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Metallurgical analysis of copper artifacts from Cahokia

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ABSTRACT

Copper artifacts from Cahokia Mounds, Illinois were analyzed from a materials science perspective to shed light on techniques used by Mississippian copper workers to deform nuggets of native copper into thin sheets. Eight small copper pieces from a copper-working site at Cahokia's Mound 34 were subjected to metallographic examination. Replication experiments thereafter recreated features of the artifacts under controlled conditions. It is concluded that copper sheets were thinned through repeated cycles of hammering and annealing performed at temperatures achievable in an open wood fire. The welding of sheets to create multilayered objects was not observed in any artifacts and could not be accomplished experimentally. Additionally, a possible cutting method used on some artifacts was identified.

1. Introduction

In the Mississippian culture, which encompassed much of present-day Southeastern and Midwestern United States from AD 1050 until European contact, copper was a key prestige good (Ehrhardt, 2009). The ceremonial and decorative copper items produced during this period, such as beads, repoussé plates, and copper-clad personal adornments, represent a zenith of prehistoric North American metalworking technique (Leader, 1988; Sampson and Esarey, 1993; Ehrhardt, 2009). Of all the major sites from this period, Cahokia, in southwestern Illinois, can be expected to have the dominant yield in copper artifacts because of its larger size, proximity to metal sources, and status as a ritual center. At present, however, it has yielded few copper objects, in contrast to the more numerous finds from Etowah, Georgia; Spiro, Oklahoma; and Moundville, Alabama; where, unlike at Cahokia, elite gravesite contexts have been examined (Ehrhardt, 2009).

Mississippian copper objects were crafted from nuggets of naturally-occurring and often highly pure native copper that were likely procured through long distance exchange from both Southeastern and Great Lakes sources (Hurst and Larson, 1958; Goad, 1980; Rapp et al., 1984), although "float" copper, found in the glacial drift across much of the Midwest, may also have been used (Halsey, 2008). The production process typically involved deforming nuggets into sheet or foil, from which objects were then fashioned via molding, embossing, perforation, riveting, and other sophisticated techniques (Cushing, 1894; Watson, 1950; Leader, 1988; Cobb and Evans, 2009; Ehrhardt, 2009). There has never been any credible evidence that Native Americans of the eastern United States employed melted metal technologies prior to European contact; instead, they relied on working (hammering) and annealing (heat treatment) to reshape copper nuggets (Schroeder and Ruhl, 1968; Clark and Purdy, 1982; Childs, 1994; Ehrhardt, 2009). Accordingly, of the two Mississippian copper sheet artifacts excavated from sites in Tennessee and examined metallographically by Schroeder and Ruhl (1968) and an additional two by Springer (2007), excavated from Moundville, Alabama, all were found to have been left in an annealed state after working. Replication experiments by Clark and Purdy (1982) suggested that thin native copper artifacts, such as these, were the product of repeated hammering and annealing cycles. Furthermore, Schroeder and Ruhl (1968) reported that the North American copper artifacts that they examined generally appeared to have been annealed at

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700–800 °C, and that one Mississippian copper object seemed to be "laminated", or composed of multiple sheets layered together. However, no technical analysis of copper materials from Cahokia has ever been conducted to verify that these broader statements about Mississippian and North American practices accurately describe the copper-working processes used at this site.

A "copper workshop house" dating to the early Moorehead phase (ca. AD 1200) was identified at Cahokia's Mound 34 by Gregory Perino in 1956 (Kelly et al., 2007; Kelly and Brown, 2010). Recent excavation by Brown and Kelly (Belknap et al., 2008; Kelly and Brown, 2010) at this site provides a uniquely valuable set of data for investigating copper production there. This study takes a materials science approach to characterizing certain aspects of the production process. Worked pieces of copper from Mound 34 were examined metallographically, and these findings were used as a baseline for several replication experiments. The specific questions addressed were: How were hammering and annealing used to thin nuggets into sheet, and what were the annealing conditions (time and temperature)? Did manipulations, such as bending, take place before or after the final anneal? Was the layering of multiple sheets used to make any of these objects? And, what method was used to produce the straight edges observed on several artifacts?

2. Materials and methods

2.1. Artifact examination

The artifacts in this study consist of eight objects recovered by Brown and Kelly at Cahokia's Mound 34, deriving from Gregory Perino's 1956 backdirt. All the items, shown in Fig. 1, are composed of copper sheet covered in black and green corrosion product. None of the objects appear to be finished pieces, but rather seem to be abandoned blanks or scraps. Some of the observed superficial features of the artifacts, listed in Table. 1, appear to be the result of specific production or manipulation techniques. Artifact 3 had been bent at a 90° angle. Artifacts 4, 6, and 8 all appear to be separating into two distinct layers in one or more places. Also, artifacts 6, 7, and 8 each display one or two distinctly straight edges, all characterized by a blunt profile with small burrs. This was interpreted as evidence that a common technique may have been used to cut all three. Because of the questions posed by these features, the following analyses focus predominantly on the artifacts described above.

Artifacts were cut across their width at areas of interest using a low-speed diamond saw (IsoMet 1000, Buehler, Lake Bluff, IL) in

