba kingdom (see Fig 1) was halted by the Fulani jihadists. No sooner had the Fulani jihadists smashed Old Oyo, in about 1830, there was a decline in iron production. The Old Oyo empire suffered the greatest collapse that drove the people back to their original homelands in the Ife area. As they migrated homewards they continued to spread the art of smelting especially in towns like Igbaja, Oyo, Isundunrin and back to Ife. Among the returning soldiers and smelters were those who had carried the technology of iron smelting with them; upon their return to the Ife area, they separated themselves from those who had stayed, and formed a separate settled area within the confines of Ife, called Modakeke. It is from this area that the slag samples studied here were taken.

It is not clear what eventually stopped the smelting operation in the region. Evidences gathered suggest the instability caused by intertribal wars, the appearance of European scrap iron, and perhaps the agrarian revolution that altered the commercial leaning of the local people, or a combination of all this factors. Lack of raw materials can be ruled out as there is still an abundance of various grades of lateritic ironstone in almost all known Yoruba towns. However, as reported by other authors (eg Miller 1995), local iron production in many African countries declined sharply with European colonization as imported iron assumed increasing economic importance.

### Site location

The site for this study is located near a disputed boundary between the Modakeke and Ife communities in the ancient town of Ile-Ife. Geologically it is situated within the basement complex of Nigeria which consist of early Proterozoic (2200 my) gneisses and schists. The schists consists of mica-garnet bearing rocks intimately associated with mafic to ultramafic rocks. These rocks have been extensively studied and characterized by several authors (e.g. Ige & Asubiojo 1991; Ige *et al.* 1998). The greenstones which are the parent rocks for the lateritic ores occur as lenses within the polydeformed migmatite-gneiss complex. Several outcrops occur along the 800 km-long greenstone belt of Nigeria. Their mineralogy consists mainly of iron-magnesium amphiboles and ore minerals such as spinel, pyrrhotite, and pyrite. The area is located within the tropical rain forest with high temperature and high humidity. A distinct characteristics of the area is the abundance of thick lateritic ironstones formed from the *in situ* weathering of high Fe-Mg ultramafic rocks.

The mining site is called *Ereta*, meaning the area where bullet-like ore nodules are found. The smelting site is about one to one and a half kilometers away from the mining site. The mine consists of a large open pit grading into tunnels and underground mines. That there are several underground mines is evidenced in the collapsed structures built above these tunnels, which can be seen in several places in Eleta. The most prevalent ore material is an aggregate of limonite and goethite derived *in situ* from greenstones.

These ores are abundant in almost all Yoruba towns and provided the needed raw materials for the operation of hundreds of mines and iron smelting centres. In other areas of Yoruba land where smelting was still in operation up until 1970, Adeniji (1977) and other oral evidences available described the major type of ore used is gravel-stone known as *oko* or *eta* stone (lateritic ironstones) derived from the breakdown of ultramafic amphibole rocks which are prevalent in the area. The *oko* stone is rich in siderite (iron carbonate), goethite (iron hydroxide) and limonite. According to oral tradition, black sand is washed from the soil during heavy rainfalls and accumulates in pockets and gutters, from where it is easily collected, and was also added to the ore. A preliminary sample of this black sand was studied and found to be rich in ilmenite and magnetite. The combination of these two iron ores, the lateritic stones and the sand, provides the furnace charge, providing the iron mineral for reduction to metallic iron as well as fluxes that will allow the formation of a good slag during smelting in the furnace.

### Mining methods

According to oral evidence and field observations, the iron smelters were prospecting for the raw materials by following the presence of iron stones on the surface; they then deduced that

more iron ore abounds under ground. They then mined the ore using two different methods

1. Open pit grading into underground or tunnel when the iron-stone is deposited vertically in the ground.
2. Trenches and channel mining if the iron-stone lies horizontally in the ground

When the ore has been mined, it is sorted into smaller pieces of gravel size and larger lumps and blocks, which are crushed to a coarse sand or gravel fraction. The ore is then carried to a stream for washing, or water is fetched from the surrounding streams. The ore is poured into baskets in small quantities and immersed in the stream, or water is poured over it and shaken repeatedly until the dirt which is sticking to the ore is washed away from it completely. Then a coarse mat is spread on the ground and the washed ore is poured on it and left there until it is bone-dry.

The following are the equipment used in preparatory work before the iron-stones can be collected: Cutlasses for clearing the bush; pickaxes for digging up the soil or digging out any roots that may hinder mining operations; heavy picks for excavation or for digging up the ground, hoes for gathering the earth and for removing the earth; baskets and small, light calabashes for removing the earth from the pit; ladders for descending into and for climbing out from inside the pit and lamps, to illuminate the underground tunnels and different kinds of food which miners consume during breaks and may eat from morning to evening.

### Iron smelting

Archaeological and ethno-archeological investigations in Modakeke have helped in the understanding of the early smelting in the community; these investigations were made possible by leaders of the town who had assisted their parents in the iron smelting. These people were persuaded to re-enact the technique for recording at the Natural History Museum, Obafemi Awolowo University, Ile-Ife, Nigeria.

#### *Ethnographic observations*

The re-enacted furnace was about 1.7 m high with constricted top which was provided with openings through which the ore is charged. The type of furnace used is a shaft furnace operated without any bellow. This furnace takes advantage of the buoyancy of hot air. The rising of this air results in a low pressure in the furnace, which in turn causes more air to be drawn in through the tuyere below, creating a natural draft with no needs to operate bellows.

Charcoal is used for smelting and it is produced from special hard wood which are distinguished by their poisonous characteristics. The most important of them is the sapwood tree (*Erythrophleum Guineense*) whose other name is *obo*. This produces hard charcoal with high combustible power.

To produce the charcoal the wood is cut into short logs and arranged in a trench about two feet deep. The branches from the trunks are sandwiched between these logs leaving small gaps between them. The space between the logs and the branches are filled with reeds and dry reed grass. The fire is then lit. Whilst burning is going on, fresh palm fronds are used to cover it, together with a layer of dry, dusty earth. Then water is mixed with dry earth until it is thoroughly wet, and this is then heaped on the dry earth to cover it.

After this, another complete layer of dry earth is added. The fire is left burning for complete three full days and nights before a section of it is uncovered to find out how well it has burned to form charcoal. In case of partial burning the remaining logs are set on fire again and covered with earth. All the charcoal which can be got from that hearth is collected for use for the smelting furnace.

The raw materials were transported for about one and a half kilometers from the mining sites to the smelting site. Much of the movement of these raw materials was done by women and children. The raw materials were charged into the 'tunnel mouth', i.e. the top of the furnace. There could be, for example, 6 baskets of gravel-size iron ore, 10 baskets of charcoal and 2 baskets of the crushed ore. Ilmenite-rich sand is also added to the ore. Exactly how much charcoal, iron ore and sand are needed to be added was governed by the skill and experience of the furnace tender who worked at the base of the furnace.

#### *Ritual and taboo*

Of course, the ancients did not understand the chemistry of burning or the physics involved in the incandescence of hot gas molecules. They were able to judge the temperature by the colour of the flames and the hot metal, but did not understand why one kind of sword needed to be thrown into a tank of water to harden it, and the other needed only to be waved around vigorously in the air. What they did learn to do was to discover elaborate rituals which maintained the procedures which controlled the carbon content of the smelted bloom, the temperature to which the finished work had to be reheated, and the rate at which it was cooled during the quenching process. Smelters and blacksmiths consider the strict observance of taboos as necessary to the successful outcome of a smelt. These taboos have been instituted long ago by the ancestral smelters to ensure success by keeping the whole process ritually pure and free from pollution or change. The first taboo is that a libation must be poured to appease *ogun*, the god of iron. The *ogun shrine* is always located at the entrance to the mining and smelting sites where the smelters worship before and after the daily operation. The offerings or sacrifices to the god of *Ogun* must be performed before mining for ironstones commences. This includes the following: palm-wine, which is poured on the mining site; life snails to be broken on the precise spot; and kola nuts, which are used to propitiate *Ogun* at this very place. These appeasements are necessary so that the miners may strike a good iron deposit and to guard against accidents.

There are also taboos that must be observed by iron miners and smelters. Among these are freedom of mind from any bitterness and must have no ill-will towards anybody; must not commit adultery or theft.

### Analytical procedure

Fourteen samples in all (11 slags, 2 ore, 1 furnace wall) have been subjected to microscopic and compositional analysis. The slag samples are here identified by the designation NG for Nigeria, followed by MO2, MO3 and MO4, and IP1 to IP5 for slags from the upper cultural layer (see above). IS1, IS2, IS2d and IS2h are from the middle cultural layer, and MO6 and MO7 are the two ore samples from the lowermost layer. MO1 is a sample from the furnace wall. It was decided to analyse all samples by optical microscopy and by bulk chemical analysis using standard XRF procedures, and to investigate a subset of representative samples using scanning electron microscopy with energy-dispersive analysis. All analyses were performed at the Wolf-

son Archaeological Science Laboratory, Institute of Archaeology, University College London. Cleaned and cut sections of the samples were crushed and ground to a fine powder in an agate mortar, oven-dried at 105 °C over night, and mixed with wax before being pressed into pellets. Bulk chemical analyses were performed by X-ray Fluorescence Spectrometry, using a Spectro Xlab 2000 and evaluating the measured values against certified reference materials for quantitative analysis.

