The Archaeometallurgical Analysis of Copper-base Artifacts from Prehistoric Nil Kham Haeng, Central Thailand:
A Preliminary Report

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Introduction
Excavations by the joint Thai Fine Arts Dept. - University of Pennsylvania Museum Thailand Archaeometallurgy Project (TAP)' in the Khao Wong Prachan Valley in central Thailand indicate that over the span of a millennium, from the mid-2nd millennium B.C. to the later 1st millennium B.C., copper producing settlements, among the largest known in Asia, had developed. This paper presents an overview of archaeometallurgical evidence from prehistoric Nil Kham Haeng, one of the large copper producing settlements, and presents the preliminary results of microstructural studies of copper-base implements from burials excavated at this site. It has been argued elsewhere (Pigott et al.; in press) that the smelting technology applied at this, and neighboring production sites, utilized the direct reduction of copper by means of oxide-sulfide interaction i.e. the process known as 'co-smelting'. The observed microstructures are compared with the results of similar microstructural studies performed on specimens obtained from laboratory-based smelting process replication experiments using a co-smelting technology as the copper extraction process (Rostoker et al.; 1989; 1991)

Nil Kham Haeng in the Khao Wong Prachan Valley (Fig.1)
The prehistoric settlement of Nil Kham Haeng, which is more than 3 hectares in size, lies equidistant between the two major copper ore sources and about three km from Non Pa Wai, a neighboring and partially contemporary copper smelting settlement of comparable proportions (ca. 5 hectares). Nil Kham Haeng was partially damaged during construction of a reservoir in
the late 1970s, but more than three hectares remain intact (Fig. 2).

**Chronology (Fig.3)**

The radiocarbon dates from Nil Kham Haeng indicate a range of activity at the site from ca. 1100 - 300 B.C. (Natapintu 1991). The site's chronology has been divided into two periods, namely basal Nil Kham Haeng I (ca. 1100-700 B.C.) and Nil Kham Haeng Period 2 (ca. 700 B.C.- 300 B.C.). Archaeological and technological evidence from the site demonstrates that the most intense metallurgical activity and associated rapid site build-up at Nil Kham Haeng occurred in Period 2, i.e. at the point where activity at Non Pa Wai was in decline.

**Stratigraphy**

Nil Kham Haeng's highly unusual, well defined stratigraphy, with its numerous sealed contexts, permit the construction of a more finely articulated chronology than most prehistoric sites in Thailand and may thus provide an important key to understanding the sociocultural and technological changes which ensued in the Valley. Habitation related activity areas at Nil Kham Haeng contain features including posthole patterns and a number of burials clearly associated with well-defined living surfaces. The presence of a series of burials over time suggests at least semi-permanent, if not year round habitation. Domestic activity is signaled by artifact classes including ceramics, shell and stone bracelet manufacture, and ample faunal remains. Industrial activity is evidenced by smelting loci termed 'hotspots', multi-layered smelting pits, probable bowl furnaces and substantial quantities of slag. The burials were a remarkable source of the products of industry in the form of finished metal artifacts in both copper and iron, as well as of the apparatus of industry, including furnace chimneys.

What is most striking about this site is its highly unusual matrix composed of multitudinous, thin layers of gravel generated from crushing copper ore and slag (Fig.4). Distributed among these strata are a number of well-defined surfaces with evidence of copper production as well as of habitation.

The rather uniform size, and graduated and lensed distribution of the crushed debris strongly suggests redeposition by water action (Fig. 5). These lensed strata may form, on a seasonal basis, from the redistribution by rainwater of individual piles of crushed ore and slag. Centuries of human effort which went into mining, transportation, and production resulted in the enormous accumulation of industrial debris (ca. 3-plus ha.- by - 6-plus m.
deep) of which Nil Kham Haeng is comprised.

