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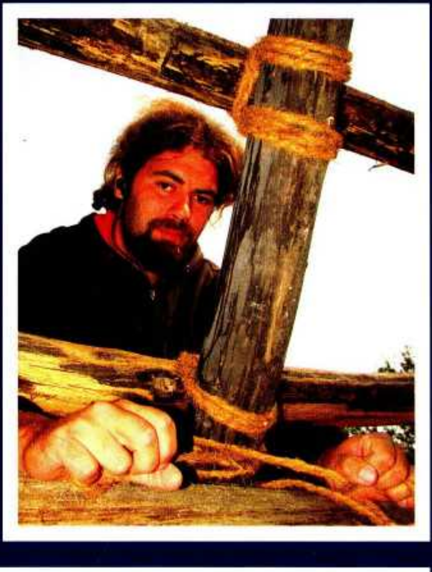

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Practical challenges of archaeometallurgy of gold found in the Thracian gold mine at Ada Tepe, Southeast Bulgaria – Analytical approaches and experimental reconstructions

Ruslan Stoychev, Petya Penkova, Margarita Grozeva

Zusammenfassung – Praktische Herausforderungen und Archäometallurgie des Golds vom thrakischen Goldbergwerk Ada Tepe, Südostbulgarien. Analytisches Herangehen und experimentelle Rekonstruktionen. *Das Grundziel des vorliegenden Berichtes ist die Darstellung der Ergebnisse einer durchgeführten Verhüttung von Goldkonzentrat. Das Konzentrat wurde experimentell von einem Quarzgang in dem in den letzten Jahren ausgegrabenen spätbronzezeitlichen und ältereisenzeitlichen Goldbergwerk am Ada Tepe, Südostbulgarien, gewonnen. Für die Rekonstruktion der Methoden, Werkzeuge und Verhüttungstechniken wurden Auskünfte von den antiken und mittelalterlichen Autoren (Diodorus, Plinius und Agricola) genutzt. Als Informationsquellen und Parallelen wurden zusätzlich auch Abbildungen von griechischer rotfiguriger Keramik und ägyptischen Wandmalereien der hellenistischen Zeit genommen. Im Gang der Forschung sind alle aufeinanderfolgenden Schritte der Vorbereitung des Experiments beschrieben. Die ersten sind mit der Herstellung der notwendigen Werkzeuge und Anlagen verbunden (Tondüsen, Gussformen, Verhüttungsstelle). Eine besondere Aufmerksamkeit ist der Physik des Verhüttungsprozesses gewidmet. Es wurden Vergleiche zwischen der im Rahmen des Experiments gewonnenen Goldschmelze und der in einem spätbronzezeitlichen Befund freigelegten angestellt. Die chemischen Analysen der beiden Goldschmelzen werden in Tabellen verglichen. Hier sind auch die Analysen einer bikonischen spätbronzezeitlichen Goldperle von Ada Tepe vorgestellt. Schließlich sind in der Diskussion Fragestellungen über die Erforschung und Rekonstruktion der Kenntnisse der alten Bergleute und Metallurgen kommentiert.*

Introduction

The archaeological investigation of Ada Tepe Site – results and questions

Ada Tepe is a hill in the eastern Rhodope Mountains with a height of 495 m. It is located 4 km south of Krumovgrad, a town in southeastern Bulgaria. Archaeological excavations at Ada Tepe began in 2001 when remains of an oval stone wall, enclosed hearths and signs of destroyed dwellings were discovered around the crest of the hill. As a result of subsequent excavations carried out over the period 2001-2006, the site was interpreted as a sanctuary or a “Thracian cult place”, dating from the end of 2nd to the middle of 1st millennium B. C. (Nekhrizov, Mikov 2002, 42-44; Nekhrizov 2006, 140-142).