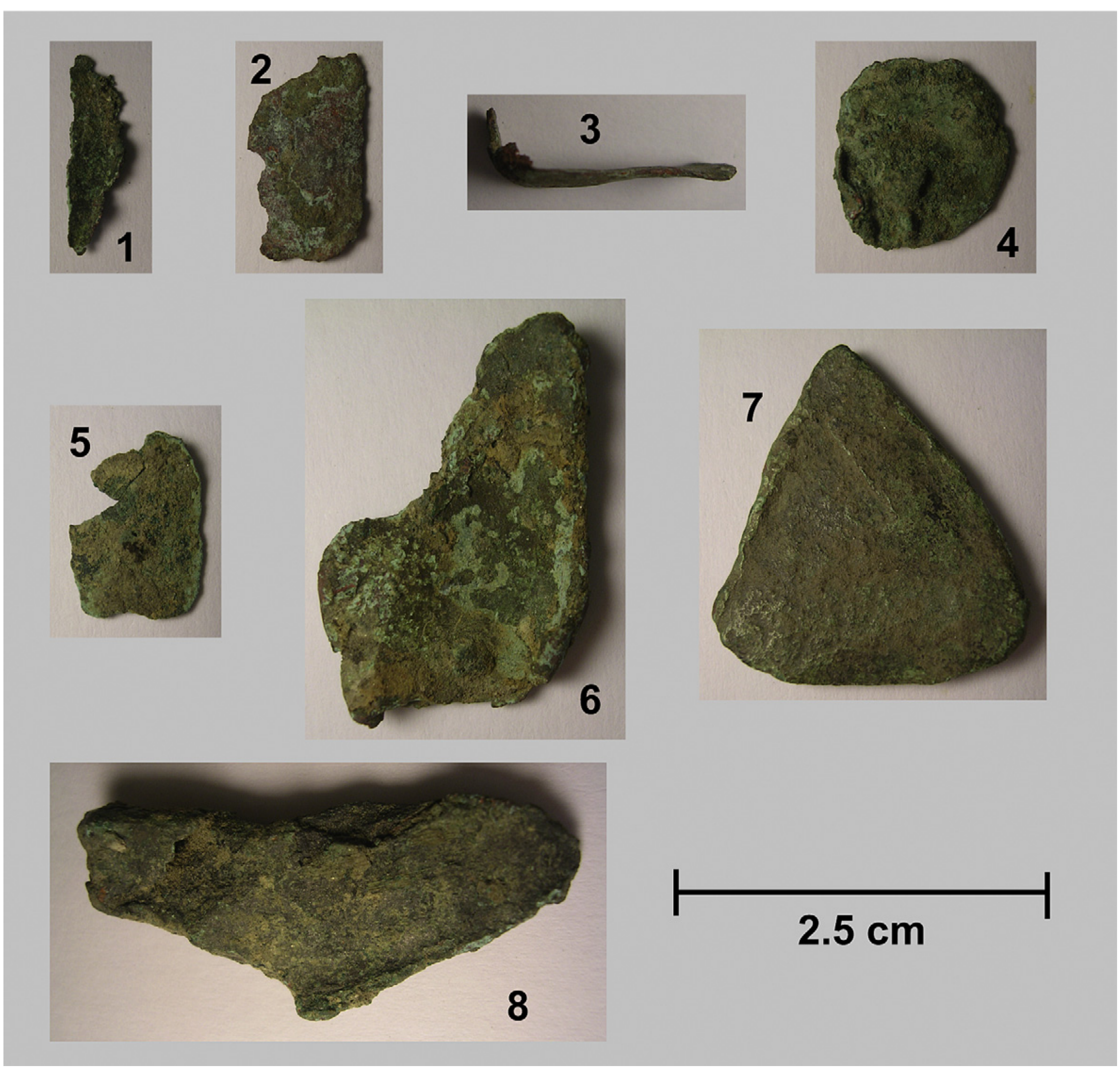

Fig. 1. Photographs of the eight copper artifacts from Cahokia's Mound 34 examined in this study.

order to expose their cross-section. Removed sections were mounted in quick-set acrylic (Lapmaster, Mount Prospect, IL), ground with grit paper, and then polished with alumina suspensions, finishing with a 0.05 μm particle size. Use of an ultrasonic cleaner during polishing was avoided, as it was found to cause pitting of the copper surface due to cavitation. Finally, mounted and polished artifact sections were etched with a 1:1 solution of 30% ammonium hydroxide (NH_4OH) and 3% hydrogen peroxide (H_2O_2) to reveal grain boundaries.

Mounted sections were examined using an inverted-light optical microscope (PMG3, Olympus, Tokyo, Japan) at magnifications between 50 and 500X. Polarized light was sometimes used to enhance grain boundary contrast, resulting in altered coloration such as that seen in Fig. 6B. Optical micrographs were used to estimate the mean spatial grain diameter ($\overline{D}$), using an intersection counting procedure detailed in ASTM Standard E 112-96 (2004).

Mounted artifact sections were also analyzed through microhardness testing on a MicroMet II hardness tester (Buehler, Lake Bluff, IL), using a Knoop indenter set to indent for 5 s at 50 g force. Indentation size was measured using the attached Buehler DigiMet micro-hardness system. Series of measurements, typically containing 5 to 20 data points, were taken in straight lines across the samples so that hardness profiles could be plotted and average hardness values calculated.

Selected artifacts were chemically analyzed using scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDX) and direct-coupled plasma optical emission spectroscopy (DCP-OES). SEM-EDX provides pinpoint elemental analysis, although the detection limit for trace elements can be poor. The S–3400N–II variable pressure SEM (Hitachi, Pleasanton, CA), operated with an acceleration voltage of 25 kV and a beam current of 110 μA, was used with an INCAx-act EDX detector (Oxford Instruments, Abingdon, UK) to measure local compositions on sections from artifacts 4 and 6. DCP-OES provides bulk elemental analysis with a detection limit of 100 ppm, roughly ten times better than that of EDX. Sections removed from artifacts 6 and 8, 1.69 and 3.55 g in mass respectively, were sent for DCP-OES analysis at ATI Wah Chang (Albany, OR).

2.2. Replication

The replicated samples were made using a native copper nugget, originating from Michigan's Keweenaw Peninsula and purchased at Dave's Down to Earth Rock Shop (Evanston, IL). The nugget had a rough exterior with many protruding knobs. This nugget was cut into sections with an Accutom-5 metallographic saw (Struers, Copenhagen, Denmark), and each section was used for a single replication attempt. Replicated samples were prepared for optical microscopy in the same manner described in the previous section.

An annealing experiment was conducted in order to observe the effects of annealing time and temperature on the microstructure of worked copper. A section of native copper, cut with two coplanar faces to ensure consistent cold work throughout the sample, was compressed to 75% percent thickness reduction in a hydraulic press (PHI, City of Industry, CA) under a 178 kN force. Nine samples of worked native copper were then cut from the pressed sheet with the Accutom-5 metallographic saw, and each was annealed in laboratory air in a temperature-controlled furnace (Barnstead Thermolyne, Dubuque, IA, USA) under a unique set of conditions. Three annealing temperatures were used: 500, 650, and 800 °C; and times ranged from 2 min to 100 min. After annealing, the samples were removed from the furnace and quenched in water.

A piece of bent sheet was made for comparison with bent artifact 3. First, a thin sheet was produced by hammering a 10 mm thick section of native copper with a steel hammer and steel anvil until it was reduced to a uniform 1 mm thickness. It was necessary to anneal the copper for 10 min at 650 °C several times during the hammering process to maintain malleability. The finished sheet was annealed again prior to being bent by hand to a 90° angle with a bend radius similar to that of artifact 3.

