

TRACING THE PYRO-TECHNOLOGICAL EVIDENCE DURING THE 3rd MILLENNIUM BC IN “SHAHDAD” THROUGH ARCHAOMETALLURGICAL REMAINS

SHAHDAD KR.E. 3. ÉVEZREDI PIROTECHNOLÓGIAI BIZONYÍTÉKAINAK NYOMON KÖVETÉSE AZ ARCHAOMETALLURGIAI MARADVÁNYOK ÁLTAL •

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Abstract

Shahdad is located on the western side of the great “Lut” desert in the south-central Iranian Plateau. Shahdad has been a major focus of archaeological research in the region due to extensive metallurgical activities, which were documented at the site and supposedly have the most abundant remains of copper metallurgy in south-eastern Iran during the Bronze Age (3rd millennium BC). Due to the archaeological studies of the vast peripheral area, the settlement was a permanently occupied city during the 3rd millennium BC. New excavations at Shahdad offer a unique opportunity to reconsider the pyro-technological remains which were probably related to metallurgical practices during this era. This research will focus on the characterization of typical Shahdad pottery styles and remains of metallurgical slags scattered across the area in association with amounts of other metallurgical remains such as copper ores, moulds, crucibles, furnaces and metallic residues. The typical characteristic style of pottery are their dense structure heavy with rough fabrication. The objects have been studied through optical microscopy, ICP-MS, and XRF to determine their chemistry, micro-chemistry, and mineralogy. The evident complexity of pottery production at Shahdad may eventually allow a better understanding of the timing of innovations and/or the adaptation of technological features observed in the overburden of Shahdad that as yet have not been scientifically documented. The scientific examination of slags and pottery sherds presented here recognizes new information regarding the microchemistry and production techniques of pottery and their possible potential application for metallurgical purposes.

Kivonat

Shahdad a nagy „Lut” sivatag nyugati oldalán, a Dél-Közép-iráni-fennsíkon található. Shahdad a régió régészeti kutatásainak egyik fókuszpontja a helyszínen dokumentált, kiterjedt kohászati tevékenységek miatt, amelyek feltételezhetően a bronzkor (Kr. e. 3. évezred) alatti rézkohászat leggazdagabb maradványait szolgáltatják Délkelet-Iránban. A kiterjedt peremterület régészeti vizsgálatai szerint a település a Kr. e. 3. évezredben állandóan lakott város volt. A Shahdadban végzett új ásatások egyedülálló lehetőséget kínálnak a – valószínűleg a korszak kohászati gyakorlatához kapcsolódó – pirotechnológiai maradványok újraértelmezésére. Ez a kutatás a jellegzetes shahdadi kerámiastílusok, a területen szétszórta található kohászati salakok és a kohászati tevékenységhez kapcsolódó maradványok (pl. rézércek, öntőformák, tégelyek, kemencék és fémmaradványok) jellemzésére összpontosít. A kerámiák stílusára jellemző a tömör szerkezet és a durva kidolgozás. A tárgyakat optikai mikroszkópia, ICP-MS és XRF segítségével vizsgáltuk, hogy meghatározzuk kémiai, mikrokémiai és ásványtani jellemzőiket. A shahdadi kerámiakészítés nyilvánvaló összetettségének vizsgálatával jobban megérthetjük az újabb innovációk megjelenését és/vagy az új technológiai jellegzetességek alkalmazását, amit eddig még tudományosan nem dokumentáltak. A tanulmányban bemutatott salakok és kerámiatörödékek vizsgálata új információkkal szolgálhatnak a kerámiák készítése technikájáról, mikrokémiájáról, illetve azok lehetséges kohászati célú felhasználásáról.