In 2005, an ancient underground ore mine (gallery) 14 meters in length together with an associated dump was investigated on the southwestern slope of the hill. It was determined by researchers to have been created by prospectors searching for gold ore during the Late Bronze Age (POPOV, ILIEV 2006, 154-156). The discoveries of 2005 changed the main interpretation of the site and initiated a methodology of research for Ada Tepe as an open cast mine dating to the Late Bronze to Early Iron Age (JOCKENHÖVEL, POPOV 2008, 251-269). Within a framework of five contiguous campaigns (2009-2013), a Bulgarian-German team registered and researched numerous structures connected with the ancient gold mining of Ada Tepe – from ore extraction of varying shapes and depth, dumps from ore processing, work stations for the separation of the extracted material, to areas connected with different types of habitation (POPOV, JOCKENHÖVEL 2010, 265-281; POPOV, JOCKENHÖVEL, GROOR 2011, 167-186).

The results of the excavations allow us to reconstruct the development of the gold extraction process, which took place at Ada Tepe. Apart from details such as the level of technical skill employed by the ancient miners, research revealed information on a whole range of activities, as well as new dates for the organization of work process and its phases (chaîne opèratoire). However, it also raised some new questions (POPOV, JOCKENHÖVEL 2010, 265-281). Perhaps the most interesting questions are connected to the processes that followed the extraction of gold ore: how was the gold separated from the crushed rock? What practices and tools were used for this and in which order? How was the raw metal created, what were the characteristics of gold metallurgy in general, and what kind of archaeological structures were connected with the whole process? Unfortunately, there was little evidence found in the field to provide clear answers to these questions, although objects such as pots, moulds, and gold slag were found, indicating that gold smelting was carried out here. Therefore, in a bid to provide some more answers, the excavator team carried out a field experiment in 2012. The purpose of this paper is to present the preliminary results of this experiment of melting gold concentrate obtained from ore excavated on Ada Tepe ore mine.

Specific characteristics of the Ada Tepe ore deposit

A comparative analysis between the archaeological investigations and the geological information indicated that the extraction and processing of gold ore from Ada Tepe were strongly connected with the geological characteristics of the gold ore deposit. For this reason, we would like to present information solely related to the elemental composition of the ore in both the centre and the periphery of the deposit, because the chemical composition of the ore is a major factor influencing work operations and the whole metallurgical process.

The Ada Tepe sector of the Chan Krum gold deposit has been systematically studied since 1953 (JELEV ET AL. 2007, 104 with bibliography). The gold in the deposit is present as electrum. It forms fine impregnations, gold emulsions, micro masses, single small gold grains and aggregates inclusions embedded in a quartz mineralization. Morphological and granulometric analyses show that the small gold grains decreased according to depth. In his geological report, Danko Jelev, one of the leading geologists in the region of Ada Tepe, provides information about the elemental composition of the gold deposit (JELEV ET AL. 2007, 104-115). Interestingly, there is a higher gold concentration and a respectively lower

Elements	Au Weight %	Ag Weight %	Cu Weight %	Fe Weight %
Native gold (Centre)	82,2	16,76	0,44	0,19
Native gold (Periphery)	81,7	17,52	0,2	0,08
Native gold (in carbonate)	82,8	16,72	0,49	0,01

Tab. 1: XRF analysis of electrum from greater depths (after Jelev et al. 2007). – XRF-Analyse des Elektrums in der Tiefe (nach Jelev et al. 2007).

№ analyze	Au Weight %	Ag Weight %	Cu Weight %	Fe Weight %
1	71,13	27,33	1,08	0
2	66,28	24,98	0,97	7,45
3	70,40	27,68	0,59	0,82
4	68,91	26,93	0,53	3,29
5	72,45	25,74	0,71	0
6	71,70	26,67	0,39	0
7	**68,11**	**29,97**	**0,58**	**0**
8	67,23	31,27	0,54	0

Tab. 2: XRF analysis of electrum from the surface (after Jelev et al. 2007). – XRF-Analyse des Elektrums von der Oberfläche (nach Jelev et al. 2007).

silver content in the veins found deep underground (*Tab. 1*).
A slightly higher silver concentration with respect to gold content was found in the veins closer to the surface (Jelev et al. 2007, 109-110) (*Tab. 2*).

