

The Rise of Metallurgy in Eurasia

Evolution, Organisation and Consumption
of Early Metal in the Balkans



Edited by

Miljana Radivojević, Benjamin W. Roberts,
Miroslav Marić, Julka Kuzmanović Cvetković
and Thilo Rehren



Miljana Radivojević holds the Archaeomaterials Lectureship at the UCL Institute of Archaeology (UK), where she graduated in Archaeometallurgy. She has spent more than 20 years publishing on early metallurgy in the Balkans and southwest Asia and the role of aesthetics in the invention of novel technologies. She continues to explore the evolution of metallurgy across most of prehistoric Eurasia as a means of uncovering the histories of metalsmiths, and the societies and environments they lived in.

Benjamin Roberts has spent over 20 years researching and publishing on European Copper and Bronze Age archaeology and frequently metallurgy and metal objects across Europe. He co-edited with Chris Thornton *Archaeometallurgy in Global perspective: Methods and Syntheses* (2014) and is currently leading Project Ancient Tin. Prior to joining the Department of Archaeology at Durham University, he was the Curator for the European Bronze Age collections in the British Museum.

Miroslav Marić is a specialist in the Neolithic-Bronze Age of the central Balkans at the Institute for Balkan Studies, Serbian Academy of Sciences and Arts, Serbia. He is the field director of the Gradište Iđoš project. His research interests include settlement archaeology, landscape archaeology, the Neolithic and Bronze Age of the Balkans, and radiocarbon dating.

Julka Kuzmanović-Cvetković was the Senior Custodian (now retired) at the Homeland Museum of Toplica in Prokuplje, Serbia. She spent more than four decades excavating the site of Pločnik, and developed a unique open air archaeo-park on the site that attracts tourists from the region, and across the globe.

Thilo Rehren is the A.G. Leventis Professor for Archaeological Sciences at the Cyprus Institute in Nicosia, Cyprus. In 1999 he was appointed to a Chair in Archaeological Materials at the UCL Institute of Archaeology in London, UK. Following a five-year secondment to establish UCL Qatar as a postgraduate training and research Centre of Excellence in Museology, Conservation and Archaeology he joined the Cyprus Institute in 2017. He places particular emphasis on the integration of archaeological, scientific and historical information, and on investigating the correlation and cross-fertilisation between different crafts and industries in the past.

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(from Pločnik, Serbia) - Julka Kuzmanović Cvetković.

Inner back cover: Reconstruction of the world's earliest copper smelting. Green flames come from the extraction of metal from malachite. Experiments at Pločnik, Serbia (2013) - Marko Djurica

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To the memory of Borislav Jovanović, our colleague, friend and inspiration

(1930 - 2015)

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Chapter 11

Belovode: technology of metal production

Miljana Radivojević and Thilo Rehren

Metal production evidence yielded during the excavation campaigns 2012 and 2013 in Belovode shows similar characteristic to the samples from the site studied and published previously (Radivojević 2012, 2013, 2015; Radivojević and Kuzmanović Cvetković 2014; Radivojević and Rehren 2016; Radivojević *et al.* 2010a). These are predominantly malachite mineral and ore samples, most likely roughly beneficiated (no samples larger than 2–3 cm in length, see Appendix B_Ch11), and very importantly, without any significant spatial pattern in the excavated area of Trench 18 or its extension (T18ext henceforth). These minerals were discovered in all areas, whether in living or economic spaces, inside the dwellings and other features, and across the excavation spits, which is why they have also been found by previous excavation campaigns (Šljivar 1993–2009).

During the 2012 and 2013 campaigns, Trench 18/T18ext, yielded c. 1300 malachite and azurite minerals, including malachite beads but excluding sherds with traces of malachite. The uncovered copper mineral samples have macroscopically similar characteristics to samples from Belovode: a prevailing number of green (malachite) minerals with black/dark specs, others that are more purely green, with an occasional blue mineral (azurite) (see Figure 1).

The initial number of 14 samples related to metal production for this settlement (See Chapter 5, this volume), was expanded and enriched, with ten new fragments of production debris: slagged sherds (B23/12 and B47/12/1), individual slags (B24/12/2 and B47/12/2), metal droplets (Bf21/12, B29/12, B47/12/3, Bf43/13 and Bf56/13) and a fragment of a metal artefact (B71/12) (Tables 1 and 2). Of these, B23/12, B24/12/2, Bf21/12 and B47/12/(1-3) were all found in the eastern corner of Trench 18, where a surface covered with ashes, charred and burnt soil (Feature 6) emerged from the base of spit 5 (where B23/12, B24/12/2 and B29/12 were found) and continued throughout spit 6 (with Bf21/12 and all B47/12 in the vicinity) (Figure 2).

This cluster also included a copper mineral B46/12, the fragment of a metal artefact (B71/12) and a sherd stained with malachite (B62/13), coming from Trench

18ext (see Appendix B_Ch11 and Table 1). Feature 6 has a distinctive bowl-shaped appearance, with initially scattered signs of burnt and charred soil narrowing down to what looks like a pit (Figure 3), c. 75 x 35 cm in size in the upper part, and 66 cm in length at the bottom. Given the spatial association of metallurgical debris from spits 5 and 6 with the work area of Feature 6, and the indicative bowl-shaped feature it is suggested that these all comprise a single unit/context. Interestingly, the whole eastern area of Trench18, including its extension, appears to be an economic area in which various activities took place throughout all Belovode horizons (see Chapter 5). Direct ¹⁴C dates are associated with Bf21/12 and all B47/12 samples (both through animal bones, see Chapter 37, Table 1; here in Table 2). In Chapter 37, Marić *et al.* model the site chronology using Bayesian statistical method, which combines both the radiocarbon dates and the relative stratigraphy recorded during the excavation. These modelled dates are presented in Table 2 and will be referenced when discussing the dating of metallurgical samples at Belovode. Of note in this context is that ¹⁴C dates for spit 6 (Bf21/12 and B47/12/1-3) are argued to be less secure than direct dates for Feature 6 (see Table 2), as the latter is a well-defined feature in the Belovode stratigraphy, and the former comes from the spit layer, and could easily have been intrusive (See Chapter 37, Figure 2).

Hence, the modelled dates for samples Bf21/12 and B47/12/1-3 are associated with Horizon 1a, which starts at 4818–4693 cal. BC (95.4% prob.) and ends at 4702–4541 cal. BC (95.4% prob.), or possibly lasts from 4776–4709 to 4689–4550 cal. BC (68% prob.). Samples B23/12, B24/12/2, and potentially B29/12, can also be closely (and more directly) associated with modelled dates for F6, which starts at 4702–4541 and ends at 4600–4404 cal. BC (95.4% prob.), or possibly has a span of 4689–4550 to 4572–4481 cal. BC (68% prob.). Overall, a rough estimate places the activities related to this assemblage in the 47th century BC. The fragment of a metal artefact, B71/12, although part of the same cluster in T18ext, is associated with a dwelling structure (Feature 3) rather than the production assemblage, and hence could be dated slightly earlier, starting at 4818–4693 and ending at 4702–4541 cal. BC (95.4% prob.), or possibly between 4776–4709 to 4689–4550 cal. BC (68% prob.).

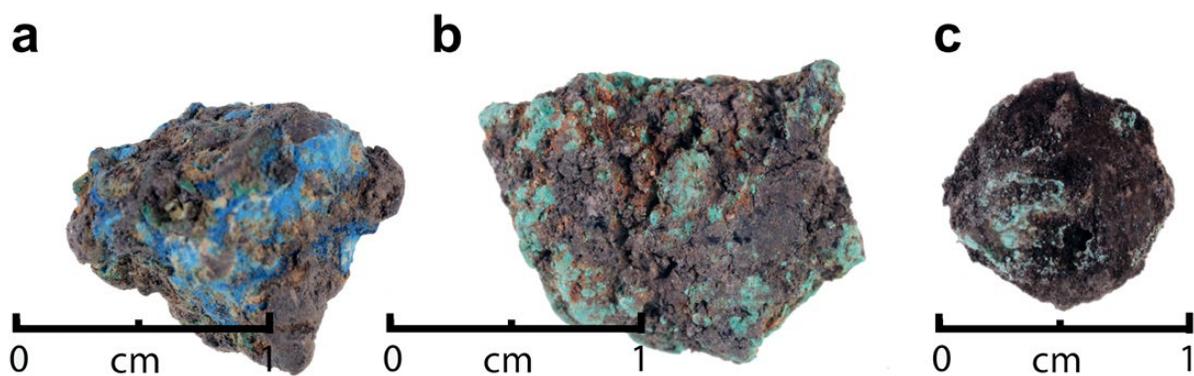


Figure 1. Typical copper minerals found in the site of Belovode: a) malachite; b) azurite and c) black and green manganese-rich copper mineral.

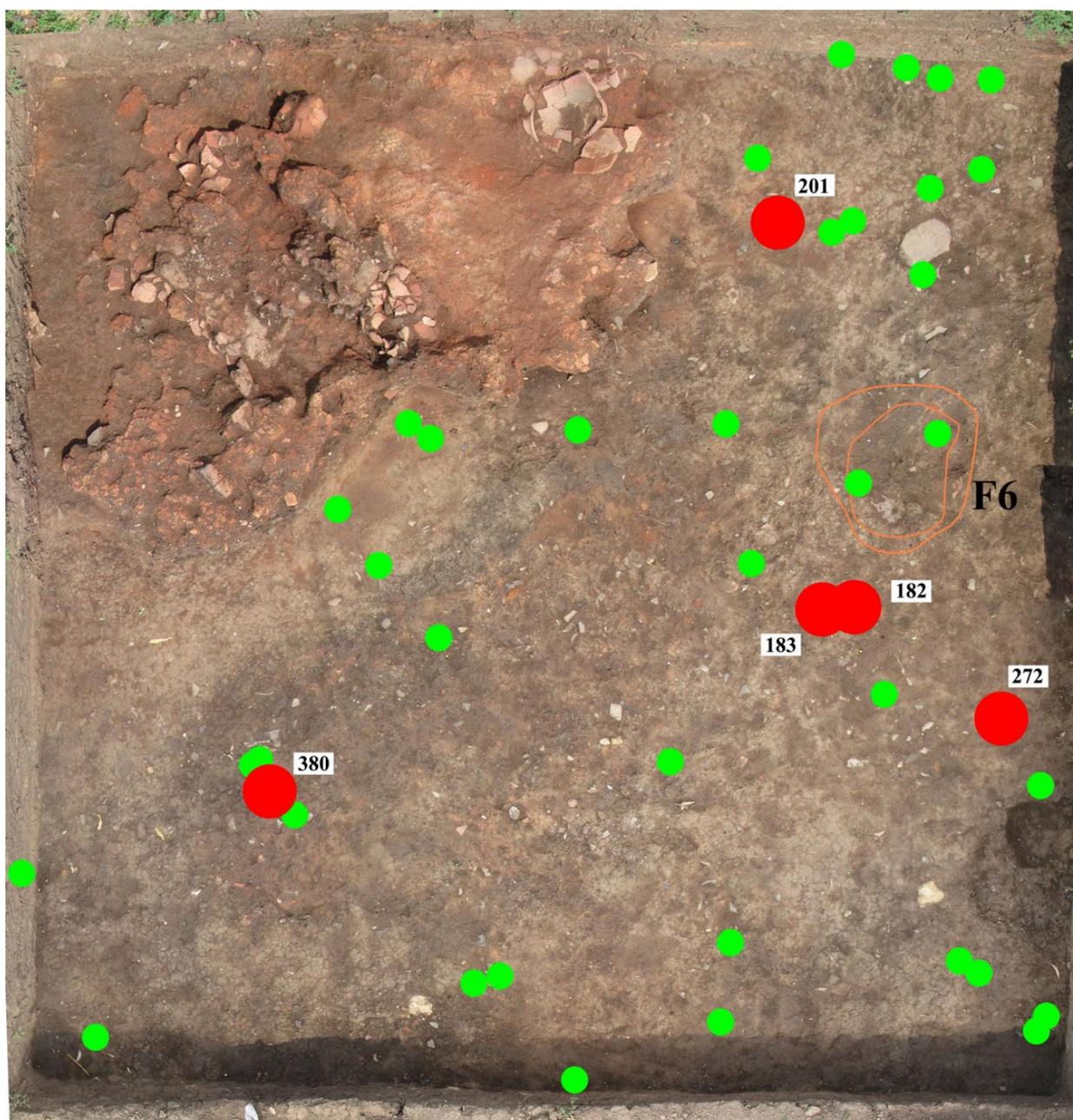


Figure 2. Trench 18 situation in spits 5 and 6, with indicated Feature 6, metallurgical finds as red spots (EDM numbers, see Table 1) and malachite finds as green. EDM 182: B23/12; 183: B24/12/2; 201: B29/12; 272: B47/12/1-3, Bf21/12; and 380: B71/12 (prepared by M. Marić).

Table 1. The list of minerals and metallurgical materials from excavation campaigns Belovode 2012 and 2013. Note an indicated subset analysed in depth with various analytical instruments.

No	trench	spit	find no.	EDM	type of material	metallurgy	OM	SEM-EDS	EPMA	LIA	NAA	LA-ICP-MS	weight (g)	reasoning for provenance analysis
B21/13	18ext	S4	1378	420	malachite (green)					X	X			top layer malachite
B23/12	18	S5	77	182	slagged sherd		X	X	X	X	X	X	11.04	slagged sherd
B24/12_2	18	S5	78	183	slag		X	X	X			X	0.42	slag
B29/12	18	S5	88	201	metal droplet		X	X					0.65	
B62/13	18ext	S5	1495	480	sherd with malachite									
Bf7/12	18	S5	70	166	malachite (black and green)		X	X					0.64	
Bf21/12	18	S6	112	272	metal droplet	X	X		X	X	X	X	3.78	metal droplet
Bf22/12/2	18	S6	116	276	mineral		X	X		X	X		5.07	workshop area mineral
Bf21/12	18	S6	111	271	haematite		X	X					1.73	
B41/12	18	S6	102	258	malachite (black and green)		X	X						
B46/12	18	S6	111	271	malachite (green)		X	X					0.66	
B47/12(1/2/3)	18	S6	112	272	slagged sherd (1) + slag (2) + metal droplet (3)		XXX	XXX	X/2	X/3	X/3	X	(1) 22.47; (3) 0.83; (2) 0.36	production evidence / metal
B52/12	18	S6	124	285	malachite bead									
B71/12	18/F3	S6	159	380	metal artefact	X	X	X	X			X		
B68/13	18ext	S6	1567	490	malachite (green)									
Bf25/12	18	S6	124	285	malachite bead									
B78/12	18/F4	S7	168	429	malachite		X	X					0.21	
B80/12	18	S7	173	434	malachite									
B171/12	18	S7	312	969	malachite bead									
B91/13	18ext	S8	1645	526	azurite									
B198/12	18	S9	1104	361	sphalerite									
Bf11/12	18	S9	1085	353	malachite bead									
B42/13	18	S11	1417	453	malachite bead									
Bf77/12	18/F9	S11	256	760	malachite bead									
Bf30/13	18	S11	1407	443	malachite bead									
Bf43/13	18/F21	S12	1492	477	metal droplet	X	X	X		X	X			metal droplet
B108/13	18	S13	1691	550	malachite (black and green)					X	X			associated with Bf43/13
C_B5/13	18ext	S13	2201	762	malachite bead									
B143/13	18	S13	1762	598	malachite									
B350/13	18ext/F39	S13	2429	881	malachite					X	X			Near ash feature 39

Table 1 continued. The list of minerals and metallurgical materials from excavation campaigns Belovode 2012 and 2013. Note an indicated subset analysed in depth with various analytical instruments.

No	trench	spit	find no.	EDM	type of material	metallography	OM	SEM-EDS	EPMA	LIA	NAA	LA-ICP-MS	weight (g)	reasoning for provenance analysis
B408/13	18ext	S13	2446	882	ash		X	X						
B409/13	18ext	S13		2266/71	pot F35									
Bf56/13	18/F21	S13	1608	506	metal droplet		X	X						
Bf111/13	18ext/13	S13	2282	803	malachite bead									
Bf125/13	18ext	S14	2350	863	silverish ore									
B95/13	18ext	S14	1532	669	malachite bead									
B155/13	18	S14	1785	612	copper mineral					X	X			a different type of a copper mineral
B361/13	18ext/F32	S15	2496	899	malachite bead									
C_B7/13	18	S17	2319	836	Malachite bead									
C-B10/13	18	S18	2510	905	green stone bead float									
B385/13	18	S19	2596	937	malachite					X	X			last malachite occurrence
B387/13	18	S19	2598	939	malachite									

Table 2. Direct dating of metallurgical materials from Belovode

No	trench	spit	find no.	EDM	horizon	type of material	associated feature	modelled C14-date 1σ	modelled C14-date 2σ	directly dated feature/spit (2σ)
B23/12	18	S5	77	182	1a	slagged sherd	F6	4689-4550 to 4572-4481 cal. BC	4702-4541 to 4600-4404 cal. BC	MAMS23380 - 4724-4555 cal. BC
B24/12.2	18	S5	78	183	1a	slag	F6	4689-4550 to 4572-4481 cal. BC	4702-4541 to 4600-4404 cal. BC	MAMS23380 - 4724-4555 cal. BC
B29/12	18	S5	88	201	1a	metal droplet	F6	4689-4550 to 4572-4481 cal. BC	4702-4541 to 4600-4404 cal. BC	MAMS23380 - 4724-4555 cal. BC
Bf21/12	18	S6	112	272	1a	metal droplet	F6 (S6)	4776-4709 to 4689-4550 cal. BC	4818-4693 to 4702-4541 cal. BC	MAMS23380 - 4724-4555 cal. BC
B47/12(1/2/3)	18	S6	112	272	1a	slagged sherd (1) + slag (2) + metal droplet (3)	F6 (S6)	4776-4709 to 4689-4550 cal. BC	4818-4693 to 4702-4541 cal. BC	MAMS23380 - 4724-4555 cal. BC
B71/12	18/F3	S6	159	380	1b	metal artefact	F3 base	4776-4709 to 4689-4550 cal. BC	4818-4693 to 4702-4541 cal. BC	MAMS23376 - 4778-4611 cal. BC
Bf43/13	18/F21	S12	477	1492	2	metal droplet	F21	4899-4798 to 4776 -4709 cal. BC	4950-4756 to 4818-4693 cal. BC	MAMS23378 - 4872-4720 cal. BC
Bf56/13	18/F21	S13	506	1608	2	metal droplet	F21	4899-4798 to 4776 -4709 cal. BC	4950-4756 to 4818-4693 cal. BC	MAMS22079 - 4847-4722 cal. BC



Figure 3. Feature 6 in Trench 18, once emptied. Note charred and burnt soil with traces of ashes (white).