### Results

The slags are predominantly composed of iron oxide and silica, in keeping with most bloomery slags analysed so far. Most remarkable, however, is the very high titania content of all of these slags, with six to eleven weight percent, except one which has just under one percent (Table 1, arranged in increasing order of titania content). The concentrations in alumina, lime, phosphorous oxide and manganese oxide are all within the typical range known from other bloomery sites. Several of the minor and trace elements, however, are present at remarkable concentrations. The zirconia content of most slags analysed here is in the range of between half a percent and one percent, as compared to typical values known from several European bloomery sites of below 400 ppm, and often below 100 ppm (e.g., Yalcin & Hauptmann 1995; Ganzelewski 2000). Similarly, there is a very high level of vanadium present, of around 2000 ppm V<sub>2</sub>O<sub>5</sub>, as compared to published values elsewhere of typically less than 200 ppm V. Strontium and the Rare Earth Elements are apparently also enriched.

#### *The slag IS 2*

Only one of the samples of slag from the middle layer of the excavation is very different from the others. While IS1 is almost indistinguishable from the other slag samples, IS2 has less than one percent titania, and the highest level of iron oxide of all slags analysed, with around 67 percent. Furthermore, the phosphorous level is much lower than with the other slags (0.3 wt% P<sub>2</sub>O<sub>5</sub> as compared to an average of 0.9 wt%), as are the levels of vanadium, manganese, zirconium, and lanthanum and cerium (the latter two not reported in the table). From this, it appears that a very different ore was used for the smelt which produced this particular slag. A subsequent screening of more slag samples from this particular layer, using XRF to analyse the cut surfaces of a further eight samples, indicated that all of them belonged to the titania-rich group, indicating that the sample IS2 is an outlier among the assemblage.

#### *Ore samples MO6 and 7*

These two samples were massive and reddish to brownish in colour, formed *in situ* from the weathering of the parental rock. One is a piece of the gravel-sized ore, while the other is a large lump which would have been crushed to gravel size before being fed into the furnace. According to microscope analysis, they both consist of an aggregation of goethite and limonite in a groundmass of decomposed anthophyllite and chlorite. The angularity of the quartz fragments indicates that the quartz was not transported geologically over a long distance, i.e. that the ore formed locally. In their chemical composition, with silica, alumina and phosphorous oxide, they resemble self fluxing ores. Most important, however, is their very high percentage of iron oxide, of up to 80 percent, and accordingly low amounts of slag-forming components. Significantly, they have only very little titania, of less than half a percent only, and low levels of zirconia and vanadia as well. Overall, the two samples are mineralogically and chemically similar enough to group them as one type of ore.



TABLE 1: XRF analyses of eleven Nigerian slag samples, two iron stone ore samples and one furnace wall fragment. Measured from pressed powder pellets. The slags are arranged in increasing order of titania content. Analyses by Xander Veldhuijzen using a Spectro Analytical Xlab 2000.

Number	Type	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	K <sub>2</sub> O	CaO	TiO <sub>2</sub>	V <sub>2</sub> O <sub>5</sub>	MnO	FeO	SrO	ZrO <sub>2</sub>	Sum
NG –	...	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	ppm	ppm	wt%
IS2	Slag	0.1	5.6	23	0.3	0.9	2.1	0.9	0.02	0.2	67	160	270	100.3
MO2	Slag	0.3	5.3	15	2.3	0.9	1.6	6.3	0.21	2.1	67	510	3380	102.2
IP1	Slag	0.3	5.2	21	0.7	0.3	0.6	6.5	0.15	0.5	62	330	6630	97.7
IS2d	Slag	0.5	7.2	27	0.9	0.3	1.4	6.6	0.22	0.5	55	520	5910	100.9
IS2h	Slag	0.4	5.8	24	0.8	0.4	1.5	7.1	0.29	0.6	59	270	6370	101.1
MO3	Slag	0.6	6.8	27	1.0	0.5	1.2	7.3	0.15	1.3	54	380	6760	100.5
IP2	Slag	0.3	5.9	19	1.3	0.6	2.7	8.2	0.16	0.5	61	780	7350	101.2
IP4	Slag	0.4	5.5	16	0.9	0.4	1.0	9.0	0.29	0.5	64	280	7230	99.0
IP5	Slag	0.4	6.0	26	0.5	0.4	1.1	9.0	0.61	0.5	53	220	6680	98.2
IS1	Slag	0.6	7.6	23	1.1	0.4	0.6	9.3	0.18	0.8	54	970	9080	98.3
MO4	Slag	0.5	6.6	21	0.8	0.5	1.6	11.1	0.10	0.5	54	130	11070	98.2
MO6	Ore	0.1	1.7	2	2.9	0.0	0.1	0.4	0.02	0.1	81	30	290	88.2
MO7	Ore	0.1	4.2	8	1.1	0.0	0.1	0.3	0.03	0.0	56	-	80	69.1
MO1	Wall	0.1	11.9	48	0.4	0.9	1.1	2.8	0.06	0.6	27	190	3080	93.9

#### Mineralogy of slag samples MO2, 3 and 4, and IP1-5

Optical metallographic studies show that the main phase present in the slags is light grey fayalite in a dark glassy matrix. The glass represents the reservoir for CaO, MgO, K<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub> and Al<sub>2</sub>O<sub>3</sub>. According to the SEM-EDX analyses, the fayalite is relatively pure Fe<sub>2</sub>SiO<sub>4</sub> with only limited substitution of manganese and calcium for iron. There is a characteristic skeletal and elongated structure of fayalite, indicating rapid cooling. This is in good agreement with their shape, indicating flow pattern typical for tap slags.

All samples show an abundance of an opaque cubic phase, often with skeletal growth pattern, identified by SEM-EDX as ulvite spinel (Fig. 2; Fe<sub>2</sub>TiO<sub>4</sub>). This mirrors the high titania content of these samples as identified by XRF analysis. Most notably, there is a marked absence of wuestite in the slags, indicating the high efficiency of the furnace. According to Tylecote *et al.* (1971) the content of wuestite in the bloomery slag provides significant clues towards the estimation of the metallurgical success of the ancient smelters. Slags with little or no wuestite indicate a more efficient process than those with higher proportions.

#### NG-IS1 and 2

NG-IS 1 and 2 are slag samples from the inner part of the furnace. One of them forms part of the titania-rich group, the other (IS2) does not (see above). Mineralogically, they consist of fayalite, glass, and spinel. The fayalite crystals are bulky and show evidence of slow cooling. While IS1 has the ulvite spinel typical of these slags, the titania-poor slag sample IS2 shows instead a much less reflective spinel (Fig. 3). According to SEM-EDX analysis, this is a solid solution between magnetite (Fe<sub>3</sub>O<sub>4</sub>) and hercynite (~FeAl<sub>2</sub>O<sub>4</sub>) with roughly equal numbers of iron and aluminium atoms in the core of the crystals, and up to three

times as many iron atoms in the outer rims. This marked zoning with an alumina-rich core and a more iron-rich rim is typical for hercynitic spinel in bloomery slags. In larger crystals, there is an epitaxial growth of ulvite around the spinel, indicating much increased titania concentrations in the residual melt when cooling and crystallisation had much progressed. Smaller hercynitic crystals are found as inclusions in fayalite, showing less evidence of coring and no epitaxial growth of ulvite. Still, there is the absence of wuestite in this sample which has been suggested as evidence for a high efficiency of the furnace.

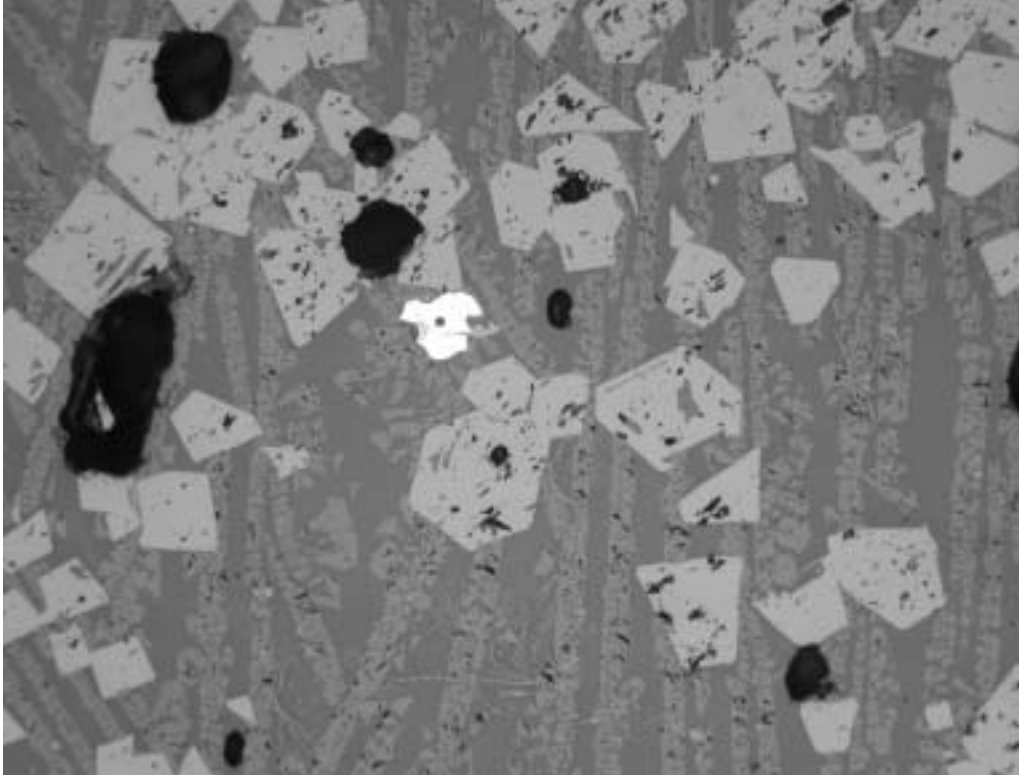
#### Furnace lining

Sample MO1 is from the furnace lining. The quartz which was originally rounded is now completely shattered as a result of intermittent heating and cooling and perhaps, the chemical attack by the melt.