To examine the hypothesis that copper sheets were welded together in layers, resulting in the "lamination" reported by Schroeder and Ruhl (1968) and possibly the layering seen in artifacts 4, 6, and 8, the joining of two copper sheets was attempted. McPherron (1967) concluded that it was not possible to weld native copper by hammering pieces together at high temperature due to the formation of surface oxides, so a modified technique was used. Two annealed sheets were made using the hammering and annealing procedure described above and ground with grit paper to produce smooth faces, free from any oxide. These two polished sheets were pressed together with a force of 178 kN in the hydraulic press, and the resulting sheet, consisting of two joined layers, was then annealed for 10 min at 650 °C.

Additionally, four techniques for replicating the cut edges observed on artifacts 6, 7, and 8 were attempted and compared. A method similar to that described by Cushing (1894) and Clark and Purdy (1982) was used for one sample: a hammered and annealed sheet was placed on soft rubber mat and embossed along a line, using a hard plastic scribe; the resulting raised line on the reverse of the sheet was then ground away with 100-grit grit paper, cutting the sheet along the line. A second sample of sheet was sheared with scissor-like steel shears (tin snips) designed for cutting sheet metal. A third was cut by hammering the copper sheet against a sharp steel corner. The final sample was bent back and forth repeatedly by hand until it cracked due to fatigue.

3. Results

3.1. Artifact examination

Figs. 2–6 show micrographs of etched cross-sections from artifacts 2–4 and 6–8, as well as the bending and cutting replication samples. All of the artifact cross-sections contained regions in which the microstructure was characterized by smooth, equiaxed grains and the presence of blunt-tipped twin boundaries within grains, as shown in Fig. 2A. For six of the artifacts, including artifacts 3 (Fig. 3A) and 8 (Fig. 6C), the entirety of the observed microstructure fit this description, although grain size and surface oxide prevalence varied. However, while artifacts 6 (Fig. 4) and 7 (Fig. 6B) largely contained this same equiaxed, twinned grain structure, each also had areas with a second type of grain structure. This second microstructure, observed to be localized near the cut edges of both artifacts, was defined by mildly distorted and elongated grains and by clusters of parallel lines on the surface of many grains (Fig. 2B). High magnification observations revealed these lines to consist of many etched, triangular pits (Fig. 2B, inset).

Artifacts 4 and 6 displayed microstructural features possibly related to the layering or welding of sheets. Artifact 6 (Fig. 4) was divided in two lengthwise by a recessed groove from which material had been selectively removed by the etchant. This groove was slightly darker than the surrounding metal and contained significantly smaller grains (Table. 1). However, the groove did not present a barrier to grain boundaries, which crossed it in many places (Fig. 4B). Fig. 5 shows two sections cut from artifact 4. The first section (Fig. 5A) contained thin gray-colored oxide inclusions that seemed to indicate a plane dividing the artifact into two distinct layers. However, the second section (Fig. 5B), cut perpendicularly to the first, clearly showed that the oxide inclusions did not form a plane or any other organized structure.

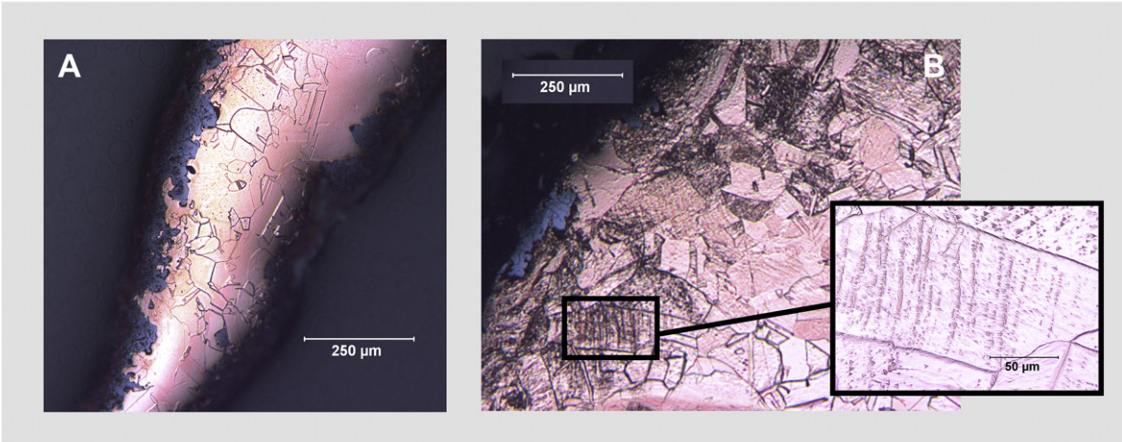

Fig. 2. Micrographs illustrating (A) the annealed microstructure of artifact 2, defined by twinned, equiaxed grains; and (B) the annealed and subsequently worked microstructure of artifact 6, which displays deformation bands (inset).

Artifact 6 (Figs. 4 and 6A) contained a suspected cut edge in addition to the etched groove described above. This edge had three distinct characteristics under the microscope: distorted and rough-surfaced grains near the edge (Fig. 6, ii), a burr extending from the edge (Fig. 6, i), and a blunt profile. Fig. 6 also shows that of the other two cut edges, artifact 7 shared the blunt profile and distorted grains (Fig. 6, iii), while the edge of artifact 8 had only the blunt profile in common, even though all three edges looked quite similar superficially.

The artifacts contained a variety of average grain sizes, as shown in Table 1. Artifacts 3 and 7 had grains over 300 μm in mean spatial diameter, and the other artifacts generally had grains less than half that size. While artifacts 1, 3, 5, and 7 contained grains of roughly uniform size, the other four artifacts displayed wide range grain size distributions, as defined by ASTM Standard E 1181-02 (2008). These wide range distributions contained a mixture of grains both much smaller and much larger than average, and minimum and maximum grain sizes are listed along with the mean in Table 1. Artifact 6 uniquely contained three distinct grain size regions, shown in Fig. 4: 64 μm within the etched groove; 137 μm in the rough, deformed grains and the neighboring equiaxed grain; and a wide range with a mean of 189 μm in the equiaxed region composing most of the cross-section.