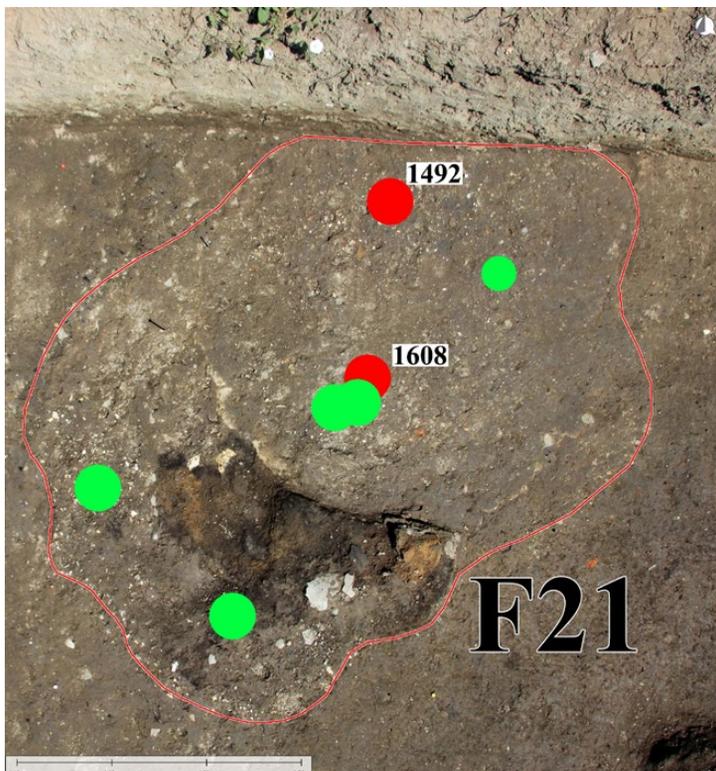


Figure 4. Feature 21. Note red dots for B43/13 (No. 1492) and B56/13 (No. 1608).

Significantly, the two copper metal droplets from Feature 21 (Bf43/13 and Bf56/13), spits 12 and 13 respectively (see Tables 1 and 2, Figure 4), are currently the earliest directly dated evidence for metal production in T18ext. They are directly associated with the dates for Feature 21, which starts at 4950–4756 and ends at 4818–4693 cal. BC (95.4% prob.), or possibly has a span of 4899–4798 to 4776–4709 cal. BC (68% prob.). In the discussion below we place these within the 49th century.

During the excavation we were not able to identify any solid structure that could have been used for metallurgical activities (such as a furnace or smelting installation). However, a vessel bottom found embedded in the soil, with traces of ashes and charred surfaces around it might have been related to a firing procedure involving copper smelting, especially if considered in the context of a nearby ash bin that partially extended under the eastern profile of the trench extension (see Figure 5). Figure 5c-d shows a fragmented vessel bottom, c. 20 cm

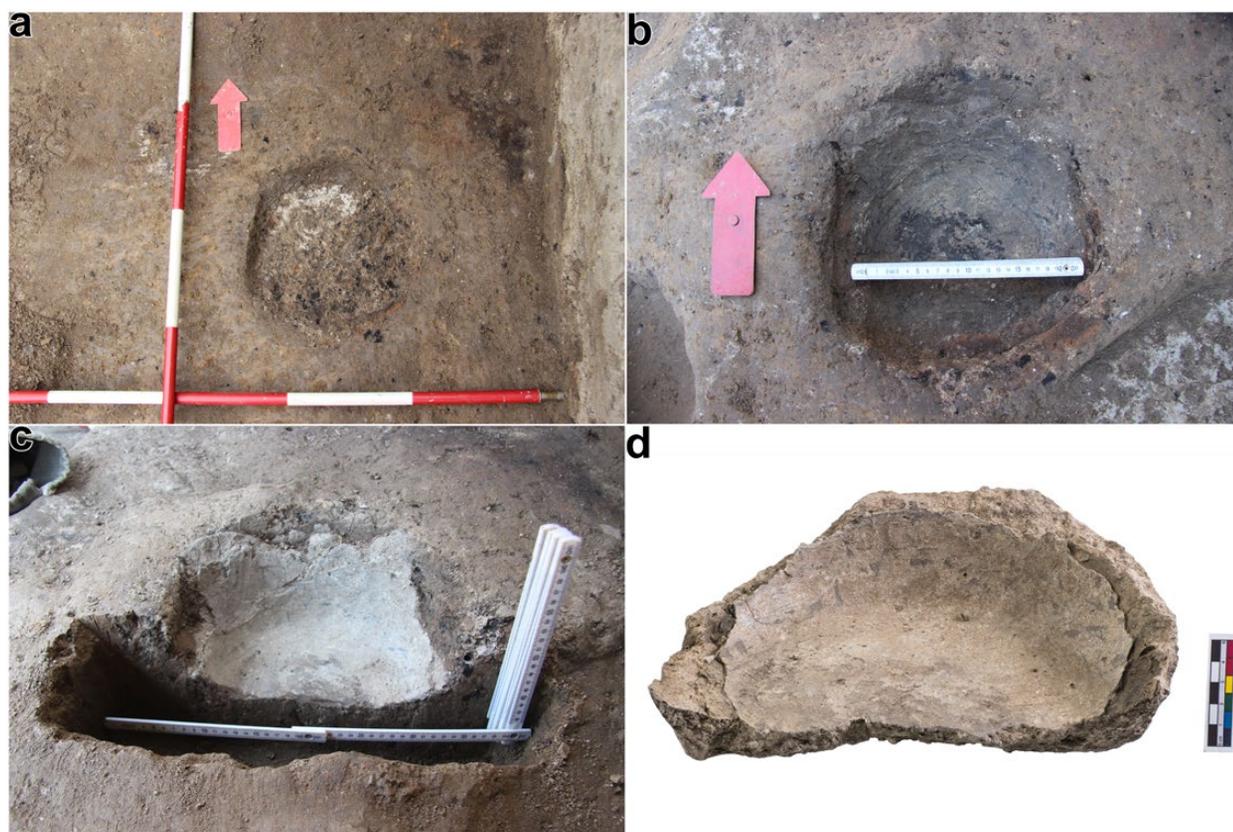


Figure 5. Feature 35, with traces of ash and charred soil (a, b) and the vessel bottom dug into the soil (c) and once excavated and cleaned (d).



Figure 6. Experimental reconstruction of Feature 35 (see Figure 5) in 2013. a) A smelting installation set up with a tuyère (not an original find but hypothesised for this experiment); b) post-smelting situation. Note no adhering slags or other remains in the vessel, despite a successful smelt taking place in it.

in diameter, as part of Feature 35, surrounded by ashes, burnt soil and charcoal (Figure 5a-b). The surface was analysed with pXRF but there were no detectable traces of metal contamination, hence its connection with the smelting process is only assumed due to the unusual field situation. Yet, the same Belovode horizon (2) and excavation spit (13) produced the earliest find of metal

production in the Trench 18 excavations: Bf56/13, a metal droplet (and associated Bf43/13 droplet). The direct date for Feature 35 indicates its start at 5003–4845 cal. BC (95.4% prob.), or possibly 4987–4857 cal. BC (68% prob.) (Chapter 37, Table 1), hence very close to the 49th century BC, the dating of the metal droplets in Feature 21 (Table 2).

In September 2013, the excavation team ran a series of copper smelting experiments and reconstructed this particular vessel bottom feature as part of the smelting installation (Figure 6a). Whilst we successfully extracted metal in this installation, there was no slag or any traces of production left attached to the vessel bottom (Figure 6b). This prompted us to conclude that such an installation could indeed have been used to smelt metal at Belovode, without exhibiting commonly encountered evidence, such as slag or green staining, either on the inside or adhering to its walls. This experiment will be reported elsewhere.

Methodology

The sampling strategy initially involved selecting materials on the basis of their visual appearance, and their response to a magnet (slag and slagged materials), in the field and laboratory. The research collection was catalogued, measured and photographed prior to sample preparation and analysis (Table 1). The following protocol for sample preparation has already been published (Radivojević and Rehren 2016) but is presented here for convenience in a shortened form. Samples selected for microstructural and compositional study were cut to size (where necessary) using a fine, diamond-coated circular saw. They were then washed with water, dried and mounted in epoxy resin. The mounted blocks were then ground using abrasive paper (1200 and 2400 grit) and polished using diamond pastes (down to 1 µm and 0.25 µm). The polished blocks were washed in an ultrasonic bath and rinsed with ethanol between each grinding and polishing stage. The initial analytical stage consisted of reflected light microscopy (OM), with photomicrographs taken on Leica and Olympus microscopes at 25x, 50x, 100x, 200x, 500x and 1000x (Table 3a). Polished blocks of artefacts (Bf21/12 and B71/12) were prepared for metallographic examination using ammonia hydrogen peroxide as an etchant, made from equal proportions of ammonia (NH₄OH), water and 3% H₂O₂.

For the following compositional analysis, samples were carbon-coated for examination under the Scanning Electron Microscope with Energy Dispersive Spectrometry (SEM-EDS, Oxford Instrument's INCA X-cite) and Electron Probe Micro Analysis (EPMA), respectively, both at an accelerating voltage of 20 kV. The EPMA was used only for the samples which contained a distinctive copper metal phase. All analyses were conducted by the first author, barring EPMA, which were conducted by Kevin Reeves, former technician at the Wolfson Archaeological Science Laboratories, UCL Institute of Archaeology, London. Table 1 includes information pertaining to samples analysed using OM and SEM-EDS analysis. The acquired data are corrected against certified reference materials (CRM), analysed

under the same conditions as the metallurgical materials on Superprobe JEOL- JXA-8600. A correction factor was applied only in cases where divergence (relative error) was higher than 10% (Table 3b). The corrected values are reported below, and uncorrected 'raw' data provided in the Appendix B_Ch11.

Where possible, EPMA was used for analysing copper metal phases, enabling the detection of elements present at the 10 ppm (0.001%) level. Seventeen elements were searched for in all samples (Table 3a), with the analytical background of these adjusted during analysis with respect to the CRM for alloyed copper. Each sample had eight to eleven runs to obtain the most precise data. In order to assess the true presence of trace elements in copper metal phases in various samples, all gained values were assessed in relation to measurements acquired for copper alloy CRM; a correction factor was applied only in cases where divergence (relative error) was higher than 5% (Table 3c). During data analysis and interpretation, the threshold for trusted values for trace elements was established at ≥ 100 ppm. Although the data are reported as µg/g (microgram per gram), we shall use ppm (parts per million) to discuss them throughout this monograph.

A subset of metal production and finished metal artefacts samples were sent to Curt-Engelhorn-Centre for Archaeometry (Mannheim, Germany) for high resolution / low threshold analysis of metal phases within these samples with a Thermo iCAP Q inductively coupled plasma mass spectrometer coupled with a Resonetics laser ablation system (Table 3a). The following protocol was followed: samples were prepared on a specimen holder. The laser system was adjusted to a spot size of 73 µm for the pre-ablation cleaning step and 58 µm for ablation at 10 Hz and an energy density of 5 mJ. Helium was used as carrier gas (600ml/min). The plasma power was set to 1400 W. Cool, Auxiliary and Argon gas flow were adjusted to 13.0, 0.7 L/min and 0.9 L/min, respectively. The following isotopes were determined: ²⁴Mg, ²⁷Al, ²⁹Si, ³¹P, ⁵²Cr, ⁵⁵Mn, ⁵⁷Fe, ⁵⁹Co, ⁶⁰Ni, ⁶³Cu, ⁶⁸Zn, ⁷⁵As, ⁸²Se, ¹⁰⁷Ag, ¹⁰⁰Mo (¹⁰⁰Ru), ¹¹¹Cd, ¹¹³In (¹¹³Cd), ¹¹⁵In (¹¹⁵Sn), ¹¹⁸Sn, ¹²¹Sb, ¹²⁵Te, ¹⁹⁷Au, ²⁰⁸Pb, ²⁰⁹Bi. Further isotopes were monitored but not quantified: ¹⁰¹Ru, ¹⁰³Rh, ¹⁰⁴Pd (¹⁰⁴Ru), ¹⁰⁵Pd, ¹⁰⁶Pd (¹⁰⁶Cd), ¹⁸⁹Os, ¹⁹¹Ir, ¹⁹⁵Pt. Line ablations with a length of 300-400µm were performed. Data were collected in time resolved mode, including around 30s integrated gas blank signal before each sample signal. The data acquisition sequence consists of blocks up to three samples each ablated five times which were enclosed by a blocks of solid reference materials. Quantification was carried out using ablation yield correcting factors with sum normalisation (Lin *et al.* 2016). A set of different external standards (BAM 211, BAM 227, BAM 376, BAM 375 and NIST 400 as well as NIST610 and NIST 612) were used to quantify major, minor and trace elements (Hawkins *et al.* 2016; Walaszek *et al.* 2013).

Table 3a: Analytical instruments used in this study, aim of analysis and relevant analytical parameters.

Instruments	Aim of Analysis	Analytical Parameters
Reflected Polarized Light Microscopy (Leica DMLM and Olympus BX60)	Phase identification and visual characterisation of microstructure	Plane polarized light and crossed polarized light were applied to examine phases in samples, their colour, homogeneity, porosity and inclusions (shape, size and uniformity). Cross-polarized light was also applied for internal reflection and identifying the composition of phases present. The microscope was equipped with a Nikon digital camera, with highest magnification of 1000x.
SEM-EDS Scanning Electron Microscopy with Energy Dispersive Spectrometry (Superprobe JEOL- JXA-8600)	1. Phase identification in samples using electron images and area/point analyses 2. Quantitative compositional analyses of observed phases 3. Observation of the relationships between phases on the basis of their atomic number contrast	Backscattered electron (BSE) imaging used. All materials analysed on JXA-8600. The accelerating voltage was 20 kV, with average dead-time of 35-40 % and working distance of 10 mm. All data are presented as normalized with stoichiometrically added oxygen, if not otherwise stated. The iron content is presented as FeO, which here stands for total iron (both valencies).
EPMA Electron Probe Micro Analysis (Superprobe JEOL-JXA-8100)	Compositional analysis of copper metal phases in all samples (down to trace element level)	All samples analysed at an accelerating voltage of 20 kV, beam current 50 nA, with average dead-time of 35-40 % and working distance of 10 mm. The following elements were checked for: Se, Zn, Cu, Fe, As, Ag, Cl, Te, S, Au, Sn, Bi, Co, Sb, Ni, Mn, Pb. All data presented as wt% and ppm, with the trusted values for the latter established at ≥ 10 ppm.
LA- ICP-MS Thermo iCAP Q inductively-coupled plasma mass spectrometer coupled with a Resonetics laser ablation system (ArF, 193 nm). Located externally at the Curt-Engelhorn-Centre for Archaeometry, Mannheim, Germany	Trace element analysis (low detection threshold) of copper metal phases in production debris and selected metal artefacts	The parameters of the ICP-MS were optimized to ensure a stable signal with a maximum intensity over the full range of masses of the elements and to minimize the formation of oxides and double-ionized species. The following elements were checked for: Co, Ni, Cu, Zn, As, Ag, Sb, Te, Pb, Bi, Mg, Al, Si, Mn, Cd, Sn, Se, P, Fe. The data is presented as wt% and ppm. Mg, Al, Si, Mn, Cd and Sn were below 10 ppm; Se below 30 ppm, and P and Fe below 50 ppm; the rest of the readings are reported in the indicated table in the text.

Results: Technology of metal making at Belovode

A total of fifteen objects were selected for in-depth analysis: five minerals/ores and ten examples of production evidence (slags, slagged sherds, metal droplets and a metal artefact fragment). All presented evidence for copper-based metallurgy, which in its nature and quantity is consistent with previous studies on archaeometallurgical materials from this site (Radivojević 2007, 2012, 2013, 2015; Radivojević and Rehren 2016; Radivojević *et al.* 2010a).

Processing: archaeological minerals

Five mineral samples were analysed (see Table 1) for microstructure and composition: Bf7/12, Bf22/12/2, B41/12, B46/12 and B78/12. All are copper based, with Bf22/12/2 exhibiting pure copper oxide (Figure 7), and the rest presenting a mixture of pure green (malachite) occasionally mixed with oolitic structures that contain high MnO content, reaching just below 40 wt% (Table 4, Figure 7). These results are in line with the findings of previous studies on Belovode minerals,

Table 3b. SEM-EDS compositional data of certified reference materials (CRM) for basalt glasses: BIR1, BCR-2, BHVO-2, given in wt%. All measured values are presented against certified average values for CRMs, with correction value calculated only for relative errors above 10% divergence. The averages of all correction values and correction factor are given in the bottom two lines; the correction factor was applied for calculating real values for analysed samples.