Ancient metallurgy: sources reviewed

A precise description of the metallurgical process of gold smelting is absent in ancient literary sources, although some information is provided by Diodorus, who gives us an idea of the techniques and materials used in melting gold in Ancient Egypt (Diodorus. III.14). At present, the best evidence for the reconstruction of ancient metallurgical techniques are the observations of the tools and structures discovered in archaeological contexts, the analysis of representations of metallurgical scenes on ancient pottery[1], and contemporary attempts to reconstruct the processes. Taken together, these various elements can be used build up a picture of ancient metallurgical operations.

Equipment and tools

The simple ancient furnace for metal smelting is a cylindrical construction made from stones and clay with dimensions between 0.30 to 0.60 m inner diameter, and a height of 2-3 m (Craddock 1995, 169-174). Strabo described even bigger constructions, which served for the processing of silver ore (Strabo. 3. 2. 8). On the basis of that information and the discoveries in Laurion, G. Conophagos suggested that some furnaces could be 3 to 4 meters in height (Conophagos 1980, 289). But there are still problems determining the right forms of early furnaces. Experiments have shown that very often these structures did not survive the smelting process (CRADDOCK 1995, 169). Preserved fragments of furnaces discovered during archaeological investigations are mostly preserved only at their base (STOYANOV, MIHAILOVA 1996, 59-60, fig. 1-2). Most of them were probably circular or oval in section, because the spaces in such an installation could create so-called "cold zones", where the melted metal could harden before the main processes took place.

A chief principle in any metallurgical process is that the flow of air through the equipment must not be obstructed, and the gas produced in the thermal reaction must be discharged outside. Oxygen is also necessary to support the combustion of the fuel and the process of oxidation and reduction which occurs during the smelting of the ore. Conversely, some of the gases are easily absorbed by the liquid metal, which can have a detrimental effect on the final product. Controlling the flow of air that is introduced into the centre of the firing vessel is the basis of all melting operations. The limited sizes of the furnaces meant it was impossible to contain a sufficient amount of air, which necessitated the introduction of more air via the use of hand-held bellows. It is believed that the earliest specimens were in use around the third millennium BC, and can provide a maximum of 200 l/min air (CRADDOCK 1995, 178, fig. 5.17; CRADDOCK 2002, 26). An example of bellows is represented in the popular scene from the Hephaestos Workshop, sculptured on the northern frieze on the Treasury of Siphnians in the Sanctuary of Apollo in Delphi (HEALY 1978, pl. 50). Because bellows were made from perishable materials, the only sure evidence for their use is the images and their clay parts (blowpipes, tuyères). These elements have been discovered during archaeological excavations of smelting sites (SHEEL 1989, 28, fig. 22; CRADDOCK 1995, 186, fig. 5.25).

The fuel

The only accessible fuel to ancient metallurgists was charcoal. Burning it creates sufficiently high temperatures and it contains no harmful inclusions, which might adversely affect the quality of melted metal (MEIGGS 1982, 90). From the present point of view, charcoal is not a robust-enough fuel source. Even at lower pressures it crumbles and turns into dust that fills the space between the pieces of ore in the furnace and so cannot allow the gaseous products of combustion to circulate (TONCHEV 2008, 25). In the first millennium B. C., the continuously expanding processing activities required huge amounts of timber, leading to the need for reforestation and cultivation of large forests near the metallurgical centers (ALLAN 1970, 9-11 with sources).

The process

Despite the fact that there are no detailed descriptions of the thermal processing of ore in ancient sources, attempts at reproduction (MERKEL 1990, 78-122), and scientific analysis of the rich remains of slag allow us to make great strides in its reconstruction. Two of the main methods of melting were closely linked to the particular characteristics of the two types of ores used. From the first type of ore (oxide, carbonate and silicate) only relatively pure metal could be produced by heating and reducing coal or wood in a suitable furnace with a controlled flow of air, and even then only a product to be further purified was produced. The second type of ore (sulfide) contained too many impurities such as sulphur, arsenic, sulfur and antimony, which, if left in large quantities in the metal after its fusion, often have a negative effect on the final quality.