The average measured hardness values for each artifact are listed in Table 1. Hardness could not be measured for artifacts 1 and 3, which were too thin to present an adequate area for indentation. Four of the remaining six artifacts had mean Knoop hardnesses between 75 and 80 $kg_f\,mm^{-2}$, with standard deviations between 10 and 15 $kg_f\,mm^{-2}$ indicating moderate variation across the exposed surface. Artifact 7, Fig. 6, which showed some deformed grains, had a mean Knoop hardness of 99 $kg_f\,mm^{-2}$ and a similar degree of variation across the surface to the previous artifacts. Several hardness profiles, including that shown in Fig. 4, demonstrated that hardness on artifact 6 differed between the three distinct zones, corresponding to the changes in grain size and appearance. The large area of equiaxed grains (Fig. 4, ii) had a mean hardness of 83 $kg_f\,mm^{-2}$; the edge region of deformed grains (Fig. 4, i), 118 $kg_f\,mm^{-2}$; and the mean Knoop hardness along the groove (Fig. 4, iii) was 135 $kg_f\,mm^{-2}$.

Multiple SEM-EDX measurements revealed that the groove on artifact 6 contained arsenic (Fig. 4D), with a peak concentration of roughly 4.5 wt%, while no arsenic was found in the bulk. Carbon and oxygen were measured on both artifacts 4 and 6. However, both carbon and oxygen can be dismissed as surface contaminants, as both elements have negligible solubility in copper (Mathieu et al., 1973; Neumann et al., 1984). SEM-EDX also found that, in artifacts 4 and 6, the black oxide inclusions contained copper; 15–30 mol% carbon, as in the matrix; 30–35 mol% oxygen, much higher than in the matrix; and no other element. This oxide is almost certainly copper (II) oxide (CuO), as the other possible oxide of copper, copper (I) oxide (Cu_2O), is red in color.

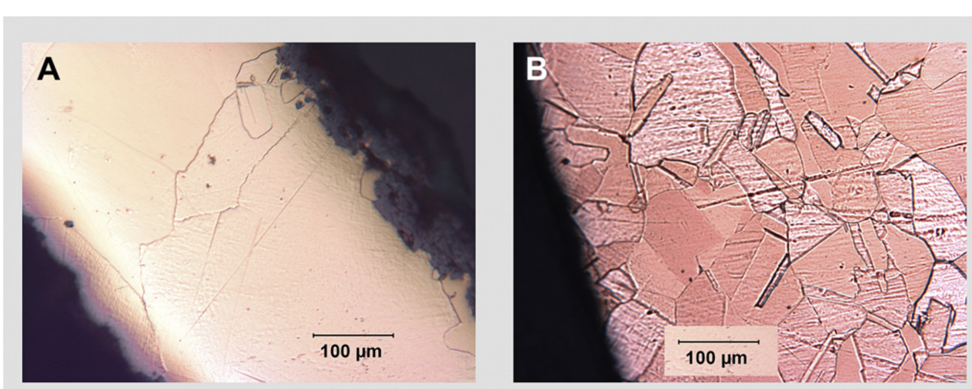

Fig. 3. Comparison between the bent regions of (A) artifact 3 and (B) the replicated sample bent by hand. Neither object shows microstructural deformation resulting from bending.

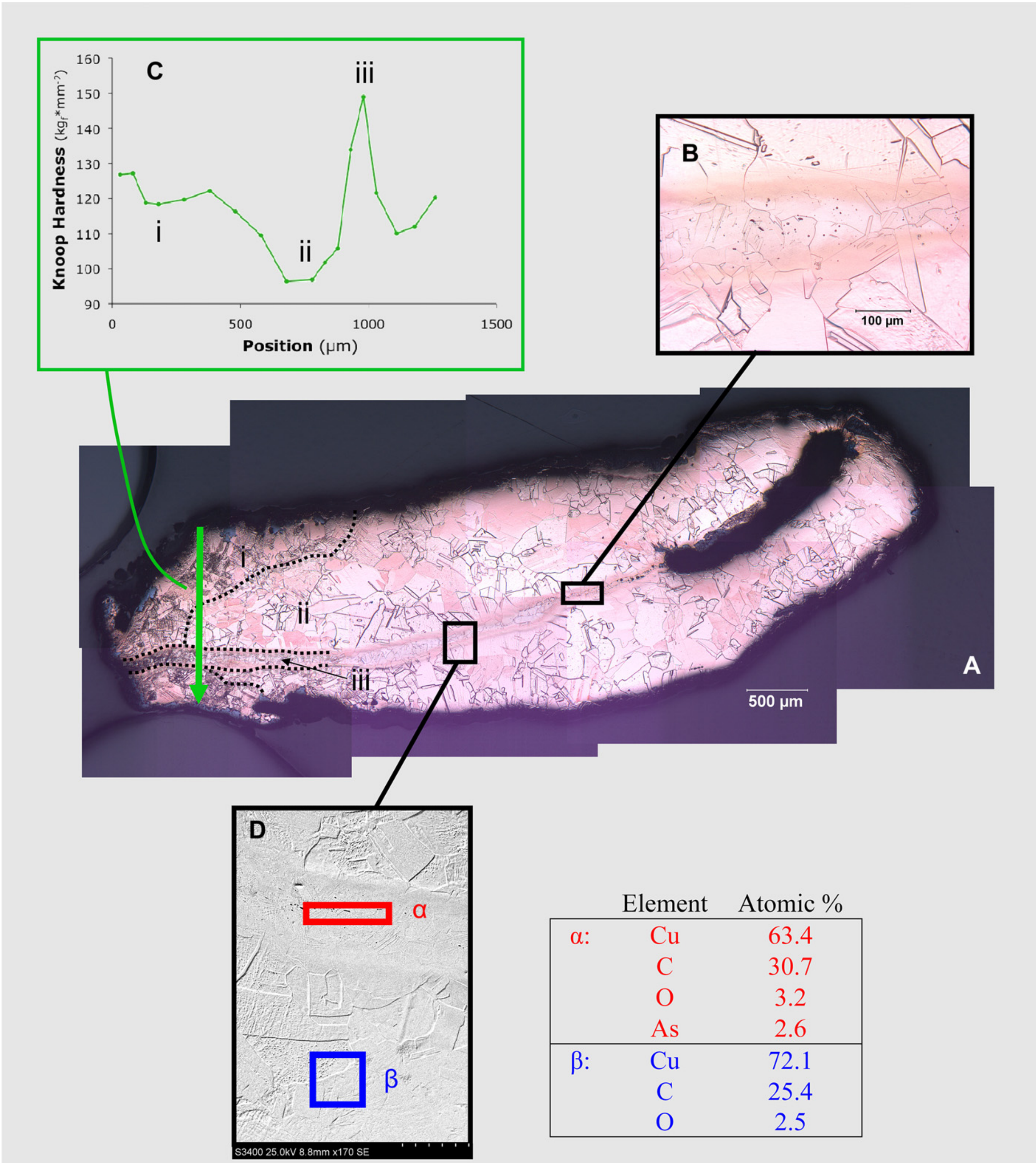

Fig. 4. This figure shows the results of a multi-technique characterization of artifact 6. (A) Optical micrograph of the etched cross-section reveals three regions: (i) deformed grains, (ii) un-deformed grains, and (iii) a recessed groove. (B) Optical micrograph of groove seen in (A) defined by smaller grains and a darker color than the surrounding material. (C) Micro-hardness profile showing that the deformed region (i) is harder than the un-deformed region (ii), and the groove (iii) is much harder than either. (D) SEM-EDX data showing that the groove (α) contains arsenic, while the bulk (β) does not.

