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Metallographic analyses from the late Urnfield period copper mining settlement at Prigglitz-Gasteil in Lower Austria

ABSTRACT: *The site of Prigglitz-Gasteil represents the largest Late Bronze Age mining settlement in Lower Austria, occupied from about 1050 to 900 BC. According to excavations from 1956 to 1958 and from 2010 to 2014, all metallurgical production steps, from copper ore to bronze, are attested at the site.*

Copper slag from an early production step is very inhomogeneous and contains large quartz inclusions. From the slag´s microstructure and composition a solidification temperature of approximately 1350 °C can be assumed. Black copper with a total sulphur content between 1 and 1.2 wt.% S consists of Cu and network-like arranged Cu_2S.

On the surface of a bronze slag a particle containing Sn and O was observed. From the slag composition a melting temperature between 1120 and 1090 °C is assumed. A corroded bronze droplet was investigated as well. The original microstructure of the metallic bronze is still visible in the corrosion products. Due to a special corrosion process about 25 wt.% Sn was observed in the corrosion products. With regard to other objects like casting molds it can be demonstrated that bronze casting took place at Prigglitz-Gasteil.

KEYWORDS: LATE BRONZE AGE; MINING SETTLEMENT; COPPER SLAG; BLACK COPPER; BRONZE SLAG; BRONZE DROPLET

Introduction

The Late Bronze Age mining settlement of Prigglitz-Gasteil is located at the easternmost fringe of the Alps in the district of Neunkirchen in Lower Austria (Fig. 1a). The site was occupied during the late Urnfield period (ca. 1050 to 900 BC). It reached a maximum extent of about 3 hectares, thus representing the largest mining settlement in Lower Austria (Trebsche, 2013; 2014a; 2015a; 2015b; Trebsche & Pucher, 2013; Haubner et al., 2015).

In contrast to the western parts of the Austrian Alps, prehistoric mining research in Lower Austria only started in the 1920s (Mühlhofer, 1935; 1952). The first direct evidence of prehistoric copper production was found in the 1950s by archaeologist Franz Hampl and geologist Robert Mayrhofer (Hampl, 1953; 1976; Hampl & Mayrhofer, 1958; 1963). The copper mining site of Prigglitz-Gasteil (Cu I) was discovered in 1955 (Fig. 1b). Hampl and Mayrhofer dug 15 narrow trenches there in 1956 and 1958. In 1959, they continued their excavation at the nearby copper smelting site of Prigglitz-Gasteil Cu II (Hampl & Mayrhofer, 1963, pp.74-75). Hampl distinguished the large mining settlement at site „Cu I" and the small copper smelting site „Cu II". If there is no precise indication, we use the name Prigglitz-Gasteil to refer to the site „Cu I". Peter Trebsche resumed fieldwork at Prigglitz-Gasteil in 2010. Altogether, in five summer campaigns from 2010 to 2014, two areas of approximately 210 m^2 were excavated on two different terrain terraces that had not been studied previously (Fig. 1c). The excavations revealed that the occupation floors were extraordinarily well preserved. The annually published excavation reports are also available online (Trebsche, 2010-2014; https://uibk.academia.edu/Peter Trebsche).

Excavations at Prigglitz-Gasteil brought to light only a few chronologically significant small finds, so the absolute chronology of the site depends on radiocarbon dating. A small series of nine C^{14} samples showed that the final occupation phase lasted from the middle of the 11^{th} to the end of the 10^{th} century BC (Trebsche, 2015a).

Thanks to its outstanding state of preservation, the site of Prigglitz-Gasteil allows for a multi-faceted approach to copper mining, bronze working and its social aspects. Fortunately, the Late Bronze Age features have not been damaged by later mining activities. The excavations from 2010 to 2014 yielded abundant material for the study of miners' daily life and nutrition, for the reconstruction of dwellings and workshops, and for the spatial analysis of a mining settlement. They also provide completely new insights not only into metal production, but also into the