	Na2O	MgO	Al2O3	SiO2	P2O5	K2O	CaO	TiO2	MnO	FeO
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
BIR1	1.45	8.71	14.35	49.86			13.54	1.24	0.08	10.76
BIR1	1.35	8.7	14.59	49.71			13.58	0.99	0.11	10.99
BIR1	1.47	8.42	14.5	49.85			13.72	1.08	0.32	10.65
<i>average measured (1)</i>	1.42	8.61	14.48	49.81			13.61	1.10	0.17	10.80
<i>certified value (2)</i>	1.82	9.70	15.50	47.96			13.30	0.96	0.18	11.30
<i>absolute error (3) = (1)-(2)</i>	-0.40	-1.09	-1.02	1.85			0.31	0.14		-0.50
<i>relative error in % (4) = (3/1)*100</i>	-27.87	-12.66	-7.04	3.71			2.30	12.99		-4.63
<i>correction value (5) = (4)/100</i>	-0.28	-0.13	-0.07	0.04			0.02	0.13		-0.05
BCR-2	2.49	3.28	12.51	55.56	0.61	1.94	7.49	2.6	0.27	13.25
BCR-2	2.65	3.15	12.44	55.52	0.45	2.06	7.52	2.57	0.25	13.38
BCR-2	2.41	3.04	12.17	56.32	0.49	1.91	7.63	2.38	0.27	13.38
BCR-2	2.56	3.36	12.55	56.06	0.46	1.89	7.47	2.35	0.2	13.11
<i>average measured (1)</i>	2.53	3.21	12.42	55.87	0.50	1.95	7.53	2.48	0.25	13.28
<i>certified value (2)</i>	3.16	3.59	13.50	54.10	0.35	1.79	7.12	2.26	0.24	13.80
<i>absolute error (3) = (1)-(2)</i>	-0.63	-0.38	-1.08	1.77		0.16	0.41	0.22		-0.52
<i>relative error in % (4) = (3/1)*100</i>	-25.02	-11.93	-8.72	3.16		8.21	5.41	8.69		-3.92
<i>correction value (5) = (4)/100</i>	-0.25	-0.12	-0.09	0.03		0.08	0.05	0.09		-0.04
BHVO-2	1.77	6.54	12.41	51.51	0.22	0.54	11.65	3.01	0.22	12.12
BHVO-2	1.73	6.42	12.53	51.39	0.22	0.64	11.7	2.95	0.23	12.19
BHVO-2	1.73	6.26	12.59	51.63	0.02	0.54	12.22	2.94	0.2	11.86
<i>average measured (1)</i>	1.74	6.41	12.51	51.51	0.15	0.57	11.86	2.97	0.22	12.06
<i>certified value (2)</i>	2.22	7.23	13.50	49.90	0.27	0.52	11.40	2.73	0.20	12.30
<i>absolute error (3) = (1)-(2)</i>	-0.48	-0.82	-0.99	1.61		0.05	0.46	0.24		-0.24
<i>relative error in % (4) = (3/1)*100</i>	-27.34	-12.85	-7.91	3.13		9.30	3.85	7.98		-2.02
<i>correction value (5) = (4)/100</i>	-0.27	-0.13	-0.08	0.03		0.09	0.04	0.08		-0.02
calculating correction factor										
	Na2O	MgO						TiO2		
BIR1	-0.28	-0.13						0.13		
BCR-2	-0.25	-0.12								
BHVO-2	-0.27	-0.13								
average	-0.27	-0.12						0.13		
correction factor ALL	1.27	1.12						0.87		

Table 3c. EPMA compositional data of certified reference materials (CRM) for copper alloys (bronzes): 32XSN6 and 33XGM29, given in wt%. All measured values are presented against certified average values for CRMs, with correction values calculated for relative errors above 5%. The averages of all correction factors are given in the bottom two lines; the correction was applied to all EPMA readings of metal phases presented below.

	Ni	Cu	Se	Co	As	Zn	Mn	Fe	Ag	Sn	Bi	S	Sb	Pb	Cd	Au
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
32X SN6 average observed (1)	0.218	87.436	0.001	0.770	0.489	1.069	0.003	0.113	1.071	7.026	0.080	0.048	0.297	1.202	0.077	0.010
certified value (2)	0.203	86.390		0.655	0.764	1.170	0.002	0.099	1.159	7.310	0.158	0.018	0.323	1.559	0.090	0.007
absolute error (3)= (1)-(2)	0.01	1.05		0.11	-0.27	-0.10	0.00	0.01	-0.09	-0.28	-0.08	0.03	-0.03	-0.36	-0.01	0.00
relative error in % (4) = (3/1)*100	6.67	1.20		14.91	-56.21	-9.43	27.27	12.39	-8.27	-4.04	-97.50	62.50	-8.94	-29.71	-16.97	24.74
correction value (5) = (4)/100	0.07			0.15	-0.56	-0.09	0.27	0.12	-0.08		-0.98	0.63	-0.09	-0.30	-0.17	0.25
33XGM29 average observed (1)	0.030	89.383	0.000	0.004	0.000	4.236	0.004	0.014	0.003	6.232	0.008	0.010	0.000	0.050	0.005	0.000
certified value (2)	0.029	89.360				4.230		0.010	0.003	6.120	0.002	0.002		0.050		
absolute error (3)= (1)-(2)	0.00	0.02				0.01		0.00	0.00	0.11	0.01	0.01		0.00		
relative error in % (4) = (3/1)*100	3.99	0.03				0.15		28.17	18.75	1.80	75.32	76.92		-0.60		
correction value (5) = (4)/100								0.28	0.19		0.75	0.77				
calculating correction factor	Ni	Cu	Se	Co	As	Zn	Mn	Fe	Ag	Sn	Bi	S	Sb	Pb	Cd	Au
average correction value	0.07			0.15	-0.56	-0.09	0.27	0.20	0.05		-0.11	0.70	-0.09	-0.30	-0.17	0.25
correction factor	0.93			0.85	1.56	1.09	0.73	0.80	0.95		1.11	0.30	1.09	1.3	1.17	0.75

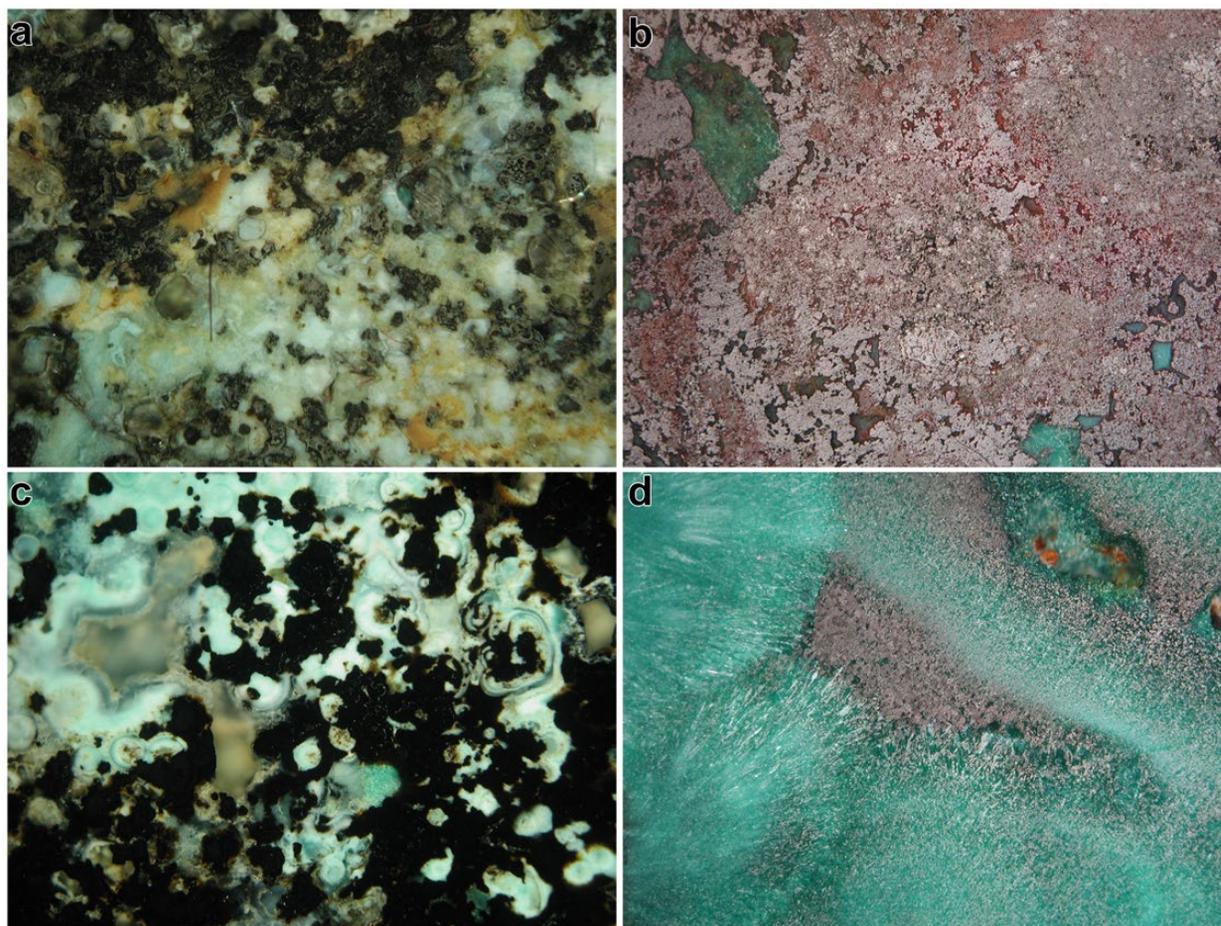


Figure 7. Photomicrographs of copper minerals from Belovode, cross polarised light. a) Bf7/12, magnification 50x, width 3.2mm. Note black (Mn-rich) and green (Cu-rich) phases; b) Bf22/12/2, magnification 50x, width 3.2mm. Note reddish/violet phase of tenorite and cuprite; c) B41/12, magnification 100x, width 1.6mm. Note black (Mn-rich) and green (Cu-rich) phases; B46/12, magnification 50x, width 3.2mm. Note pure green (copper carbonate – malachite) phase.

Table 4. SEM-EDS compositional data for oolitic (dark) phases in minerals Bf7/12 and B41/12. All values are averages of ten to fourteen analyses of each sample / phase and normalised to 100%.

	Al ₂ O ₃	SiO ₂	P ₂ O ₅	K ₂ O	CaO	MnO	FeO	CoO	NiO	CuO	ZnO
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
Bf7/12	9.0	15.8	2.7	0.2	2.1	39.3	0.9	0.1	0.1	28.6	1.2
B41/12	6.9	14.0	2.6	0.0	2.0	38.0	0.2	0.0	0.0	36.1	0.1

where manganese-rich black and green copper minerals feature prominently in the collection of beneficiated ores used for copper smelting on the site (Radivojević 2007, 2012, 2013, 2015; Radivojević and Rehren 2016; Radivojević *et al.* 2010a) and beyond, in Vinča-Belo Brdo. Previous analyses have also shown that sulfidic minerals are also a common inclusion in secondary copper ores from Belovode.

Production: slagged sherds and slags

Installations/slagged sherds

The two slagged sherds (B23/12 and B47/12/1) show close technological and spatial association with the cluster of production debris around the Feature 6 area in Trench 18 (Figure 2, Table 2). Given these links, we are

inclined to ascribe the entire assemblage to Horizon 1b (see Chapter 10), which is in line with the foundations of Feature 3, or the dwelling feature in Trench 18. Both finds are from the bottom of spit 5 and the beginning of spit 6; given the artificial nature of excavation spits, the association is without doubt. Also, the microstructural and compositional relationship presented below strengthens our assumption that these belong to a single or two closely associated smelting events.

Visual inspection of both objects (Figures 8 and 9) shows that the ceramic bodies were unaffected by heat treatment exceeding the initial firing temperature, apart from the single sections in both examples that exhibit a bloated surface stained with a mass of green and grey appearance. These stained surfaces are spread across the broken sections, implying that the sherds were fragmented prior to the contact with

metallurgical activities. This is the same scenario already observed for four slagged sherds discovered in previous excavations (see Chapter 5, this volume) and extensively reported elsewhere (e.g. Radivojević 2013, 2015; Rehren *et al.* 2016). Hence, as with these previous examples, these sherds most likely formed a lining for a hole-in-the-ground smelting installation, leading to some localised burning and bloating of the ceramic and droplets of slag and metal adhering to them. This is further corroborated by the presence of ashes, burnt soil, clay and charred surfaces in the charred pit, Feature 6, associated with these finds; in Figure 3 we see that Feature 6 was deep enough to possibly represent such a hole-in-the-ground installation. Two slagged sherds found in its vicinity strengthen this hypothesis.

The ceramic fabric of Belovode 23/12 and 47/12/1 appears optically dark grey, well kneaded and

tempered with abundant quartz grains, amounting to almost 50 vol% (Figures 10a and 10d). The dense paste looks 'dry', with little indication of potential collapsing. Most of the quartz grains closer to the area of intense vitrification have lost their angularity and decomposed (Figure 10b); this is followed by coarse bloating pores. Noteworthy is the formation of slag on top of these sherds; while in some areas it is clear that the slag had only a short liquid contact with the ceramic surface (Figure 10c), others exhibit a stronger interaction of the slag and ceramic body (Figures 10b and 10d). This is yet another indicator that these sherds were not used as a crucible, but for lining a hole in the ground; the surfaces that show close interaction were most likely closer to the 'hot spot' in the metallurgical installation.

Bulk chemical analyses of the ceramic bodies indicated the use of similar clay for both samples (Table 5), where the consistent ratio of silica to alumina (approximately 5:1) and similar readings of iron oxides, lime, potash and magnesia suggest a common origin for the clay. These were, however, parts of two different pottery vessels,



Figure 8. Slagged sherd B23/12. Note green stain mass adhering to the top and cross sections.



Figure 9. Slagged sherd B47/12/1. Note green staining on one section only.

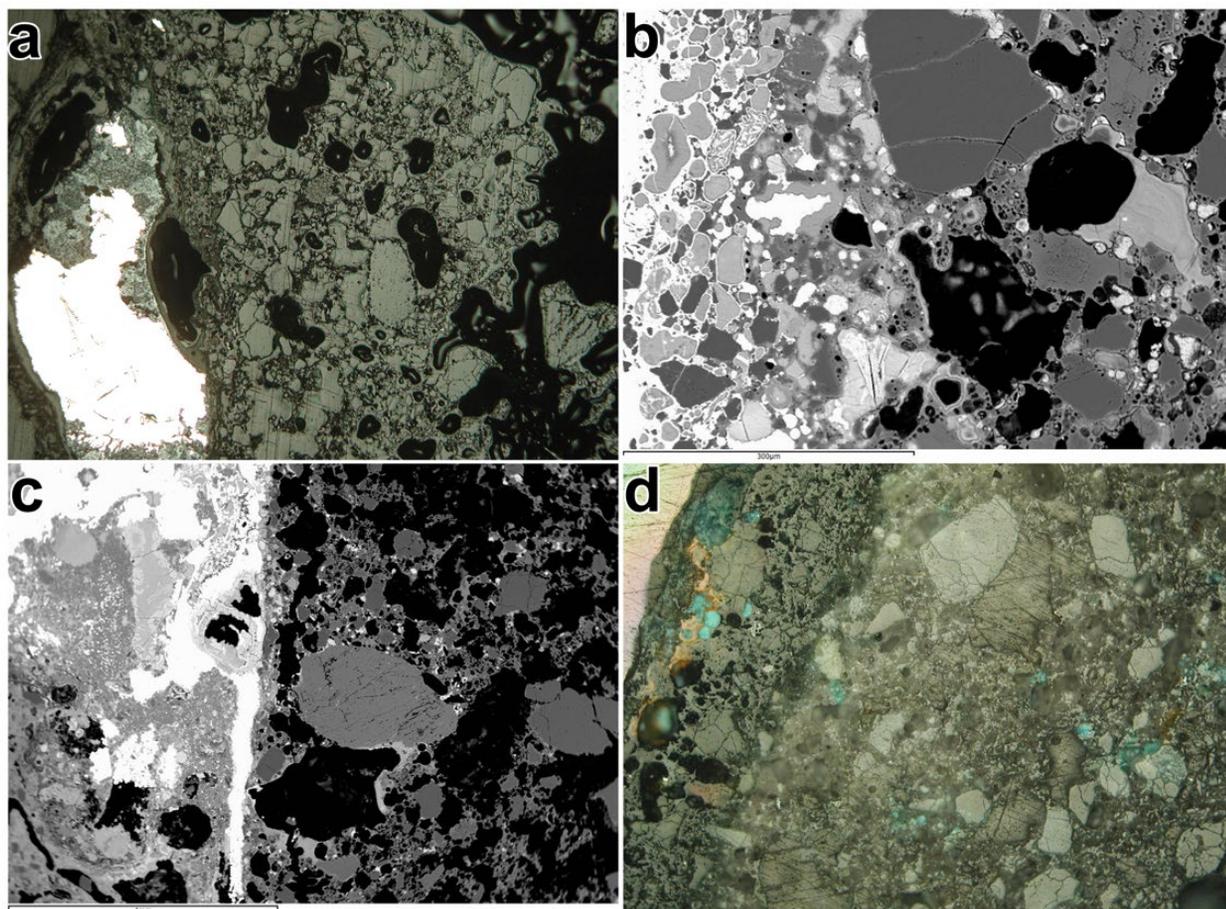


Figure 10. a) Photomicrograph slagged sherd B23/12 taken under plain polarised light, 50x magnification, 3.2 mm width. Note bright metal phase stuck on top of the 'cold' ceramic body; b) Backscattered electron image of a section of B23/12. Note highly reactive ceramic body (right, with decomposing grey quartz grain) and slag (left, with bright metal phases); c) backscattered electron image of a section of B23/12. Note poor reaction of metal phase with ceramic body; d) Photomicrograph of slagged sherd B47/12/1, taken under plain polarised light, 50x magnification, 3.2mm width. Note 'cold' ceramic body moderately fused with slag (left, with green corrosion products).

and were not found in association in the field. The 'hot' or bloated sections of the sherds (Table 5) exhibit a slightly different picture, with greater variability of the silica to alumina ratio (on average 4:1, though it varies from 3:1 to 7:1). Elevated readings of copper (up to 6.8 wt% CuO) and iron oxides (up to 12.4 wt% FeO⁺) and sulfur (only in B47/12/1) represent the ores included in the smelt, while increased potash and lime concentrations would have been due to fuel ash contamination.