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KULCSSZAVAK: ARCHEOMETALLURGIA, RÉZ, SALAK, KERÁMIA, ÁSVÁNYTAN, SHAHDAD, IRÁN

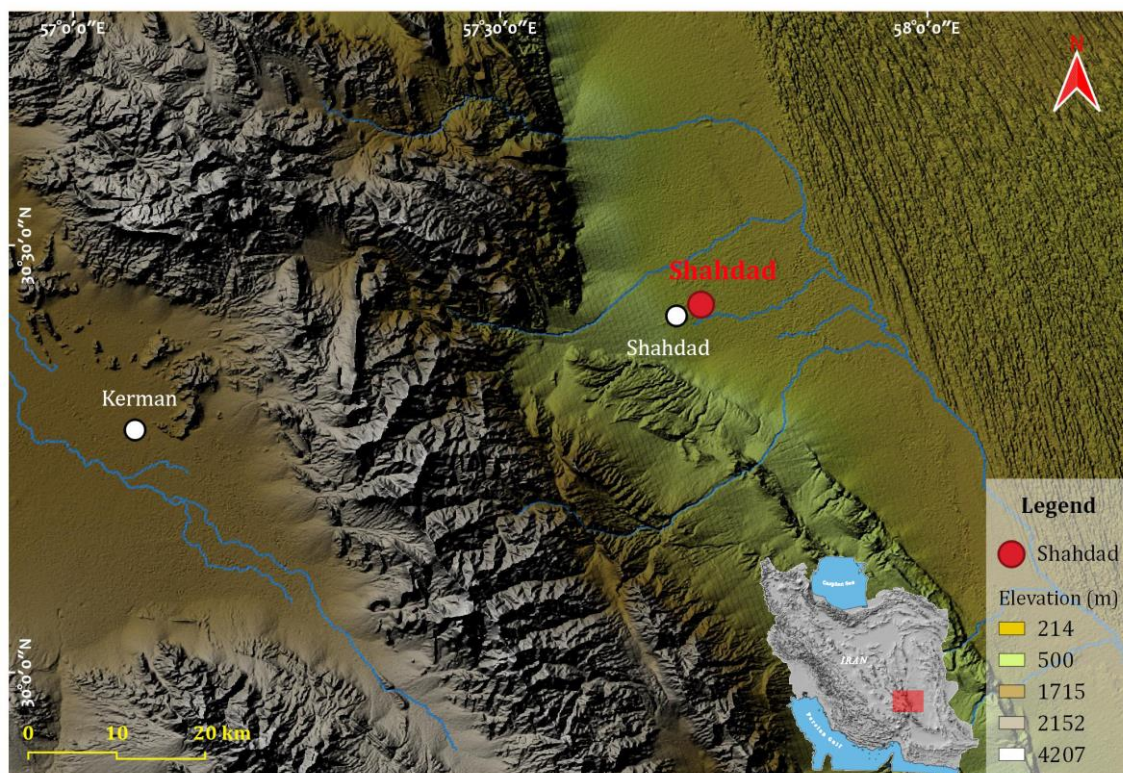


Fig. 1.: Geography and location of Shahdad in the Central Iranian Plateau

1. ábra: Shahdad földrajzi elhelyezkedése a Iráni-fennsík központi területén.

Introduction

One of the aspects of the early metallurgy and metal extraction in the Central Iranian Plateau compared to the neighbouring regions of the Indus Valley in the east to the Mesopotamia in the west is the abundance of metal finds. Although there are many ore deposits and metal-bearing geological formations in the central part of Iran, as is the case in the Kerman region, mainly Shahdad which has been outcropped with diverse metalogenic compositions. The Early Bronze Age civilizations and re-excavated sites in the highlands of Iran had direct access to the ores and mines (Helwing 2021). The importance of the Shahdad in the southern part of the Lut Desert in the manufacturing and trade of metal in central Iranian archaeology was reflected in textual archives of the ancient archaeological peripheries which were well described as Aratta Culture (Majidzadeh 1976) (**Fig. 1**). The ancient texts of Ebla in the second half of the 3rd millennium BC (Steinkeller, 2016) and the archives of Kanesh and Mari in the first half of the 2nd millennium BC, debated the dynamic role of lands east of Mesopotamia (e.g., Elamite cities, in particular Susa) (Weeks 2016) in the trade of metal ores and artefacts in western Asia via various transit

and commercial hubs in West Asia such as Uruk. The Iranian sites offer a rich collection of metal artefacts and a long history of metal utilization in the south-central Iranian plateau (Larsen 1967; Joannes 1991). Results of lead-isotope analyses from Byblos and Tell Arqa at the eastern shore of the Mediterranean, however, have indicated that metal-bearing ores from the Iranian plateau had reached the southern Levant on the western border of Mesopotamia (Morr et al. 2013).