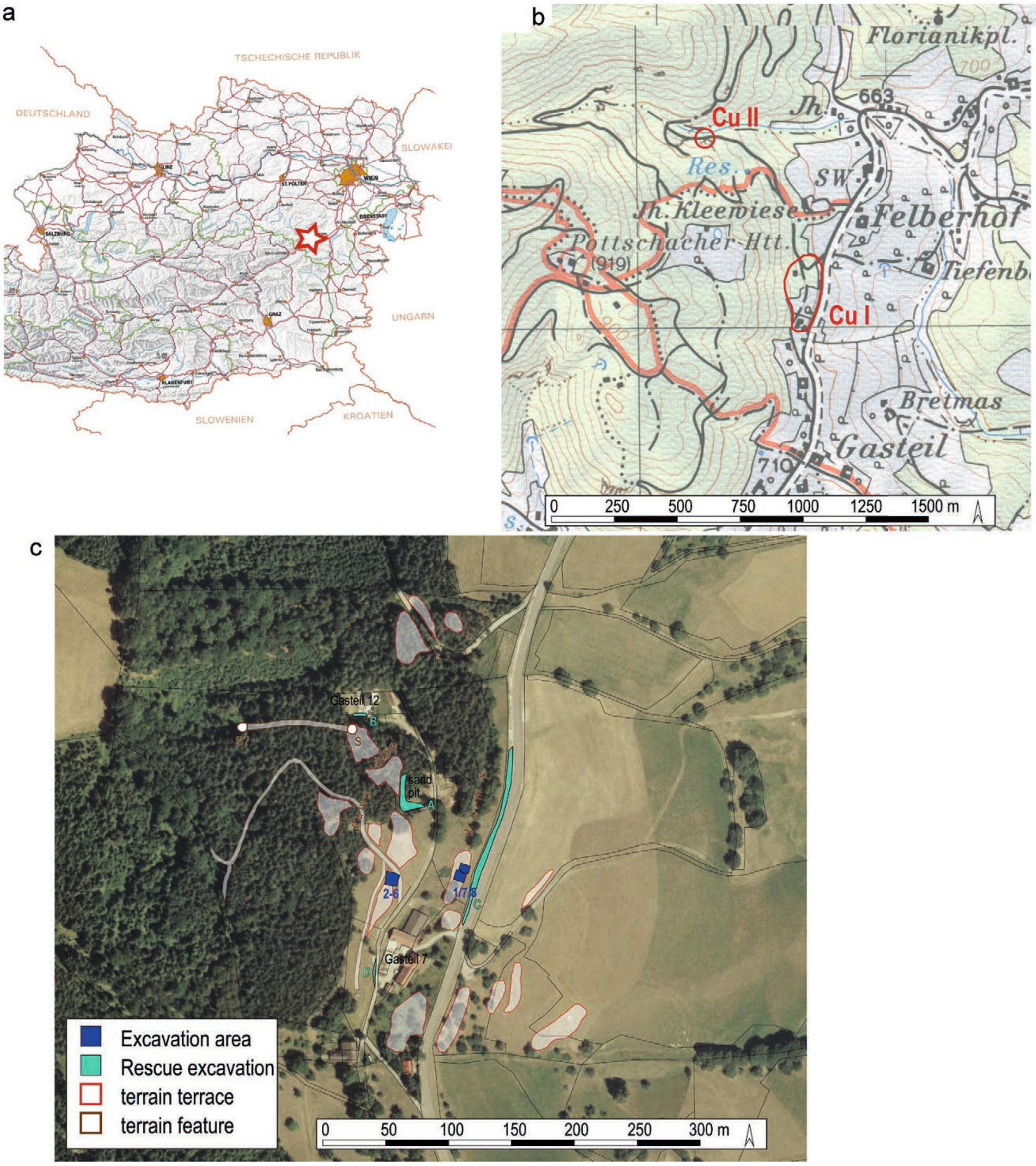

Fig. 1: Prigglitz-Gasteil (a) Location of the site in Austria; (b) topographic map of sites Cu I and Cu II in the cadastral area of Prigglitz; (c) plan of site Prigglitz-Gasteil Cu I.

operation and supply of a mine (butchering, manufacture of tools, timber procurement etc.). Archaeozoological analysis of the animal bones from the Prigglitz-Gasteil site shows striking parallels with pork processing at the contemporaneous Hallstatt salt mine (Trebsche & Pucher, 2013).

During the excavations, only indirect evidence for copper ore mining was found such as coarse dump layers and miners' tools (antler picks and hammers). The main local ores are chalcopyrite, pyrite and siderite. At present, it is unclear which mining technique (open cast mining, open-trench extraction or underground working) was employed at Prigglitz-Gasteil. Unfortunately, there are no terrain features such as pits or sunken shafts visible at the site. Although several prospection techniques have already been employed (archaeological field surveys, aerial photography, analysis of LiDAR terrain models, geomagnetic surveys), no direct traces

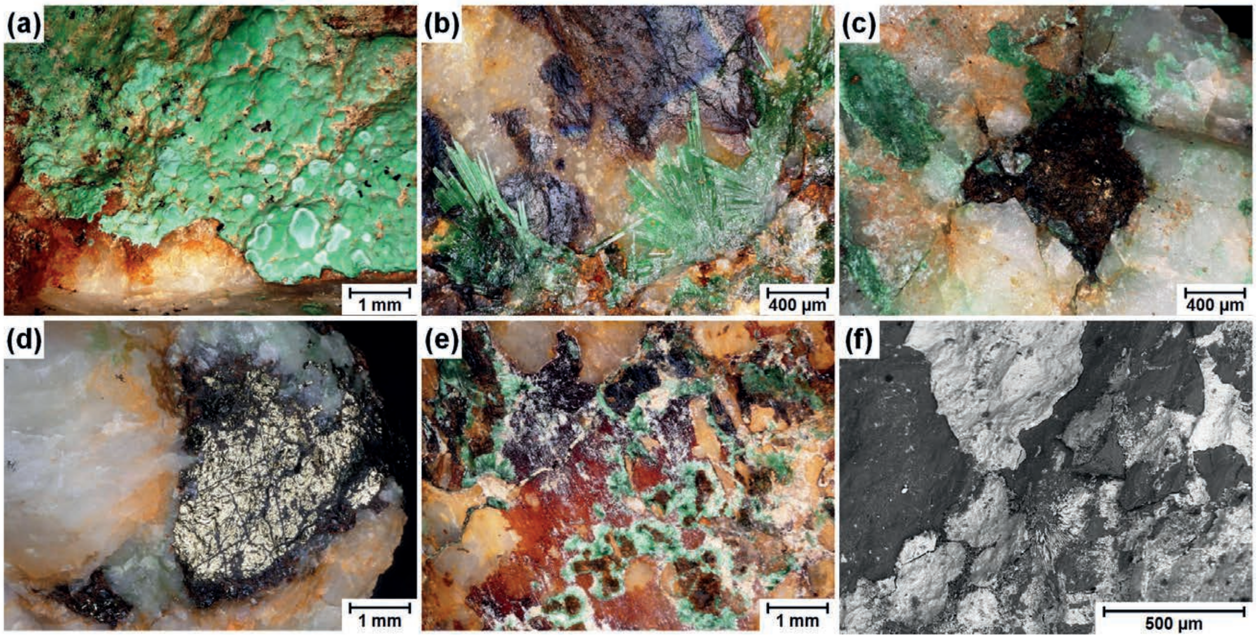

Fig. 2: Ore samples from Prigglitz-Gasteil Cu I in 3D-OM; (a) nodular malachite and quartz; (b) needle-like malachite; (c) limonite inclusion, (d) chalcopyrite in quartz; (e) mixture of malachite, cuprite and limonite; (f) SEM-BSE, cupreous regions are bright.

of copper mines were detected. In 2013 and 2014, 18 percussion drillings were conducted to a maximum depth of 10 m which was not enough to reach the underlying bedrock (Trebsche, 2014a; 2014b; 2014c). Therefore, geoelectric prospections and core drillings into greater depth should be conducted in the future in order to investigate the stratigraphy of the mining dumps and to localise the underground workings and the copper vein (in case there is some copper ore left).