**Mining and Ore Processing**

The crushed debris at Nil Kham Haeng signals a technological change over time in the approach taken to the production of copper in the Valley. In earlier contexts at neighboring Non Pa Wai there is no indication of ore crushing on the scale and thoroughness seen at Nil Kham Haeng. The suggestion is that the crucible smelting practiced at Non Pa Wai may have utilized larger and most probably richer ore pieces in the smelting process, i.e. there was no need to crush ores to the extent to which they were being crushed at Nil Kham Haeng. The layered, crushed gravel component which comprises Nil Kham Haeng is the result of continuous, intensive ore crushing usually associated with a high gangue (waste-rock) component of the ore, i.e. low grade ore. Thus, in the Valley while the objective of producing copper endured over time, different production strategies were implemented which were appropriate, continuous and productive.

It is doubtful that the hectares of production debris at Non Pa Wai and at Nil Kham Haeng resulted from the exploitation of the same source of copper ore, namely Khao Tab Kwai, the most accessible deposit (see Fig. 1). Perhaps the metalworkers at Non Pa Wai, while continuing to utilize the ores at Khao Tab Kwai, began to exploit deposits of more difficult access located high in the upper reaches of the mountain of Khao Phu Kha. Nil Kham Haeng's proximity to these deposits suggests that they were the primary source of copper ores for that site's industry.

At both ore sources, miners grubbed out the rich surface expressions and then found it necessary to mine deeper following rich, oxidic veins. With the advent of shaft mining more effort had to be expended, first in the act of mining itself, and then in beneficiating the mined ores on-site by dressing and hand sorting to obtain the richest pieces of ore. Shaft mining may have ensued in response to increasing exhaustion of surface deposits combined with a greater demand for ore; indications are that this may have occurred sometime after the mid-2nd millennium B.C. and the start of industrial activity at Non Pa Wai. As shaft mining proceeded deeper into the deposit it brought miners into closer contact with the less oxidized, sulfur-rich ores - more complex in composition and harder to smelt. Over time, exhaustion of sources along with deeper mining and the extraction of potentially lower grade and sulfide
ores may have initiated changes in processing techniques. Thus, it may be that the vast amount of crushed ore and slag at Nil Kham Haeng suggests that local miners in the early 1st millennium B.C. were mining deeper lying, lower grade ores which necessitated more dressing to extract the copper. The crushing would also facilitate the re-use of slag in smelting both as a flux and as an additional source of copper. There is no doubt that quality and composition of the copper ores were major factors in the technological strategies, but TAP research suggests that metalworkers were successfully able to apply simple, but effective smelting techniques to achieve consistent copper production.

**Copper Production**

At Nil Kham Haeng the crushed ore gravel is present in the basal deposit of the site, but not in the volume per cubic meter that is seen in the upper levels of the deposit. The volume of crushed ore does appear to increase significantly with the passage of time. While crucible fragments are in evidence at Nil Kham Haeng, their presence is dramatically reduced at this site where the technique of bowl furnace smelting appears to have been practiced. The quantities of crucible fragments so abundant at Non Pa Wai are missing at Nil Kham Haeng. At Nil Kham Haeng fragments of what have been termed 'furnace chimneys' were quite common. Fragmentary, but entire, chimneys were included as grave goods in the several Nil Kham Haeng burials. As a result we have been able to assemble one complete chimney (Fig. 6). The height and diameter of such chimneys will aid in the measurement of furnace volume which in turn will assist us in our attempts to estimate the amount of copper produced on a per furnace basis. It would appear that from early in the site's history, bowl furnace smelting, which is described below, was practiced, perhaps exclusively at Nil Kham Haeng.

How was copper being produced? At Nil Kham Haeng crucibles are almost non-existent so instead of smelting crucibles, the metalworkers, we believe, used a smelting furnace - a small bowl ca. 20 cm in diameter easily dug in the gravel at the site's surface. The loose gravel necessitated that the bowl then be lined with a chaff-tempered refractory clay. A chimney was placed over the bowl completing the furnace structure. Ore, fuel and recycled slag were then charged in layers into this furnace. Our reconstruction suggests that the charge was comprised of a mixture of oxidic and sulfidic ores. Fuel could have been dry wood and bamboo which need not have been converted to charcoal prior to the smelting
process because the co-smelting reduction process is primarily sulfur not carbon driven. The slag served several purposes, as a flux, a source of additional copper and sulfur, and to blanket the charge, helping to retain the sulfur-rich gases which drive the co-smelting process. Thin fragments of chaff-tempered ceramic, bowl furnace lining vitrified during smelting were ubiquitous at Nil Kham Haeng. Such furnaces were easily built and then easily broken up to extract the product, most probably a circular piano-convex ingot of variable thickness. No such ingots are known from the site.