Shahdad as a centre of metal extraction in Iran is located in the highlands of Kerman and the southern part of the Lut Desert, and it provides a long and unique sequence of metallurgical remains and artefacts from the beginning of the fourth to the middle of the 3rd millennium BC (Eskandari 2017, 2021b). The context and later chronology of the material culture of Shahdad, and in particular the substantial finds from early excavations, are still unknown and that is also true for metallurgical remains. 24 years after the publication of Curtis and colleagues (2000), which was established based on many archaeological excavations, research, and publications in Iran and Mesopotamia, today we can reassess the chronology of previously dated objects as well as suggest dates for some of the

undated artefacts in south-central Iran (Curtis et al. 2000). This paper focuses on the study of metallurgical remains and artefacts collected from Shahdad. Pottery and slags have been selected covering the results from the late 3rd millennium BC to the middle of the 2nd millennium BC.

Due to the lack of an organised settlement sequence and also the lack of absolute C14 dating, it is hard to present an absolute chronology of Shahdad. The comparative analysis of the funerary goods from the cemetery of Shahdad reveals that the site dates to the middle 3rd millennium BC and lasts until the late 3rd millennium BC. This dating is based on the comparative studies on pottery, chlorite and marble vessels, bronze objects and seals of the site with the contemporaneous regions of south-eastern Iran and its neighbours such as Shahr-i Sokhta, Jiroft, Bampur, Tepe Yahya, Mundigak, Umm-al Nar, Susa and the sites of central Asia (Eskandari 2019, 2021a; Eskandari et al. 2021). Furthermore, the existence of a linear Elamite inscription in Shahdad (an old Iranian writing system related to the second half of the 3rd millennium BC) confirms this relative dating of the site.

Ever subsequently the emergence of remaining archaeomaterials such as pottery, slag, and crucibles, followed by smelted metal in the late 3rd Millennium BC in Shahdad, high-temperature

industrial processes performed a fundamental role in any subsequent technical and economic development. Despite all these remains, systematic studies and recent insight into past societies are strongly required towards burials, and finalized artefacts, looking to the expertise and creativity that left multiple and often complex features of production behind.

Material and Methods

The ancient periphery of Shahdad is about 9 km² and is covered with metallurgical remains and pottery sherds. Ten pieces of slag and three ores in addition to the slags with five pottery sherds have been considered and studied in this paper. The styles of the pottery in Shahdad are the same and all of them are crashed sherds with coarse-grained additives and reddish color due to the exposed heating to the fabrication. In accordance with their form and typology, these sherds can be clarified as crucibles. Typical samples which belong to the same style are shown in **Figs. 2** and **3**. Concerning the chemical aspects and elemental composition related to eventual copper extractions, slag and ores have been assembled from the region and studied via XRF and ICP-MS. It should be mentioned that all the studied artefacts (pottery and slags) in this paper were certainly manufactured in the Shahdad area during the 3rd millennium BC.