Considering the extraordinary size (3 ha) of the copper production site at Prigglitz-Gasteil, it seems possible that not only local ores were processed. To test this hypothesis, several surveys of copper ore deposits in the region (district of Neunkirchen) have been conducted by Michael Götzinger (Institute of Mineralogy, University of Vienna) and Uwe Kolitsch (Mineralogical Department, Museum of Natural History of Vienna) since 2012, supported by local mineral collectors (Niedermayr et al., 2014, pp.121-124).

Following copper ore extraction, the next steps in the chaîne opératoire are ore beneficiation, smelting, alloying and casting of bronze objects. All these steps are attested at the site of Prigglitz-Gasteil, with more than 146 copper or bronze fragments, droplets and intact bronze artefacts as well as approximately 400 pieces of different slag types. However, these finds cannot be interpreted by macroscopical investigation alone but require archaeometallurgical analyses. Considering the great number of metallurgical finds, archaeometallurgical analyses are planned in three steps: The first step aims to investigate from which stage of the copper production process these remains originate (Eibner, 1992). The second step is to characterize the local copper ores and ore specimens from neighbouring deposits with the aid of trace elements and lead isotopes. This should provide the basis for identifying products of the Prigglitz-Gasteil copper mine in regional or supra-regional contexts. The third step is to investigate local bronze working techniques using metallographic analyses.

In this paper, the results of four metallographic analyses will be presented. These samples were chosen from different find categories and allow for a preliminary orientation. In the future, a representative number of samples will be selected to further investigate each copper production step at the site of Prigglitz-Gasteil.

Archaeological context of the analysed samples

One piece of coarse slag (no. 1, inventory number UF-11901, Fig. 3) was selected for archaeometallurgical analysis from the site Prigglitz-Gasteil Cu II. This site probably represents a small smelting site contemporaneous with the large mining settlement at site Cu I. It is located approximately 700 m northwest of the site Cu I in the so-called Klausgraben (Fig. 1b) and was investigated in 1959 by Franz Hampl (Hampl & Mayrhofer, 1963, pp.74-75).

The piece of black copper (no. 2, Fig. 4) was found by Reinhard Lang near the farmstead Gruber in the area of the site Prigglitz-Gasteil Cu I in the years 2000–2010. Lacking a stratigraphic context, this surface find cannot be dated with absolute certainty, but most probably it originates from the Late Bronze Age as the vast majority of finds from the site does.