A section of artifact 6 containing the groove was submitted for DCP-OES analysis; silver was found at a concentration of 220 ppm by weight and no other elements surpassed the instrument's detection limit. This was unexpected, since it represented a failure to identify the arsenic measured with SEM-EDX in the same artifact. A section of artifact 8 was also analyzed with DCP-OES, and the only detectable trace elements were silver, at 140 ppm, and silicon, at 67 ppm by weight. All three of the detected trace elements

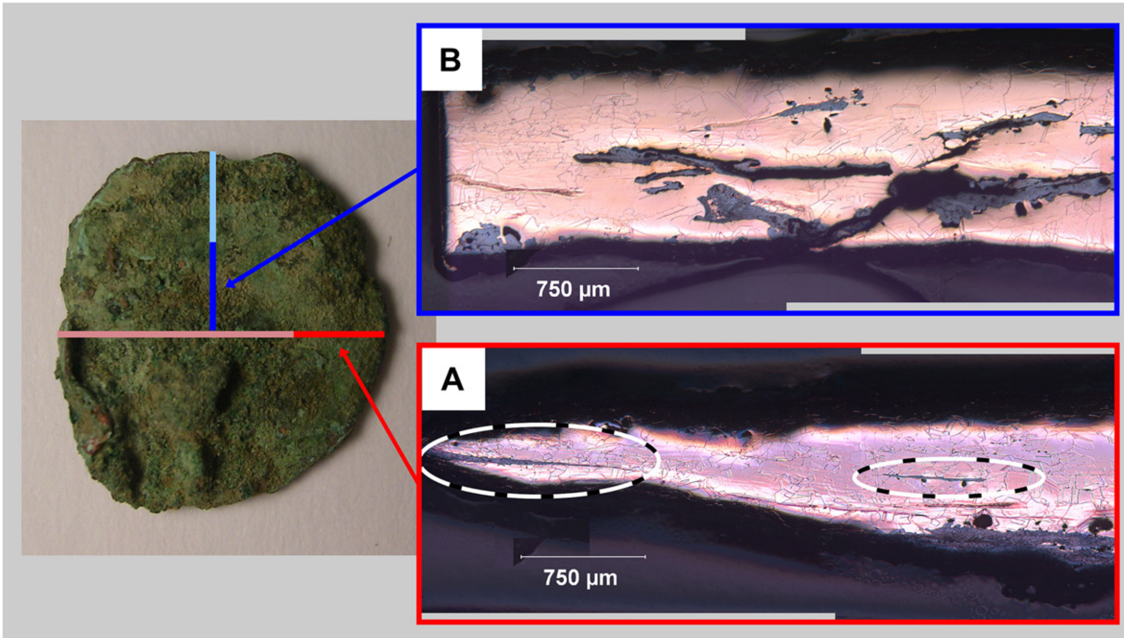

Fig. 5. Optical micrographs of cross-sections from artifact 4: (A) showing planar oxide inclusions (circled) that appeared to suggest layering. (B) taken from a perpendicular cut on the same artifact, containing disordered oxide inclusions, likely indicative of flattened surface protrusions rather than intentional layering.

(arsenic, silver, and silicon) are known to commonly occur in native copper (Broderick, 1929; Craddock, 1991; Hurst and Larson, 1958; Rapp et al., 1984, 2000).

3.2. Replication

It proved infeasible to reduce a 10 mm thick piece of native copper to a 1 mm thick sheet using hammering alone. The copper became noticeably less malleable after cold hammering to ~30% reduction, which was achievable with several minutes of hammering. After ~50% reduction, the copper was extremely difficult to work, and cracks began to appear at the edges of the piece. Annealing the worked copper at this stage for 10 min at 650 °C returned the piece to its original malleable state. Four or more cycles of hammering and annealing were needed to achieve the desired 90% reduction in thickness. Hammering produced debris consisting of millimeter-sized flakes of copper. A black oxide layer formed during annealing, covering the surface of the copper. During air cooling or quenching, this oxide flaked off, producing a large amount of fine black powder.

The measured grain sizes from the annealing experiment are shown in Table 2. Grain size increased with both annealing time and temperature. The sample annealed at 500 °C for 15 min, which represented the lowest temperature and the shortest anneal at that temperature, contained grains sufficiently elongated to prevent measurement of a mean size. Four other samples (500 °C for 40 and 100 min, and 650 °C for 6 and 15 min) displayed wide range grain size distributions. Differently sized grains appeared to be distributed randomly within each sample, rather than grouped together. The remaining four samples, annealed longer or at a higher temperature, all had larger, more uniform grain sizes. Additionally, all of the samples annealed at 650 °C and 800 °C contained extensive twinning, while few to no twins were visible in the samples annealed at 500 °C.

The microstructures of the bent regions of both artifact 3 and the replicated bent sheet are compared in Fig. 3. Neither object showed any apparent signs of grain distortion. The grains in the replicated sample were significantly smaller than those in artifact 3.

Several samples of replicated layered sheet were successfully created by using the hydraulic press to force together two polished, annealed sheets. However, none of the samples maintained their bond after being annealed. Annealing-induced oxide formation along the interface between the two layers caused every sample to separate back into two sheets when air cooled or quenched.

Figure 6 compares the microstructures of cut edges of three artifacts (6, 7, and 8) with microstructures of the four replicated cut samples. As stated in section 3.1, artifact 6 had three features apparently associated with the cut edge: a blunt edge profile; distorted, banded grains adjacent to the edge; and a small burr extending from the cut face. Among the replicated samples, only the one formed by repeatedly flexing to produce fatigue cracking shares the defining blunt profile with the artifacts (Fig. 6G). Like artifact 6, the fatigued sample also displays both a burr (Fig. 6, vi) and distorted, banded grains at the cut edge (Fig. 6, vii). The other three replicated samples (Fig. 6D–F) all have much sharper edges, as well as different grain structures. The sample embossed and ground following Cushing (1894) (Fig. 6D), shows unaltered grains, while both the sample cut with steel shears (Fig. 6E) and that hammered against an edge (Fig. 6F) show some grain elongation (Fig. 6, iv and v).