Chemically, the furnace lining is remarkably rich in iron oxide (27 wt%) when viewed as a ceramic material. Similarly, its level of titania (just under three weight percent), zirconia and rare earths elements is much closer to the dominant slag group than to the limonitic ore samples. This probably reflects the composition of the black sand, which is reportedly washed from the soil during heavy rain falls; the local earth, being used for the building of the furnace, will contain a significant amount of such heavy minerals.

## Discussion

Apart from oral evidence, scattered excavations and ethnographic data, not much has been known previously about the technological process of iron smelting in south-west Nigeria. However, extensive work has been done on the archaeometallurgy



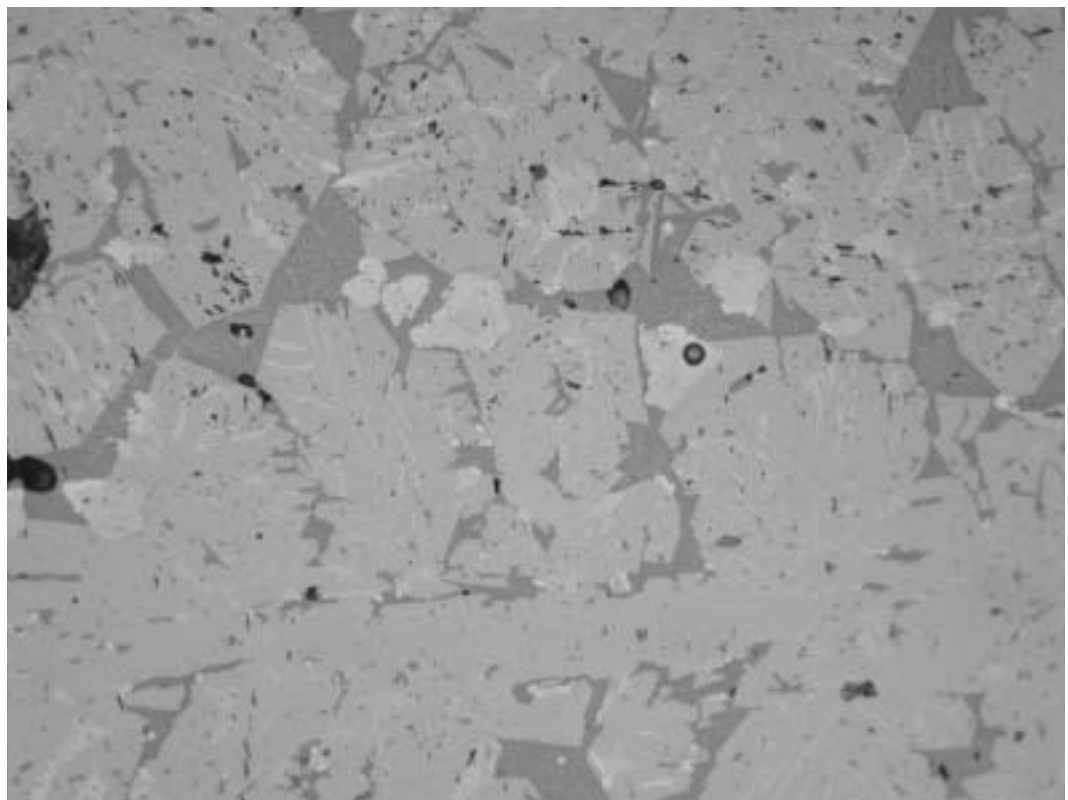
**Fig 2**  
Ulvite spinel crystals (light grey) and skeletal fayalite chains (medium grey) in a glassy matrix. Bright white is iron metal (centre), porosity is black. Sample NG MO4, reflected light micrograph, width of image ca. 1 mm.

data confirms the regular use of a blended ore for the smelting operation. The high titania content of around ten weight percent of most slags is most likely due to the addition of ilmenitic black sand to an otherwise goethite or limonite rich ore. The two ore samples of this latter type are very low in titania, and could not possibly have resulted in the production of the slag found. Ilmenite ( $\text{FeTiO}_3$ ), which has around 50 wt% titania, will have been the major source of the titania in the slag to account for the current composition. It is impossible to estimate from the current data the relative proportions of black sand and limonitic ore used in the smelting, because we have no reliable data for the average composition of the ore, or the black sand. The ratio of silica to alumina in the slag, however, is higher than in the two ore samples, indicating that at least a certain amount of the silica in the slag

was derived from the black sand, likely as quartz sand contamination. Similarly, the higher concentration of phosphorous oxide in the titania-rich slags as compared to the titania-poor sample IS2 indicates that phosphorous may have been introduced together with the black sand, but probably not as apatite since the calcium oxide levels are on average only slightly higher

of the Nsukka, about 250 km to the east (Okafor 1992), and at Taruga about 600 km northeast of the study area (Tylecote 1975). Our own work aims in the long run to contribute to filling the gap, to then enable comparative studies of the skills and methods used during Late Iron Age iron smelting.

The analytical study of the Modakeke slag and ore samples demonstrates a high level of skill of the ancient iron smelters, in that they left no free iron oxide (*ie* wuestite) in the slag. This shows that they exploited all the iron metal from the ore that was possibly extractable using the bloomery process, producing a rather lean slag with on average only 50 percent iron oxide. In addition, the chemical



**Fig 3**  
Hercynite crystals (centre, medium grey) and blocky fayalite (slightly darker) in a glassy matrix. Sample NG IS2, reflected light micrograph, width of image ca. 1 mm.

than the phosphorous oxide levels. More likely, the black sand contained a certain amount of monazite and similar minerals, since also the level of rare earths elements is much higher in these slags than in the ore, or the slag IS2. Alternatively, the phosphorous could have come as iron phosphate together with the limonitic ore, in which case the slag IS2 would derive from an altogether different ore. In all of these assumptions, no allowance has been made for any contribution of the furnace wall or the fuel ash, both of which may have contributed considerably to the slag forming (Crew 2000).

Overall, there are apparently several advantages from using a blended ore rather than just one or the other. Pure black sand would probably have been not rich enough in silica to produce a suitable slag in the presence of large amounts of titania. David *et al.* 1989 report about 15 wt% silicate minerals in magnetite sand smelted by the Mafa in northern Cameroon, but in this process, the single internal tuyere contributed critically to the slag formation. Unfortunately, no chemical analyses are available for the ore and slag from that process. The limonitic ore on the other hand could possibly have been smelted on its own, as is indicated by the slag sample IS2. However, the overall content of iron oxide in that one slag sample is much higher than in the others (67 wt% vs. an average of 58 wt%), suggesting that the mixture did smelt better than the pure ore. It also appears that the black sand was easily obtained, at least after heavy rain fall, and thus preferred over the more tediously mined limonitic iron nodules.

Furthermore, the iron oxide content of the various slag phases does not differ much; fayalite ( $\text{Fe}_2\text{SiO}_4$ ), the hercynitic spinel ( $\text{Fe}(\text{Fe}, \text{Al})_2\text{O}_4$ ) and ulvite ( $\text{Fe}_2\text{TiO}_4$ ) all have between 60 and 70 weight percent iron oxide in the EDX analyses, with ulvite being the lowest in terms of iron oxide content. Thus, allowing the formation of ulvite rather than fayalite through the introduction of ilmenite instead of quartz as a gangue mineral is beneficial for the efficiency of the process; each silicon atom of the silica ( $\text{SiO}_2$ ) will require two iron atoms to form fayalite ( $\text{Fe}_2\text{SiO}_4$ ), while each titanium atom in ilmenite ( $\text{FeTiO}_3$ ) needs only one more iron atom to form ulvite ( $\text{Fe}_2\text{TiO}_4$ ). In effect, the yield of iron metal increases with increasing formation of ulvite. This is reflected in the composition of the titania-rich slag which is rather lean in terms of bloomery slags. Furthermore, the absence of wuestite indicates that the iron produced in the smelting operation was probably steel and not soft iron.

## Conclusion

The iron smelting process at Modakeke in south western Nigeria was based on the smelting of a blended ore, mixed from limonitic ore nodules mined from the subsoil, and black sand gathered from the surface after heavy rain falls. The chemical composition and the mineralogy of the slag samples corroborates this ethnographic information. The yield of the process

must have been very high, with an ore grade of around 80 percent iron oxide and a relatively lean slag with less than 60 percent iron oxide. The efficiency of the smelting was accordingly very high, leaving not more iron oxide in the slag than absolutely necessary for slag formation. One may assume that the metal produced under these strongly reducing conditions was probably steel rather than soft iron, allowing for the manufacture of good quality implements and weapons. However, the current conclusions are based on a few initial samples only, and clearly require a much more in-depth study before more general and representative conclusions concerning the state of iron metallurgy in the Ife region can be drawn. Similarly, it is clearly necessary to obtain more archaeological information from the excavated site in order to assess the real age of the smelting site, which at present can only most broadly be placed in the Late Iron Age, probably prior to the colonial period.

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# New excavations in the Chalcolithic Mine T of Timna

## Preliminary report of the excavations March-May 2001

Alexandra Drenka<sup>1</sup>

### Introduction

The Timna Valley (previously Wadi Mene'iyeh) is located about 30 km north of the shores of the Gulf of Aqaba-Eilat. It is a large, semi-circular, erosional formation of some 70 km<sup>2</sup>. It is bounded by the Timna Cliffs, up to 470 m high, except at its east side, where it is open to the Arabah Rift Valley. The upper parts of the Timna Cliffs are limestones and dolostones of the Upper Cretaceous Judea Group, and the lower parts are sand-

stones of the Lower Cretaceous Kurnub Group (Segev *et al.* 1992). At the foot of the Timna Cliffs, the whitish sandstone of the 'Avrona - Amir' formation hosted most of the copper deposits which were mined in ancient times.