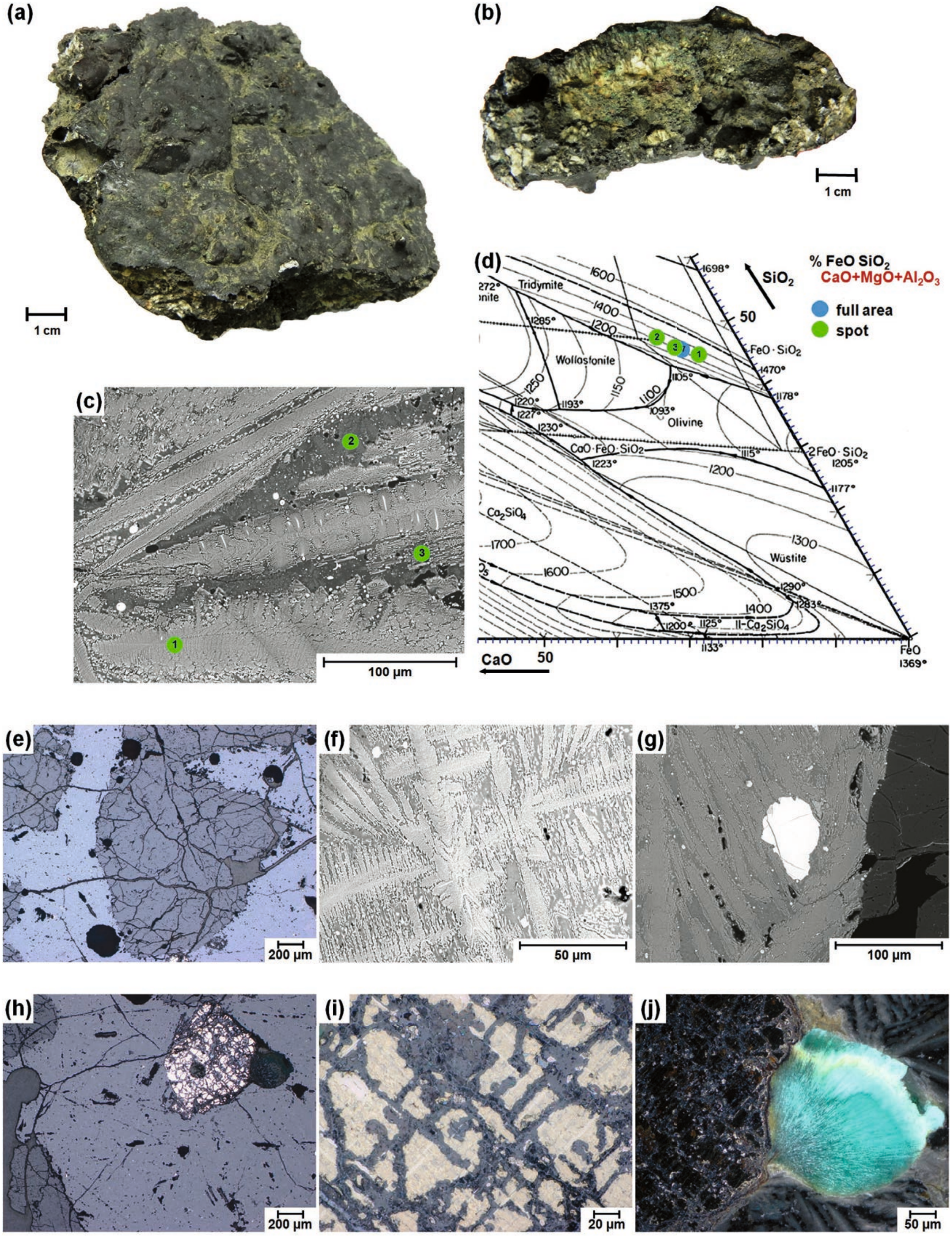

Fig. 3: Slag sample from Prigglitz-Gasteil Cu II; (a) front view and (b) side view; (c) SEM image of the slag with marked spots for the EDX analysis; (d) FeO-SiO_2-CaO(MgO-Al_2O_3) phase diagram showing the elemental composition of the EDX measurements shown in (c); (e) LOM image showing the distribution of darker quartz and brighter slag; (f) dendritic slag microstructure in SEM; (g) chalcopyrite inclusion in the slag (SEM); (h-j) copper and cupreous inclusions (LOM).

The fragment of a platy slag (no. 3; inventory number UF-22692.1896. Fig. 5) and a bronze droplet (no. 4; inventory number UF-22692.1778, Fig. 6) also come from site Prigglitz-Gasteil Cu I. They were found during the 2013 excavation campaign and belong to stratigraphic unit 916 in excavation area 6 located on the upper terrain terrace. The stratigraphy of the upper terrain terrace can be divided into four phases: in the first phase, fine-grained dump layers indicate the beneficiation of copper ores; during the second phase, these dumps were levelled horizontally to construct a working platform or terrace. Several hearths and postholes were documented on this terrace which was covered by a sequence of different layers. The older layers in this sequence provide evidence for copper or bronze metallurgy, including the fragment of a stone casting mould for a bronze knife (see Trebsche, 2015a, pp.49, fig. 2/7). Copper based artefacts and platy slags concentrated mainly in a greyish layer with lots of charcoal (stratigraphic unit 916) from where the two mentioned samples were selected. Later, the use of this working terrace changed and large amounts of animal bones were deposited. The whole sequence comprised at least 23 charcoal layers indicating (local) fire events. In the following third phase, fine-grained material from ore beneficiation was dumped again covering the mentioned terrace. After that, a cultural layer with postholes belongs to the fourth phase. A layer of coarse dump material was deposited onto the cultural layer. Finally, in the fifth phase, a working pit was dug into the terrace. While the first four phases can be dated to the late Urnfield period by radiocarbon samples, the fifth and last phase belongs to the medieval period (Trebsche, 2015a, pp.48-51).

Archaeometallurgical analyses

Copper ore samples, slags and metallic copper alloys were analysed by metallographic methods.

For metallographic investigations the different samples were cut and vacuum infiltrated with epoxy resin. After plane grinding, the samples were fine grinded and polished using 9, 3 and 1 µm diamond suspensions. The prepared samples were finally examined by an inverse light optical microscope (LOM).

Further investigations of original and polished samples were carried out with a FEI QUANTA 200 K scanning electron microscope (SEM) which was equipped with an energy dispersive X-ray detector (EDX). For SEM images, only the back scattered electron mode (BSE) was used. A digital 3D optical microscope (Keyence, 3D-OM) was used to investigate the sample´s surface.

Copper ores

During the excavation campaign in 2014, several ore samples were collected by Roland Haubner from the excavated material. At first, the surfaces of the samples were investigated by 3D-OM and afterwards samples were crushed to sizes which were acceptable for the SEM-EDX analysis.

The images in Fig. 2 reveal different copper ores. Generally, the gangue associated with the copper ores in the investigated samples was quartz. In some areas the quartz is covered by nodular malachite (Fig. 2a). In voids the malachite forms needle-like crystals (Fig. 2b). Due to long-time storage of the samples in the mine dump, original ore and oxidized material are mixed. In Fig. 2c a limonite inclusion and in Fig. 2d a chalcopyrite ore inclusion can be seen. A mixture of malachite, cuprite and some limonite is shown in Fig. 2e. Using SEM-EDX in backscattered mode, the cupreous regions are bright in contrast to the darker quartz. Additionally, other minerals such as pyrite, oxidized pyrite (limonite), titanite ($CaTiO_3$) and cinnabarite (HgS) were locally observed. Similar results were described by Michael Götzinger and Uwe Kolitsch (cf. Niedermayr et al., 2014, pp.121-124).