Optical microscopy and SEM-EDS analyses revealed a heterogeneous structure of slag and copper-rich materials in both samples (23/12 and 47/12/2). The slag contains newly formed phases and metal prills in a glassy matrix, with corrosion products developing on the edges and inside porosity holes (Figure 11). The main inclusions suspended in the slag matrix are copper 'dross', delafossite and iron-rich spinels (the latter two only for B23/12), suggesting at least the primary smelting nature of the process related to B23/12 (Figure

11a). Both samples are dominated by copper oxide and copper metal prills (Figure 11b and 11c). The chemical analyses of the bulk glassy slag matrix were conducted through area analysis in B23/12, while in B47/12/2 this was not possible due to the uneven distribution of slag and its small volume on this sherd's surface. Spot analyses were conducted to aid the understanding of slag chemistry in both samples, in areas relatively free of copper-based components (Table 6).

The chemistry of the green-stained and bloated outer surface of all slagged sherds revealed significant contamination with copper (up to 27 wt%, with increased levels of MnO and ZnO in B23/12) and fuel ash (elevated readings of CaO, P₂O₅, K₂O, MgO), clearly different from the ceramic composition (Table 5). These data correlate well with the already published slagged sherd and slag samples from Belovode, which include significant readings of MnO and ZnO, and increased values of FeO (Radivojević 2013, 2015; Radivojević and Rehren 2016; Radivojević *et al.* 2010a). Analyses

Table 5. SEM EDS compositional data for ceramic body of slagged sherds B23/12 and B47/12/1, sections unaffected (cold) and affected (bloated) by high temperatures. All values are averages of two to twenty-seven analyses of each sample / phase and corrected with factors based on CRM analysis; the uncorrected data is reported in the Appendix.

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂	MnO	FeO	CoO	NiO	CuO
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
B23/12 cold	2.2	1.5	14.8	71.6	0.1	0.0	3.0	1.4	0.7	0.0	5.3	0.0	0.0	0.0
<i>stdev s</i>	0.6	0.2	1.3	2.5	0.3	0.0	0.4	0.4	0.4	0.0	0.7	0.0	0.0	0.0
B47/12/1 cold	1.4	1.5	12.8	73.5	0.4	0.0	3.0	1.0	0.7	0.0	6.2	0.0	0.0	0.0
<i>stdev s</i>	0.1	0.3	0.2	2.4	0.5	0.0	0.0	0.3	0.3	0.0	1.5	0.0	0.0	0.0
	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂	MnO	FeO	CoO	NiO	CuO
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
B23/12 bloated	4.1	1.3	19.9	63.9	0.0	0.0	4.8	2.5	0.4	0.0	3.8	0.0	0.0	0.2
<i>stdev s</i>	1.7	1.1	3.1	3.5	0.0	0.0	1.2	1.1	1.0	0.0	3.2	0.0	0.0	0.4
B47/12/1 bloated	1.8	1.7	14.7	66.5	1.3	0.4	2.6	1.6	0.7	0.0	6.1	0.0	0.0	3.1
<i>stdev s</i>	0.5	0.3	1.7	5.0	1.3	1.0	1.2	0.4	0.4	0.0	1.9	0.0	0.0	1.8

Table 6. SEM EDS compositional data for slag matrix in slagged sherds B23/12 and B47/12/1, both as spot analysis (relatively free of inclusions) and as area analysis (bulk slag analysis in areas of c. 100 x 100 microns in B23/12). All values are averages of three to twenty-nine analyses of each sample / phase and corrected with factors based on CRM analysis; the uncorrected data is reported in the Appendix.

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂	MnO	FeO	CoO	CuO	ZnO
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
B23/12 spot	0.7	1.9	10.3	47.2	1.7	0.0	4.8	12.0	0.3	3.4	4.2	0.2	13.3	0.4
<i>stdev s</i>	0.7	0.8	3.8	10.1	2.4	0.0	2.0	7.0	0.3	4.1	2.2	0.5	7.0	0.7
B23/12 area	1.4	1.4	13.8	70.6	0.2	0.0	5.1	1.0	0.8	0.0	3.8	0.0	2.3	0.0
<i>stdev s</i>	0.4	0.3	3.4	8.0	0.3	0.0	2.6	0.3	0.6	0.0	0.3	0.0	0.6	0.0
B47/12/1 spot	0.0	1.8	11.6	45.4	1.2	0.0	3.3	7.9	0.6	0.0	6.6	0.0	21.7	0.0
<i>stdev s</i>	0.0	0.2	1.9	6.7	2.2	0.0	1.1	9.0	0.1	0.0	3.8	0.0	2.8	0.0

presented here reinforce the hypothesis of a preference for manganese-rich black and green ores for metal extraction, both at Belovode and across the Vinča culture sites.

The difference in (bulk) chemical composition between the two sherds is also reflected in the formation of phases in the slag matrix. While slagged areas in B47/12/1 are commonly rich in copper 'dross' suspended in slag matrix (Figure 10c), B23/12 also presents manganese- and iron-rich spinels and delafossite (Table 7, Figure 11a). Iron-rich spinels commonly form grey cubic crystals embedded in a glassy matrix, corresponding to the general formula AB₂O₄, where A could be magnesium, zinc, or manganese; B may be aluminium, iron or chromium; and O is oxygen. Judging by its composition, the phase that formed in B23/12 is similar

to the mineral franklinite, (Zn, Mn²⁺)(Fe, Mn³⁺)₂O₄. This is the same as seen in the slagged sherd Belovode 31b, recovered from Trench 3 and published previously (Radivojević 2013: 22).

Delafossite (Cu¹⁺Fe³⁺O₂) is commonly recognised optically as straight grey lathes (Figure 11a). In nature, it is not usually found as a primary mineral, but rather near the base of the oxidised zone of copper deposits. The co-appearance of delafossite and cuprite is indicative of oxidising conditions during the smelt, at around the partial oxygen pressure required to reduce copper from cuprite (Bachmann 1982; Müller *et al.* 2004: 40). The debate on whether delafossite indicates melting or smelting conditions has bodies of evidence supporting both arguments but previously published metallurgical debris from Belovode strongly suggests

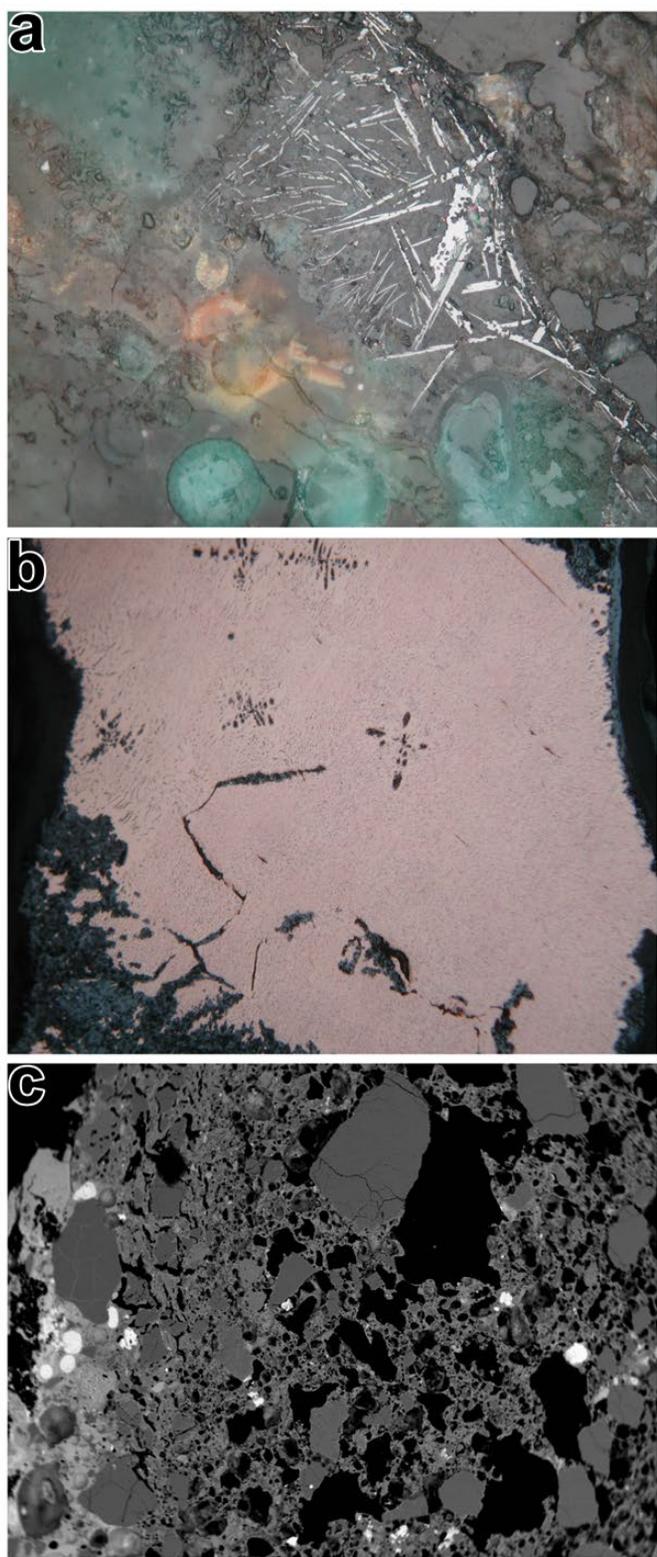


Figure 11. a) Photomicrograph of slagged sherd B23/12 taken under cross polarised light, 500x magnification, 0.34 mm width. Note light grey lathes of delafossite suspended in the glassy slag matrix; b) Photomicrograph of copper metal in slagged sherd B23/12 taken under cross polarised light, 200x magnification, 0.85 mm width. Note the copper-copper oxide eutectic texture and some dendritic cuprite; c) Backscattered electron image of slag section (left, with bright metal prills) in B47/12/1. Note slag forming from fused ceramic with decomposing quartz grains.

moderately reducing conditions indicative of copper smelting (Radivojević 2013; Radivojević and Rehren 2016). The presence of these newly formed phases further corroborates the argument that B23/12 had contact with slag forming in a process that involved manganese-rich copper ores, with relevant readings of zinc and nickel, as in previously published evidence (Radivojević 2013: 22, Table 6). Sample B47/12/1 does not offer the same indication and may have been either part of a different metallurgical process (but not a crucible) or, more likely, simply farther from the ‘hot spot’ in the lined hole-in-the-ground smelting installation in Feature 6 in Trench 18.

Finally, the copper metal phase in both samples demonstrates the nature of the metal produced (Figures 11b and 11c). In B23/12 we see a smaller size of dendritic structures in the metal phase with a pronounced copper–copper oxide eutectic, which could indicate a faster cooling rate (Figure 11b). The very presence of fully molten copper saturated with oxygen suggests exposure to temperatures of around 1070 °C or above for both slagged sherds. In addition, electron microprobe examination of pure copper metal phases in the slag matrix of B23/12 revealed a significant string of trace elements that might be helpful in discerning the type of copper ores used (Table 8). Notable readings indicate S, Ni, Mn, Sn, Bi and Pb and, less significantly, iron content in the copper metal phase, the significance of which to understanding of the slagging process and reducing conditions of the smelt has been discussed at length (Craddock 2001; Craddock and Meeks 1987; Tylecote *et al.* 1977). Concentrations of manganese of up to 150 ppm, nickel reaching 200 ppm and tin on average 70 ppm indicate a distinctive compositional pattern of the copper ore charge, the origins of which will be discussed in Chapter 41. The LA-ICP-MS analysis (Table 8a) are largely consistent with these readings, most notably the prominent Ni content.

In summary, the slagged sherds present further evidence for being used as the lining for a hole-in-the-ground smelting installation, reinforcing previous archaeometallurgical research on this site. Strengthening this argument is the firm contextual association with Feature 6, which includes burnt soil, charred surfaces and ashes, shaped in a bowl-like structure in the excavation layers (Figures 2 and 3). The conditions of the smelt were moderately reducing, as determined by the valency of newly formed copper and iron phases in the glassy matrices of slagged sherds:

Table 7. SEM EDS compositional data for MnFe spinels and delafossite in slagged sherd B23/12. All values are averages of two to nine analyses of each sample / phase and normalised to 100%.

	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂	MnO	FeO	CoO	NiO	CuO	ZnO
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
B23/12 MnFe spinel	2.9	3.7	1.0	0.0	0.0	0.1	0.8	0.0	28.3	53.1	3.8	0.4	3.8	2.5
<i>stdev s</i>	0.0	1.1	0.5	0.0	0.0	0.2	0.4	0.0	5.3	7.3	0.1	0.5	1.7	0.3
B23/12 delafossite	0.1	1.3	4.6	0.7	0.0	0.1	0.7	2.7	4.9	32.0	0.0	0.0	52.6	0.0
<i>stdev s</i>	0.3	0.6	5.9	1.1	0.0	0.3	0.7	0.4	2.2	4.3	0.0	0.0	4.1	0.0

Table 8. EPMA compositional data of different metal prills in the slag matrix of B23/12 slagged sherd (selected significant trace element values), given in wt%. Values above c. 0.01 wt% (100 ppm) are considered reliable based on CRM measurements; values below this are indicative only. All data are corrected for values obtained from the reference material, using a procedure reported in the methodology section. Values sought but not found at levels above c. 0.01 wt% were indicated as not detected (n.d.).

	S	Mn	Fe	Co	Ni	As	Ag	Sn	Te	Pb	Bi	Analytical total
	wt%											
B23-12	0.009	0.015	0.002	0.001	0.008	n.d.	n.d.	0.033	n.d.	0.007	0.032	100.80
B23-12	0.007	n.d.	0.004	n.d.	0.010	0.084	n.d.	n.d.	n.d.	n.d.	n.d.	100.94
B23-12	0.014	n.d.	0.002	0.003	0.010	n.d.	n.d.	0.011	0.012	n.d.	n.d.	100.71
B23-12	0.000	n.d.	n.d.	n.d.	0.001	n.d.	n.d.	n.d.	n.d.	0.038	n.d.	100.97
B23-12	0.006	n.d.	0.004	n.d.	0.021	n.d.	n.d.	0.004	0.014	n.d.	n.d.	100.18
B23-12	0.015	n.d.	n.d.	0.001	0.001	n.d.	n.d.	0.001	0.024	n.d.	n.d.	100.74
B23-12	0.003	0.007	n.d.	0.003	0.012	n.d.	n.d.	n.d.	n.d.	n.d.	0.008	100.32
B23-12	0.005	0.007	n.d.	0.006	0.000	n.d.	n.d.	n.d.	n.d.	0.004	n.d.	100.59
B23-12	0.007	0.013	0.001	n.d.	0.013	0.031	0.004	0.011	n.d.	n.d.	0.027	100.44
B23-12	0.001	0.004	0.002	n.d.	0.007	n.d.	n.d.	0.010	n.d.	0.022	n.d.	99.44

Table 8a. LA-ICP-MS analysis of copper metal phases in production debris and artefacts. Values sought but not found above the indicated detection limit were treated as not detected (n.d.).

	Cu	Co	Ni	Zn	As	Ag	Sb	Te	Pb	Bi
	%	µg/g								
<i>detection limit</i>		1	20	5	37	4	5	2	2	1
B23/12	100.0	10	67	n.d.	n.d.	6	n.d.	n.d.	3	n.d.
B24/12	100.0	16	38	69	n.d.	7	n.d.	n.d.	n.d.	n.d.
Bf21/12	100.0	14	89	15	n.d.	12	n.d.	n.d.	n.d.	n.d.
B47/12	100.0	15	97	12	n.d.	12	n.d.	n.d.	n.d.	n.d.
B71/12	100.0	n.d.	84	23	n.d.	7	n.d.	n.d.	n.d.	n.d.

dross including cuprite (Cu₂O), tenorite (CuO) and delafossite (CuFeO₂). The formation of spinels with a strong presence of manganese, and manganese, nickel and tin in trace element analysis of the copper metal all provide further arguments for primary extraction of copper ores, as well as being indicative of the composition of gangue minerals in the smelted ores.