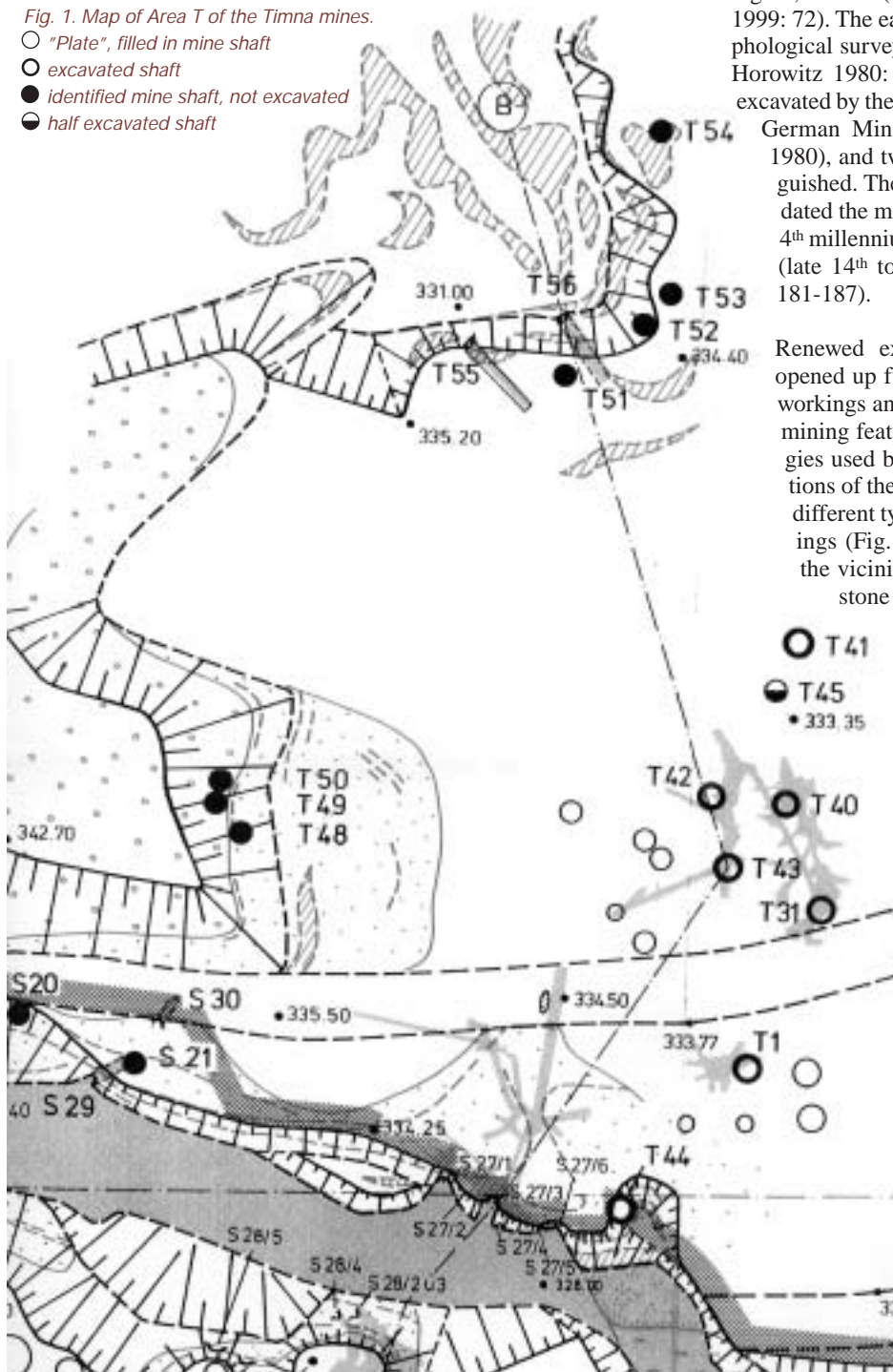
Mine T (Fig. 1) was discovered in 1976 by Beno Rothenberg's team during trial excavations of enigmatic mining relics ("plates"), located on one of the earliest terraces in the mining region, Area T (Conrad & Rothenberg 1980: 28; Rothenberg 1999: 72). The early terrace was identified during the geomorphological survey of the Timna mining region (Hauptmann & Horowitz 1980: 57-68). Mine T was subsequently partially excavated by the 'Arabah Expedition' in collaboration with the German Mining Museum Bochum (Conrad Rothenberg 1980), and two periods of ancient mining could be distinguished. The archaeological finds from these excavations dated the mining activities to the Chalcolithic Period (5<sup>th</sup>-4<sup>th</sup> millennium BC) as well as the Egyptian New Kingdom (late 14<sup>th</sup> to mid-12<sup>th</sup> centuries BC) (Rothenberg 1980: 181-187).

Renewed excavations in Mine T (March-May 2001) opened up further extensions of the ancient underground workings and contributed to a better understanding of the mining features as well as the different mining technologies used by Chalcolithic miners and by mining expeditions of the Egyptian New Kingdom. From tool marks of different types, preserved on the walls of the mine workings (Fig. 2) and from broken stone tools scattered in the vicinity of the mines - in addition to the complete stone tools and metal chisels found in the previous excavations (Figs. 3 and 4) (Conrad *et al.* 1980: Abb. 73) - we knew about the different methods employed for copper ore mining during periods separated by thousands of years. However, it was mainly from our own personal underground experience and observations that we learned about solutions for many of the technical problems which the Chalcolithic miners of the area must have faced. Extremely difficult working conditions forced us to solve many of these problems in a way that we believe our predecessors did, using not only common sense, but all their senses.

The excavations in 2001 were on behalf of the Institute for Archaeo-Metallurgical Studies (IAMS), Institute of Archaeology, University College London, directed by Beno Rothenberg, in collaboration with C.T. Shaw, Royal School of Mines, Imperial College London, with Alexandra Drenka acting as chief field supervisor of the excavation.<sup>2</sup>

Fig. 1. Map of Area T of the Timna mines.

- "Plate", filled in mine shaft
- excavated shaft
- identified mine shaft, not excavated
- ◐ half excavated shaft



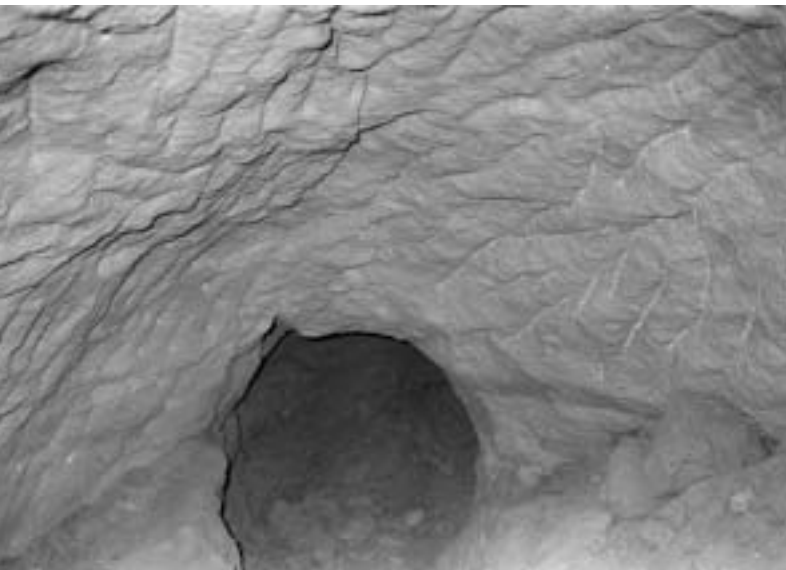


Fig. 2. Tools-marks in Mine T: left - Chalcolithic hammer-marks; at the right - New Kingdom chisel-marks.

**The excavations in Mines T and T1<sup>3</sup>**

Six weeks of digging and the removal of tons of sand deposits from underground workings revealed new galleries and chambers, eight new shafts (T1000 - T1007), a large connecting tunnel between Shaft T42 and newly-discovered Shaft T1002, and the most impressive part of Mine T1: the ‘Green Chamber’ (marked in Fig. 5 as Room B).

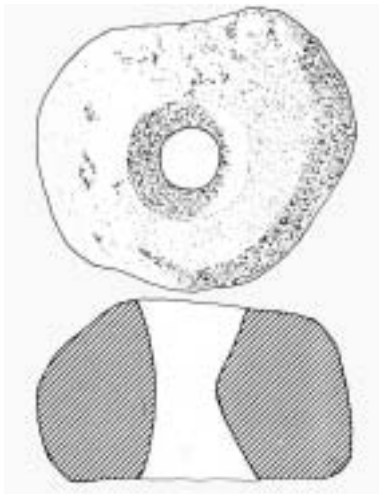


Fig. 3. Chalcolithic mining hammer with typical bi-conic shafthole.