But, examples of another type of ingot are known. Some evidence for the use of so-called 'shallow molds' (Fig. 7f) occurs at Nil Kham Haeng. Fragments of such molds have been excavated. Surface finds in the disturbed areas of the site did yield thin, circular disk ingots of a type also known from other two other sites in the region, Tha Kae and Wat Tung Singto. Such ingots would have been cast in the shallow molds and their find spots at Nil Kham Haeng suggest they had been placed in the graves which were disturbed by bulldozing in the 1970s. These ingots may have been cast from the primary furnace products, the piano-convex ingots, which were melted down for the purpose.

Ingots, regardless of type, would have had to have been melted down in crucibles and cast into molds in order to produce the copper, cordiform implements, an artifact type thought to be characteristic of the production taking place at Nil Kham Haeng. These implements are the subject of an analytical program at MASCA and Lehigh University, the preliminary results of which are described below.

At Nil Kham Haeng, metal artifacts are present and these are known only from burial contexts. By far the most common artifacts are socketed, cordiform, blunt-ended implements of very thinly cast copper - ca. 2mm in thickness (Fig. 8). Their true function is unknown, but speculation ranges from projectile points, to digging spades, to ingots. Clusters of these artifacts are known from several Nil Kham Haeng burials - one cluster contained about 60 such artifacts. From these finds it is clear that by the 1st millennium B.C. some locally produced metal was being selected for burials.

Casting

The casting of these artifacts at Nil Kham Haeng was undertaken using bivalve molds often bearing external linear incised designs just as at Non Pa Wai (Fig. 9). From the evidence at Nil Kham Haeng, it appears that casting may have been somewhat restricted to the cordiform
implements. If frequency of occurrence is any indicator, the use of bivalve molds generally at Nil Kham Haeng was nowhere as significant as it was at Non Pa Wai.

While these cordiform implements were common grave goods at Nil Kham Haeng they were often, however, mis-cast. In such mis-cast artifacts the molten metal, when cast into the bivalve mold did not flow to completely fill the mold. These implements were cast copper and such unalloyed metal does not flow freely even at casting temperature. In fact, it has been suggested that the molds in which the copper implements were cast may have been pre-heated to a very high temperature in order to facilitate such thin castings.

In the bivalve molds used to cast such implements, the mold impression is clearly quite shallow. These molds yielded castings of remarkable thinness - so thin that one might question the utility of these socketed implements. The frequency of mis-cast artifacts suggests, along with their fragile construction, that these implements may not have been produced to perform a specific mechanical task. Furthermore, the microstructural analysis of these implements indicates that they were simply cast to shape and no further hot or cold working attempted. The edges might have been sharpened or cold-worked to increase their hardness, but none of this was done. Was the mis-casting due to inefficient techniques of casting or did the ultimate function of these implements have little to do with their physical integrity? Perhaps these implements constitute a type of 'ingot' - a standardized unit typical of the Valley copper industry and therefore easily recognized, counted, transported and melted - very useful in the exchange networks in which the sites in the Valley participated. But this is, however, mere supposition.

The Laboratory Program

PIXE analysis of the 15 cordiform implements which are under study clearly indicates that the smelted copper, even having been remelted for casting, is quite rich in sulfur (see Fig. 10). Furthermore, the smelting slag itself occurs in vast quantities at the site and is also sulfur-rich. Thus, we have argued elsewhere, that co-smelting of oxides and sulfides was being practiced at Nil Kham Haeng (Pigott et al.: in press). Rostoker et al. (1989; 1991) have shown that co-smelting in a crucible is a viable method for the production of copper metal. The current laboratory program underway at MASCA and Lehigh University is analyzing a group of copper artifacts from Nil Kham Haeng with express intent of determining if their structure and composition is consistent with that of metal which is a product of the co-smelting process.