This fact necessitated the use of so-called polyphase processing. The first stage is the "firing" of the ore in order to remove as much of the harmful impurities as quickly as possible. Each subsequent part of the process aims to further purify the metal to the required quality (FORBES 1964, 18). Major operations and processes are identical in the processing of both types of ores.

First, the furnace is heated to approximately 1000°C by burning charcoal for about an hour. If the facility is newly built from clay, then heating is continued until the inside is completly dry. After, the ore is placed in the furnace, together with more fuel and some additional impurities necessary for the formation of well liquefied slag[2].

All this can be placed separately or together, but always in proportion. Ratios undoubtedly vary from metal to metal and ore to ore, and also depend on the construction of the furnace (CRADDOCK 1995, 199). As a rule, even after enrichment, the ore was still composed of a large amount of extra minerals (between 60 and 80% silicates and/or iron oxides) that have a very high melting point, and would quickly suffocate the process. Specifically, the endothermic medium in the silicates and iron oxides are connected together to form compounds with a much lower melting point, so that it can be removed from the furnace as liquid slag. In some cases, the minerals in the ore had the exact proportions of silicates and iron in order to remove any impurities in the form of slag. Usually, however, there was a discrepancy. In this case, in order to make and maintain a liquid slag, if the ore contained iron silicates, then silica must be added, or vice versa.

Reactions observed in experiments with antique furnaces can be divided into four vertical zones (CRADDOCK 1995, 200 with references). In the upper part of the furnaces, the ore, flux and fuel is dried and heated. The second area is located opposite the flow of air, here the oxidation of the fuel creates the required temperature and at the same time, the combustion creates carbon monoxide (CO), which "wraps around" the hot mineral and strongly reacts with the metal oxides (i.e. reduce it). Slag is formed in this area due to the interaction between the silica and iron oxide. Thus, during the process, the metal ore is converted to droplets of molten metal and the excess material – into liquid slag. Just below the reaction

Fig. 1: Experiment setup. Preparing replicas of clay tuyères and pots. – Experimentaufbau. Vorbereiten der Lehmdüsen- und Topfnachbildungen.

Fig. 2: Preparing blowpipes from the Sorghum plant, which were necessary to provide an additional air flow in the hearth. – Vorbereiten der Blasrohre aus Sorghum, die nötig sind, um einen zusätzlichen Luftstrom in der Feuerstelle zu liefern.

Fig. 3: First stage of the experiment. Melting of silver-gold alloy "Doré". – Erster Abschnitt des Experiments. Schmelzen der Silber-Gold-Legierung „Doré".

zone the heavier metal droplets drop down through the slag and accumulate at the bottom of the furnace, forming a shapeless mass of metal. Slag is periodically tapped through a hole made in the wall of the furnace and the smelting continues by adding more ore and fuel. Experiments have shown that a treatment process probably lasted between 5 and 10 hours, in which up to a few tens of kilograms of metal accumulated in the bottom of the furnace (Merkel 1990, 120). It is assumed that the processing took place almost always at night because of the lower temperatures, and because it was the best time to control the conditions inside the furnace itself (Craddock 2002, 28).

So these steps form the basic principles of processing metals such as copper, lead and tin. There are some additional steps required, depending on the art and and nature of the materials. Gold is almost always found in metallic form in the deposits and can be removed by crushing the rock in which the veins of the precious metal are trapped. Then, by washing the mixture of crushed rock, the heavier gold particles can be separated out, which can then be melted in the furnace. Apparently, already in Roman times, gold iron pyrite was processed, where gold was extracted at an almost molecular level. In the process of melting, a known amount of silicate is added to the iron pyrite in order to produce slag, which allows the removal of the concentrated gold (Craddock 2002, 29). This method is also mentioned by Pliny (Plin. XXXIII. 69 and 79), and the piles of slag from such processing are

Fig. 4: Melt of silver-gold alloy "Doré". – Schmelzen der Silber-Gold-Legierung "Doré".