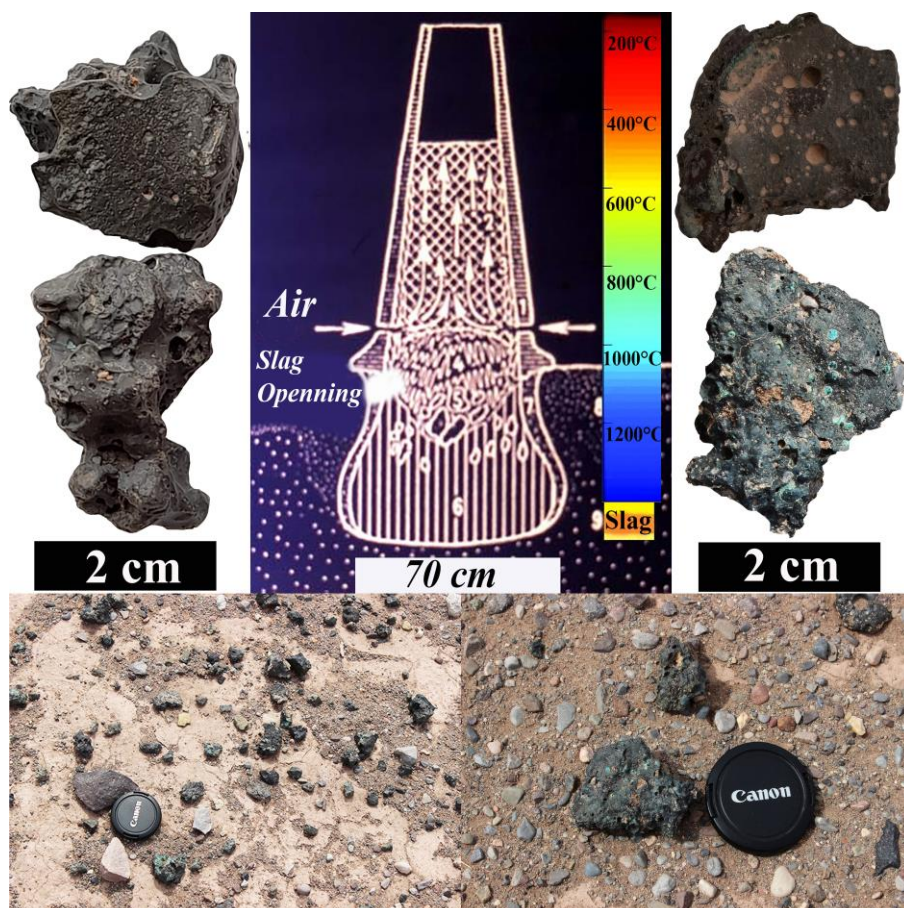


Fig. 2.: Some slag artefacts from the region and an illustration of the place of their production within the kiln.

2. ábra: A területen talált néhány salakmaradvány és a kemencén belüli képződésük helyét mutató modell.