Thermodynamics of Co-Smelting:
It has been generally assumed that the copper smelting process consisted of either the carbon reduction of oxide ores or the "matte" smelting of sulfide ores. However, based on TAP research the use of a relatively simple technique of co-smelting has been proposed as another technique by which copper was smelted in antiquity. The co-smelting process involves the inter-reaction between copper oxide and copper sulfide or copper-iron sulfide minerals. The relevant chemical reactions and their free energies for co-smelting at 1500°K are (Rostoker et al. 1989:73):

1) \[3\text{Cu}_2\text{O} + \text{FeS} = \text{FeO} + \text{SO}_2 + 6\text{Cu}\]  \[F = -42.63\ \text{Kcal}\]
2) \[5\text{CuO} + \text{CuFeS}_2 = \text{FeO} + 2\text{SO}_2 + 6\text{Cu}\]  \[F = -113.5\ \text{Kcal}\]
3) \[3\text{CuO} + \text{FeS} = \text{FeO} + \text{SO}_2 + 3\text{Cu}\]  \[F = -65.23\ \text{Kcal}\]
4) \[2\text{CuO} + \text{S} = \text{SO}_2 + 2\text{Cu}\]  \[F = -49.43\ \text{Kcal}\]

The generally large negative free energies of reactions establish that all the above reactions can proceed at reasonable copper smelting temperatures. In a series of laboratory experiments performed by Rostoker, copper metal was extracted directly from mixed oxide and sulfide ores (Rostoker et al. 1989; 1991). As a result of this research it was determined that the operating conditions necessary to smelt the complex weathered copper-iron sulfide ores in the presence of oxides are simply a combination of high temperature and a sulfur gas-rich reducing atmosphere. Rostoker's research demonstrated that the co-smelting technique for copper extraction is not only thermodynamically possible, but can also be replicated experimentally.

**Microstructural Analysis**

The fifteen copper artifacts from Nil Kham Haeng analyzed by PIXE are under study by optical microscopy and by scanning electron microscopy (SEM) using a JEOL 6300F at Lehigh University. Two of these artifacts, an axe (Fig. 11,T5126) and a spearpoint (Fig.12,T5109), were shown by PIXE and SEM-EDS to be tin-bronzes. They will be discussed later. The other 13 artifacts are all cordiform implements at least 6 of which are of similar composition. Comparison of the analytical results on these implements with Rostoker's co-smelting replication experiments indicates that they have microstructures similar to those produced in Rostoker's experiments in which malachite and chalcopyrite ores were co-smelted. The photomicrographs (Figs. 13, 14, 15) were selected to show the common microstructural features which appear to be characteristic of most of the copper artifacts. Fig.13 shows a low magnification optical photomicrograph of the microstructure of one of the cordiform implements (T4892). The matrix (base metal) is copper and there is no indication of any hot or cold working. A large
number of round (gray) copper sulfide (matte) inclusions are observed dispersed in the base metal. Further investigation of these inclusions by imaging and X-ray energy dispersive spectroscopy (EDS) via the SEM indicates that metallic iron has precipitated within the sulfide inclusions. Fig. 14 shows a higher magnification SEM picture of a two-phase inclusion particle. EDS results indicate that the dark phase in the secondary electron image is metallic iron and the light gray phase is copper sulfide. From the Cu-Fe-S equilibrium phase diagram at 1250°C (Fig.20), one of the notable features of this system is that liquid copper can coexist with the matte and solid iron. Therefore, the presence of copper matrix with copper sulfide inclusions and metallic iron in a copper matrix is a possible equilibrium situation for this system. The other necessary condition for the presence of metallic iron in the copper matrix is that a strongly reducing atmosphere was used during the copper smelting process. The iron was reduced to metallic iron rather than segregating to the slag phase. However, there are some samples in which metallic iron was not observed in the copper matrix. The reason for the absence of iron is that the less iron component ore might be used in the copper co-smelting process. According to the phase diagram in Fig.20, iron as separate phase would be expected only when the total iron concentration is about 20wt% or higher, and this would be only under reducing conditions. Thus, when copper ore which has less iron was used for copper smelting, the iron would not present as a metallic state in the copper matrix.