4. Discussion

4.1. Microstructural interpretation

The deformation of copper resulting from cold working is marked by a series of changes at the microstructural level. Metals flow plastically through the deformation of grains. Individual grains deform via the formation and motion of dislocations, linear defects in a grain's crystal structure. Metals are said to work harden as the concentration of dislocations increases, impeding the motion of additional dislocations. Dislocations appear in an etched metallographic section as small pits where the etchant has preferentially removed the material at the intersection of the dislocation line and the surface. Since dislocations concentrate along preferred crystallographic slip planes, these pits are grouped in clusters of parallel

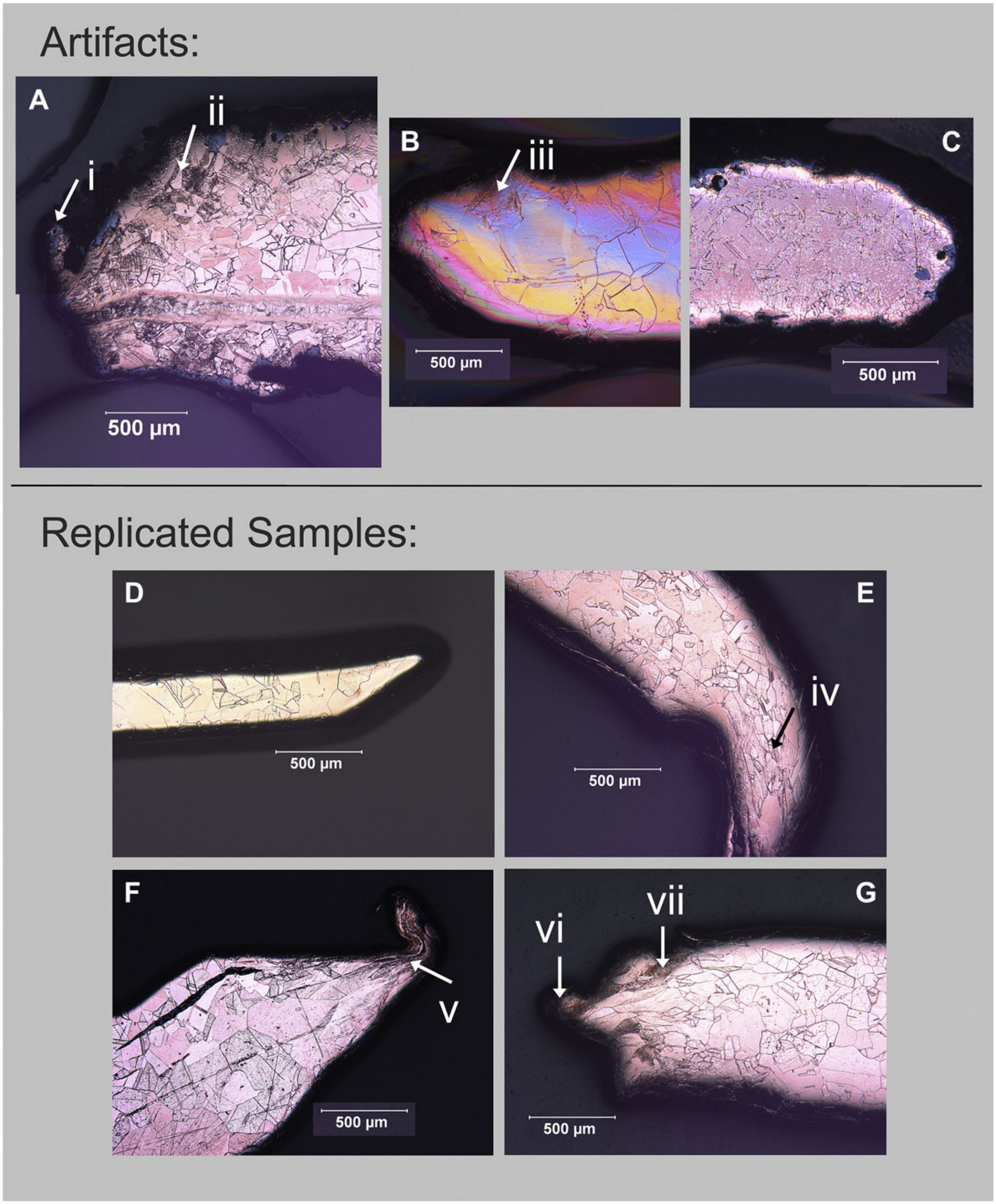

Fig. 6. Micrographs comparing the apparently cut edges of three Mound 34 artifacts (A–C) with the four cut replication samples (D–G). Artifact 6 (A) shows shear burrs (i) and deformation bands (ii), while artifact 7 (B) shows only deformation bands (iii), and artifact 8 (C) shows neither. The replicated samples represent embossing and grinding (D); shearing with steel shears (E), which produced elongated grains (iv); hammering against a corner (F), which also shows grain elongation (v); and repeated bending (G), which displays shear burrs (vi) and deformation bands (vii), much as in artifact 6 (A).

lines called deformation bands. Pitted deformation bands from artifact 6 are shown in Fig. 2B. A history of light to moderate cold working in copper is evident by distorted grains and the presence of deformation bands, as well as by increased hardness, all of which were observed in artifacts 6 and 7 (Table. 1 and Fig. 6). With more extreme cold working, deformation bands become long, bent flow lines that extend across grain boundaries, and grains become increasingly elongated until grain boundaries are no longer discernible (see Vernon, 1985, 1990). No instances of this extremely worked structure were seen in any of the examined artifacts.

When deformed copper is heated during annealing, the effects of cold working are reversed through a series of three processes. In the first stage, called recovery, dislocations are eliminated or redistributed by diffusion of copper atoms. Next, during recrystallization, new equiaxed grains nucleate and grow, consuming material from the distorted grains. Once the distorted grains have been replaced, larger new grains grow at the expense of smaller new grains, establishing the grain growth stage. All three of these processes serve to make the copper softer by removing barriers to dislocation motion. Additionally, faults in the stacking of

Table 1
Observations and measurements of the eight copper artifacts from Cahokia's Mound 34.