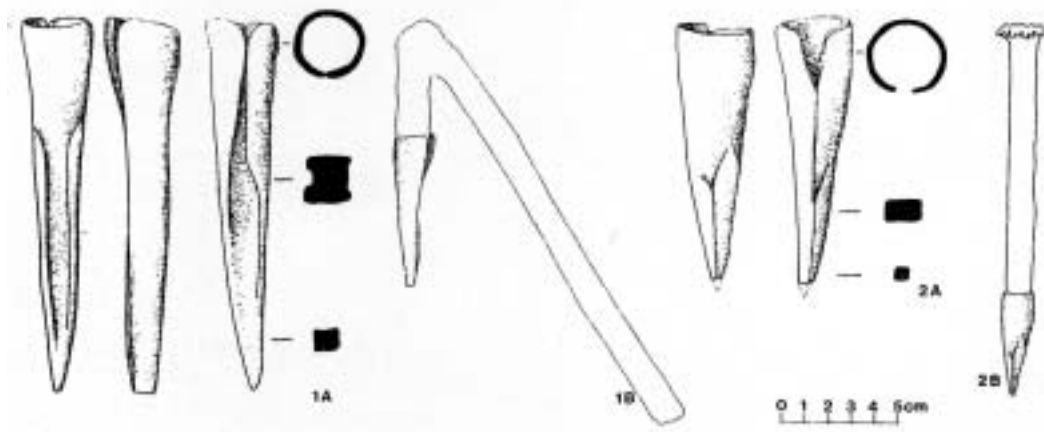


Fig. 4. Two metal mining tools found in Timna, with proposed reconstruction of complete mining tools.

The starting point of our excavation was an opening in the north wall of Shaft T40. This was the point where the team of the German Mining Museum Bochum in 1976 stopped their excavations in this area. Continuing north, we cleared another 16.70 m, but we did not reach the end of these workings. However, based on our very detailed investigations, though in a limited section of the mine, we are reasonably confident that we recognized and established the characteristics and features of the

Mine T  
survey: A. Frumkin, G. Danon, R. Porat, A. Rekin  
ICRC 2002

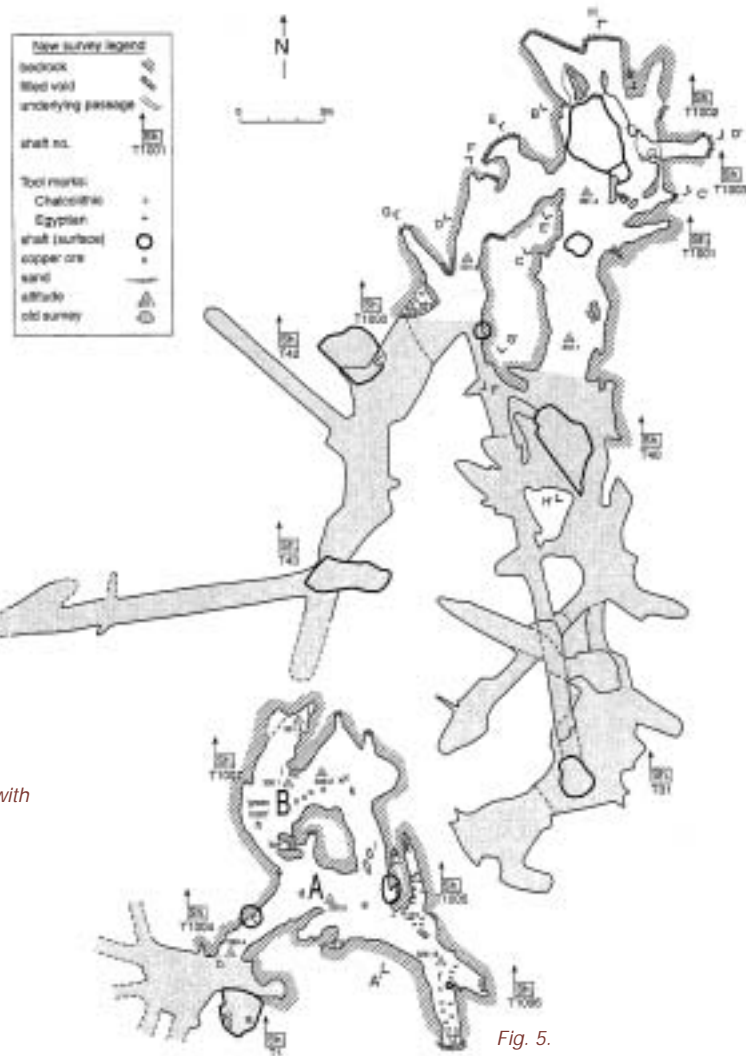


Fig. 5. Plan of Mine T and T 1 after excavation in 2001.



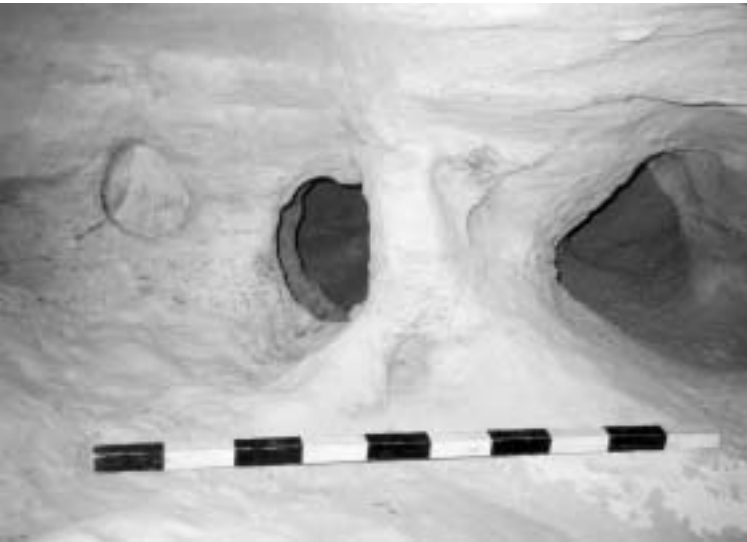


Fig. 6. Supporting pillar. The chisel marks are of the New Kingdom.

mining technology used by the earliest underground miners at Timna. It is, therefore, significant to describe in detail the progress of our work, with the emphasis on the working conditions which our team experienced, especially during the first days of digging, since here and then we learned our first lessons of mining. This brought us close to the ancient miners we actually kept following in our excavation.

The north gallery (1.70 m wide, measured from the floor, its height 1.72 m) was roughly cut into the soft whitish sandstone formation and was found completely filled with wind-carried yellowish sand mixed with small pebbles. At a distance of about one meter from where we started to excavate, the channel widens slightly on its right side, forming a niche with numerous small cavities. The latter are the result of the miners simply following the copper vein - the copper ore nodules were mostly found in narrow veins of about 5-15 cm - and extracting the ore. At this spot, the ancient miners had decided to leave part of the rock intact and mined all around it, leaving it as a supporting pillar (Fig. 6). On the surface of this pillar are numerous long deep chisel-marks associated with Egyptian New Kingdom mining at Timna (Rothenberg 1980: 182). Such Egyptian presence is attested by their typical toolmarks in many places of the Chalcolithic Mine T. Their intrusive character is usually also recognizable in expended niches or in places very rich in ore; in many of them it is still possible to follow the veins of copper mineralisation. Supporting pillars are also present at other locations in the mine, presumably wherever further work could endanger the safety of the mine. These pillars evidently played a very important role in early mining, providing stability to the underground galleries and safety for the miners.

With further progress in removing the fill deposit in the gallery, our working conditions kept deteriorating and our enthusiasm started to slow down. There was very limited space for the person clearing the fill and with each additional bucket conditions were aggravated. When Shaft T40 was 5 meters behind us, these difficulties had intensified by lack of air and light. This was the moment to think about the people who were here a few thousand years ago. How did they overcome these very severe limitations? How did they keep digging and what was the power - or knowledge - which helped them to overcome human basic needs? Though they were less spoiled than we are and pro-

bably far better-trained in this kind of work, we soon recognised evidence that the ancient miners had the same difficulties just above us. On the ceiling of the tunnel we noticed an irregular circular feature filled with sand and small pebbles, and with no doubt that it was just what we needed - a filled-in ancient shaft - we started to clear it. However, the fill deposit was very compact and very hard to remove. Falling sand and the lack of air and light made such work almost impossible. We therefore decided to try and open the shaft from the surface above.

The first few attempts to locate the shaft on the surface were unsuccessful, as it was not sufficient to determine underground the distance from the entrance through the gallery and its orientation. At this point, we found it difficult to understand why the compass and ruler could not solve our problem. Obviously the approximate position of the shaft could not help us much. Finally, the only tools left were our senses. With ears to the ground, we listened while knocking from below, and with our hands slowly moving on the surface, we tried to feel the point of the strongest vibration. We were amazed by the result. Once we had determined the point, we started digging. With confidence and a large pick, we cut through the thin conglomerate cover down to bedrock and soon the outline of a shaft (Shaft T1001) became clearly visible (Fig. 7). A short while afterwards, we could see the darkness of our gallery below.

During this season of our excavation, we opened five more shafts using the same method (Shafts T1000 - T1005). It was notable that the ancient miners had often used natural fractures



Fig. 7. Shaft T1001 - typical for the irregular Chalcolithic ventilation shafts of Mine T.



in the rock as the most convenient location to sink a shaft, and moreover that the shaft was cut on one side of the fault whilst the surface of the natural fault formed the other side of the shaft so that the rock there was left almost intact. As a result of leaving untouched one side, and following the face of the sloping fault, this type of shaft is not precisely vertical; this was the reason why we could not locate the shaft on the surface using only our underground measurements and orientation.

We very carefully cleaned the shaft walls in order to preserve possible tool-marks, which are of decisive significance for the understanding of the mining technology and for the dating of the mine workings. Indeed, we found numerous round and shallow hammer-marks on the inner walls of Shafts T1004 and T1005, indicating the type of the tools used for sinking these shafts (*cf.* Fig. 3). However, the bottom of Shaft T1005, as well as its immediate vicinity, is densely covered with long and deep chisel-marks (*cf.* Fig. 4). Evidently we witness here the overlapping of two different mining technologies: the earlier, using hafted stone hammers, practiced by the Chalcolithic miners of the 5<sup>th</sup> to 4<sup>th</sup> millennium BC, and the later, using metal chisel and hammer ('mallet and gad') typical for the Egyptian New Kingdom intrusive workings of the 14<sup>th</sup>-12<sup>th</sup> centuries BC.