Fig. 5: Melting a gold concentrate extracted from the Ada Tepe ore. – Schmelzen eines Goldkonzentrates extrahiert aus dem Ada Tepe Gold.

found near the Roman mines of Tres Minas in Northern Portugal (Bachmann 1993, 157).

Experiment setup

Although we decided on a plan of action after having reviewed a number of articles and written descriptions of the process dating back to Antiquity, the experiment raised a lot of questions and presented further problems to be solved.

Among the archaeological material and structures from Ada Tepe, there are none which could be identified as the remains of a large metallurgical furnace or portable hearth, similar to that were used for smelting copper, tin or lead. Was this because such installations were not necessary, as the substance to be melted was a gold concentrate in the form of powder? Finds of crucibles indicated the delicate character of equipment needed for melting gold concentrate, and the ancient gold workers probably found that

Fig. 6: Unmelted gold concentrate. – Ungeschmolzenes Goldkonzentrat.

Fig. 7: Adding a flux (borax) to the gold concentrate. – Hinzufügen eines Flussmittels (Borax) zum Goldkonzentrat.

a more suitable structure for melting gold was a simple hearth inside a clay vessel. They needed nothing more than a shallow hollow in the terrain into which could be placed a ceramic vessel full of charcoal, with the gold concentrate placed in a crucible in the middle of charcoal (BRANIGAN 1974, 68).

In order to build our hearth, we chose an open area to utilize the natural draught. To provide additional air flow to the hearth, we then made tuyères of clay mounted at the end of the hollow stems of the Sorghum plant. Three different types of clay with different compositions and amount of temper were experimented with when creating the tuyères and crucibles (*Fig. 1-2*).

Experiment

The experiment took place in November 2012 on the eastern slope of Ada Tepe. The temperature of the soil and the air was relatively low. We began by digging a small pit in the ground and placing a fired

Fig. 8: Maintaining the temperature in the hearth. – Aufrechterhalten der Temperatur in der Feuerstelle.

ceramic vessel in it to serve as a fireplace. A small hole was drilled at the lower part of the vessel and a tuyère was placed in it. We started the fire using thick branches and logs. Once the fire died down to embers, we added some charcoal.

The fire temperature was constantly measured by three thermocouples: one commercial platinum-rhodium type B (maximum temperature 1800°C) and two laboratory-made chromel-alumel (maximum temperature 1350°C) type K thermocouples. They all were connected to a MasTech MY-64 multimeter, displaying the temperature directly in °C. The platinum-rhodium thermocouple was placed at the bottom of the ceramic vessel. The other two were placed at the surface of the crucible and below it. As they were in direct contact with the embers, the temperature reading of these two thermocouples was not stable and depended strongly on the blowing intensity and the state of the burning charcoal. So we considered their readings as tentative and assumed only the reading of the platinum-rhodium thermocouple as a real working temperature.

In the first stage of the experiment, we decided to melt 5.1 g. filings of a silver-gold alloy "Doré" (with 0.4% gold) because of its lower melting point ~962°C. We placed the filings into the crucible, then covered it with a lid and positioned the crucible in the fire (*Fig. 3*). Burning charcoal was piled up all around it. However, the maximum temperature we could reach with an open fire was 940°C, as indicated by the platinum-rho-