Artifact Number	Catalogue Number	Object Description	Dimension, *l x w x t* (mm)	Mass (g)	Microstructure Description	Mean Spatial Grain Diameter, $\overline{D}$ (μm)	Mean Knoop Hardness (kg_f mm^{-2})
1	34-59-31-3	Rough edges; heavily corroded; slightly dished shape	14 × 4.1 × 0.56	0.061	Equiaxed grain structure with blunt twins	81	N/A[a]
2	34-58-21-8	Small, flat sheet; rough edges	13 × 7.4 × 1.1	0.256	Equiaxed grain structure with blunt twins	90[b] (36–680)	77
3	34-61-18-5	Bent at 90° angle; 2–3 mm bend radius	15 × 7.4 × 0.66	0.299	Equiaxed grain structure with blunt twins	323	N/A[a]
4	34-55-21-8	Significant corrosion; appears to be separating into two sheets at one corner	12 × 11 × 1.5	0.896	Equiaxed grain structure with blunt twins	113[b] (49–540)	76
5	34-53-25-16	Small, flat sheet; cracked	12 × 8.6 × 0.79	0.184	Equiaxed grain structure with blunt twins	89	79
6	34-59-31-2	One straight and apparently cut edge with burrs; appears to be separating into two sheets at several places	25 × 17 × 2.4	3.42	Three distinct regions: equiaxed grains, heavy deformation bands near cut edge, groove created by selective etching along the center of the cross-section	Equiaxed: 189[b] (82–670) Deformed: 137 Groove: 64	Equiaxed: 83 Deformed: 118 Groove: 135
7	34-37-38-12	Triangular shape; surface shows smoothing; two straight edges display small burrs and appear to have been intentionally cut	22 × 20 × 1.8	3.81	Equiaxed grains with some deformation bands	328	99
8	34-74-7-pp.27	One straight and apparently cut edge with burrs; appears to be separating into two sheets at several places	34 × 14 × 2.3	4.94	Equiaxed grain structure with blunt twins	116[b] (39–410)	79

[a] Object was too thin for accurate hardness measurement.
[b] Wide Range grain size distribution, size range in parentheses.

crystallographic planes occur during recrystallization and grain growth, appearing as rectangular twin boundaries within grains called annealing twins. Copper that has been worked and then annealed can therefore be distinguished by equiaxed grains, annealing twins, and low hardness—these microstructural traits, visible in a micrograph from artifact 2 (Fig. 2A), characterize most of the cross-sectional area of artifacts 6 and 7, and the entirety of the observed sections from the remaining six artifacts (Table 1). Heavy working prior to the anneal results in smaller grains in the annealed material, since the higher number of defects provides more sites for nucleation during recrystallization. However, apart from this effect, annealing can be said to generally obscure evidence of previous manipulation in terms of grain morphology.

4.2. Artifact working process

The equiaxed, twinned grain structure (Fig. 2A) composing regions of artifacts 6 and 7, and all of the other six artifacts corresponds clearly to copper that has been worked and then annealed. Local variations in grain size, as in artifact 4 (Fig. 5), indicate differing amounts of deformation prior to the final anneal, which can be due to an uneven initial cross-section of the nugget or to uneven hammering. The second type of microstructure (Fig. 2B), seen on parts of artifacts 6 and 7, contained distorted grains and etched lines, identifiable as deformation bands by the presence of pitting (Fig. 2B, inset). This structure indicates areas of localized deformation, which must have been moderate in degree due to the lack of flow lines or dramatically elongated grains. Hardness data corroborates the identification of annealed and deformed regions. Table 1 shows that, for all artifact regions identified as having fully annealed structures, mean Knoop hardness is near a baseline of 80 kg_f mm^{-2}. The mean hardness of all sampled points on artifact 7, which showed light deformation, is ~100 kg_f mm^{-2}, and that of the apparently worked region of artifact 6 is ~120 kg_f mm^{-2}. The microstructures and hardness values indicate that all eight artifacts were annealed prior to being abandoned, and artifacts 6 and 7 were lightly to moderately worked after annealing. Both objects contained deformed material associated with a cut edge, and artifact 7 also showed evidence of working near a tapered area that appeared to have been hammered.

In order to describe the working practices at Cahokia's Mound 34, several factors must be considered. First, none of the artifacts were finished objects; all appeared to have been abandoned somewhere between being an unworked nugget and being a completed sheet or blank. Therefore, these artifacts only allowed the process of transforming a nugget into a sheet to be described, and provided no data about further finishing techniques. The microstructures showed that hammered artifacts were annealed at least once, but since only the most recent hammering and annealing cycle can be deduced from the microstructure, the total number of cycles used to reduce the artifacts from nuggets to their present thicknesses could not be directly determined.

Table 2
Measured grain sizes in native copper samples compressed to 75% height reduction and annealed under controlled conditions.

Anneal Temp. (°C)	Anneal Time (min.)	Mean Spatial Grain Diameter, $\overline{D}$ (μm)
500	15	N/A[a]
500	40	174[b] (40–511)
500	100	257[b] (64–600)
650	6	131[b] (57–970)
650	15	258[b] (91–980)
650	40	452
800	2	299
800	6	537
800	15	616

[a] Elongated grains precluded meaningful measurement.
[b] Wide Range grain size distribution, size range in parentheses.

Reductions of over 90% have been demonstrated on unannealed float copper through cold hammering alone (Schroeder and Ruhl, 1968; Clark and Purdy, 1982). However, achieving this would be extremely laborious, and the copper would be at a high risk of cracking. Repeated cycles of hammering to 30% reduction and then annealing were found to be much more practical. Furthermore, even though the artifacts differed widely in thickness and apparent degree of completion, all eight artifacts were left in a predominantly annealed state, with none showing more than light, localized deformation. This is strong evidence that annealing was conducted multiple times during the thinning process and that the artifacts therefore derive from a process characterized by repeated cycles of hammering and annealing.

Finally, since such a procedure was apparently used, most of the artifacts examined could potentially have been hammered and annealed for many subsequent cycles. The artifacts were in no way “used up” or “exhausted”. This suggests that they were not discarded because of degraded material properties. While it seems likely that some of the objects may have been by-products from trimming the margins of a larger sheet, others may have been abandoned for as-yet unclear reasons.

4.3. Annealing conditions

The purpose of the annealing experiment was both to select a standard annealing time and temperature for replication experiments and to identify a possible range of annealing times and temperatures to which the artifacts had been subjected. Exact determination of annealing conditions was not possible since only a single level of cold work (75%) was studied and since trace element concentrations were not measured for all artifacts and samples.