Slag lump from copper smelting (no. 1)

The surface of the slag in Fig. 3a has a comparatively homogeneous appearance but the side view shows a mixture of slag and quartz (Fig. 3b). The quartz particles reach a dimension of several mm. After metallographic preparation, the darker quartz is clearly distinguishable from the brighter slag (Fig. 3e). In LOM many cracks are observed in the quartz and some cavities in the slag. We assume that this piece of slag was formed when molten slag was removed from the furnace and accidentally came into contact with quartz sand outside the furnace. In this case, the quartz cannot be related to the copper ore which was filled into the furnace. This is plausible, because XRD analysis showed no cristobalite which should be formed during the high-temperature treatment in the furnace. Additionally the microstructure show no distinct reaction zones between quartz and slag. The slag´s microstructure consists of fine dendrites and the interdendritic space is filled with a glass phase (Fig. 3f). To get information about the melting temperatures of the slag local SEM-EDX measurements are necessary (Haubner et al., 2017; Haubner & Strobl, 2017). Thus, the full area of Fig. 3c and several spots were measured and the calculated results were plotted in the conventional FeO-SiO_2-CaO phase diagram (Fig. 3d), note that for the calculation MgO and Al_2O_3 were added to CaO (Haubner, Strobl and Klemm, 2017). For comparison all EDX results of the discussed copper slag and a bronze slag, which will be described later, are summarized in Table 1. The full area and the single spots are located in the tridymite and FeO-SiO_2 region. Melting temperatures above 1200 °C are realistic for this slag. The brighter dendritic material (spot 1) shows the stoichiometry of $FeSiO_3$ containing some impurities. In the phase diagram this spot is located right from the full area spot. Spot 2 was measured in the

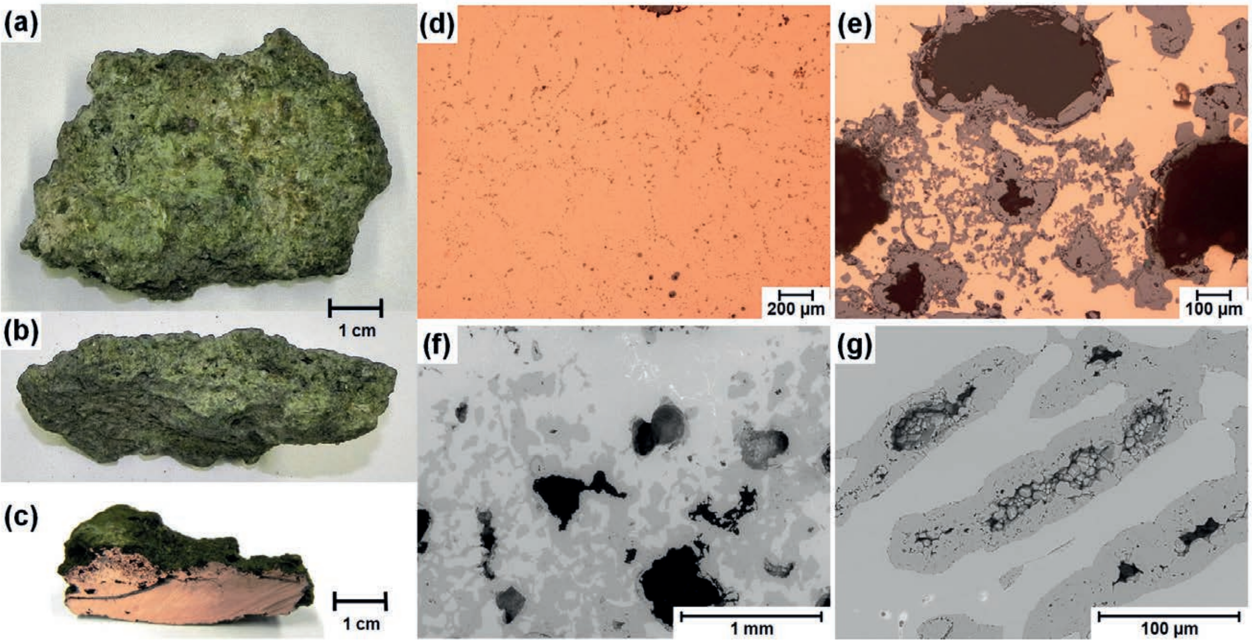

Fig. 4: Black copper lump Prigglitz Gasteil Cu I; (a) front view; (b) side view; (c) cross section; (d) homogeneous copper with Cu_2S inclusions (LOM); (e) inhomogeneous region containing holes, sulfides and oxides (LOM); (f, g) SEM images showing the inclusions in metallic copper.

Mas. %	slag from copper smelting					slag from bronze production			
	area	spot 1	spot 2	spot 3		area	spot 1	spot 2	spot 3
Fe	38,9	41,9	35,3	37,7		40,7	46,6	36,9	38,2
Si	23	22,8	24	23,1		17,1	16,1	18,5	17,2
Ca	2,3	--	4	3,3		4,5	2,6	5,8	5,4
Mg	1,3	2,3	--	0,8		1,2	3,1	--	--
Al	2	1,8	2,9	2,3		2,8	--	3,3	3,3
Mn	--	--	--	0,8		--	--	--	--
K	1,1	1,4	1,3	1		1,4	--	1,7	1,9
S	0,6	0,3	0,9	0,6		0,8	--	--	--
O	29,5	29,4	31,6	30,3		28,1	27,9	30,1	28,8

Tab. 1: Original EDX results from area and spot measurements of slag samples.

darker glass phase area and contains higher amounts of CaO and Al_2O_3. This spot is located left of the full area spot. As assumed, spot 3 is located between spot 1and 2 and the elemental composition is near the full area spot.

The composition $FeSiO_3$ suggests the presence of pyroxene crystals, but it was not possible to identify this phase by crystallographic methods. During solidification the crystallization of fayalite (Fe_2SiO_4) and additional quartz is assumed. From the phase diagram a slightly higher solidification temperature (about 1350 °C) for the FeO-SiO_2 mixture can be assumed compared with the glass phase solidification temperature (about 1320 °C).