Most of the newly discovered shafts, except Shafts T1002 and T1003, are characterised by the same general features: an irregular oval shape with a diameter of 0.65 m to 0.85 m and a depth of 0.80 m to 2.50 m. The distance between the shafts varies from 1.80 m to 5 m. We assume that all these shafts were used for ventilation and light, and some probably also for the transport of copper ore. These ventilation shafts are quite different from the shafts used for the movement of the miners, which often show rough steps, as for instance Shaft T 31 (Fig. 8).



Fig. 8. Shaft T32 - typical for shafts used for the movement of miners and ores.

Shafts T1002 and T1003 are somewhat different (Fig. 9). Shaft T1002 was found at a distance of 3.30 m from Shaft T1001. It is a large irregular cavity, roughly hammered out, with dimensions of 3.40 m by 2.40 m (measurements taken from the surface) and depth of 2.00 m to 2.30 m (measured from the surface to the bottom of the mine). This shaft was in fact finally reopened from below, since the surface was very close - only 0.25 m. When Shaft T1002 was partly cleared, we noticed on its southeastern corner a feature which, after complete clearing, turned out to be another attached shaft, T1003. This shaft is very carefully cut, oval in shape, its diameter 0.80 m and its depth 0.85 to 1 m. Chisel-marks are visible all around its walls. After completely clearing these two shafts and exposing their outlines and toolmarks, it was possible to conclude that Shaft T1002 was opened and used by Chalcolithic miners as an opening to their mining 'galleries', probably also for ventilation and light, and perhaps also for transport of copper ore. Shaft T1003, of later date, was an Egyptian New Kingdom prospecting shaft, which led into the earlier underground workings, and these the Egyptians simply expanded in search of so far undetected copper ore veins. Further clear evidence of the overlapping of these two completely different mining techniques are the toolmarks in the large niche just below Shaft T1003 and at several places in the gallery between Shafts T40 and T1002, newly cleared by our team. Perhaps the best-preserved wall showing both stone-hammer marks and chiselmarks is located in a large niche, approximately 0.80 m north of Shaft T1001. Very intensive mining activities left here numerous small- to medium-sized cavities in the rock. A large portion of the ceiling is covered with roundish, shallow hammermarks, and just below these are deep



Fig. 9. Shaft T1002, irregular Chalcolithic shaft, and shaft T1003, regular, oval shaft of the New Kingdom.



*Fig. 10. Copper ore nodules in the ceiling of Mine T1 (in the Green Chamber).*

and long chiselmarks. Evidently this part of the mine workings also saw intensive activities during both the Chalcolithic period and the Egyptian New Kingdom.

The most impressive discovery of our excavation is the 'Green Chamber' in Mine T1. Here we found large lumps of green copper ore sticking out of the white sandstone, mainly on the ceiling of the 'Green Chamber' (Fig. 10). We could only wonder why the ancient miners left behind what they were actually looking for. We must assume that this treasure of copper ore was not exposed in ancient times and was hidden by a layer of sandstone which has eroded with time. If this is the case, we are left with a further question: why has this process not happened in other places of Mine T, or in the other mine workings previously explored by the 'Arabah Expedition'?

The 'Green Chamber' (Room B on Fig. 4) is 3.45 m long, 2.50 m wide and, at the highest point, 1.30 m high. It is important to emphasize that this is the largest mined-out space we have seen in Mine T. Its floor slopes northward, and we observed two openings, most probably the entries into further mining galleries. We also observed a possible shaft (T1007), again against a rock fracture, which we decided not to open at this stage.

The only access to the 'Green Chamber' is from Room A, where we exposed all known features associated with the early mining of Timna: a supporting pillar, numerous irregular cavities of different size, a shaft for ventilation and ore transport (Shaft T1005) (the latter densely covered with the typical early

hammermarks), and on the wall just below it, typical chiselmarks of the New Kingdom. More evidence for Egyptian workings was found under Shaft T1005, and on its right side a beautifully carved, very narrow channel, approximately 0.25 m in diameter and at least 2 m long. There are chiselmarks all around its walls, cut along green mineralised copper veins. Further removal of the sand deposit in Room A revealed a transverse tunnel with ceiling and sidewalls covered with chiselmarks. Nowhere in the workings of Mine T did we feel as strong an Egyptian presence as in this room. Although we noticed in the ceiling a small roundish opening next to a crack in the rock, marked on our plan of the mine as Shaft T1006, we were unable to open it. The heavy underground air forced us to discontinue our work here, but we hope to complete our investigation of these workings in the near future.

In light of the excavations in Mine T and additional surveys in 2001-2002, it seems evident that underground mining in the Chalcolithic period already existed on a fairly large scale, and that it extended over quite a large area around Mine T. North of the excavated Mine T, the workings go on, as shown by the gallery openings T55 and T56 bearing early hammermarks, with several shafts nearby. There is also further evidence for Chalcolithic mining in Area S (Conrad & Rothenberg 1980: 103-150) on both sides of the wadi south of Mine T, again mostly reworked by Egyptian New Kingdom miners. As already established by the excavations in the 1970s (Rothenberg 1980: 170-176), Mine T and the other early mine workings in Timna bearing the typical hammermarks, could be dated by archaeo-

logical evidence, pottery, and flints, to the 5<sup>th</sup> to 4<sup>th</sup> millennium BC. These are the earliest copper mines so far discovered in the Levant. Networks of mining galleries and chambers with shafts, the latter being used as mine entrances, for ventilation and light, and for ore transport, represent the very beginnings of underground mining technology, which in later times gradually developed to sophisticated shaft and gallery mining. This is still the principle of mining today.

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## Notes

1. At the suggestion of Beno Rothenberg, the following preliminary report was written by Alexandra Drenka, in charge of the field work, as a personal report for the director about the excavation of the mine workings, with emphasis on the personal experience of the excavators in the difficult underground workings, clearly reflecting on the problems the ancient miners must have experienced. Once written, it was decided to publish this unique documentation as the supervisor's preliminary report. A comprehensive report of the excavations in Area T will be published separately (B.R.)
2. We are grateful to Asaf Holzer, the local archaeologist, for his important assistance in running this excavation.
3. In 1976, Mine T1 was excavated as a separate unit (starting with shaft T1), though it was assumed that it was actually a part of adjacent Mine T. For reasons explained in the following, we did not yet succeed to connect these two units.



# Carinated and knobbed copper vessels from the Narhan Culture, India

Ashok Kumar Singh & Pranab K. Chattopadhyay

## Abstract

*Agiabir is a new site in eastern Uttar Pradesh of India in the Narhan series of cultures, excavated by Banaras Hindu University. A cache of ten large copper objects, two iron swords and an iron lamp stand were discovered in the excavation of 2000-2001. Archaeologically these objects are dated to be of circa 4<sup>th</sup> to 5<sup>th</sup> century BC. Two of these copper objects from Agiabir and one from Narhan have been analysed. All these have been obtained from Period III of the Narhan Culture, associated with NBP Ware.*

## Introduction

In 1984, discoveries at the small village of Narhan (26°19' N, 83°24' E), situated at the left bank of the river Ghaghara, in the Gola tehsil of district Gorakhpur, Uttar Pradesh, led to the identification of a new culture in the archaeological map of India (Singh 1994) (Fig. 1). During the subsequent two decades



Fig. 1. Map of Eastern India, showing Chalcolithic and NBP sites.

almost two dozen ancient settlements of this culture were discovered in the surrounding districts. Amongst these, the most recent one is from Agiabir (25°13' 52" N, 82°38' 41" E). The site is located in district Mirzapur, Uttar Pradesh on the left bank of the sacred river Ganga. The site was discovered in 1998-99 by the first author (Singh 1999), and the Banaras Hindu University conducted three sessions of excavations (Singh and Singh 1999-2000, 2001 and 2002).

The Chalcolithic culture of the Middle Ganga Valley is characterised by the use of copper and lithic artefacts associated with Black-and-Red Ware. The pots were fired in an inverted position, so that after firing the interior and a portion of the top exterior turn black under reducing condition, whereas the remainder of the exterior turns red, being exposed to the oxidizing furnace atmosphere. The Narhan culture is basically a pre-iron Phase Chalcolithic culture with the principal ceramic assemblages of white painted Black-and-Red Ware. The Period I of the Narhan culture is similar to period IIB of the Ferro-chalcolithic culture of Pandurajar Dhibi of West Bengal (De & Chattopadhyay 1989). The Chalcolithic culture of both these sites is significant due to the presence of iron. Period II is Pre-NBP with presence of iron. Period III is associated with NBP Ware.

In Period III of Narhan culture, the major pottery assemblage is the Northern Black Polished (NBP) Ware. NBP is made of well-levigated clay on a fast wheel. Thickness of the ware is generally uniform, and it was well-fired. Typologically, NBP can be classified into several categories, such as bowls with straight sides, bowls with convex sides, handis with sharp carination, and dishes with closing featureless rims. NBP is a deluxe pottery, and it is represented by many shades, including golden, pink, silvery, and steel blue. Red Ware is also known in this period and in some of those assemblages, the presence of closing featureless rims with rounded base, known as carinated handis (cooking pots), were noted. This shape in pottery is further adopted in large copper vessels.

The copper objects recovered from these cultural sites were studied to reveal the alloying pattern, manufacturing techniques, and their correlations with other sites. The copper objects from Narhan, Period I, includes one ring and one fishhook, Period II included two hairpins or so-called antimony rods, a nail-parer, four bangles, one fishhook, and indeterminate copper objects. Period III, on the other hand, originated one carinated copper vessel and a bead. Analytical studies have been made of some of these objects elsewhere (Singh, Merkel & Singh 1996-97).