The wide range grain size distributions in the lower temperature annealed samples were not a product of variable prior deformation since large grains were evenly distributed in the cross-sections and deformation was uniform. Instead, a wide range distribution appeared to be related to the stages of recrystallization represented by samples annealed at 500 °C for 40 and 100 min and those annealed at 650 °C for 6 and 15 min, while longer or hotter annealing resulted in more uniform grains. The spatial grouping of extreme grain sizes in the Cahokian artifacts suggested that both uneven prior deformation and variable annealing conditions contributed to the wide range grain size distributions. It was not possible to directly match annealed experimental samples to artifacts since the artifacts mostly had grains smaller than any of the annealed samples. However, the presence of a wide range grain size distribution indicates that some artifacts had not been annealed sufficiently for grains to reach uniform size, suggesting an annealing temperature of 650 °C or lower. Additionally, the abundance of annealing twins in the artifacts and the lack thereof in all three samples annealed at 500 °C suggests that the annealing temperature of the artifacts must have been above 500 °C in order to permit the formation of annealing twins. While this is a rough estimate at best, annealing at 650 °C for around 10 min was found to produce a reasonable approximation of the microstructures seen in the artifacts and was accordingly used as the standard annealing condition for the bending and cutting replication experiments.

This annealing experiment bears on the artifacts’ processing histories in two ways. First, the observed artifact microstructures were shown to be achievable with an annealing temperature at least as low as 650 °C. Since open-pit wood fires can reach peak temperatures of 730 °C or more (Clark and Purdy, 1982), temperatures above those attainable in an open fire were not required. Second, the artifacts were likely annealed for times on the order of minutes to tens of minutes, rather than seconds or hours.

4.4. Bending

Neither in artifact 3 (Fig. 3A) nor the replicated sample (Fig. 3B) was the microstructure in the bent region distinguishable from an as-annealed microstructure. Introducing a 90° bend by hand in a 1 mm thick annealed copper sheet does not leave microstructural evidence. Therefore, it is not possible to determine whether artifact 3 was bent before or after being annealed.

4.5. Layering

The selectively etched groove on artifact 6 (Fig. 4) at first appeared to be evidence of intentional layering. However, SEM-EDX revealed the presence of arsenic in the grooved region. The 4.5 wt% arsenic concentration in the groove is similar to that of some arsenic bronzes, which are significantly harder than pure copper (Lechtman, 1996). This would explain the high Knoop hardness measured along the groove. Furthermore, the lack of arsenic in DCP-OES does not necessarily contradict the SEM-EDX results since the section sent for analysis was large enough that arsenic concentration for the whole section could still fall below the 100 ppm detection limit, even if the arsenic-containing band extended several hundred micrometers into it. Since arsenic is known to occur in native copper from Michigan (Hurst and Larson 1958)—sometimes with elevated concentrations in individual nuggets (Rapp et al., 2000)—and alloyed metal technology requiring melting was almost certainly unknown at Cahokia, the groove can confidently be attributed to a naturally-occurring, arsenic-rich heterogeneity that was flattened into a thin band when the artifact was hammered.

Another suspected indication of layering was planar oxide inclusions in artifact 4 (Fig. 5A) that appeared to have been trapped between two layered sheets. However, a second section from the same artifact (Fig. 5B) clearly showed that this was not the case. These planar features appeared to have instead been formed by the flattening of natural pores, inclusions, and surface knobs often found on native copper nuggets, a phenomenon also noted by Cobb and Evans (2009). These flattened features created the illusion of intentional layering both superficially, as apparent layer separation at corners visible with the naked eye, and in some polished cross-sections, in the form of planar oxide inclusions.

McPherron (1967) demonstrated that native copper sheets could not be welded by hammering at high temperature, due to rapid oxidation of the surfaces. Our replication experiments showed that oxidation also prevented welding by hammering two oxide-free copper sheets together and then annealing the composite sheet. While some level of adhesion was achieved between the pressed sheets before annealing, this was not considered to be successful layering as the stress applied to join the sheets rendered the copper much too hard and brittle to be shaped further without annealing. Therefore, it does not appear to be possible to weld native copper in open air through heat and hammering alone.

4.6. Cutting

Artifact 6 defined the three micrographic features of the cut artifacts: a blunt profile, distorted grains along the cut edge, and burrs. Even though three artifacts had remarkably similar superficial evidence of cutting, the micrographs from the edges were quite different (Fig. 6). The microstructure of artifact 7 was possibly altered near the cut edge by hammering that occurred after the final anneal. Artifact 8 contained only annealed grains at the edge, apparently having been fully annealed after the cut was made. Additionally, neither artifact 7 nor 8 was sectioned at a location where a burr was revealed in the cross-section, though burrs were clearly visible on all three artifacts.

All three defining features of the cut artifacts are clearly present in the replicated sample cut with fatigue bending, while the remaining three replicated samples display very dissimilar micrographs to the artifacts. Unequivocal identification of a technique through replication is not possible since an unlimited number of possible methods remain to be tested. However, the uncanny similarity between the fatigued sample and artifact 6 strongly suggests that this artifact, and possibly 7 and 8 as well, were cut using a fatigue-based technique, such as repeated bending or scoring and snapping.

5. Conclusion

These findings have characterized several aspects of the methods used to transform native copper nuggets into the thin sheet copper artifacts found at Cahokia's Mound 34. The realities of working native copper by hand and the largely annealed microstructures of all the artifacts, regardless of thickness or apparent completion, indicates that these artifacts were probably the product of a process in which larger pieces of native copper were reduced into thin sheets through repeated cycles of hammering and annealing. It was demonstrated that the annealing observed in the artifacts could have been accomplished by heating for a matter of minutes to temperatures obtainable in an open wood fire. Such an anneal was found to produce black copper (II) oxide powder residue. The use of this process would have allowed the artifacts to be reshaped through further hammering and annealing, but instead these artifacts were abandoned without further work.

The existence of the previously suggested practice of creating layered sheet was not supported by this study. The microstructures of the artifacts did not provide any sign of the joining of layers, though it was found that the flattening of natural features of the nuggets during hammering readily creates the illusion of intentional layering. Furthermore, it was confirmed that welding together layers of copper with compression and heat was not possible in air because of surface oxidation. Finally, it was also shown that, while a variety of cutting techniques may have been used at Cahokia, at least one edge was cut through repeated bending or a similarly fatigue-based method.

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