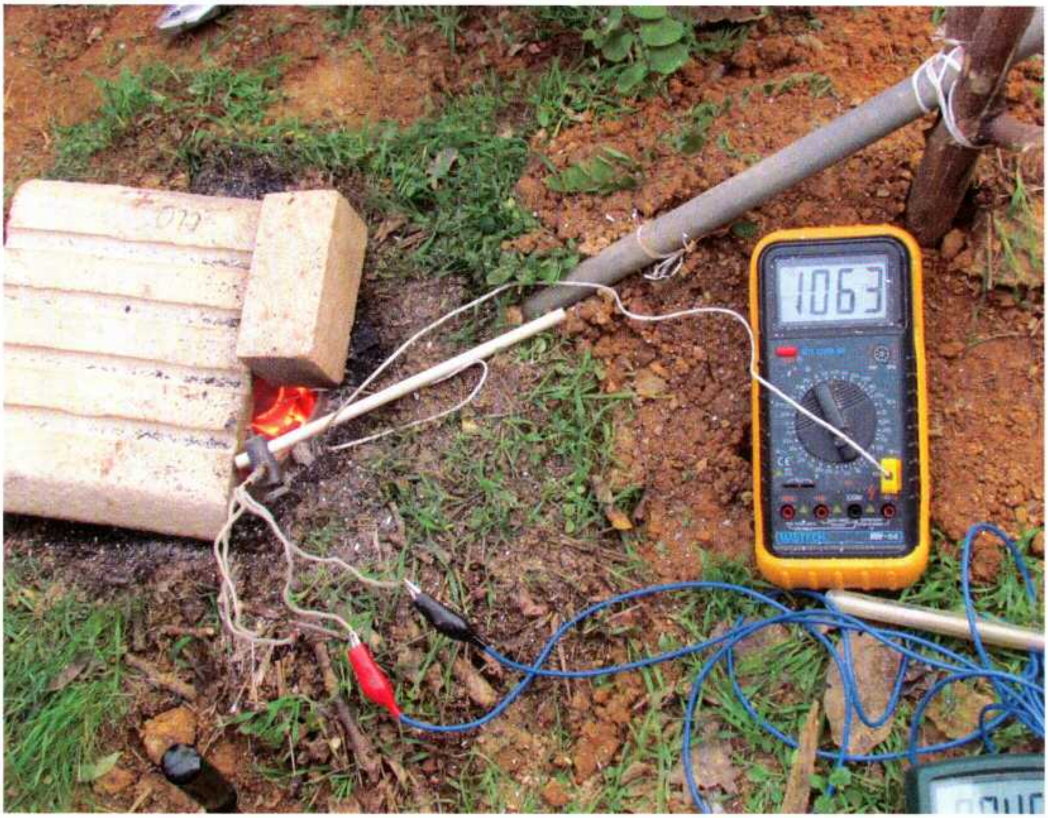

Fig. 9: Measurement of the temperature in the hearth. – Messen der Temperatur in der Feuerstelle.

dium thermocouple, and it was a problem to keep even this temperature stable. After covering the ceramic vessel filled with charcoal with fireproof bricks, the temperature rose slowly. Now we could increase the temperature and keep it steady for few minutes at ~1000°C in order for the metal filings to melt (*Fig. 4*).

The second stage of the experiment was the melting of the gold itself. The 0.9 g. gold concentrate extracted from the Ada Tepe ore veins acquired during the previous experiment was placed in a crucible (*Fig. 5*). We covered the crucible with a lid and positioned it in the fire as previously described. The temperature rose slowly, but barely reached ~1100°C and we could keep it at this level only for few seconds, despite delivering air via the bellows. We took the crucible out of the fire, and after opening the lid, we discovered that the gold had not actually melted, although something like a slag was noticed (*Fig. 6*). After our first attempt we came to the realization that we needed to use flux. In our so-called "gold concentrate", there were small fractions of quartz, which prevented the gold melting at its natural melting point (1064.18°C). We added some borax ($Na_2B_4O_7$) (also known as sodium borate, sodium tetraborate, or disodium tetraborate) as this was the only flux available to us at the time (*Fig. 7*). The temperature rose to ~1100°C and to maintain that level for a while, we had to intensify the delivery of air onto the coals (*Fig. 8*). As a result, after a few minutes, we removed the lid of the crucible and found a spherical golden nugget (*Fig. 9-10*).

Fig. 10: Gold melt. – Goldschmelze.