Free slag pieces

Two free slag pieces (B24/12/2 and B47/12/2) were discovered associated with the two slagged sherds described above, B23/12 and B47/12/1 respectively (Figures 12 and 13). Both are therefore part of the production debris assemblage related to Feature 6. The

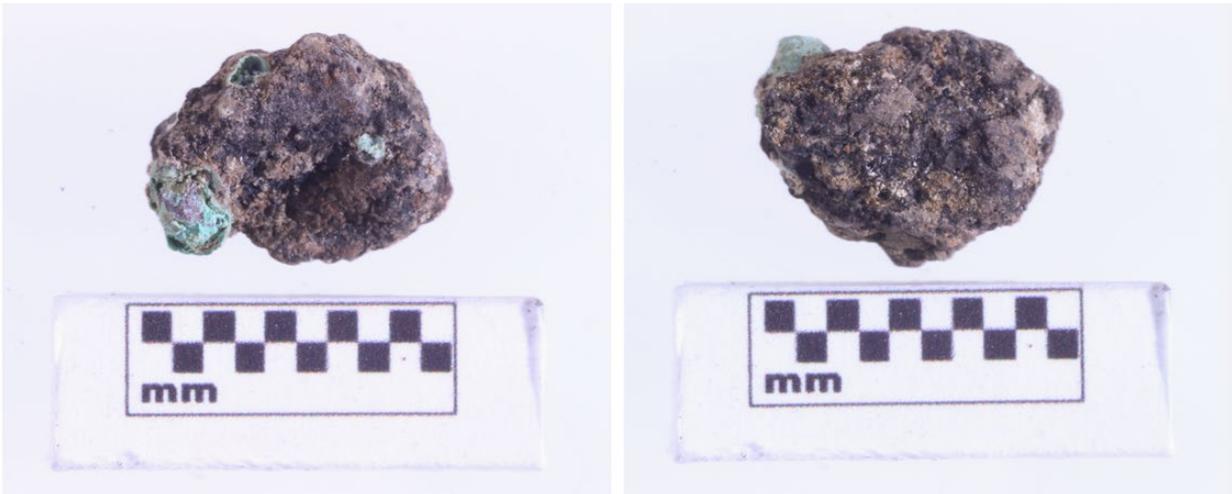


Figure 12. Free slag sample B24/12/2. Note green staining on top of (red) copper metal phase.

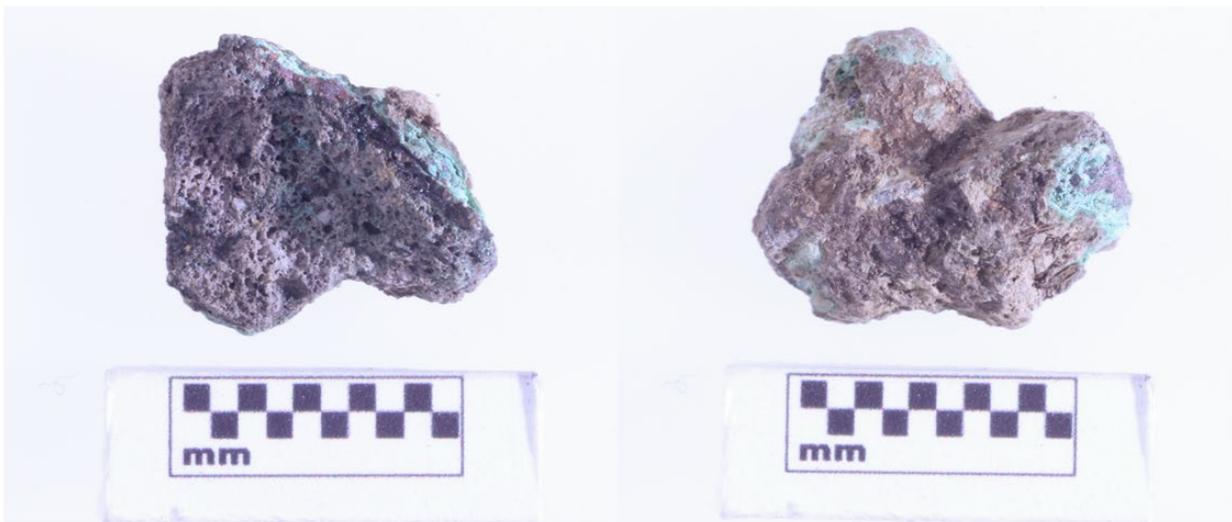


Figure 13. Free slag sample B47/12/2. Note green staining and porous outer surface.

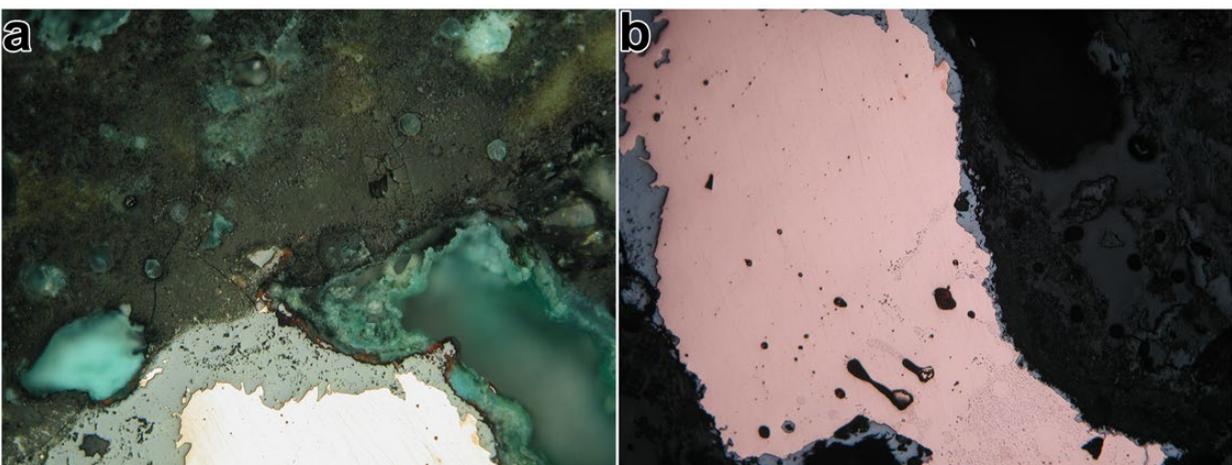


Figure 14. a) Photomicrographs of slag B24/12/2 taken under cross polarised light, magnification 100x, width 1.6mm. Note bright yellow copper metal phase (bottom) surrounded by copper oxide (grey), corrosion products (green), and glassy slag matrix (dark grey) with small grey particles dispersed in it (spinel); b) Photomicrographs of bright metal phase in slag B24/12/2 taken under plain polarised light, magnification 100x, width 1.6mm. Note the well-developed copper-copper oxide eutectic.

Table 9. SEM-EDS compositional data of slag matrices in B24/12 and B47/12/2, both as spot analysis (relatively free of inclusions) and as area analysis (bulk slag analysis in areas of c. 100 x 100 microns). All values are averages of two to twenty analyses of each sample / phase and corrected with factors based on CRM analysis; the uncorrected data is reported in the Appendix.

	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	CaO	TiO ₂	MnO	FeO	CoO	CuO
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
B24/12/2 spot	0.0	0.8	5.2	38.6	7.8	0.0	0.0	5.6	0.0	3.4	11.9	0.0	0.0
stdev s	0.0	0.1	0.7	1.9	0.0	0.0	0.0	1.5	0.0	1.3	2.0	0.0	2.3
B24/12/2 area	0.0	1.3	7.2	33.8	14.3	0.0	0.2	8.1	0.1	3.3	10.7	0.4	0.0
stdev s	0.0	0.4	1.3	5.3	2.4	0.0	0.4	1.8	0.2	2.3	3.9	0.5	3.0
B47/12/2 spot	0.2	2.4	13.7	49.3	0.1	0.4	2.6	9.4	0.7	0.1	4.7	0.0	0.0
stdev s	0.5	0.4	4.0	3.6	0.3	1.1	1.6	11.9	0.5	0.2	1.9	0.0	6.5
B47/12/2 area	1.2	1.4	12.5	59.5	0.5	0.0	2.9	1.9	0.9	0.0	4.7	0.0	0.0
stdev s	1.1	0.1	0.7	3.9	0.6	0.0	0.2	0.7	0.5	0.0	0.8	0.0	5.4

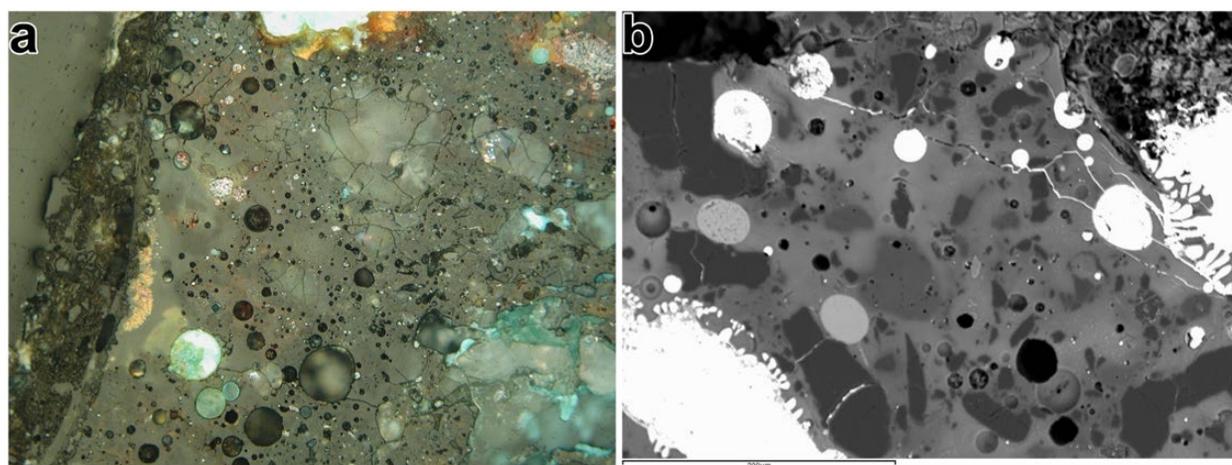


Figure 15. a) Photomicrograph of slag matrix in slagged sherd B47/12/1, taken under cross polarised light, 100x magnification, 1.6 mm width. Note green corrosion developing in place where rounded metal prills of copper formed; b) Backscattered electron image of glassy slag section in B47/12/1. Note bright metal phase and decomposing dark grey quartz grains.

slag specimens are vitrified, strongly magnetic and green-stained droplets, not exceeding 1 cm in length (Figures 12 and 13). They solidified from a highly viscous melt into amorphous grey samples with light green stains, gas holes, and corroded metal in places. Their combined weight is less than 1 gram (B24/12/2 is 0.46 g and B47/12/2 0.36 g), which fits the current knowledge of the appearance and volume of Belovode free slag pieces as published previously (Radivojević and Rehren 2016; Radivojević *et al.* 2010a).

Microstructural and compositional analyses revealed heterogeneous slag matrices and their newly developed phases (Figures 14 and 15). These samples consist primarily of copper 'dross' and copper metal phases/prills and manganese- and iron-rich spinels (only B24/12/2) suspended and unevenly distributed throughout a mostly glassy slag matrix (Figure 14a). The bulk slag glass composition is relatively even in both area and spot analysis mode (Table 9), with one

difference being elevated lime averages in the spot analysis for B47/12/2. This slag is consistent overall with the slag in the slagged sherds, with a ratio of silica to alumina of 4–5:1. The major difference between these two slag pieces is the significantly enriched matrix with fuel ash components (particularly P₂O₅) and ore charge (manganese, copper and iron oxides). Both samples present high readings of fuel ash (magnesia, potash and lime) in comparison to the 'cold' unreacted ceramic bodies of slagged sherds (Table 5) and have copper contamination rising to 27 wt%; the evidence is clearly indicative of these samples being molten pieces of ceramic mixed with ore gangue material fluxed by fuel ash.

Both free slag samples contain areas rich in copper 'dross': copper oxide phases varying from tenorite and cuprite to corrosion products (Figures 13 and 14). Distinguished by its typical bright red / orange internal reflection, cuprite is mostly present as convoluted

Table 10. SEM-EDS compositional data of MnFe spinels slag B24/12/2. All values are averages of four analyses of this phase and corrected with factors based on CRM analysis; the uncorrected data is reported in the Appendix.

	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	CaO	TiO ₂	MnO	FeO	CoO	NiO	CuO	ZnO
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
B24/12/2 MnFe spinel	6.4	6.3	1.4	1.1	0.0	0.0	0.8	0.3	20.6	56.6	2.6	0.0	2.9
stdev s	2.5	2.1	1.3	1.6	0.0	0.7	0.3	2.9	3.8	0.9	0.0	1.6	0.6

Table 11. EPMA compositional data of different metal prills in slag matrices of B24/12/2 and B47/12/2 free slag samples (selected significant trace element values), given in wt%. Values above c. 0.01 wt% (100 ppm) are considered reliable based on CRM measurements; values below this are indicative only. All data are corrected for values obtained from the reference material, using a procedure reported in the methodology section. Values sought but not found at levels above c. 0.01 wt% were indicated as not detected (n.d.).

	S	Mn	Fe	Co	Ni	As	Ag	Sn	Te	Pb	Bi	Analytical total
	wt%											
B24/12/2	0.013	0.003	0.003	0.006	0.008	0.062	n.d.	n.d.	0.024	n.d.	n.d.	100.31
B24/12/2	0.020	n.d.	0.002	0.005	0.006	n.d.	0.007	n.d.	n.d.	0.014	0.017	100.23
B24/12/2	0.012	n.d.	0.015	0.009	n.d.	n.d.	0.004	0.023	n.d.	0.027	0.027	99.01
B24/12/2	0.011	0.001	0.001	n.d.	0.001	n.d.	0.007	n.d.	n.d.	n.d.	n.d.	98.88
B24/12/2	0.005	0.001	0.002	0.005	0.011	0.012	n.d.	n.d.	n.d.	0.004	n.d.	100.22
B24/12/2	0.005	n.d.	n.d.	0.009	0.009	n.d.	0.007	n.d.	0.005	n.d.	n.d.	98.64
B24/12/2	0.013	0.007	n.d.	0.003	n.d.	0.009	n.d.	n.d.	0.010	0.042	n.d.	99.29
B24/12/2	0.005	0.001	0.008	0.004	0.004	n.d.	n.d.	n.d.	0.008	n.d.	n.d.	99.93
B24/12/2	0.009	0.005	0.005	0.002	0.014	n.d.	0.004	0.014	n.d.	n.d.	0.004	100.76
B24/12/2	0.008	0.009	0.002	n.d.	0.004	n.d.	n.d.	0.024	0.031	n.d.	n.d.	100.63
B47/12/2	0.008	n.d.	0.062	0.009	0.015	n.d.	0.007	n.d.	0.011	n.d.	0.003	100.24
B47/12/2	0.009	n.d.	0.042	0.001	n.d.	n.d.	0.008	0.008	n.d.	0.035	n.d.	99.80
B47/12/2	0.013	0.001	0.018	0.004	0.004	n.d.	0.008	0.006	0.034	n.d.	0.006	99.79
B47/12/2	0.004	0.014	0.001	n.d.	0.007	n.d.	n.d.	n.d.	0.016	n.d.	0.019	100.04
B47/12/2	0.013	0.003	0.022	n.d.	0.008	n.d.	0.005	0.002	n.d.	n.d.	n.d.	100.32
B47/12/2	0.006	n.d.	0.012	0.001	0.007	n.d.	0.003	0.006	n.d.	0.025	n.d.	100.20
B47/12/2	0.018	n.d.	0.012	n.d.	0.018	n.d.	n.d.	n.d.	0.009	n.d.	n.d.	99.17
B47/12/2	n.d.	n.d.	0.004	n.d.	0.028	100.26						

agglomerations or dendritic crystals; these appear yellow and red under cross polarised light (Figure 15a). Manganese- and iron-rich spinels (Table 10) appear only in sample B24/12/2 and are compositionally consistent with the same phase observed in slagged sherd B23/12/2 (Table 7), with relevant readings of copper, cobalt and zinc oxides. Finally, the copper-copper oxide eutectic (Figures 14b and 15b) in both free slag samples confirms a high temperature process in excess of 1070 °C. Electron microprobe examination of these pure copper metal phases in B24/12/2 and B47/12/2 (Table 11) reveals a fairly consistent intake of sulfur, nickel, manganese, cobalt, tin and iron. The presence of iron in copper metal, as mentioned above, indicates sufficiently reducing conditions to facilitate this process (cf. Craddock and Meeks 1987). These readings are also consistent with the trace element

pattern seen in associated slagged sherds B23/12 and B47/12/1, overall indicating a specific signature of the ore charge, with the consistent selection of manganese-rich copper ores that contain nickel and cobalt, some remnants of primary copper minerals (iron, sulfur) and potentially associated with polymetallic deposits that contain arsenic, tin, lead and bismuth.

The closest geological formations that fit this description are located in eastern Serbia, in the wider area of the Bor mining region, with dominant types of copper ore including porphyry copper, and massive sulfide deposits with significant Pb, Zn, and Au mineralisation (Janković 1967; Jelenković 1999; Monthel *et al.* 2002; Neubauer and Heinrich 2003). The porphyry deposits at Bor also carry enargite (copper-arsenic-sulfide), a common mineral for this locality (Sillitoe 1983).

Making and working: Copper mineral and metal artefacts

The total assemblage of artefacts from Belovode includes 12 mineral ornaments, two metal droplets and, for the first time on this site, a fragment of a finished artefact. They are consistent with copper-based metallurgy and, very importantly, metal smelting, melting and working activities, thus completing the evidence for the full metallurgical *chaîne opératoire* at Belovode (*sensu* Ottaway 2001).