In the slag some copper and cupreous inclusions were observed (Fig. 3g) and a large bright particle was identified as chalcopyrite. Other inclusions are partly reduced to copper and contain mixtures of metallic copper and chalcopyrite (Fig. 3h, i). During long-time storage of the slag in the dump, malachite had been formed in some areas by oxidation (Fig. 3j).

Black copper after smelting (no. 2)

We assumed that the lump shown in Fig. 4a, b (front and side view) contains metallic copper due to its relatively high weight of 274 g. After sectioning the lump it was identified as metallic copper (Fig. 4c). The lower part has a homogeneous appearance but at the left side there is a crack and the material at the top appears porous and inhomogeneous.

The homogeneous copper shown in Fig. 4d contains fine, network-like arranged, scarlet inclusions which were

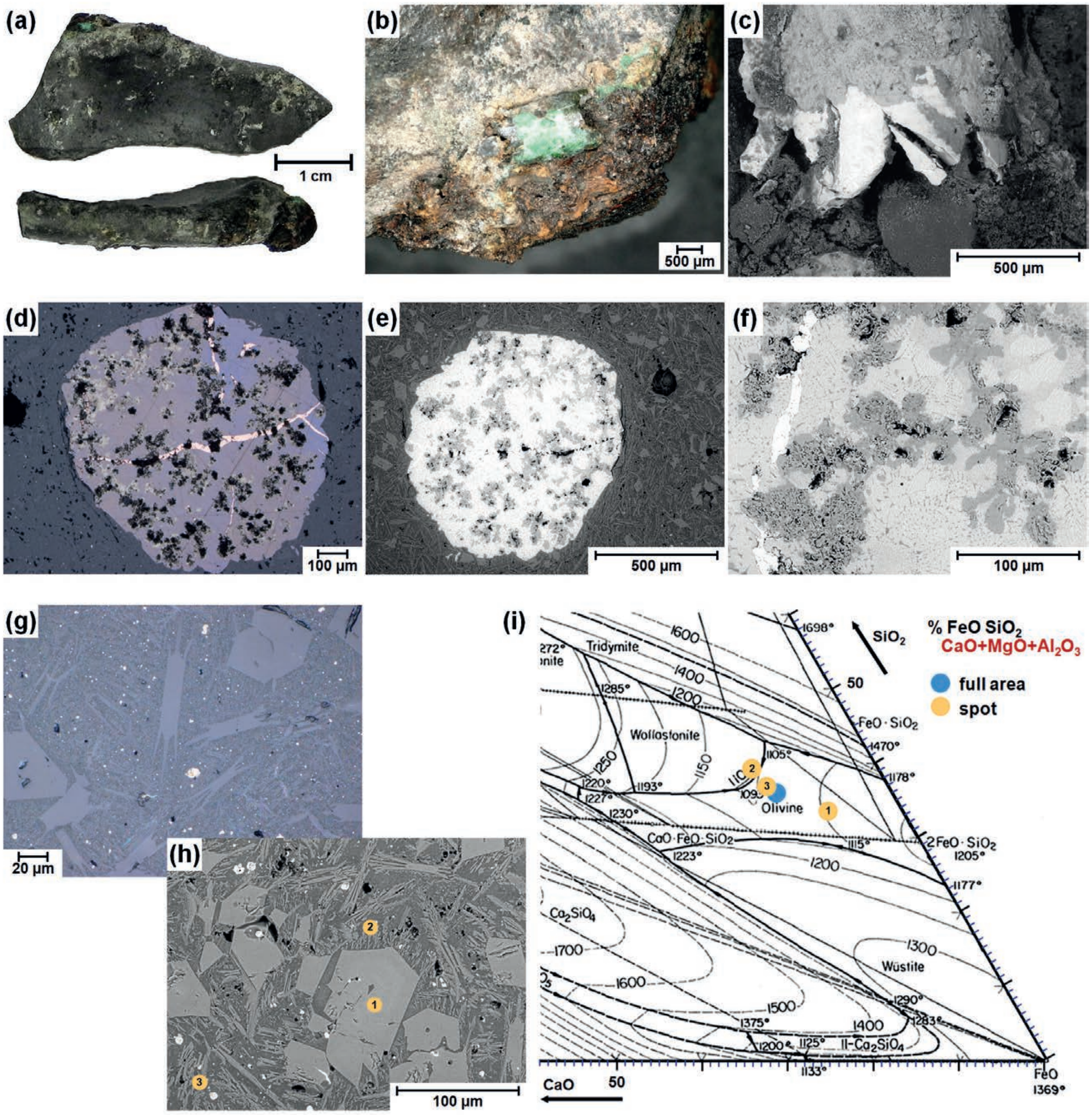

Fig. 5: Platy slag from Prigglitz-Gasteil (Pr 1896); (a) front and side view; (b) surface with Sn rich particle (3D-OM); (c) Sn rich particle in SEM); (d) copper rich inclusions (LOM); (e, f) SEM-BSE pictures of the Cu-rich inclusion; (g) slag microstructure in LOM; (h) SEM picture with indicated spots for EDX measurements; (i) phase diagram showing the elemental composition of the EDX measurements shown in (h).

identified by EDX as Cu_2S. The total sulphur content for the full area is between 1 and 1.2 wt.% S in Cu. The microstructure is characteristic for a Cu-melt during solidification where pure Cu is crystallizing first and S is concentrated in the melt. At last Cu_2S and Cu are crystallizing according to the Cu-S phase diagram (Massalski, 1990; Schumann, 1967).