After etching, the mounted copper samples, which all contain substantial matte, generally appear to possess a large grain size (Fig.15, T4894). This could indicate that they were exposed to high temperature during the smelting process. (Low temperatures and a large fraction of matte inclusions would tend to produce a smaller grain size.) These observations are very similar to those obtained by Rostoker from specimens produced in his laboratory replication experiments. In his experiments, copper metal was extracted at 1250°C in a sulfur-rich reducing atmosphere. Optical micrographs of the laboratory replicated samples indicate that the copper matrix also has a large number of matte inclusions and possesses a large grain size (Fig.16 and Fig.17). Metallic iron was not observed in this sample, but in his report of other replication experiments metallic iron was noticed (Rostoker et al. 1989:69).

The microstructural investigations for the copper production at Nil Kham Haeng are consistent with the practice of a co-smelting technology. This type of single-stage bowl furnace co-smelting process might therefore represent a phase in the gradual technological
transition from the processing of oxide ores to that of sulfides exclusively.

**Concluding Observations**

1. Thermodynamic analysis indicates that a co-smelting process is possible.
2. The replication experiments show that it is not difficult to obtain copper metal directly by such a co-smelting process.
3. The microstructure of the copper objects from Nil Kham Haeng is consistent with the features expected of co-smelted metal: the presence of a high volume fraction of residual sulfides, the large grain size of the copper matrix and the presence of metallic iron. The first two features are indication of high temperature, and the last suggests a reducing environment.
4. The combination of the high sulfur content of the cordiform implements and their unique shape will assist in future attempts to identify these artifacts as products of the Khao Wong Prachan Valley copper industry when they are discovered outside of its confines. Two such finds have recently been made of topologically similar artifacts, one at a site some 60 km north of the Valley (Surapol Natapintu: personal communication). A second find of several implements of somewhat similar shape has occurred in the excavations at Nong Nor in the Bang Pakong Valley south of Bangkok (Charles Higham: personal communication). While no analyses are available on these finds, their discovery alone strongly suggests that the copper from the Khao Wong Prachan Valley was reaching, at a minimum, regional consumers.
5. In the study of archaeometallurgical remains from prehistoric Southeast Asia prior to the TAP excavations, analysis of copper-base artifacts have revealed only the presence of tin-bronze. In the Khao Wong Prachan Valley, at least the results from Nil Kham Haeng, indicate that the opposite is thus far true, with but two exceptions among the 15 copper artifacts in this study (Fig. 11, T5126; Fig.12, T5109). These two artifacts, which are tin-bronce have microstructures and compositions totally different from the 13 copper cordiform implements (Fig.18, T5126; Fig.19, T5109). The microstructures of the two tin-bronzes indicate that they were fabricated by a very different smelting technique than the other artifacts in this study; e.g. the tin content, different grain size, and the markedly small quantity of matte inclusions, as well as their topology combine to suggest that these artifacts might have been imported to this region along the same exchange networks by which Nil Kham Haeng copper moved out from this Valley. This copper may well have been exchanged for other commodities with peoples who dwelt near tin deposits and
had the requisite technological understanding of how to manufacture bronze.

Notes
1. The fieldwork and research of the Thailand Archaeometallurgy Project has been funded, in part, by the National Geographic Society, the National Science Foundation, the American Philosophical Society and the University of Pennsylvania Museum. The generous support of Mrs. Graham Cummin and Ms. Maude de Schauensee is also acknowledged.
2. PIXE analysis (proton-induced x-ray emission spectroscopy) was undertaken under the auspices of MASCA by Dr. Stuart J. Fleming and Dr. Charles P. Swann at the Bartol Research Institute (University of Delaware).