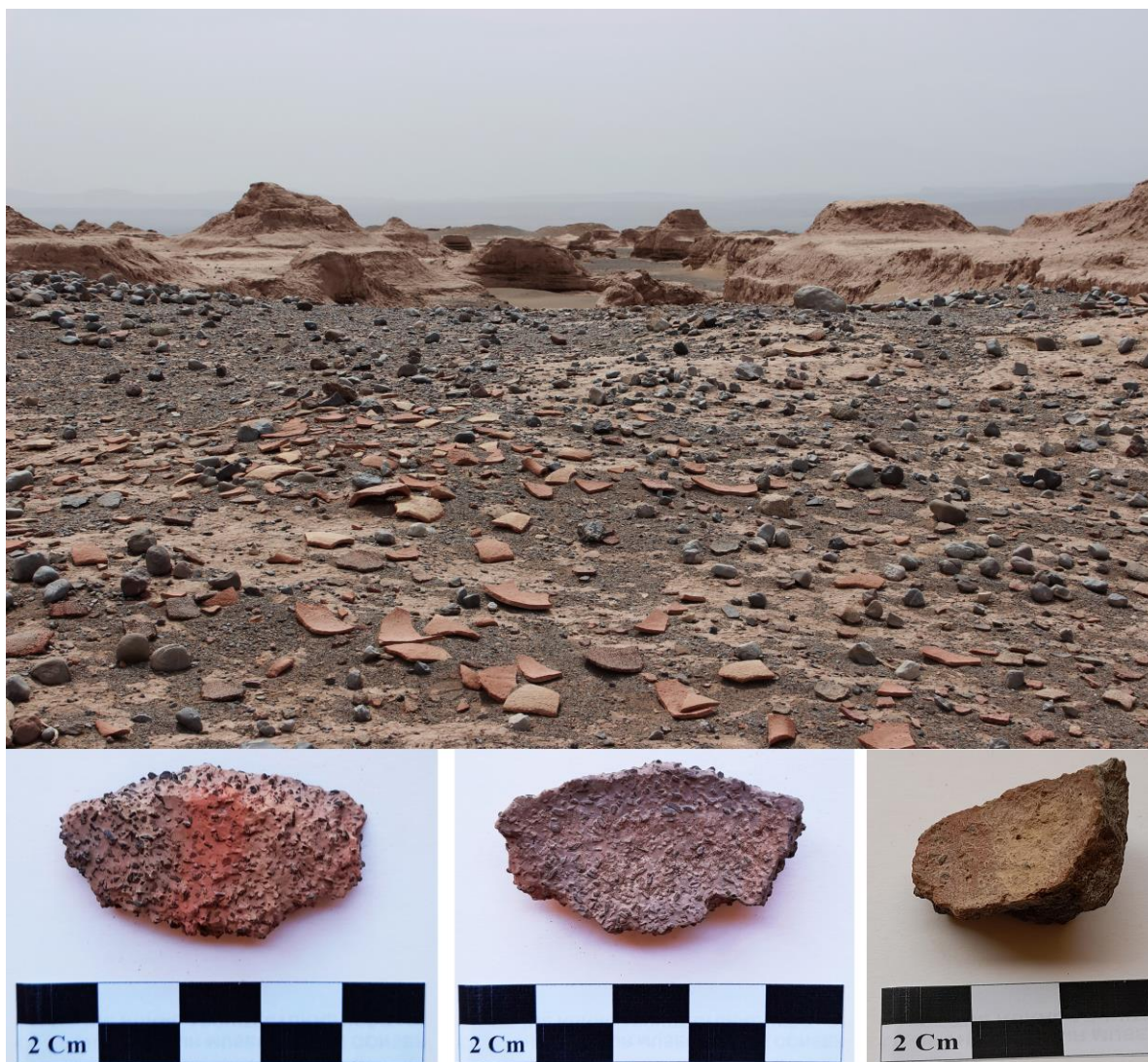


Fig. 3.: Pottery-type collection from Shahdad. All the sherds scattered on the surface belong to the same style
3. ábra: Shahdadi kerámiatípusok. A szétszórta található töredékek mind egy típushoz tartoznak.

The materials have been studied via mineralogical-chemical methods. Microscopic investigations and observations to define textural properties of slag and pottery (petrography and petrology of the finds) have been completed with an Olympus microscope (model X51), at the Department of Chemistry and Structure of Novel Materials at the University Siegen, Germany. The bulk chemical composition of the slags and ores has been studied with an XRF spectrometer (ARL model 8420). UniQuant software has been utilized for qualitative and quantitative measurements. For measurement of the loss on ignition (LOI), the samples were exposed to heat at about 1050 °C for one hour. Trace element concentrations have been done by PerkinElmer's NexION 1000® ICP mass spectrometers at the Zaminrizkavan Co. Ltd., Tehran, Iran. The instrument is an ultimate high-throughput system for routine, multi-elemental, trace-level analyses that meet regulatory standards.

Results and Discussion

Chemical composition of the slags

Artefacts associated with copper-extraction activity have some added elements as the signature of the ore deposit to the charge. The chemical composition of the slags shows that they can be classified into three groups I, II, and III regarding SiO₂ constituents in the system SiO₂-CaO-Fe₂O₃ (Table 1. and Fig. 4). Group I can be described as high silica slag (30.7–53.1 w%) compositionally enriched with iron and calcium that led to the formation of olivine-type texture through green birefringence color of these minerals (fayalite slag) (Fig. 4A). The lower amount of MgO consents to the presence and building of the kirschsteinite (monticelite) type of olivine. Due to the rapid cooling rate olivines are crystallized in skeletal form and are very fine in texture (Hezarkhani &

Keesmann 1996). Group II is classified as pyroxene slags because it forms more iron-rich pyroxene with hedenbergite composition (more Ca-Fe rich) (**Fig. 4B**). Due to the higher portion of the metal to silica (Me:Si), which is responsible for pyroxene building within the matrix, the melting point of these groups is higher than the others. Group III, however, can be mentioned as the ore-bearing

mother rocks with more silica-calcium rich composition, e.g., dolomite-bearing copper impregnations. The olivine, as well as pyroxene-type slags, have possibly smelted from Ca-rich melt (10.2–24.5 w%). MgO amounts in analysed slag vary from 2.0 to 6.5 w%, and these can be reflected in different color within the lath-crystals of olivine in different samples (Addis et al. 2016).