The copper objects from Period I's first excavation season at Agiabir comprised a single item, namely a fishhook. Period II was free from copper, though about thirty iron objects were recorded. In the first season of excavation, ten specimens of copper, including wire, hairpins, bangles and a few objects of indeterminate use were recovered from Period III (NBP). The excavation of 2000-2001 has obtained a cache of copper and iron objects in a room, buried in layer (12), on the top of layer (13) from trench YE-6 III, at a depth of 6.60 m below datum (Singh & Singh 2001). These objects were recovered from Period III and can be chronologically dated to the middle phase of NBP (Fig. 2).



Fig. 2. Cache of Period III copper and iron objects in situ excavated at Agiabir, including carinated bowls, a mirror, knobbed vessels, copper objects and an iron lamp stand.

The cache comprised ten large copper objects. These include two cooking vessels (*handa*), a globular vessel, two bowls, two carinated handis, a copper knobbed vessel and a mirror. All these objects are complete and kept upside down, and found in a highly corroded state with thick bluish-green corrosion. An analytical study of two carinated handis of Agiabir and Narhan

(Sp. Nos. 1 & 2) (Fig. 3) and the knobbed vessel from Agiabir (Sp. No. 3) (Fig. 4) are presented here. A thin core of about 0.8 mm uncorroded metal was detected in the first two specimens. A metallic core was also detected in the third one, but it was rather thin. The dimensions and measurements of the vessels are recorded in Table 1.

TABLE 1

Sp. No.	Site	Diameter of the mouth (cm)	Diameter of the body (cm)	Thickness of the rim/ body (cm)	Height (cm)
1.	Agiabir	19.0	30.8	0.28	9.1
2.	Narhan	23.5	37.0	0.30	12.9
3.	Agiabir	27.6	33.6	0.57	3.2

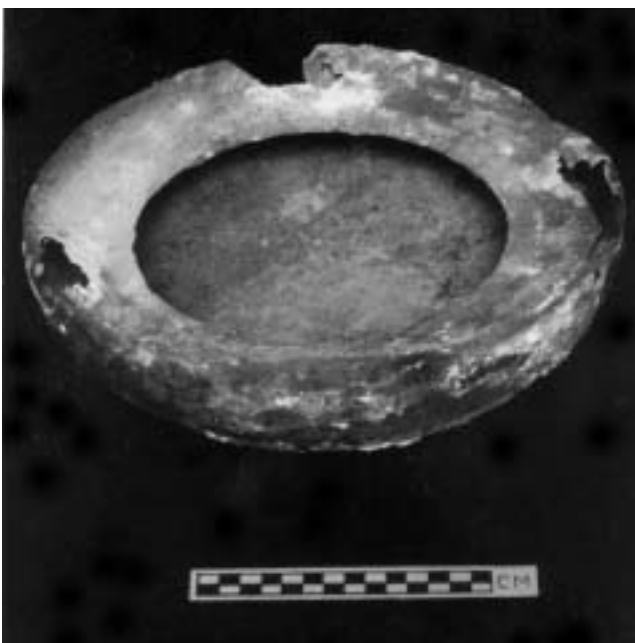


Fig. 3. Carinated handi from Agiabir, Period III (NBP), circa 4<sup>th</sup> to 5<sup>th</sup> century BC.

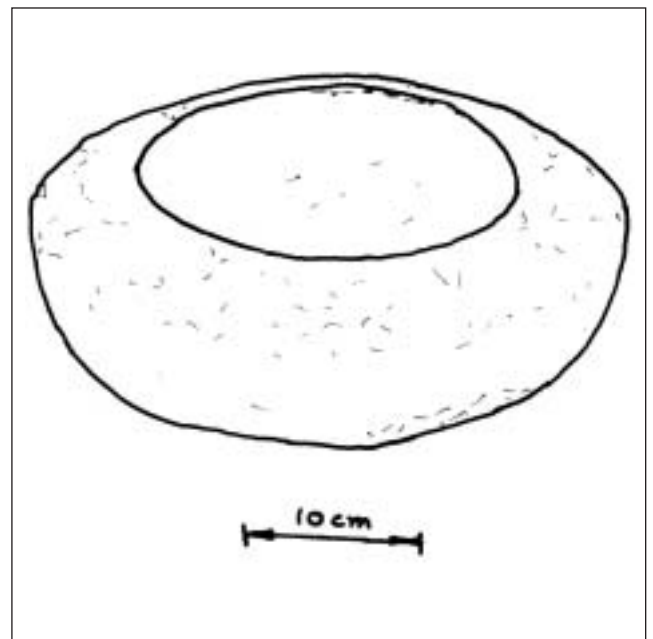


Fig. 4. Sketch of carinated handi from Narhan, Period III.

### Analytical procedures

Initially the surfaces of the three specimens were inspected through a microscope. The carinated bowl from Agiabir (Sp. No. 1) was clean at its internal surface but was found to have soot marks at its outer surface as well as a coating of clay and ash. One may observe in villages still today the use of clay at the outer surface of the cooking pots. This clay layer protects the vessel from the soot produced in the fire of woods. The application of this protective clay layer indicates the use of the same tradition over thousands of years. The outer surfaces of all these three objects were found fully converted into malachite [ $\text{CuCO}_3 \cdot \text{Cu}(\text{OH})_2$ ] and other complex corrosion products, a common feature on copper-based objects (Chase 1979).

The chemical analyses of the three specimens are made to identify the constituents and alloying, if any, with the help of an Atomic Absorption Spectrophotometer, Perkin Elmer 238.

### Analysis of objects

The first analyses of the copper objects from Narhan had thrown new light on the inception of copper technology in this site (Singh, Merkel & Singh 1996-97). Analyses of a fishhook and

a bangle from Period I and a bead of Period III clearly indicated that there is no evidence of using pure copper for manufacturing artefacts, whereas use of alloy were recorded as low tin bronzes with the addition of tin from 3.81 to 9.86 wt%. In the contemporary copper bronzes of Bihar and West Bengal similar observations were recorded by Emission Spectrograph analysis, where evidences of tin were found around 10 wt% (Chattopadhyay 1992). The results are shown in Table 2.

TABLE 2

No	Site	Cu	Sn	Ag	Pb	Fe	Zn
1.	Agiabir	98.50	<0.1	0.02	<0.1	<0.1	<0.1
2.	Narhan	99.02	<0.1	0.005	<0.1	<0.1	<0.1
3.	Agiabir	35.83	17.98	0.02	<0.1	<0.1	<0.1

The specimens Numbers 1 and 2 from the above table clearly indicated that there is no evidence of alloying in the two carinated vessels from Agiabir and Narhan. The specimen Number 3, the knobbed vessel of Agiabir, was specifically searched more



Fig. 5.  
Knobbed  
vessel from  
Agiabir,  
Period III.

precisely for copper, tin, lead and zinc. The oxidized contents, *i.e.* the presence of non-metallic constituents, discouraged the analysis to 100 wt%. The ratio of metallic constituents of the object clearly identifies it as high tin bronze. Chase (1979) provides an explanation for the increase in tin as due to selective copper dissolution from the corrosion products. The silver content in these three specimens indicates the presence of it in copper ores.

### Metallography

(Sp. No. 1): A sample was taken from the copper vessel of Agiabir and mounted on ebonite. The sample was polished and

observed through a metallurgical microscope. The specimen was found to have a pitted surface, due to corrosion. Subsequently it was etched with ferric chloride and ammonium hydroxide solutions. Fine twins were revealed in the microstructure. A few intergranular grey crystals have been observed, which may be Cu-Cu<sub>2</sub>O or PbS inclusions.

(Sp. No. 2): Similar observations were made with a specimen from the copper vessel of Narhan. In this specimen corrosion pits were observed in the polished mount but in lesser amounts than the previous sample. However, after subsequent polishing it was almost minimized. After etching, the microstructure revealed twins.

(Sp. No. 3): A fragment of the knobbed vessel with a thickness of 2 mm was polished across its cross-section, and solid core was revealed, with a heavily-corroded outside surface. Subsequently it was etched with ferric chloride and ammonium hydroxide and was observed through a metallographic microscope to reveal its manufacturing technique. The microstructure was quite different from specimens 1 and 2. There was no evidence of dendritic cast structure or twins. Thus the use of casting alone or annealing after mechanical working could not be established.

### SEM-EDX Analysis

For more precise and in-depth analysis, detailed identification of inclusions, matrix, and the remaining non-corroded core, all three specimens were further scanned and checked with SEM-EDX, with Leica S440 scanning electron microscope at Pal. Div. II of Geological Survey of India. In addition, the distribution of tin in the knobbed vessel of Agiabir was analysed. The observations clearly indicated its oxidized state. The semi-quantitative average values of the specimens (in atomic percent) are shown in Table 3. Two different types of inclusions have been observed during scanning in the carinated vessels.

### Observations

(Sp. No. 1 and 2): The above studies clearly indicate that the inclusions noticed in the two carinated objects from the two sites are of two types. Bright inclusions in the structure are lead sulphide, perhaps indicating contamination of galena with the cop-

TABLE 3

No	Site/ Item	Cu	Sn	Pb	Fe	S	O	Sb
1.	ABR Carnt Vessel Matrix	95.16	0.21	-	0.59	-	4.04	-
2.	ABR Carnt Vessel Inclusion-bright	32.10	-	57.04	-	10.85	-	-
3.	NRN Carnt Vessel Matrix	99.62	-	0.03	0.35	-	-	-
4.	NRN Carnt Vessel Matrix	99.37	0.30	-	0.29	-	-	-
5.	NRN Carnt Vessel Inclusion -bright	46.34	0.15	15.37	4.92	33.21	-	0.01
6.	ABR Knobbed Vessel matrix	16.76	18.63	-	0.82	-	60.70	1.48
7.	ABR Knobbed Vessel matrix	5.62	20.21	-	0.89	-	71.42	-
8.	ABR Knobbed Vessel matrix	7.48	19.57	-	0.83	-	70.70	-



per ore. The grey inclusions are similar to chalcopyrite, as indicated by the presence of sulfur and copper in them (Figs. 6, 7 & 8). It is quite probable that sulphide ores, i.e. chalcopyrite, were used to smelt the copper.

