

**MINERALOGY MEETS ARCHAEOLOGY:
EXPERIMENTAL ARCHAOMETALLURGY OF BRONZE AGE SN AND CU SMELTING**

by

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Abstract

The main goal of the archaeometallurgical short course at the Butser Farm in Hampshire (UK), was to conduct experiments concerning Bronze Age Sn and Cu smelting techniques. In this context, three different smelting experiments were carried out. In the first experiment a shaft furnace was used to smelt ca. 1000 g cassiterite. The output of the experiment was only 150 g of metallic Sn since high furnace temperatures of $\gg 500^\circ\text{C}$ led to the vaporization of liquid Sn during smelting. Temperatures above 1200°C were easily reached with a bag bellow. The second experiment was carried out in the same furnace with a slightly different design. In this experiment ca. 1000 g of malachite were smelted. The output of 425 g of metallic Cu was relative high. In both experiments very low charcoal was used. This fact can be explained with the furnace design because the shaft was relatively high (80 cm in experiment 1, slightly less high in experiment 2) and had a small diameter of 20 cm. The third experiment was designed to use a bowl furnace for simultaneous Cu (malachite) and Sn (cassiterite) smelting. This furnace-type was characterized by high charcoal consumptions, low temperatures and high oxygen fugacities. This experimental setup produced neither metallic Cu nor metallic Sn. Mineralogical investigations of the Cu experiments yielded the assemblage glass + olivine + spinel + feldspar \pm metal drops in the slags. Based on the occurrence of Cu metal and/or cuprite (Cu_2O) and the composition of the metals variations in the oxidation/reduction conditions could be deduced within the furnace assemblages. The uppermost part of the slags contained no metallic Cu inclusions but large amounts of cuprite (Cu_2O), which indicates oxidizing conditions. Deep in the furnace near the bottom, Cu is reduced to metallic Cu which appears as large agglomerations as well as small droplets in the oxide slags. Therefore these mineralogical observations provide informations about the efficiency of the furnaces used.

Introduction

Experimental archaeology represents the intersection between archaeology and natural sciences. Experiments are an indispensable tool when interpreting archaeological remains as well as when it comes to reconstructing human technologies.

Experimental results can help to interpret archaeological and scientific observations and thus allow establishing or abolishing certain ideas (BELL & HOSFIELD, 2009). A short course dealing with experimental archaeology, held at the Butser Ancient Farm in Hampshire, UK, was attended by the senior author and provided the basis for these archaeometallurgical investigations. The main goal was to familiarize participants with experimental techniques in archaeology, the organization and design of specific metallurgical experiments, the interpretation of the obtained experimental products and a discussion concerning arising problems in the course of the experiments. In order to achieve these goals field-based metal smelting experiments together with short theoretical talks and discussions were carried out (CRADDOCK & TIMBERLAKE 2005). The main attention was turned to prehistoric mining and smelting on the British Isles. At the beginning of every experiment, archaeological evidence and information from related sciences should be gained. In the case of early metal mining and smelting it is necessary that besides archaeological evidence contemporaneous mineralogical investigations and mining site explorations are required in order to obtain the maximal input information for the experimental design. The most important ore minerals of the British Isles used in Prehistory were Cu-carbonates such as malachite ($\text{Cu}_2[(\text{OH})_2|\text{CO}_3]$), Cu-sulfides such as chalcopyrite (CuFeS_2) and Sn-oxides such as cassiterite (SnO_2) (TIMBERLAKE, 2007). Before starting the construction of the smelting furnaces, some basic questions have to be considered:

Which metal should be produced?

It is important to know which raw material depending on the archaeological/geological context we can use and from where the material can be obtained. The most realistic reproduction can be obtained by smelting ores which were mined in the studied area. For instance TIMBERLAKE (2007) collected raw ore for malachite-azurite copper smelting experiments from a vein which was exploited in prehistoric times. In nature ores rarely occur pure and hence they are a mixture of ore minerals, gangue minerals and host rock. Smelting of this mixture yields products such as metals and slags which have a defined mineral assemblage (and hence chemical composition) which is typical for a specific deposit. Therefore the furnace design has to be adapted to the ore used for smelting.

How can metal be extracted from oxide- and sulfide minerals?