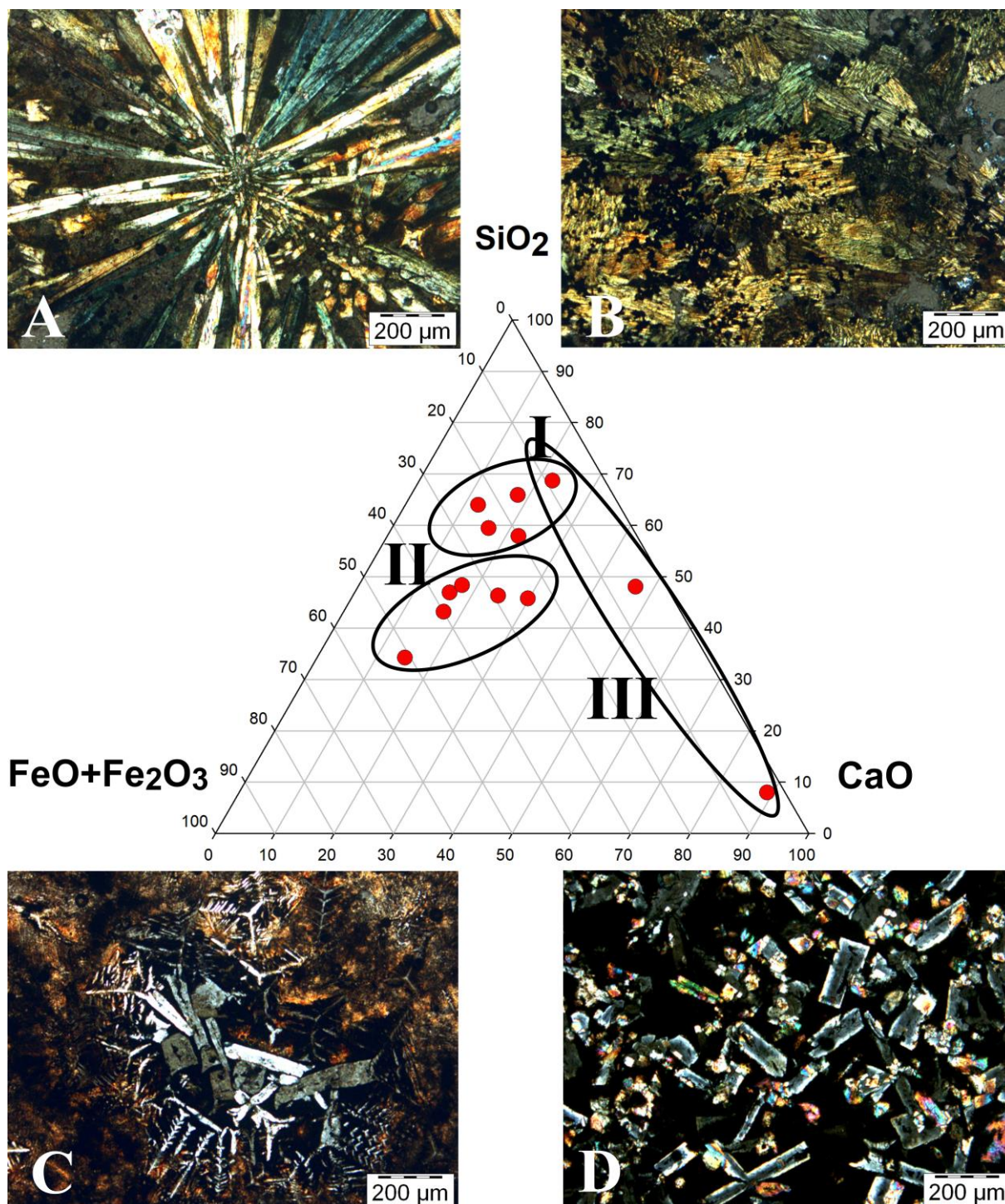


Fig. 4.: The chemical composition of the slags and their relationship to the silicate minerals within their texture
4. ábra: A salakok kémiai összetétele és a szerkezetükben lévő szilikátásványok.