The porous and inhomogeneous region shows large holes surrounded by a dark phase in metallic copper (Fig. 4e).The SEM-EDX analyses identify the large grey areas as Cu_2O (Fig. 4f, g). Additionally, in the copper and in the Cu_2O fine Cu_2S particles were observed, but the absence of Cu corrosion products like malachite indicates that the Cu_2O was formed during solidification.

In the copper phase several bright spots are visible, where Sb and O were identified (Fig. 4f, g), which means that some fahlore was used for Cu smelting. In this black copper sample, no As was detected, although As is another typical element in fahlores (Kharbish et al., 2007) and copper ingots produced from fahlores (Ertl et al., 2017; Haubner et al., 2017).

Slag from bronze production (no. 3)

A platy slag excavated 2013 near a fireplace was investigated (Fig. 5a). In LOM a green particle with approximately 3 mm length and 1 mm width was observed near the edge of this slag sample (Fig. 5b). SEM-EDX analyses show that the particle´s core is Sn- and O-enriched and is covered by malachite (Fig. 5c). It was not possible to identify this particle as SnO_2 ore, but it was a first hint that this slag was a by-product of the Sn bronze production. This extraordinary observation requires further investigations of much more platy slags which will be performed in the next stage of our research.

This slag is quite compact because during the metallurgical process it was completely molten. After metallographic preparation, LOM and SEM images of the cross sections are shown in (Fig. 5g, h). Area and spot measurements by EDX allow the appraisement of the melting point from the phase diagram in Fig. 5i. The microstructure is characterized by large, compact olivine crystals with mainly Fe- and small amounts of Mg- and Ca-silicate (Fig. 5i spot 1). The elongated crystals are olivine as well but they crystallized later together with the glass phase (Fig. 5i spot 2 and 3).

As shown in the FeO-SiO_2-CaO phase diagram, the melting temperatures for the different slag phases are between 1120 and 1090 °C. But this is only a rough estimation because the other trace elements Mg, Al, K and S in the slag also have an influence on the melting respectively solidification temperatures.

In the slag cupreous inclusions were additionally observed (Fig. 5d, e). In the SEM-BSE image (Fig. 5f) the white veins were identified as Cu, the light grey areas are chalcopyrite and the dark grey areas are FeS (pyrrhotite).

Bronze droplet (no. 4)

During the excavation period of 2013, approximately 100 copper alloy droplets were found near a fireplace (Trebsche, 2014b). For analytical investigations, a droplet with corrosion products on its surface was selected (Fig. 6a). In SEM-BSE on the droplet´s surface the corroded microstructure of Sn-bronze was already visible (Fig. 6b). The corrosion products are greyish and the metallic phase is bright. EDX measurements showed that the different areas vary considerably in their Cu and O content, in contrast the Sn amount with 25 wt.% is almost constant. An explanation for this Sn-enrichment is that reactions of Sn and Cu occurred: Sn forms insoluble SnO_2 and Cu forms Cu^+ ions, which are soluble and transported to other locations (Haubner et al., 2017).

For metallographic investigations the droplet was mounted in araldite and in a first step the surface area was polished. Mixtures of metallic and corroded areas became visible in LOM (Fig. 6e) and SEM (Fig. 6d). The original microstructure of the bronze was dendritic, but currently the copper dendrites are corroded - forming malachite - and the interdendritic Sn-enriched areas remain metallic. Using polarized light in LOM, the metallic areas can be distinguished well from the green malachite and the red Cu_2O (Fig. 6f).

After further polishing of the droplet it became clear that this droplet was fully corroded and no metallic bronze core was observed (Fig. 6b). But at the rim of the droplet metallic areas were detected beneath a scale layer (Fig. 6g). Using polarized illumination in LOM, the distribution of malachite (green) and Cu_2O (orange-red) is well visible (Fig. 6h). Why metallic phases are found at the surface but not in the core of the droplet is yet unknown. The corroded core of the droplet shows a dendritic structure, which had been formed during the bronze solidification (Fig. 6i). In polarized light the oxidized copper dendrites are orange-red, the Sn-enriched interdendritic areas are scarlet and near the surface Cu_2O reacted to green malachite by further oxidation (Fig. 6i).

Conclusions

At the archaeological site Prigglitz-Gasteil the metallurgical production steps of copper alloy, from ore to bronze, are proven. This investigation presents analytical results from cupreous ores, copper slag, black copper, bronze slag and bronze droplets.

Ores: In the investigated samples, the gangue associated with the copper ores was quartz which contained nodular or needle-like malachite and cuprite. Limonite and chalcopyrite inclusions were found occasionally. Other minerals such as pyrite, titanite ($CaTiO_3$) and cinnabarite (HgS) were locally observed.

Copper slag (no. 1): The slag lump is very inhomogeneous and contains large quartz inclusions. This is typical for primary copper smelting slags (“slag cakes”) where unmolten quartz relics from the gangue are to be expected.The slag´s microstructure consists of fine olivine dendrites, and the interdendritic space is filled with a glass phase. SEM-EDX measurements showed that the SiO_2 content is high in this slag. From the phase diagram solidification temperatures of approximately 1350 °C can be assumed.

Black copper (no. 2): This piece of metallic copper contains network-like arranged Cu_2S inclusions, and the total sulphur content is between 1 and 1.2 wt.% S. Locally, holes surrounded by Cu_2O were observed. The absence of Cu corrosion products like malachite indicates that the Cu_2O was formed during solidification. In the copper phase Sb was identified. This indicates that fahlore was used for Cu smelting. As to our present knowledge, at Prigglitz-Gasteil fahlore occurs only very rarely compared with the dominating sulphidic copper ores. The only fahlore specimens were found during core drillings in 2017. It cannot be excluded, however, that the piece of black copper was transported from somewhere else into the mining settlement.