Extracting Cu from oxide- and carbonate minerals is the simplest way to produce metal. This process involves the reduction of Cu-oxides according to the model reaction (METTEN, 2003):



Thus the oxygen fugacity in the furnace is of particular interest. The most important furnace variable though is the temperature which is responsible for the viscosity of the coexisting oxide melt (the subsequent slag) and the metal melt. A low viscosity of an oxide melt favors the agglomeration of small metal droplets into one large metal aggregate. For instance the reduction of Sn oxides to metal is more complicated due to the boiling and evaporation of metallic Sn at relatively low temperatures ($> 500^\circ\text{C}$). This fact requires a special furnace design in order to remove the metallic, molten Sn from the hottest zone of the furnace.

Sulfide ores are even more complicated to smelt. In a first step S₂ (sulfur) has to be removed from the system. This happens by “roasting”, which represents the oxidation of sulfide minerals, such as chalcopyrite, and leads to the formation of Cu- and Fe oxides as shown by reactions (1) and (2) (METTEN, 2003).



and



This process takes place at high firing temperatures and high oxygen fugacity. However it is currently not clear how sulfides were roasted in prehistoric times. The Cu- and Fe oxides produced by this process have to be reduced in a second step (reaction 1). The main problem that arises when smelting chalcopyrite is the production of Fe-free, metallic Cu. The control of oxygen fugacity in the furnace is therefore of fundamental importance. Too low fugacities lead to the reduction of Fe to metallic Fe so that a Cu-Fe alloy will be produced. Too high oxygen fugacities lead to the oxidation of Cu and thus no metal will be produced. Hence it is important that the oxygen fugacity is adjusted below the Cu oxidation reaction



and above the Fe reduction reactions



and



(O’NEILL, 1988). In this context, Si, present as SiO₂, can be used to flux the Fe-oxides by adding quartz to the experiments according to the following reaction (METTEN, 2003; O’NEILL, 1988):



Which smelting tools are necessary for the experiments?

In addition to the understanding of the physical and chemical processes involved in smelting, archaeological finds in context of metal smelting are very important for the reconstruction and replication of prehistoric metal smelting processes. Ore smelting can be carried out in different ways. If one chooses some kind of bank furnace with high chimneys some kind of tuyère for firing is needed (TIMBERLAKE, 2007). For metal production high temperatures in excess of 1100°C are fundamental. This can not be achieved with normal firing and requires a system that allows air circulation within the furnace.

Tuyères are made of ceramics and represent the interface between the inside of the furnace chamber and some kind of bag bellows on the outside (CRADDOCK & TIMBERLAKE, 2005). Ceramic collecting pots are inserted at the bottom of the furnace in order to “collect” the molten metal. If one uses blow pipes for firing, the bag bellows and tuyères can be replaced by human lungs, bamboo pipes and smaller kinds of tuyères, which protect the bamboo pipe from burning near the furnace chamber (TIMBERLAKE, 2007). A further possibility is crucible smelting, where the ore and charcoal are filled into a ceramic crucible and closed with a ceramic lid. If conserved, pottery (tuyère, collecting pots and crucibles) and hardware (bag bellows) should be replicates of archaeological finds (CRADDOCK & TIMBERLAKE, 2005). However this is rarely the case and in the case that smelting pottery and particularly bag bellows do not exist, which is the most common case, etymological studies provide the basis for the experimental construction of these devices and thus can be seen as an interpretation of how these artifacts could be designed (BROWN, 1995).

How should the furnace be constructed?

Which furnace design fits the archaeological evidence? The furnace design is of fundamental importance in experimental archaeometallurgy. The archaeological field evidence of furnaces is in most cases scarce or not present at all. Even if relics are preserved the real design only represents an interpretation. The design is crucially for the interplay between the two important variables, temperature and atmospheric conditions. Therefore, several furnace designs were discussed and built during the short course, namely the bowl furnace, the post-hole furnace, and the earth bank shaft furnace (TIMBERLAKE, 2007). The furnace types are described in the following chapter.

What to do with metal?

Metal was an important material, which was used for different technical as well as decorative purposes. Casting of metal is an important procedure for the production of artefacts. This leads to questions concerning the materials used and the characteristics of the casting moulds and technologies (TIMBERLAKE, 2007).

In order to answer some of the questions above, during the short course, three groups, each consisting of two people performed different smelting experiments. The first experimental step was the production of pottery for metallurgical purposes. Each group produced a small number of tuyères, crucibles, lids, collecting pots and casting moulds (Figure 1). The raw material for the pottery was London clay, mixed with sand, sheep dung and straw. This mixture should assure tenacity even at high temperatures. However the used clay was of bad quality due to high Fe contents, so that after drying and burning most products were cracked. Therefore it was recommended to use other clays for better pottery quality for future experiments.