Copper mineral artefacts (ornaments)

A total of 12 mineral ornaments were found, of which all but one (C-B10/13, green stone ring bead) are made of malachite of various levels of purity (Figure 16). Typologically, the malachite beads can be roughly divided into three distinctive categories: circular or cylindrical (B52/12, B171/12), flat disc (Bf77/12, Bf30/13, C_B5/13, Bf111/12, Bf111/13, B361/13) and ring beads (B42/13, B95/13, C_B7/13) (cf. Wright *et al.* 2008; Wright and Garrard 2003). As such they are

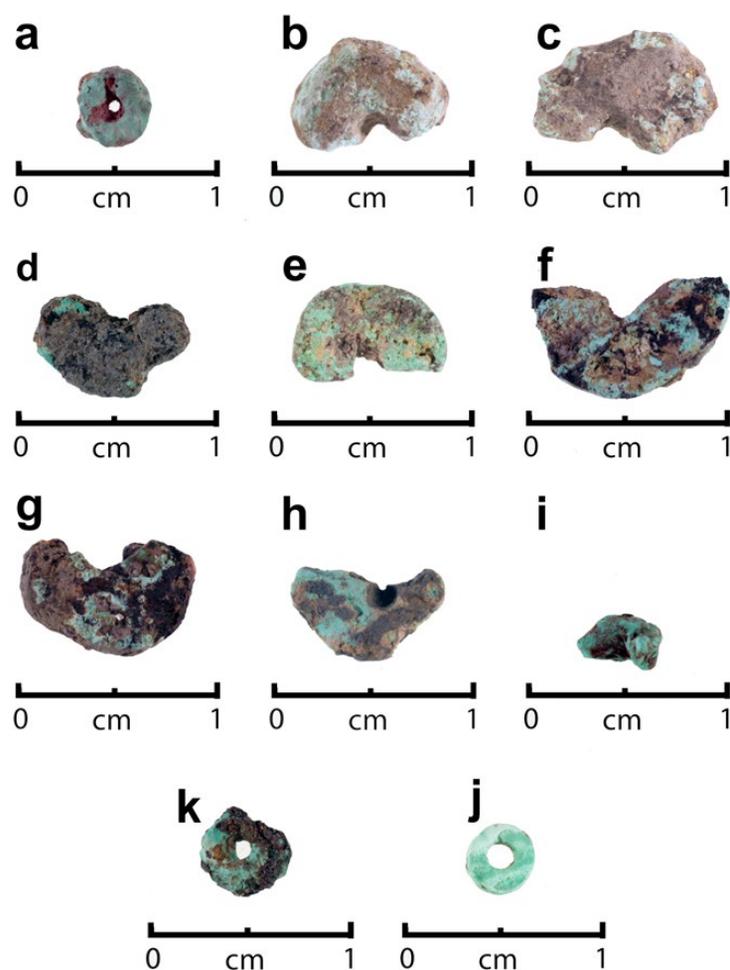


Figure 16. Malachite beads. Circular a) B52/12; b) B171/12; Flat disc c) Bf77/12; d) Bf30/13; e) C_B5/13; f) Bf111/12; g) Bf111/13; h) B361/13; Ring beads i) B42/13; j) B95/13; k) C_B7/13.

consistent with previously discovered and studied beads from the site of Belovode (Radivojević 2007, 2012). Visually, all beads exhibit a thick, light green corrosion layer, with occasionally macroscopically visible oolitic formations on the surface, as in Bf111/12, Bf111/13 and B95/13 (Figures 16f, 16g, and 16j). Only a few complete beads survived, such as C_B7/13 (Figure 16k). Although we have not conducted compositional analyses of these artefacts, macroscopic observation suggests that at least three out of 11 (Bf111/12, Bf111/13 and Bf30/13) might have relevant manganese oxide readings (dark phases), which is not uncommon, given the abundant use of black and green copper minerals and ores in Belovode.

Copper metal droplets

The droplet category of metallurgical debris can include semi-molten and fully molten pieces of ore/metal, and technically covers a wider range of activities that could have produced them. Droplets can be the result of anything from an attempt to smelt metal to accidentally lost debris from smelting, melting or casting. We treat them here as a single category as they are unified by one aspect: they have not been worked and hence occur in nebulous forms with many noticeable porosity holes; in the field they occur covered in green corrosion products, just as any other copper metal artefact (Figure 17). Five copper droplets from excavation campaigns of Trench18/T18ext were found in two distinctive clusters: one directly associated with the assumed metallurgical pit, Feature 6, and the other with another distinctive and well defined feature, Feature 21, which is interpreted as a sealed refuse pit (see Chapter 10). The former includes Bf21/12, B29/12, B47/12/3 (Figure 17), of which Bf21/12 and B47/12/3 were recovered from the same (EDM) spot (see Tables 1 and 2), while the latter yielded Bf43/13 and Bf56/13, together with pieces of malachite and a piece of obsidian (Chapters 10 and 19).

In the first group of droplets (Bf21/12, B29/12 and B47/12/3), B29/12 is morphologically and structurally different (Figures 17c-d) from the other two, which both have a thoroughly developed green patina on their surface (Figures 17a-b and 17e-f). B29/12 contains predominantly dross and some remnants of what seems to have once been a fully developed metallic phase (Figure 18, Table 12). Optically, this dross is also confirmed with bright

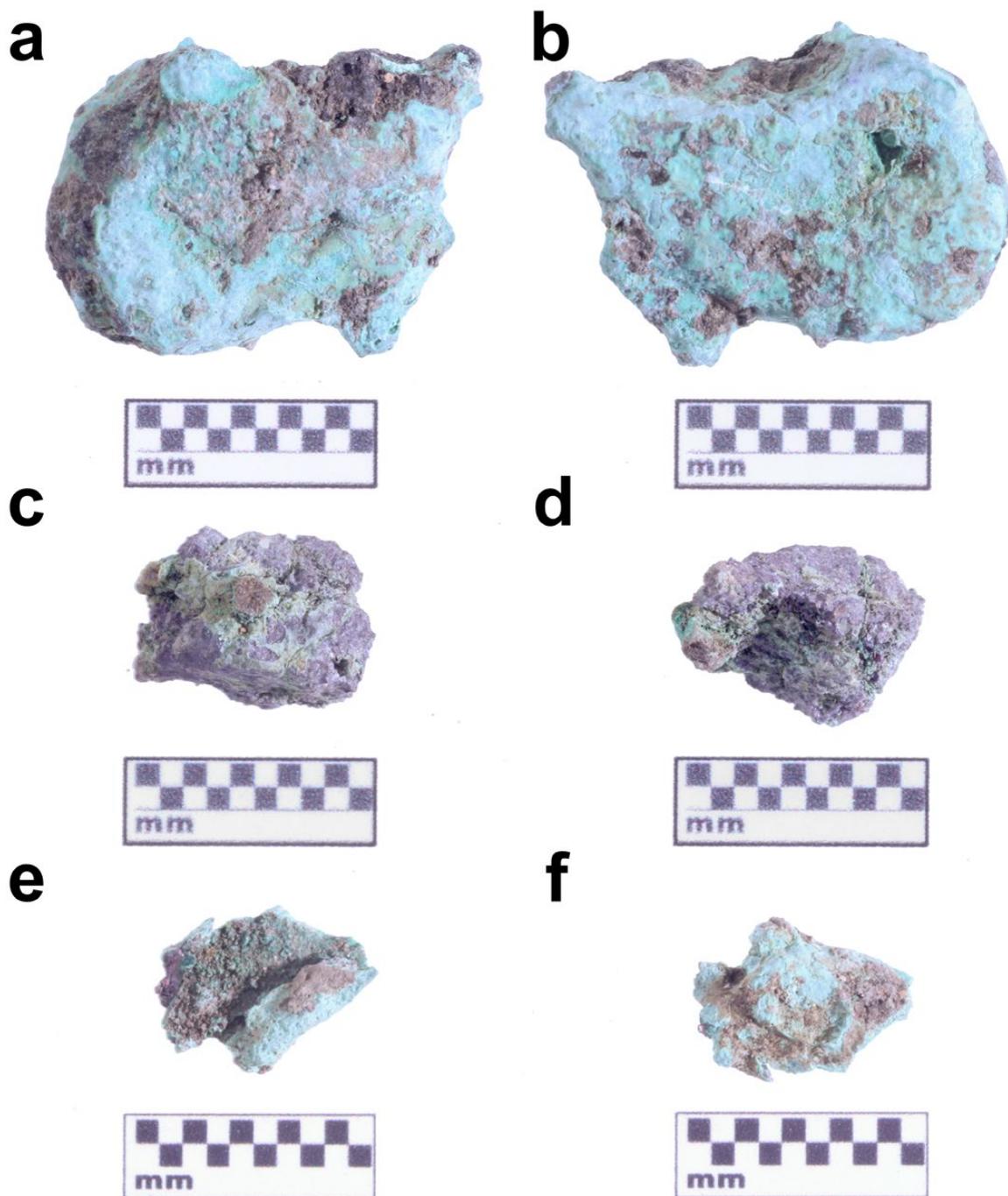


Figure 17. Copper metal droplets associated with Feature 6. a-b) Bf21/12; c-d) B29/12; e-f) B47/12/3. All are classified as droplets following the criteria outlined above.

red internal reflections under cross polarised light, characteristic of cuprite (Cu_2O) (Figure 18b). B29/12 could thus be interpreted as resulting from either a melting event (as indicated by the lack of iron reading as seen in Table 12), or a smelting event of copper ore of high purity, which was followed by full oxidation leading to the dross formation. Experiments with copper smelting and refining have yielded evidence of this form of droplet, attached to the crucible wall

due to insufficiently high temperatures (i.e. only just above the melting point), or the presence of impurities (S. Timberlake, personal communication). Artefacts of this shape are also known from previous analyses of metallurgical debris from Belovode (Radivojević 2013; Radivojević and Rehren 2016) (see Chapter 5).

Bf21/12 and B47/12/3 both present fully molten copper metal bodies, with no traces of working. Their

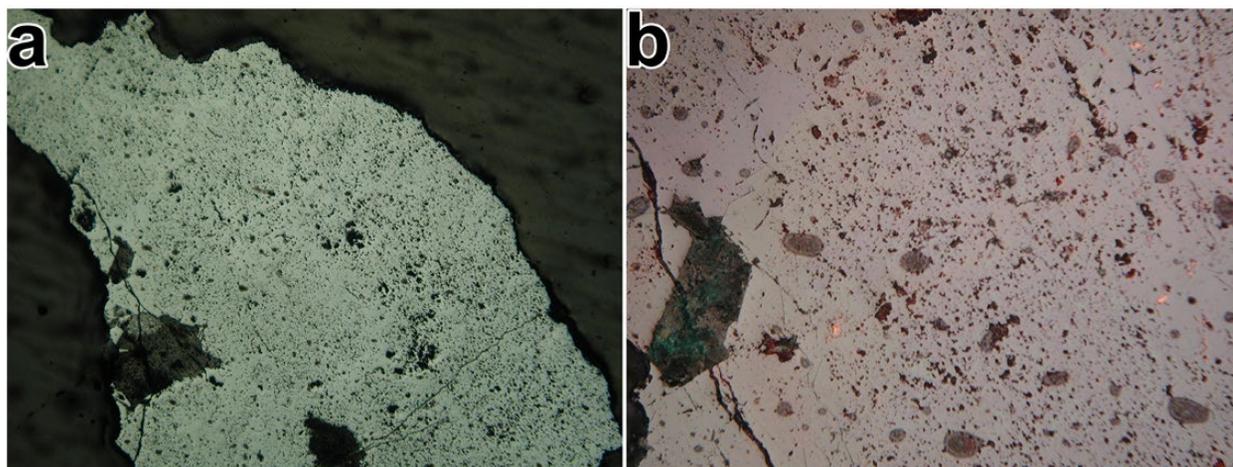


Figure 18. a) Photomicrograph of fully oxidised copper metal droplet B29/12 taken under plain polarised light, 25x magnification, 6.4 mm width. Note grey phase of predominantly copper oxide ('dross'); b) Photomicrograph of copper metal droplet B29/12 taken under cross polarised light, 100x magnification, 1.6 mm width. Note small bright specks of copper metal (pale pink) in the dross.

Table 12. SEM-EDS compositional data of copper rich phases in Belovode B29/12, B47/12/3 and Bf43/13. All values are averages in at% of two to thirteen analyses of each sample / phase.

	O	S	Fe	Cu
	at%	at%	at%	at%
B29/12 metal phase	2.5	0.0	0.0	97.5
B29/12 dross	32.3	0.0	0.0	67.7
B47/12/3 metal phase	3.9	0.0	0.0	96.1
B47/12/3 dross	31.8	0.0	0.0	68.2
Bf43/13 metal phase	2.2	0.0	0.0	97.8
Bf43/13 dross	30.6	0.0	0.0	69.4

micrographs (Figure 19) both exhibit a pale yellow/orange copper (metal) body, with green corrosion products developing on its edges. The optically bright phase shows a residual as-cast structure, preserved in the microstructure of the copper-copper oxide eutectic with well-developed α grains of copper. These alpha grains are, in both cases, characterised by their highly reflective bright colour and dendritic shape in the as-cast eutectic ($\text{Cu}+\text{Cu}_2\text{O}$) structure with grey oxide particles within a bright matrix (example in Figure 19a). In Figures 19a-d we do not see any deformation, but rather the opposite; the α grains are present, unaffected by any mechanical action and preserving the fully developed dendrites. The porosity and gas holes in Figures 19a-d are consistent with the oxygen-rich nature of the metal, which could have been a result of discarding these pieces of molten metal in ambient air while still hot. Bf21/12 and B47/12/3 are therefore candidates for being interpretation as casting debris, and not only because of their association with other metallurgical finds. This assumption is, in the case of Bf21/12, further strengthened by the analyses

indicating a very low presence of iron (15 ppm on average; Table 13), which for freshly-smelted metal would have been higher, as seen in the earlier analyses presented above (cf. Craddock and Meeks 1987). Their association with the smelting evidence of B47/12/1 and B47/12/2 is therefore interesting and either indicates the widely varying redox conditions typical of early metallurgical operations, or they represent debris from different metallurgical activities that were taking place on the same spot (smelting, melting and casting), in the pit, Feature 6.

The two metal droplets, Bf43/13 and Bf56/13, mentioned above, were associated, within a sealed refuse pit (Feature 21) and are securely dated to c. 49th century BC, which makes them the earliest metal artefacts discovered in the excavated section of Trench 18/T18ext. Similar to the case of copper metal droplet Belovode M14 (see Chapter 5) (Radiojević 2013), these were initially assumed to be copper minerals, due to their blocky shape and a thick, light-green patina covering their surfaces (Figure 20). Both samples are about 1 cm long and exhibit a rich dark-red phase in the cross section (see Bf43/13). The matrices of both samples share general characteristics: predominantly grey copper-rich dross with preserved speckles of bright yellow copper metal and porous corrosion products on the edges (Figure 21). The porosity and cracks throughout the polished sections in both cases suggest an intensive reaction with the atmosphere during cooling, which could indicate, as was likely the case with B29/12, that they were discarded while the metal was still hot. They could also have originated from a smelting event of rather pure copper ores, or refining (melting).

Compositionally, these two samples differ. While Bf43/13 consists only of copper metal and dross (copper oxides)

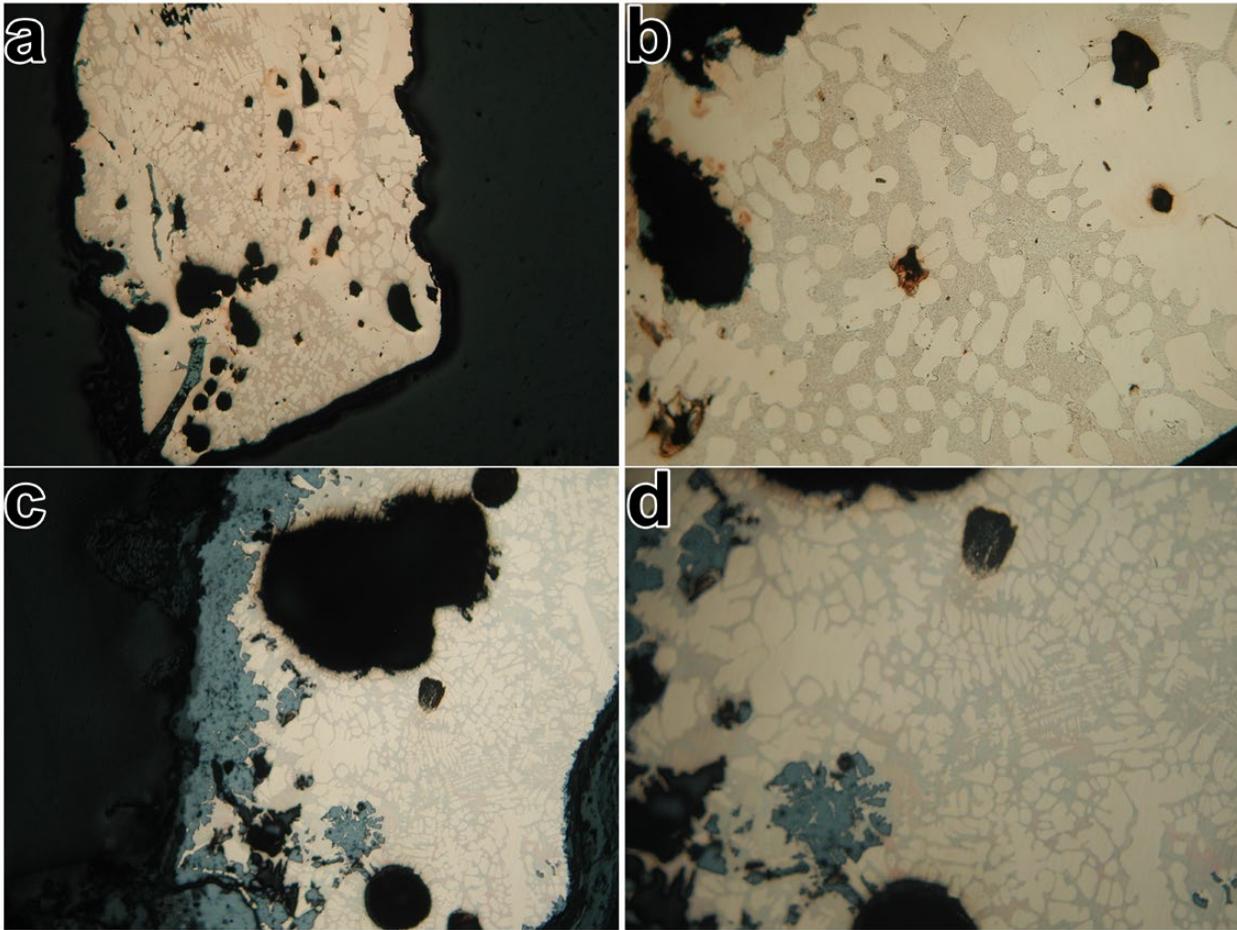


Figure 19. a) Photomicrograph of Bf21/12 taken under plain polarised light, magnification 25x, 6.4 mm width, etched with ammonia hydrogen peroxide. Note bright yellow alpha grains of copper surrounded by copper – copper oxide eutectic; b) Photomicrograph of Bf21/12 taken under plain polarised light, magnification 100x, 1.6 mm width, etched with ammonia hydrogen peroxide; c) Photomicrograph of B47/12/3 taken under plain polarised light, magnification 100x, 1.6 mm width; d) Photomicrograph of B47/12/3 taken under plain polarised light, magnification 200x, 0.85 mm width.