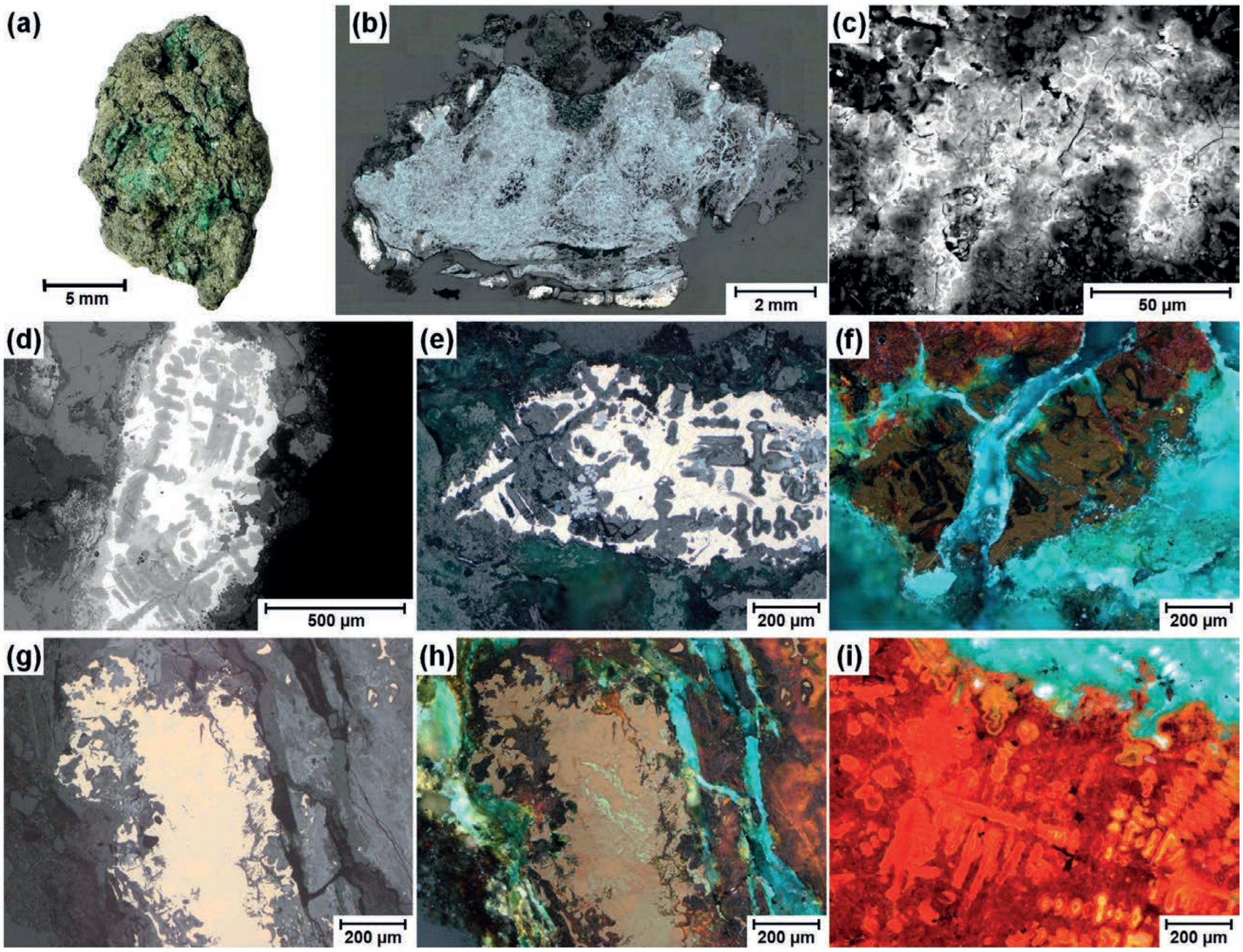

Fig. 6: Bronze droplet (P1778); (a) front view; (b) metallographic cross section (LOM); (c) droplet surface (SEM-BSE); (d – f) microstructure near the droplet surface (SEM and LOM); (g – i) cross section near the droplet center (LOM) – (g) metallic bronze (h) same location in LOM with polarized light (i) corroded bronze in the center of the droplet.

Bronze slag (no. 3): On the surface of this slag a particle containing mainly Sn and O was found. This platy slag is compact and was completely molten during production. The microstructure is characterized by large, compact and elongated olivine but the elongated crystals solidified later together with glass phase. According to the phase diagram, the melting temperatures for the different slag phases are located in the olivine area and are between 1120 and 1090 °C. In the slag inclusions of cupreous phases, chalcopyrite and FeS were identified.

Bronze droplet (no. 4): On the droplet´s surface the corroded microstructure of Sn-bronze is already visible. While the Cu- and O-content varies, the Sn amount with 25 wt.% is almost constant. The original microstructure of the bronze was dendritic, but currently the droplet is corroded, forming mainly Cu_2O. The dendrites are still visible due to the interdendritic Sn-enriched areas showing different colours. Using polarized light green malachite, formed by further oxidation, and red Cu_2O can be distinguished.

The platy slag (no. 3) and the bronze droplet (no. 4) from layer 916 on the upper terrace confirm that bronze was cast at the site of Prigglitz-Gasteil. Therefore in the mining settlement, not only copper was produced but also copper alloys and bronze objects. The fragment of a stone casting mould (see Trebsche, 2015a, 49 fig. 2/7) for knife production provides the evidence of bronze casting. Interestingly, bronze casting took place between two periods of ore beneficiation as attested by the local stratigraphy in the upper terrain terrace. At present, it remains unclear whether bronze casting was a regular or an occasional activity at the Prigglitz-Gasteil site, and whether the bronze objects were cast for local use or for regional trade.

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