The simplest way to ventilate fire in a furnace is using blow pipes, but bellows are more comfortable and secure. Two possible construction principles are possible, namely bowl bellows and bag bellows. The design of the two types can be highly variable (BROWN, 1995). Prehistoric archaeological evidence concerning bellows is almost unknown. However it is reasonable to expect that bellows were used. It was decided to construct one bag bellow for each group (Figures 2a-b). The bag bellow was reconstructed after Craddock and TIMBERLAKE (2005).



Figure 1

a) Mixing of the ceramic raw materials for the production of metallurgical equipment. Ingredients used: clay from a local pit, straw, sheep dung, and sand. b) Finished crucible with a clay lid and filled with raw ore consisting of malachite and chalcopryite and charcoal powder ready for the experiment. c) Setup of the ceramic equipment before firing. d) The same setup after the firing process in an open bonfire.

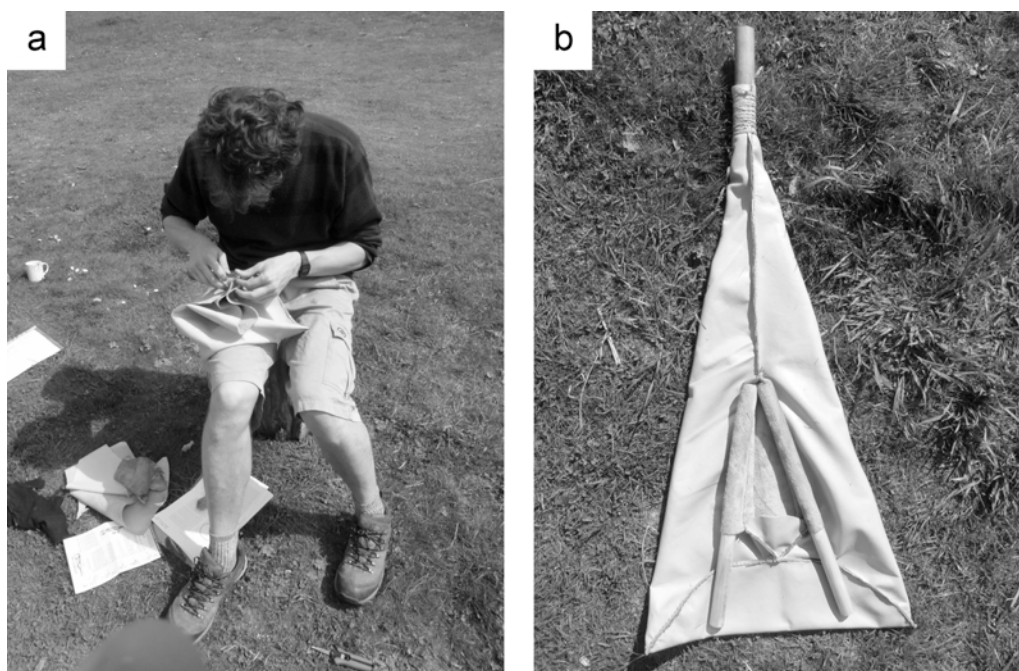


Figure 2
 a) Sewing a bag bellow made of cow leather. b) A finished bag bellow.

Experiments

Experimental practice and outcomes were further discussed based on the investigations of CRADDOCK & TIMBERLAKE (2005), TIMBERLAKE (2007) and CRADDOCK et al. (2007).

Experiment 1

The goal of the first experiment was to smelt Sn in a bank furnace. As mentioned above, metallic Sn is strongly volatile at high temperatures. The furnace was designed to produce low (500-600°C) temperatures at the bottom, where molten, metallic Sn can accumulate without undergoing strong evaporation. For this reason, the tuyère was mounted a little bit higher from the bottom (at a 1/4 of the furnace height from the bottom). At the bottom, a sealed clay tube was inserted to tap the metallic Sn. Three thermocouples were inserted to measure the temperature at the top, near the tuyère and at the bottom of the furnace. The furnace was dug into a natural earth bank. The shaft and bottom were sealed by a double layer clay smear.