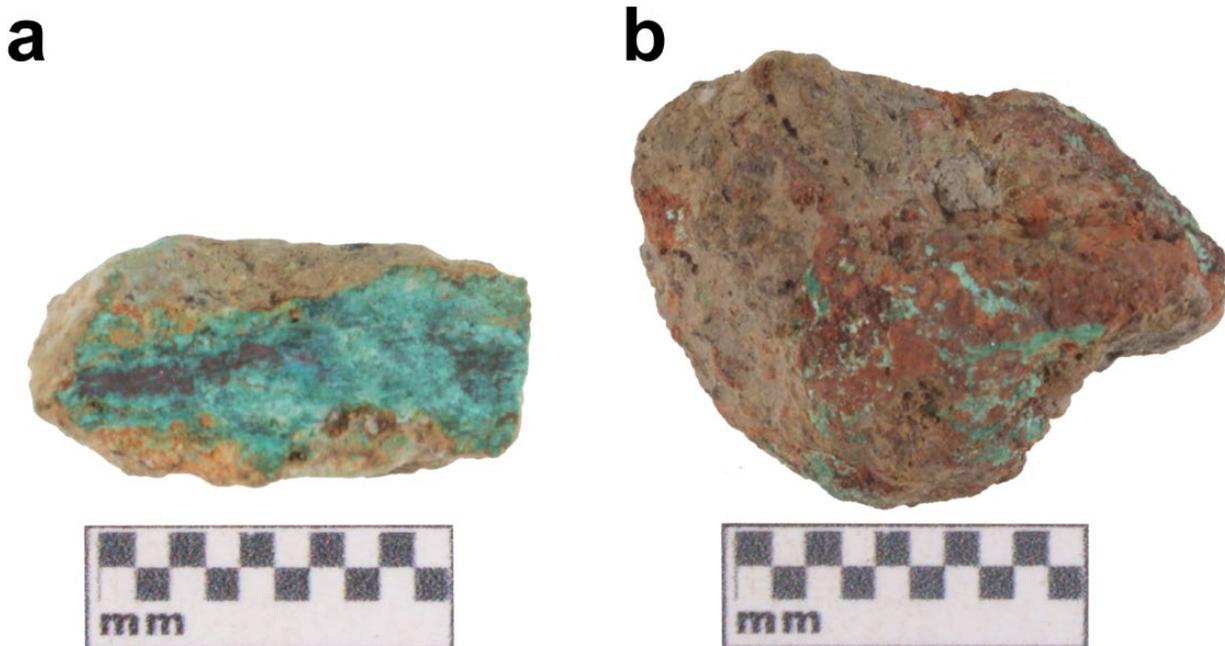


Figure 20. Copper metal droplets associated with Feature 21. a) Bf43/13; b) Bf56/13.

Table 13. EPMA compositional data of copper metal phase in Bf21/12 (selected significant trace element values), given in wt%. Values above c. 0.01 wt% (100 ppm) are considered reliable based on CRM measurements; values below this are indicative only. All data are corrected for values obtained from the reference material, using a procedure reported in the methodology section. Values sought but not found at levels above c. 0.01 wt% were indicated as not detected (n.d.).

	S	Mn	Fe	Co	Ni	As	Ag	Sn	Te	Pb	Bi	Analytical total
	wt%											
Bf21/12	0.005	0.006	0.003	0.003	0.012	n.d.	n.d.	n.d.	n.d.	n.d.	0.030	99.75
Bf21/12	0.005	n.d.	0.002	n.d.	0.016	n.d.	n.d.	0.001	n.d.	0.014	0.026	99.82
Bf21/12	0.004	0.005	0.002	0.002	0.013	0.129	n.d.	n.d.	0.005	0.021	n.d.	100.25
Bf21/12	0.003	0.001	0.002	0.003	0.025	0.095	n.d.	0.015	0.016	n.d.	n.d.	99.56
Bf21/12	0.008	0.002	n.d.	0.009	0.016	n.d.	n.d.	n.d.	0.015	n.d.	0.001	99.28
Bf21/12	0.009	0.008	0.002	0.001	0.017	n.d.	n.d.	0.053	n.d.	0.005	n.d.	99.08
Bf21/12	n.d.	0.012	n.d.	0.001	0.003	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.	99.38
Bf21/12	0.001	n.d.	0.001	0.002	0.018	n.d.	0.003	0.017	n.d.	n.d.	n.d.	98.77
Bf21/12	n.d.	0.007	n.d.	0.005	0.006	n.d.	n.d.	0.014	0.008	n.d.	n.d.	99.39
Bf21/12	0.004	0.001	0.005	0.006	0.012	0.017	n.d.	n.d.	n.d.	0.022	0.038	99.36
Bf21/12	0.011	n.d.	n.d.	n.d.	0.011	n.d.	0.001	n.d.	n.d.	n.d.	n.d.	98.18

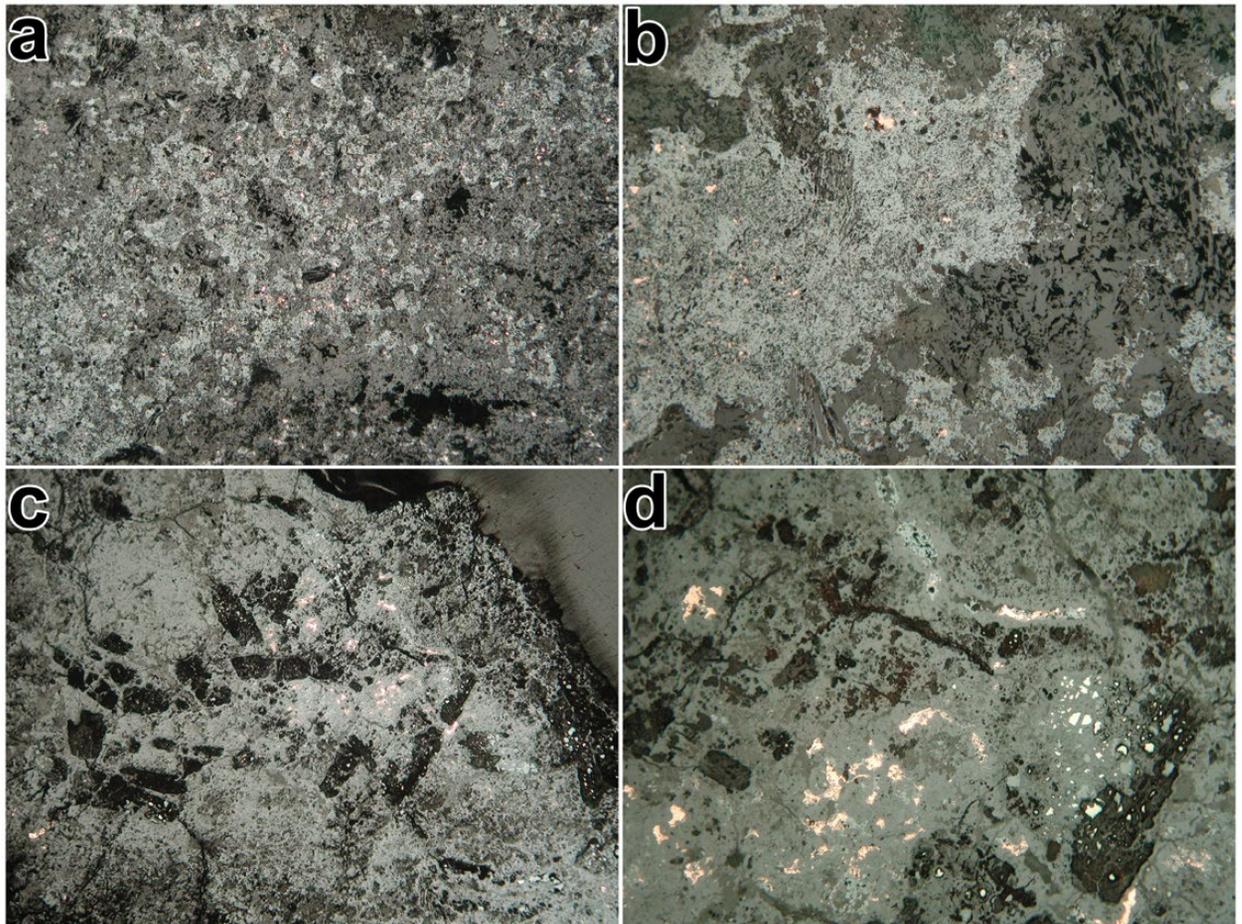


Figure 21. a) Photomicrograph of almost fully oxidised copper metal droplet Bf43/13 taken under plain polarised light, magnification 25x, 6.4 mm width. Note bright yellow speckles of copper metal in grey copper dross; b) Photomicrograph of copper metal droplet Bf43/13 taken under plain polarised light, magnification 200x, 0.85 mm width. Note bright yellow prills of copper metal in grey copper dross; c) Photomicrograph of the almost fully oxidised copper metal droplet Bf56/13 taken under plain polarised light, magnification 25x, 6.4 mm width. Note bright yellow speckles of copper metal in grey copper dross; d) Photomicrograph of copper metal droplet Bf56/13 taken under plain polarised light, magnification 200x, 0.85 mm width. Note bright yellow phase of copper metal in grey copper dross, and a cluster of white pale angular and partially rounded phases of copper sulfide.

Table 14. SEM-EDS compositional data of various copper-rich phases in Bf56/13. All values are averages in at% of one to seven analyses of each sample / phase.

	O	S	Fe	Cu
	at%	at%	at%	at%
Cu metal phase	8.1	0.0	2.7	89.2
Cu ₂ O	33.3	0.0	2.5	64.2
CuO, tenorite	50.0	0.0	1.7	48.3
FeS ₂ , pyrite	0.0	66.6	32.9	0.5
Cu ₂ S, chalcocite	0.0	35.7	3.5	60.8
CuFeS ₂ , chalcopyrite	0.0	52.2	16.5	31.4

(Table 12), Bf56/13 contains various sulfur-rich phases alongside molten copper metal (Figure 21d, Table 14). These diverse phases indicate that the copper ore was a mix of primary and secondary copper minerals (such as chalcopyrite, chalcocite). The dominant presence of chalcocite, for instance in rounded prills, rather than its natural angular form, and its embeddedness in copper dross favours of this interpretation. It is not clear if this sample was intended for any practical use; the presence of sulfur-rich inclusions would have rendered the copper metal brittle and unusable, so this artefact may have been discarded early in the process. It is notable is that similar sulfur-rich examples of early experimentation with smelting copper were identified at Belovode in previous studies (Radivojević 2013). They were then identified as demonstrating 'slagless' copper smelting, and this interpretation also can be applied to Bf43/13 and Bf56/13, as well as B29/12.

Copper metal fragment

Sample B71/12 is the only worked copper metal artefact ever found in the site of Belovode. Although it originates from the same spit (6) as the rest of the metal production debris, it is associated with Feature 3, which is a dwelling (Figure 2). The object itself is around 1 cm long and is identified as a fragment (Figure 22a). Metallographic examination (Figure 22b-d) shows a pale yellow/orange copper metal body, with green corrosion products developing on its edges. This bright metal phase shows as-cast structure preserved in the microstructure of the copper-copper oxide eutectic (bright with grey dots) with α grains of copper (bright). The elongation of the α grains of copper and the eutectic is due to mechanical deformation during working of the artefact. This elongation pattern persists all along the length of the sample (Figure 22c) and partially at right angles across the width, indicating locations where the heaviest force was applied. Based on the criteria given by Rostoker and Dvorak (1990: 16), the reduction of thickness could have been around 20%, suggesting at least several cycles of cold reduction working followed

by annealing. The reduction in thickness indicated by the deformed grains and eutectic seems not to have caused intolerable brittleness of this object (Figure 22d).

The electron microprobe examination of this object (Table 15) shows broadly a similar trace element pattern for both this artefact and Bf21/12 (Table 13), but also includes other metal phases of production debris (Tables 8, 11). Bismuth, arsenic, tin and sulfur feature as predominant trace element (c. 100 ppm on average), followed by lead, nickel and iron, although in slightly lower quantities. This is probably because B71/12, as the finished artefact, must have gone through more cycles of re-melting than the droplets. The relevant readings of sulfur provide an indicator of the primary copper ore mix with the secondary minerals representing the choice of ore charge.

Discussion and conclusion

The metallurgical evidence from the site of Belovode presents all main elements of the *chaîne opératoire* of metal making, from ore selection, via experiments and successful smelts, to melting, re-melting and working an artefact. The highlight from the assemblage is the association of slagged sherds, slags and metal droplets within Feature 6, which may come close to reflecting how the Vinča culture smelting installations might have looked (Figures 2 and 3). The slagged sherds were not found lining the feature but the analysis indicates that they were exposed to high temperatures (reaching c. 1100 °C), and Feature 6 has the closest indication of such temperatures in association with metallurgical debris. Given the ephemeral nature of these early hole-in-the-ground installations, even in experimental settings (see Chapter 4), a burnt pile of soil and charcoal is the best evidence likely to be found from the onset of metallurgical practice at the site of Belovode, or elsewhere.

The sealed refuse pit, Feature 21, with two metal droplets (Bf43/13 and Bf56/13) and dated to the 49th century BC, is another important element for understanding metallurgical activities at Belovode, as it is closest to the earliest dated evidence for copper smelting at the site (Radivojević *et al.* 2010a). Radivojević *et al.* (2010a) made the most of the available absolute dating in their article by associating the pit in which the slags were found with the stratigraphically closest data point in an adjacent trench, hence the stated uncertainty. Our new dates (Chapter 37) are directly associated with metallurgical remains, and the fact that Feature 21 can be securely dated within the 49th century BC (see Table 2) is reassuring for the validity of the c. 5000 BC date previously proposed.

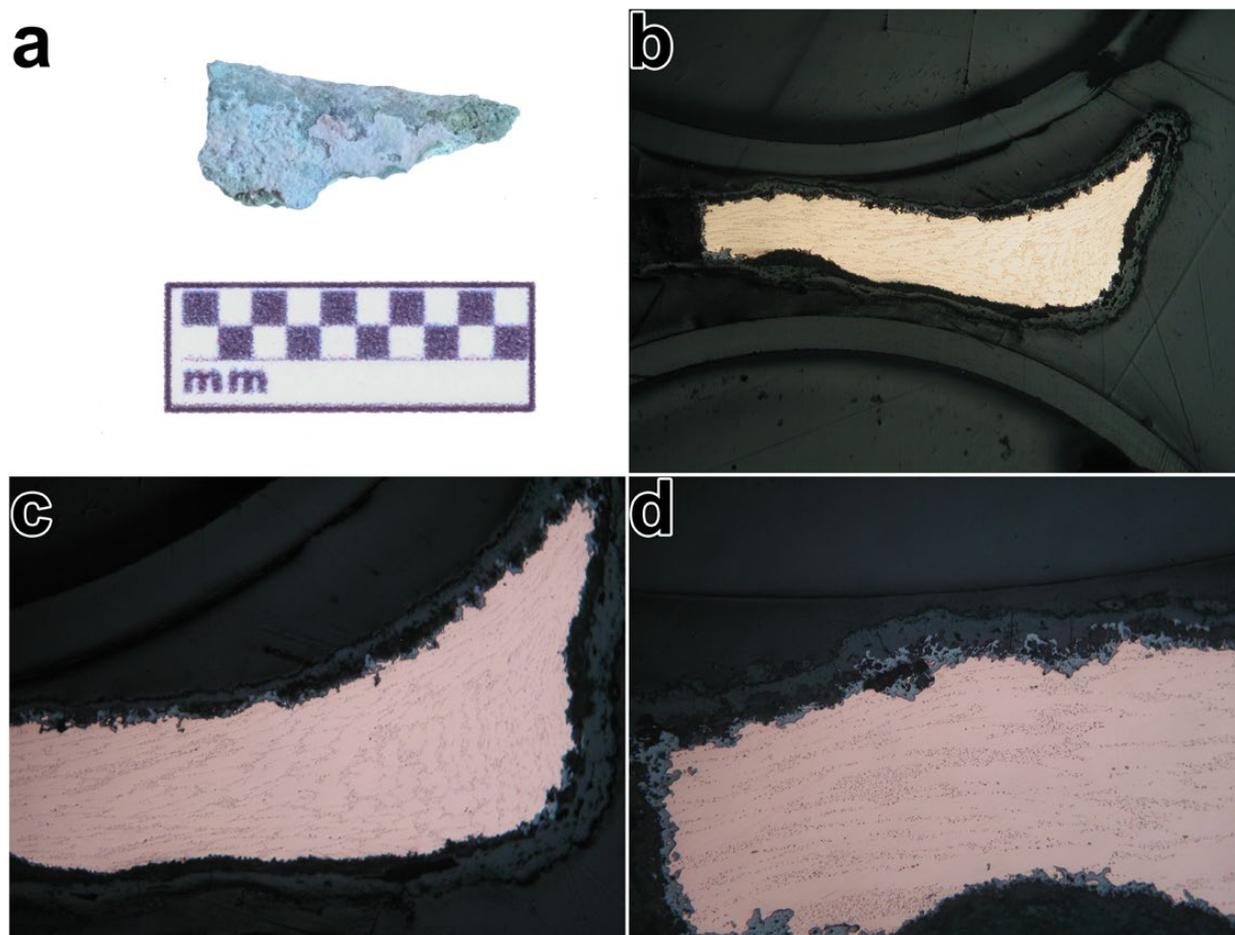


Figure 22. a) Fragment of a copper metal artefact, B71/12; b) Photomicrograph of B71/12 taken under plain polarised light, magnification 50x, 3.2 mm width, etched with ammonia hydrogen peroxide; c) Photomicrograph of B71/12 taken under plain polarised light, magnification 100x, 1.6 mm width, etched with ammonia hydrogen peroxide. Note elongated bright alpha grains of copper suspended in copper-copper oxide eutectic; d) Photomicrograph of B71/12 taken under plain polarised light, magnification 200x, 0.85 mm width, etched with ammonia hydrogen peroxide. Elongation of alpha copper grains and copper-copper oxide eutectic reveals mechanical deformation by working.