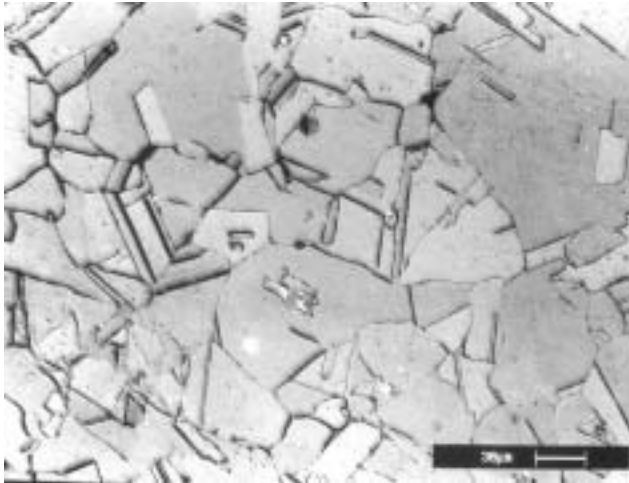


Fig 6. Microstructure of carinated copper handi of Agiabir, showing twinned copper grains, bright lead and grey chalcopyrite inclusions. Scale bar 30  $\mu\text{m}$  SEM-EDX. Etched; ferric chloride and ammonium hydroxide.

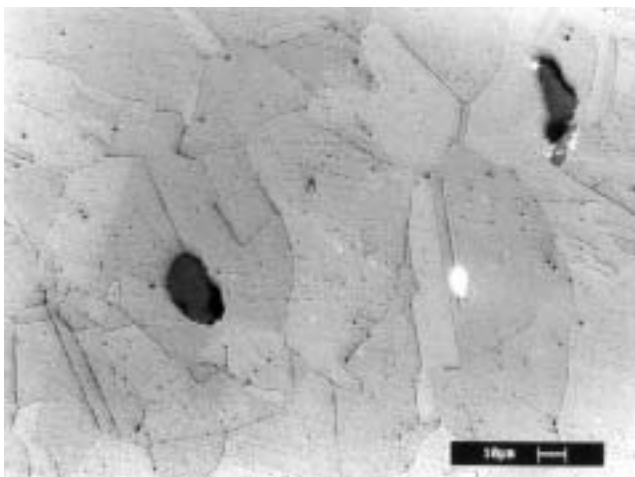


Fig. 7. Microstructure of carinated copper handi from Agiabir, showing twinned bright lead and grey chalcopyrite inclusions. Scale bar 10  $\mu\text{m}$  SEM-EDX. Etched; ferric chloride and ammonium hydroxide.

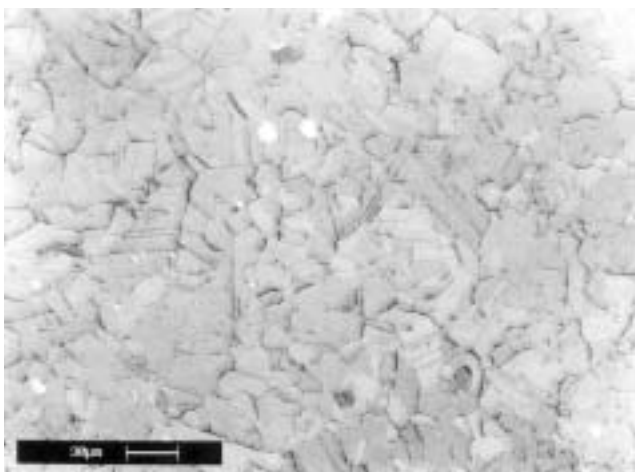


Fig. 8. Microstructure of carinated copper handi from Narhan, showing twinned copper grains, bright lead and grey chalcopyrite inclusions. Scale bar 30  $\mu\text{m}$  SEM-EDX. Etched; ferric chloride and ammonium hydroxide.

**(Sp. No. 3):** The knobbed vessel, though highly corroded, exhibits unaltered microstructures. The vessel consists of around 19.5 percent tin, and definitely has been accepted as high-tin bronze. The microstructure as revealed through SEM-EDX indicates that quenching was performed. The structure (Figs. 9 & 10) clearly indicates that quenching was closest to pure  $\beta$  phase. In one region of its structure martensite was found with random dislocations and stacking faults.

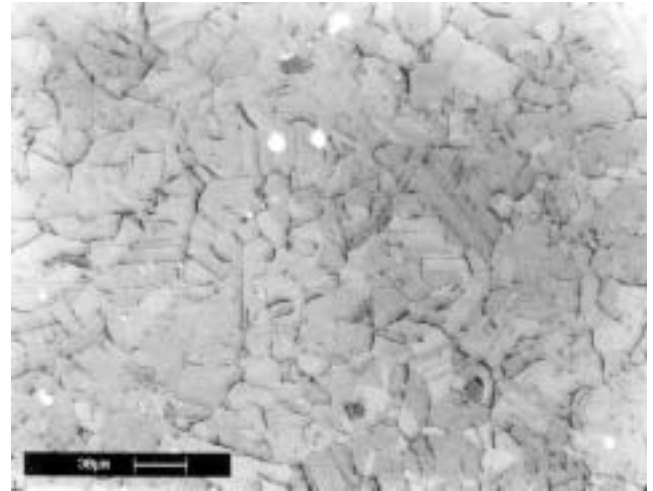


Fig. 9. Microstructure of knobbed vessel from Agiabir. Martensite indicates that quenching was closest to pure  $\beta$  phase. Scale bar 30  $\mu\text{m}$  SEM-EDX. Etched; ferric chloride and ammonium hydroxide.

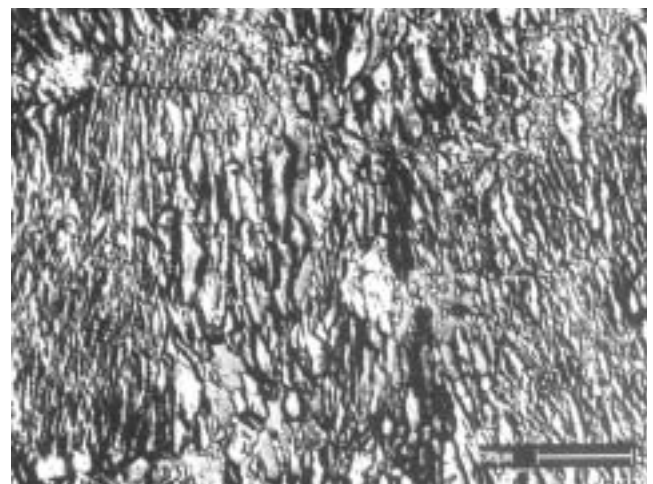


Fig. 10. Microstructure of knobbed vessel from Agiabir. Martensite in another region shows random dislocations and stacking faults. Scale bar 20  $\mu\text{m}$  SEM-EDX. Etched; ferric chloride and ammonium hydroxide.

The specimen, analysed in the present context, has a different composition and structure. To reveal the manufacturing of the knobbed vessel the traditional bronze making techniques were studied through ethnoarchaeological context. The basic practices in Kerala and also in West Bengal are similar in nature (Srinivasan 1998; Srinivasan & Glover 1998; Chattopadhyay 2002b). Tin is added to liquid copper in a crucible to give the desired ratio. After melting the liquid is poured into sand moulds and an ingot is made. After reheating it was hammered into a vessel by alternate heating and forging. There is every possibility of traditional continuity in the present day practices of high tin bronze making.

Finishing processes, perhaps, made by hammering with wo-oden mallets and subsequently heating and quenching was carried out. The non-existence of as-cast dendrites and twins on the one hand, and on the other hand the internal structure of martensite, clearly indicates the quenching after hot working.

## Conclusions

The presence of high tin bronzes has not yet been detected in a Chalcolithic context in eastern India. It may be tentatively concluded that the use of high tin bronze began in the early historic period. The mirror from Chandraketurgarh highlights the stages of the metal craft of the early historic period in Eastern India (Chattopadhyay 2002a). The copper and bronze objects of Agiabir, on the other hand, highlight the stage of copper and bronze metallurgy during the 5<sup>th</sup> to 6<sup>th</sup> century BC. Detailed studies of high tin bronze vessels and mirrors have been made elsewhere (Srinivasan 1998; Srinivasan & Glover 1998).

The shape and microstructures of the two vessels revealed that they were manufactured by forging, i.e. cold working from the original cast. The grains break into smaller sizes and after reheating, i.e. annealing, twinned grains are formed. In manufacturing vessels of this shape the methods of sinking and raising are applied. In sinking, the vessels are hammered from the internal surface by placing them over a wooden groove, whereas raising is a process of working by placing them over a dome headed stake and hammering from the outside surface. In most cases, both sinking and raising are applied simultaneously. Based on the identical shape, composition, similar cultural context and contemporaneity of the two carinated vessels, the present authors presume that both were manufactured at the same location. The analysis of two carinated vessels also establishes the use of copper, in purer form without any alloying, also a new finding in Eastern India during the 5<sup>th</sup> to 4<sup>th</sup> century BC.

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# MSc in the Technology and Analysis of Archaeological Materials

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