As raw material 1006 g impure cassiterite with approximately 60 wt.% SnO₂ was used. The fine-milled powder was mixed with charcoal, sheep dung and water to form small Sn balls. This yielded 14 Sn balls with approximately 100 g weight. Then the bag bellow was mounted in front of the tuyère and the bamboo nozzles were fixed at the base to avoid any movement and stress on the tuyère. The furnace was fired and filled with charcoal and after 14 minutes 1100°C were reached near the tuyère. After 20 minutes 7 Sn balls were charged, followed by the other seven Sn balls 20 minutes later. After 2 hours and 11 minutes the Sn was tapped, however no metallic Sn flowed out. The metallic Sn was trapped in the oxide slag, and after milling the slag, 150 g of metallic Sn droplets could be collected.

The charcoal consumption was low and did not exceed 6 kg (4 buckets) for the whole experiment. The temporal temperature development in the furnace is shown in Figure 3. It can be seen that temperatures in excess of 1000°C were reached immediately after filling the furnace with charcoal (ca. 15 minutes after firing the furnace). The high bottom temperatures at the beginning can be explained with the starting procedure of firing, where in the furnace a small “bonfire” was set. After filling charcoal into the furnace, temperatures sank below 500°C to rise again after 40 minutes to over 700°C. This peak shows a clear negative correlation with the temperature near the tuyère. This effect probably represents the beginning of cassiterite smelting where cold material (cassiterite balls) came from the top of the shaft towards the high-T region near the tuyère. Afterwards, the bottom temperature sank slightly below 700°C. However bottom-T measurements were taken slightly above the “real” bottom of the furnace so that slightly lower temperatures had to be expected near the clay tube at the bottom where metallic Sn should accumulate.

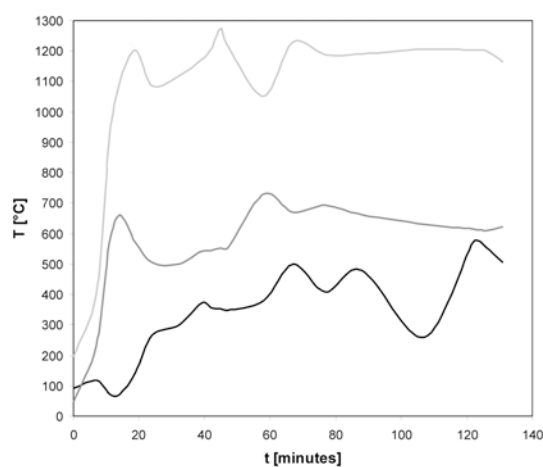


Figure 3
The temperature development in experiment 1. The bright grey curve is the temperature near the tuyère (TC2), the grey curve corresponds to the bottom temperature (TC3), the black curve represents the temperature at the top (TC1).

Experiment 2

The cassiterite smelting furnace was re-activated for a second experiment but the design was completely changed. The aim of this experiment was to smelt malachite in a bank furnace. The design changes affected the bottom of the furnace where a collecting pot was installed. The tuyère was mounted onto a cone, so that it was possible to change the angle of the tuyère relative to the furnace shaft. Cone and tuyère were mounted as close as possible to the bottom, in the vicinity of the collecting pot, in order to obtain the highest temperatures. The primary clay lining inside the furnace was repaired. Four minutes after starting up the furnace, temperatures in excess of 1200°C were already reached. The raw material was 1000 g of coarsely grinded malachite gravel (African malachite). After reaching smelting temperatures, 500 g of malachite were added in intervals of 20 minutes. Bluish flames at the top of the furnace indicated carbon oxide burning and thus highly reducing conditions. The experiment lasted 2 hours and 36 minutes and afterwards the furnace cooled down during the night. The highest reached temperatures near the tuyère were > 1370°C which even led to a burnout of the thermocouple. The overall charcoal consumption was also very low, only ca. 4.5 kg charcoal were used (3 buckets). After opening the furnace the next day (Figure 4a), no metallic Cu was found in the collecting pot at the bottom but below the input of the cone, a massive metallic Cu agglomeration was found instead (Figure 4b). Above the position of the cone small amounts of glassy slag were found. A total of 425 g of metallic Cu were produced in this experiment.