Table 15. EPMA compositional data of copper metal artefact B71/12 (selected significant trace element values), given in wt%. Values above c. 0.01 wt% (100 ppm) are considered reliable based on CRM measurements; values below this are indicative only. All data are corrected for values obtained from the reference material, using a procedure reported in the methodology section. Values sought but not found at levels above c. 0.01 wt% were indicated as not detected (n.d.).

	S	Mn	Fe	Co	Ni	As	Sn	Te	Au	Pb	Bi	Analytical total
	wt%											
B71/12	0.005	0.000	0.005	0.000	0.018	0.000	0.000	0.012	0.000	0.000	0.027	100.55
B71/12	0.012	0.000	0.000	0.003	0.001	0.000	0.000	0.019	0.000	0.000	0.010	100.43
B71/12	0.007	0.000	0.000	0.000	0.004	0.000	0.008	0.019	0.011	0.005	0.000	100.49
B71/12	0.008	0.012	0.010	0.003	0.000	0.000	0.000	0.000	0.000	0.018	0.026	100.70
B71/12	0.017	0.000	0.004	0.000	0.025	0.033	0.005	0.000	0.000	0.034	0.030	100.70
B71/12	0.002	0.000	0.000	0.002	0.000	0.075	0.015	0.000	0.000	0.000	0.000	100.58
B71/12	0.005	0.002	0.000	0.000	0.016	0.000	0.044	0.046	0.000	0.000	0.043	100.86
B71/12	0.025	0.004	0.000	0.006	0.005	0.000	0.000	0.000	0.020	0.000	0.000	100.63
B71/12	0.010	0.005	0.007	0.000	0.010	0.000	0.019	0.002	0.000	0.005	0.027	100.76
B71/12	0.000	0.000	0.002	0.002	0.004	0.005	0.000	0.000	0.009	0.000	0.000	98.84

Also reassuring is that the conclusions from previously conducted studies on Belovode metallurgy (Radivojević 2007, 2012, 2013, 2015; Radivojević and Rehren 2016; Radivojević *et al.* 2010a) are fully consistent with the microstructural and chemical examination of the assemblage as presented in Table 1. There are, however, variations regarding the composition of ores, most strongly indicated by the results of EPMA analysis of metals and metal phases in slags in Trench 18/18ext. This is visible in comparisons with data published by Radivojević and Rehren (2016: 223, Table 4) where sulfur appears either only sporadically in slags, or is not detected at all, while in B24/12/2 and B47/12/2 it has a consistent presence of up to 150 ppm (Table 11). A similar situation is shown for B23/12 (Table 8). A novel element not previously detected is tin (Sn), which is present in all EPMA-analysed samples (Tables 8, 11, 13, 15). It is therefore suggested that the batch of ore used in association with the excavated household in Trench 18/18ext was a 'standard' one, with black and green manganese-rich copper ores, which additionally contained primary copper minerals (hence the sulfur content), as well as some concentrations of tin, amongst other trace elements. Significantly, all but one of the samples examined with the electron microprobe come from the same horizon (1b), the exception being associated with Feature 6.

The known preference for black and green manganese-rich copper ores is demonstrated in the ternary plot of CaO/MnO/K₂O, together with previously published Belovode metal production data (Figure 23). There are

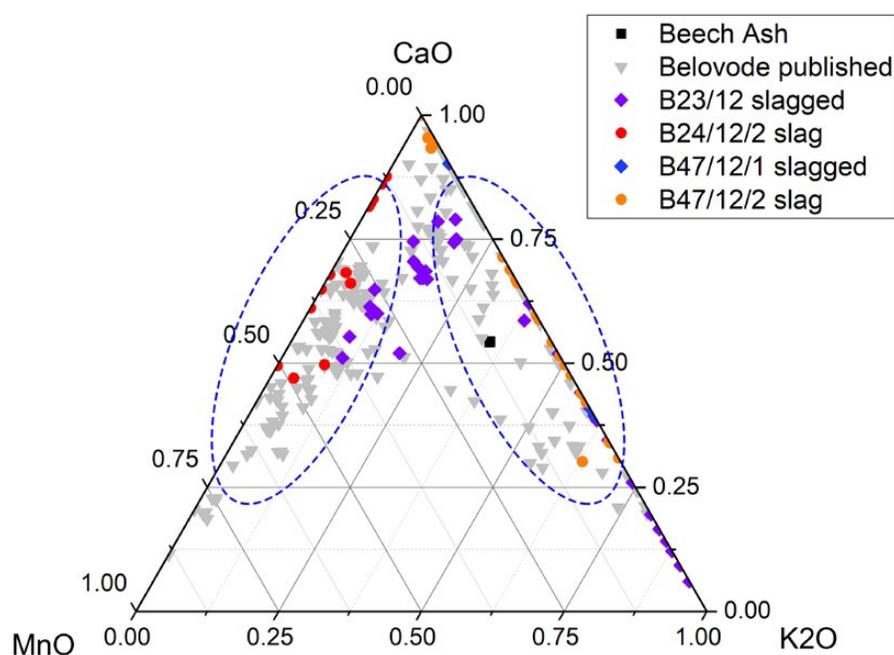


Figure 23. The K₂O-CaO-MnO plot of slag matrices in Belovode 23/12, 24/12/2, 47/12/1, 47/12/2 and all previously conducted comparable Belovode data (Radivojević 2007, 2012). Beech ash data from Jackson and Smedley (2004: 39, Table 4) to simulate fuel ash.

two distinctive clusters: one (Ellipse 1) that associates lime readings with potash, and the other (Ellipse 2), characterised by the manganese-rich chemical signature in slag which comes from the black and green ores mentioned above. Ellipse 1 reflects the fuel contamination, with beech ash readings used to estimate what the fuel might have contributed to slag formation (data from Jackson and Smedley 2004: 39, Table 4). It is interesting to note that most readings for B47/12/1 and B47/12/2 fall into the fuel ash group, while free slag B24/12/2 shows the greatest relative contribution from manganese. A similar conclusion has been reached in previous studies, pointing to the depletion of ore signature in slagged sherds in comparison to free slag samples, due to a higher fuel ash and ceramic contribution to the slag (Radivojević 2012; Radivojević and Rehren 2016: 224, Fig. 11).

The ternary plot of components understood to represent typical pottery (SiO₂/Al₂O₃/TiO₂), fuel ash (CaO/MgO/P₂O₅/K₂O) and ore (FeO/MnO/ZnO/NiO/CoO/As₂O₃/SnO₂/Sb₂O₃) contamination in the glassy slag matrices (re-cast as Cu-free) in Figure 24 illustrates two points: the difference of slag composition from the ceramic composition, and the technological similarity of early metal making in Belovode, and across the Vinča culture. The data used cover all analyses of Vinča culture slag matrices conducted thus far (Radivojević 2007, 2012), and includes previous analyses of Belovode pottery ('cold' sections of slagged sherds) and here presented 'cold' sections of slagged sherds B23/12 and B47/12/1 (Table 5). Figure 24 shows that the new Belovode

analyses are consistent with previously analysed Belovode samples, as well as with slag matrices from the sites of Vinča-Belo Brdo and Gornja Tuzla. The strong cluster of all glassy matrices in the silica+alumina+titania corner implies that they were predominantly formed by these acidic oxides. This in effect explains why the slags (and specifically the free slag samples) are highly viscous, despite the significant input of basic oxides such as lime, iron and copper oxides (Davenport *et al.* 2002: 63). It is also apparent that some readings in the newly analysed Belovode assemblage have much stronger intake from ceramic elements, something that has been observed already

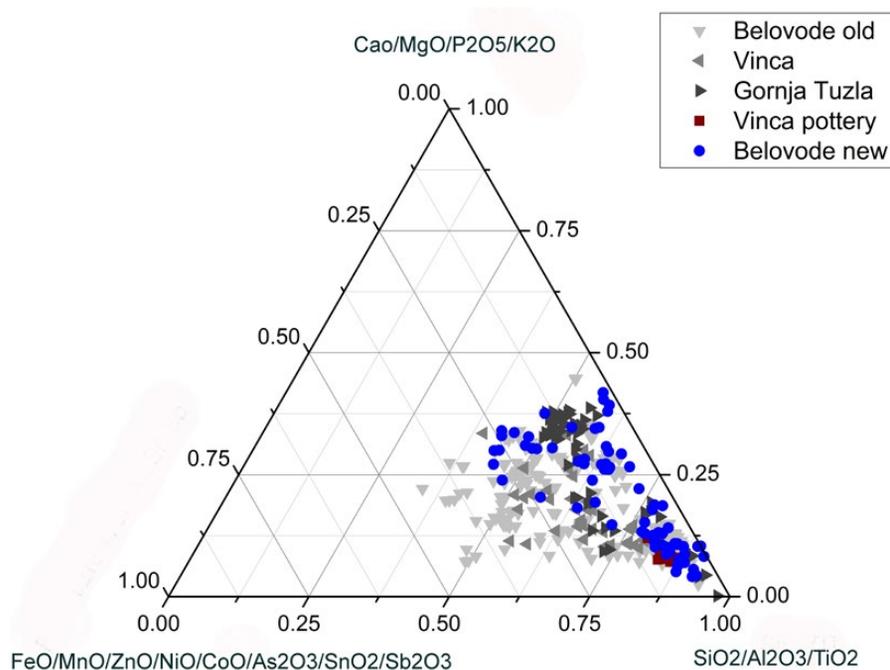


Figure 24. $\text{SiO}_2/\text{Al}_2\text{O}_3/\text{TiO}_2$ - $\text{CaO}/\text{MgO}/\text{P}_2\text{O}_5/\text{K}_2\text{O}$ - $\text{FeO}/\text{MnO}/\text{ZnO}/\text{NiO}/\text{CoO}/\text{As}_2\text{O}_3/\text{SnO}_2/\text{Sb}_2\text{O}_3$ ternary plot for all Belovode and Vinča culture metallurgical slag matrix data and ceramic values for previously studied Belovode pottery and ‘cold’ pottery studied here (data from Radivojević 2007, 2012).

in the analyses of slag on slagged sherds (see Tables 5 and 6). Equally, the stronger fuel ash/ore intake is predominantly related to free slag samples.

One of the most significant aspects revealed by this ternary plot is the similarity of technological parameters used in copper smelting across the studied Vinča culture sites. The slags are highly viscous, though different from ceramic composition, and products of the smelting process of this nature of persist throughout c. 600 years in the Vinča culture. The exceptions to the rule are, however, virtually slagless smelting events preserved in the structure of B29/12, Bf43/13 and Bf56/12. Interestingly, B29/12 and Bf43/13 did not contain iron or manganese (Table 12), which are crucial

for the generation of slag (Bachmann 1982).

The newly analysed assemblage from Belovode fits well into the known picture of early copper smelting practices in this part of the world, using the same type of manganese-rich black and green copper ores and employing the same principles of smelting, with temperatures kept just above the threshold required for metal extraction (1100 °C). The latter is demonstrated by the evidence for molten copper metal giving a minimum temperature of around 1070 °C for the copper-copper oxide eutectic, and the high viscosity, heterogeneity and incompletely liquefied

slag glassy matrices limiting the range towards higher temperatures, which has already emerged as the benchmark of the earliest copper smelting technology.

Appendix

Appendix available online as part of Appendix B at https://doi.org/10.32028/9781803270425/AppendixB_Ch11



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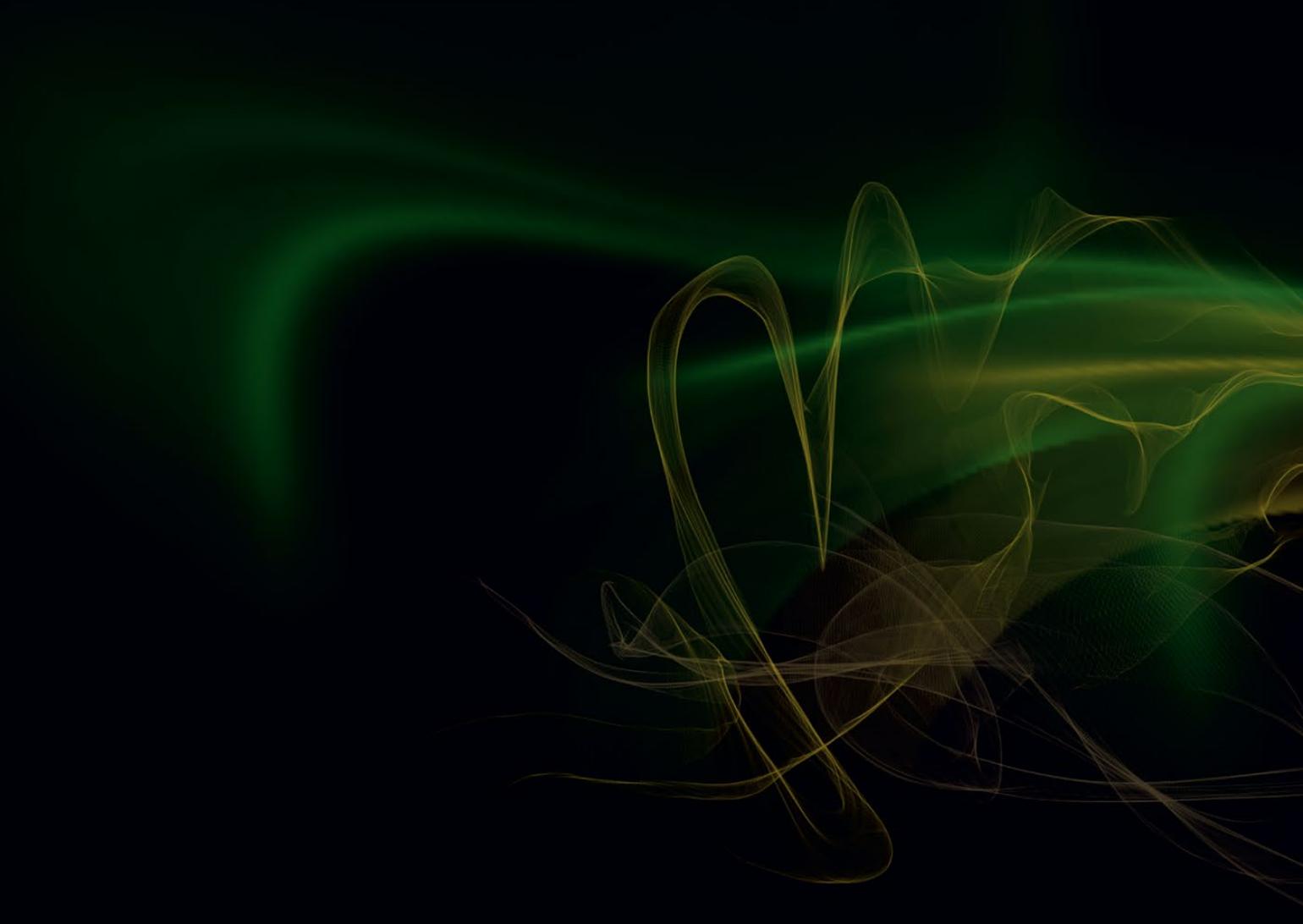
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The Rise of Metallurgy in Eurasia is a landmark study in the origins of metallurgy. The project aimed to trace the invention and innovation of metallurgy in the Balkans. It combined targeted excavations and surveys with extensive scientific analyses at two Neolithic-Chalcolithic copper production and consumption sites, Belovode and Pločnik, in Serbia. At Belovode, the project revealed chronologically and contextually secure evidence for copper smelting in the 49th century BC. This confirms the earlier interpretation of c. 7000-year-old metallurgy at the site, making it the earliest record of fully developed metallurgical activity in the world. However, far from being a rare and elite practice, metallurgy at both Belovode and Pločnik is demonstrated to have been a common and communal craft activity.

This monograph reviews the pre-existing scholarship on early metallurgy in the Balkans. It subsequently presents detailed results from the excavations, surveys and scientific analyses conducted at Belovode and Pločnik. These are followed by new and up-to-date regional syntheses by leading specialists on the Neolithic-Chalcolithic material culture, technologies, settlement and subsistence practices in the Central Balkans. Finally, the monograph places the project results in the context of major debates surrounding early metallurgy in Eurasia before proposing a new agenda for global early metallurgy studies.