Analysis results

The actual ancient objects made from gold found on the Ada Tepe site are comprised of a single bi-conical bead and one melted spherical gold prill. The analysis of these two archaeological finds and the gold prill from our experiment are an on-going process. However, we can present our preliminary SEM analysis results (*Tab. 3*).

As visible in *table 3*, the bi-conical bead and the Late Bronze Age melt have a very close elemental composition. Also apparent is the fact that our newly-melted gold shows a different elemental composition. The difference in the elemental composition was visible even to the naked eye because their colour was not the same.

Initially, we were at a loss to explain this discrepancy. One possibility was that the bi-conical bead and the Bronze Age melted prill were brought here from another location – it is possible that they are not produced with gold from Ada Tepe – but this is the least probable hypothesis. It is not a logical assumption that the ancient miners would bring melted gold from another place if they already had it here. The other possibility is that the gold was refined (Rapson 1992, 203-212; Ramage, Craddock 2000; Rehren 2003, 207-215) – but still we have a quite high concentration of silver in the sample, as well as the fact that copper is absent altogether.

Then we tried to find a possible explanation in the ore deposit itself. As we mentioned above, the elemental composition of the samples deriving from the greater depths of Ada Tepe as described in the geological report of Danko Jelev

	Au Weight %	Ag Weight %	Cu Weight %	Pb Weight %
Bi-conical bead	83,39	15,61	1,01	-
Melt (LBA)	86,90	12,14	0,95	-
Melt (2012)	69,46	28,86	-	1,68

Tab. 3: SEM analysis of the finds and the recently produced gold melt. – SEM-Analyse der Funde und der jüngst produzierten Goldschmelze.

have a higher gold concentration. In our case the gold concentrate is derived from a vein near the surface.

Discussion

The results from our experiment reflect the fact that these are preliminary investigations and findings. We need to improve the precision of our technique and also the instruments and equipment used. In terms of the equipment used, it could be seen that the clay with a greater amount of and larger-grained temper had better refractory properties. The crucibles with thicker walls are more resistant to high temperature, heat slowly, but retain more heat which is very important for the success of the procedure. The covering of the hearth also affects the retention of heat around the pot, and the rapid heating of the material.

We also learned that we need to add flux, which was clearly proven by the experiment. But we also need to find out what kind of flux people from the Late Bronze Age used. As we have learned from this experiment and from examining the modern technical literature on gold melting, many natural forms of gold contain impurities, so flux is required to collect and remove them from the gold. A higher percentage of impurities requires using more flux and some small amount of flux should be used even when the material appears to be pure. We also discovered a crack in one of the crucibles most likely caused by the fact that it had been heated too fast, so we need to improve this aspect of the experiment as well.

One question we still have no answer to is: why was there lead but no copper in our melt?[3]

So as you can see we have many questions and also some problems to be solved, but our work shows that experimental archaeology can give new opportunities to check working hypotheses. By use of modern analytical methods, experimental archaeology can make information about ancient mining and smelting techniques much more accessible.

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Notes

[1] On the Classical Greek vases (Craddock 2002, 26, fig. 12) and frescoes from Egyptian tombs from Hellenistic age (Sheel 1989, 23, fig. 14).

[2] The so-called metallurgy "flux"-silicate materials containing silica or limestone crystals and lumps melting release substances that bind to existing iron oxide in the ore and help shape the slag. Perhaps Aristotle (or Pseudo-Aristotle)

describes a similar process when he talked about the production of iron in halibite "... they repeatedly sailed what remains after the first driftwood, and burn together, hurl stones called πυρίμαχος (fire-proof)" (Aristotle. 833b. 48). Agricola mentions the use of fluxes made from crushed marble, limestone slate and lead (Agricola IX). If the ore is very clean then it requires very little flux (CRADDOCK 1995, 199).

[3] According to Prof. Clemens Eibner, one of the possibilities is that the lead have been introduced via the charcoal used in the heating process. We are thankful to all participants for the discussion during the EXAR Conference in Linz.

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