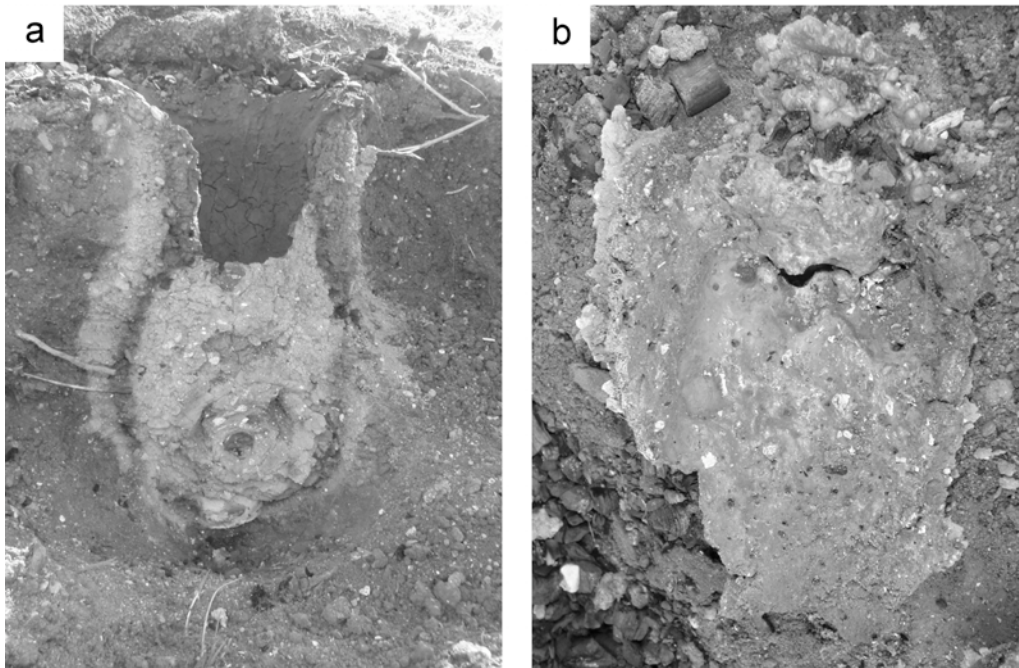


Figure 4

a) Cross-section of the furnace after smelting. The slagged cone is easily visible.

b) Extracted slag from experiment 2. At the top of the picture a massive agglomeration of metallic copper is visible.

Experiment 3

This experiment was completely different from the experiments described above. The bank shaft furnace was abandoned and a simple bowl furnace was constructed. With this design three simultaneous experiments could be carried out. The aim of these smelting experiments was to compare the thermal and atmospheric behavior between bank shaft- and bowl furnaces as well as the comparison between different Sn smelting techniques. As raw materials, cassiterite of good- and bad quality was used. In the first crucible 80 g of fine-milled cassiterite (good quality) and charcoal were mixed and sealed with a clay lid. The second crucible was filled with 80 g of fine-milled cassiterite (bad quality) and charcoal and sealed with a clay lid as well. Although the two experiments contain the same mass of raw material, they should show a difference in their metal output since different quality raw materials were used. Bad quality cassiterite consisted of approximately 60-70 wt.% SnO₂. A third charge was loaded for comparison, using the experimental set-up of the shaft furnace from experiment 1. This charge consisted of 300 g of fine-milled cassiterite (good quality), which was again mixed with sheep dung, charcoal and water to form four Sn balls. The evaporation of metallic Sn at high temperatures posed no problem since crucibles with sealed lids were used. In order to minimize this effect for the Sn ball experiment, the bottom of the bowl furnace was shaped in a way that molten metallic Sn could flow below the clay seal into a region where the soil was much cooler. The balls and the crucibles were arranged in the bowl near the input of the tuyère to obtain the highest smelting temperatures.

Near this position the thermocouple was then mounted. During this experiment some interesting observations were observed. Since the temperature difference between the tuyère (hottest zone) and the backside of the crucibles was large, a temperature gradient within the crucibles was expected. Smelting temperatures > 1100°C could only be kept by intense pumping with the bellows. The charcoal consumption was very high and after only one hour two wheel barrows of charcoal were already consumed. The metal production of the experiment suffered from too high oxygen fugacities, which prohibited the reduction of Sn. Unfortunately, due to tight time constraints, alloying and casting of Cu, Sn and bronze from the experiments 1 and 2 was not possible.