References


Figure Captions

1. Map of the prehistoric sites and some ore deposits in the Khao Wong Prachan Valley in central Thailand

2. Contour map of the prehistoric site of Nil Kham Haeng

3. Working chronology for the sites of Nil Kham Haeng and Non Pa Wai

4. View of excavation at Nil Kham Haeng showing its unusual stratigraphy

5. Close-up view of gravel lens stratigraphy at Nil Kham Haeng

6. Furnace chimney assembled from fragments excavated in burial at Nil Kham Haeng (T5347)

7. Variety of ceramic mold types from Khao Wong Prachan Valley sites. Shallow Mold (f)

8. Copper socketed cordiform implement, a number of which were excavated from burials at Nil Kham Haeng. The bronze axe head (T2852) is on the far left with the crescent-shaped blade. The other 2 are copper (T3065)

9. Ceramic bivalve mold for the casting of small cordiform implements from Nil Kham Haeng (T5291)

10. Table of results of PIXE analysis of copper-base artifacts from Nil Kham Haeng.

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11. Tin-bronze axe from Nil Kham Haeng burial (T5126). This may have been mis-cast as there is no hollow socket. For such an axe not to have a socket is virtually unknown in prehistoric Southeast Asia.

12. Socketed tin-bronze spearpoint from Nil Kham Haeng burial (T5109).

13. Optical micrograph of copper sulfide inclusions dispersed in copper matrix (x 100). Cordiform implement (T4892)

14. SEM micrograph of two phase particle of iron dendritic on copper sulfide (x4000). Cordiform implement (T4892)

15. Optical micrograph of large grain size of copper matrix after etching (x250). Cordiform implement (T4894)

16. Optical micrograph of copper sulfide inclusions dispersed in copper matrix from replication experiment (x 100)

17. Optical micrograph of large grain size of copper matrix after etching (x 100)

18. SEM micrograph of tin-bronze axe (x720). Cordiform implement (T5126)

19. Optical micrograph of tin-bronze spearpoint (x100). Cordiform implement (T5109)

20. Copper-iron-sulphur equilibrium phase diagram, 1250°C (Krivsky and Schuhmann, 1957)

end
Fig. 1. Map of the prehistoric sites and some ore deposits in the Khao Wong Prachan Valley in central Thailand.
Fig. 2. Contour map of the prehistoric site of Nil Kham Haeng
Fig. 3. Working chronology for the sites of Nil Kham Haeng and Non Pa Wai
Fig. 4. View of excavation at Nil Kham Haeng showing its unusual stratigraphy
Fig. 5. Close-up view of gravel lens stratigraphy at Nil Kham Haeng
Fig.6. Furnace chimney assembled from fragments excavated in burial at Nil Kham Haeng (T5347)
Fig. 7. Variety of ceramic mold types from Khao Wong Prachan Valley sites. Shallow Mold (f)
Fig. 8. Copper socketed cordiform implements, a number of which were excavated from burials at Nil Kham Haeng. The bronze axe head is on the far left with the crescent-shaped blade (T2852). The other 2 are copper (T3065)
Fig. 9. Ceramic bivalve mold for the casting of small cordiform implements from Nil Kham Haeng (T5291)
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<th>Fe</th>
<th>S</th>
<th>Pb</th>
<th>Zn</th>
<th>Ag</th>
<th>Sb</th>
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<td>&lt;0.44</td>
<td>0.195</td>
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**Fig.10.** Table of results of PIXE analysis of copper-base artifacts from Nil Kham Haeng.
Fig. 11. Tin-bronze axe from Nil Kham Haeng burial (T5126). This may have been mis-cast as there is no hollow socket. For such an axe not to have a socket is virtually unknown in prehistoric Southeast Asia.
Fig. 12. Socketed tin-bronze spearpoint from Nil Kham Haeng burial (T5109).
Fig. 13. Optical micrograph of copper sulfide inclusions dispersed in copper matrix (x 100).
Cordiform implement (T4892)
Fig. 14. SEM micrograph of two phase particle of iron dendritic on copper sulfide (x4000).
Cordiform implement (T4892)
Fig. 15. Optical micrograph of large grain size of copper matrix after etching (x250).
Cordiform implement (T4894)
**Fig. 16.** Optical micrograph of copper sulfide inclusions dispersed in copper matrix from replication experiment (x 100)
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