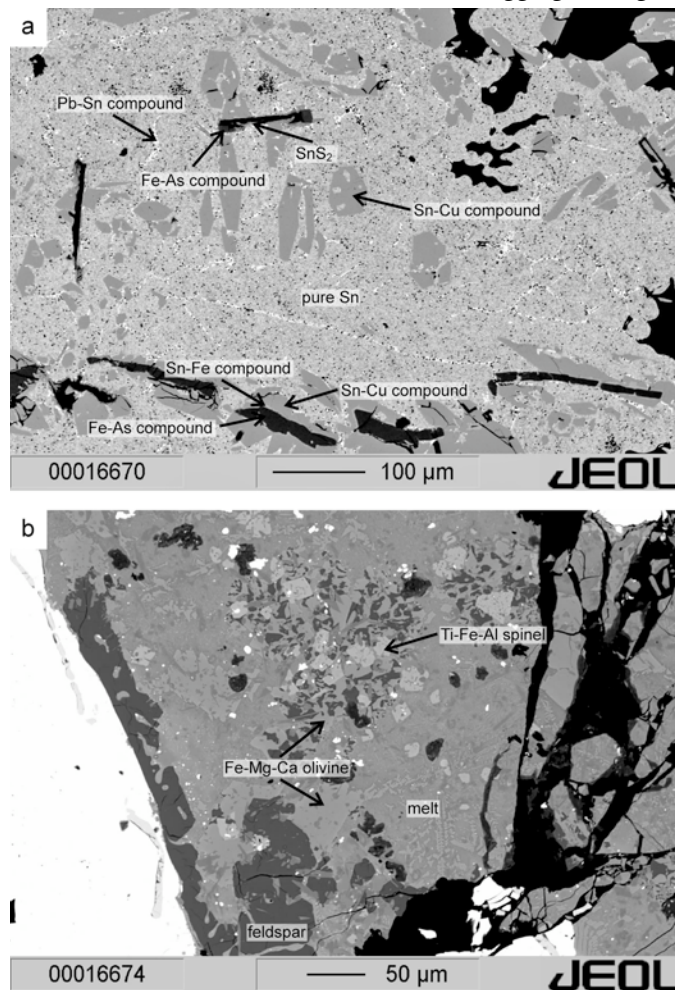
Results

Mineralogical observations in the experimental run products

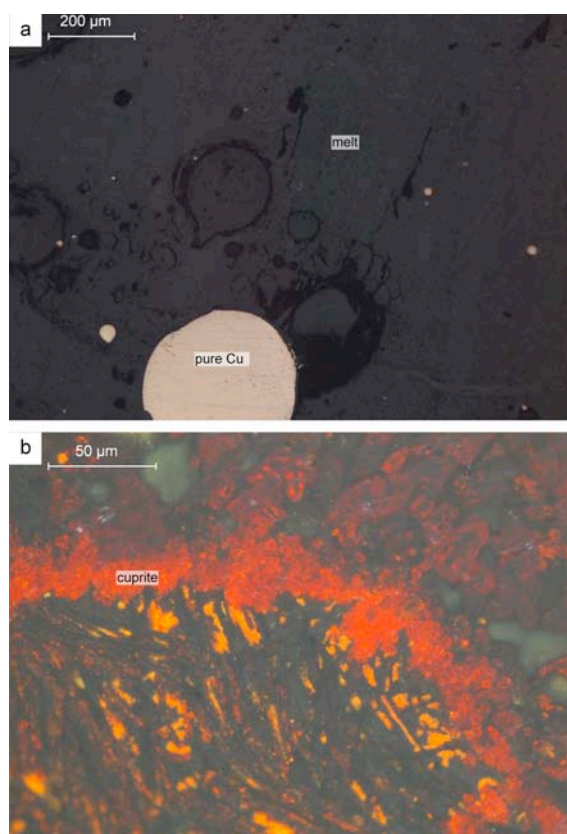
The Sn smelting experiment 1 in the earth bank shaft furnace produced impure metallic Sn. The chemical system can be described in the system Sn-Cu-Pb-As-Fe. However the largest part of the metallic Sn droplets consisted of metallic Sn with inclusions of a Cu-Sn aggregate (Figure 5a). Within the metallic Sn, small, dispersed veins of a Pb-Sn compound were visible. Within the cores of the Cu-Sn aggregates, Fe-As and Fe-Sn compounds were observed (Figure 5a). Oxide slags which host metallic Sn-droplets consisted of glassy melt, spinel, olivine and feldspar. EDS (energy-dispersive) electron microprobe analysis revealed that spinel consists mainly of Ti, Fe and Al. Olivine is primarily a forsterite-fayalite solid solution, but also contains small amounts of Ca (monticellite-kirschsteinite solid solution). Feldspar was a ternary Ca-Na-K solid solution (Figure 5b).

Figure 5

a) Backscattered electron image of a Sn droplet of experiment 1 (bad cassiterite quality). b) Backscattered electron image of an oxidic slag of experiment 1 (bad cassiterite quality).



Based on the mineralogy and the composition of the metals and slags from the Cu smelting experiments different oxidation/reduction condition could be deduced. The uppermost part of the slag contained no metallic Cu inclusions but large amounts of cuprite (Cu_2O), which indicates oxidizing conditions above the Cu oxidation reaction (4). Deep in the furnace near the bottom, Cu is reduced to metallic Cu which appears as large agglomerations as well as small droplets in the oxide slags. This glassy slag does not contain any Cu-oxide phases and hence reducing conditions below reaction (4) prevail. Polished samples of slags and metals obtained from



experiment 2 (Figure 6a and 6b) also show two distinct zones with respect to f_{O_2} . The first zone is represented by a glassy slag and metallic Cu droplets (Figure 6a) and represents a highly reducing environment, the second zone is characterized by a glassy slag without any metallic Cu droplets but with lots of cuprite crystals which represents again an oxidizing environment (Figure 6b). While the first zone can be localized slightly below the tuyère the second zone is above the air-flow entrance of the tuyère.

Figure 6

a) Microphotograph of the reducing part of the malachite smelting experiment 2 containing a drop of metallic Cu. b) Microphotograph of the oxidized portion of the slag where Cu_2O occurs.

Interpretation of the experimental results

The preparation and the results of these experiments revealed the complexities concerning metal smelting and the reproduction of prehistoric techniques. 1) The pottery suffered from improper clay. 2) The resulting metal products of experiments 1 and 2 were satisfying; however the furnace in experiment 1 was not running correctly, since no Sn flowed out at the bottom, indicating non-ideal temperature gradients. The bottom design of the furnace of experiment 2 was also not ideal. Metallic Cu did not flow into the collecting pot, but produced a large agglomeration between the tuyère and collecting pot instead. 3) Experiment 3 suffered from too high oxygen fugacities and thus no reduction occurred and 4) due to insufficient time alloying and casting of the metallic products was not possible. Despite these complications, problems and limits it was possible to illustrate the possibilities and the importance of experimental archaeology for a better understanding of prehistoric smelting techniques.

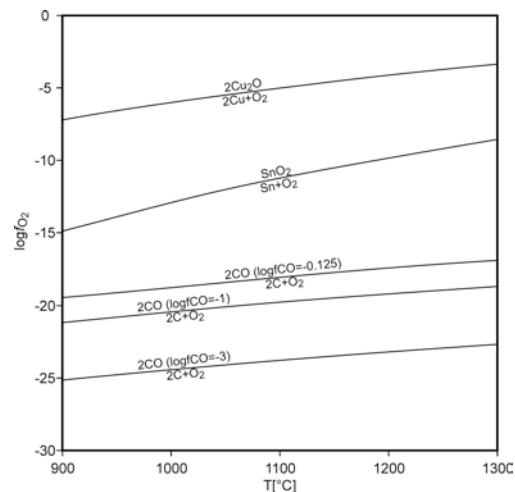
The comparison between the shaft furnace and the bowl furnace clearly show the advantage of the small, high and isolating shaft design over the large, bulky design of the bowl furnace since: 1) the charcoal consumption was very low and 2) temperature was much more evenly distributed and the highest temperatures were reached without excessive bag bellow pumping. The mineralogical observations of experiment 1 show highly reducing conditions below the



reaction (Figure 7). This indicates an oxygen fugacity of -10 ($\log f_{\text{O}_2}$ at 1200°C) when calculating reaction (8) using the thermodynamic data of ROBIE & HEMINGWAY (1995). The reduction of cassiterite to metallic Sn follows the reaction



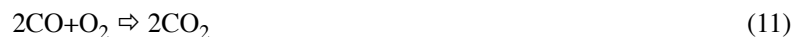
Figure 7
T-log f_{O_2} diagram showing the position of important oxidation reactions in the Sn and Cu system as discussed in the text. The reactions were calculated using the thermodynamic data of ROBIE & HEMINGWAY (1995). The production rate of carbon oxide molecules strongly depends on the presence of carbon (assumed to be in excess for metal smelting) and oxygen as illustrated by varying $\log f_{\text{CO}}$.



The occurrence of carbon oxide (CO) results from the presence of carbon (C) in charcoal, which acts as fuel and as reduction source and oxygen (O_2), which is put into the system by pumping the bag bellow. The by-products of charcoal consumption are thus CO and CO_2 , where CO reduces the ores to form metals. The following equations therefore depend on the oxygen-, carbon oxide- and carbon dioxide fugacities (Figure 7):



and



Too high oxygen fugacities lead to a complete oxidation of CO (reaction 11) so that the reduction of cassiterite is impossible and/or already reduced metallic Sn becomes oxidized and forms cassiterite again. In the experiment Sn oxide compounds were never found.

As mentioned before the malachite smelting experiment can be subdivided into a more reducing and a more oxidizing zone. The oxidizing zone, without metallic copper droplets, can be attributed to high oxygen fugacities above -4 ($\log f_{O_2}$ at 1200°C) (ROBIE & HEMINGWAY, 1995) while the zone with lots of metal droplets is below this value. The reduction of Cu oxides and/or decomposed Cu carbonates (malachite) follows the reactions:



and



It is expected that malachite, ($\text{Cu}_2[(\text{OH})_2\text{CO}_3]$), decomposes and dehydrates by “sinking down” the shaft of the furnace with increasing temperatures. Therefore in the hottest zone no or only relict malachite should be present. In this region the largest part consists of cuprite (Cu_2O) and if oxygen fugacity is very high, tenorite (CuO). The appearance of Cu in an oxidized state in the area above the air flow of the tuyère suggests that in this region the highly oxidizing air flow creates atmospheric conditions which are not suitable for the formation of metallic Cu. But immediately below this zone Cu is present as metal. This zone is also characterized by lower temperatures. At the bottom of the shaft temperatures were clearly below the Cu melting point of ~1100°C and no molten, metallic Cu flowed into the collecting pot on the bottom.

Limitations of experimental archaeology in recreating prehistoric technologies

Experimental archaeology has become very important in the interpretation of archaeological records and artefacts and the reconstruction of prehistoric technologies. The experimental results can be compared with archaeological evidences and thus allow to reconstruct an overall picture of past human activities and habits. Experimental archaeology is also important when it comes to abandon certain views and ideas that did not work out. The band width of experiments spans the whole field of archaeological sciences. However experimental archaeology is only as good as its input and outcome are questioned and critically discussed. Despite its importance it is limited by several factors in a way that the single factors are interrelated so that one limitation influences another. For example, if a prehistoric smelting site was excavated and fragments of furnaces, fireplaces, hearths, tools and slag and metal artifacts were recovered (in many cases much less is recorded), the reconstruction of part of the whole smelting operation can be carried out by experimental smelting. But several problems arise due to the following limitations:

1) The most important limitation is the experimentalist itself. Scientists dealing with prehistoric technologies, although familiar with the matter, are modern human individuals, which live in a modern world and are influenced by modern thinking and ideas. This means he is familiar with physical, chemical and technological knowledge, with which prehistoric humans were not. It is very difficult to abandon our modern thinking and knowledge. Therefore the reconstruction and completion of archaeological relicts and artifacts as well as technologies point in a outcome which probably does not represent the real prehistoric situation.

2) Smelting activities were always related to mining activities, which raises questions concerning the provenance of the raw material. In most cases local deposits were probably exploited. However a prehistoric smelter could have obtained ores from other deposits or the sought minerals of the local deposits change during times due to intensive exploitation.

3) Prehistoric miners probably obtained mostly ores from the Earth's surface which are in many cases no more preserved and thus cannot be accessed by archaeometallurgists.

4. The initial treatment of the ore is hard to reconstruct, due to the absence of relevant artefacts and it also strongly depends on the type of ore (sulfides or oxides). For instance, if sulfide ores were smelted, roasting was necessary. Does the archaeological record of a roasting bed exist, is it not preserved anymore, or did they not use any roasting bed? This question is strongly related to the raw material question.

5) The next complications arise when it comes to the assessment the furnace design. Furnaces are rarely found and in most cases badly preserved. If smelting was carried out in crucibles, the evidence is not much better. The furnace design not only governs the temperature distribution in the furnace, which is directly connected with the tuyère position but also the atmospheric conditions in the furnace such as the oxygen fugacity. According to these fundamental variables the composition and the output of metals and slags is closely related to these variables. Therefore quantification of temperatures and oxygen fugacity during slag formation can be carried out by petrological investigations of slags and metal, using relevant phase diagrams. These mineralogical data in turn provide valuable informations about the efficiency of a furnace, e.g. the smelting process.

Overall, the experimental investigations in combination with mineralogical investigations strongly push archaeological sciences forward in their goal of reconstructing prehistoric processes but one needs to bear in mind that the obtained answers are usually far from being definitive!

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