Table 1.: Bulk chemical composition of the slags and ores in w%. LOI is measured after heating the samples to 1050 °C for one hour. The sample order is stated based on the found numbers ascending and the types of them as slag and ore.

1. táblázat: A salakok és az ércek kémiai összetétele tömeg%-ban megadva. Az izzítási veszteséget (LOI) egy órán át tartó, 1050 °C-ra való hevítés után mértük. A minták sorrendjét a leltári számok alapján növekvő sorrendben, a típusokat pedig salakként és ércként adjuk meg.

Sample	058-1	067-0	070-4	063-2	055-1	059-14	050-14	057	061-1	058-0	061-0	059-2	059-13
Na ₂ O	1.5	0.84	0.72	0.56	0.76	1.2	0.58	0.64	0.74	0.97	1.3	0.58	0.26
MgO	4.8	2.9	2.3	2.0	2.0	3.0	2.9	2.6	6.4	2.9	3.9	4.5	2.7
Al ₂ O ₃	9.7	7.8	5.5	6.3	7.3	8.1	6.3	9.2	5.6	9.6	12.9	7.0	2.1
SiO ₂	37.6	40.6	40.1	30.7	53.1	54.1	39.9	43.4	35.3	45.0	47.4	31.5	7.3
P ₂ O ₅	0.45	0.29	0.29	0.16	0.31	0.29	0.38	0.41	0.44	0.29	0.48	0.20	0.14
SO ₃	0.044	0.27	0.58	-	1.1	0.61	0.25	1.4	0.56	1.3	1.6	0.061	0.83
Cl	0.020	0.53	0.64	0.11	0.72	0.51	0.30	1.0	0.55	0.79	0.031	0.012	0.015
K ₂ O	0.29	0.20	0.13	0.15	0.21	0.16	0.14	0.22	0.17	0.19	0.40	0.24	0.052
CaO	24.5	14.6	13.7	13.3	10.2	14.8	21.1	11.9	13.8	17.2	15.5	30.7	81.7
TiO ₂	0.42	0.36	0.27	0.20	0.37	0.40	0.30	0.40	0.25	0.49	0.58	0.46	0.14
Cr ₂ O ₃	0.034	0.095	0.063	0.024	0.024	-	-	-	-	-	-	-	-
MnO	0.045	0.067	0.036	0.063	0.049	0.066	0.098	0.046	0.032	0.13	0.036	0.014	0.23
Fe ₂ O ₃	20.1	28.8	31.7	45.7	19.7	13.2	25.2	17.7	32.7	15.5	6.1	3.4	2.8
CuO	0.21	2.4	3.7	0.44	3.9	3.3	2.3	8.8	3.2	5.4	9.49	21.20	1.50

Table 2.: Trace element analysis of five slag samples by ICP-MS in ppm.

2. táblázat: Öt salakmintá ICP-MS módszerrel mért nyomelem összetétele, ppm-ben megadva.

Sample	57	059-14	061-1	063-2	067-0
Ag	103	16	5	<5	8
Al	41408	39927	29257	30819	39209
As	1018	246	285	160	1091
Ba	979	289	370	497	631
Be	<5	<5	<5	<5	<5
Bi	<5	17	<5	<5	<5
Ca	70818	88011	88060	92130	92275
Cd	13	<5	<5	<5	5
Co	64	28	54	58	119
Cr	130	138	132	394	1405
Cu	129787	56066	51893	9363	37523
Fe	105326	75954	200741	273996	172175
Ga	12	9	20	29	18
K	11250	7877	9632	8691	10935
La	16	15	12	30	15
Li	39	31	27	22	38
Mg	12327	16798	35653	12722	15834

Sample	57	059-14	061-1	063-2	067-0
Mn	1233	1749	976	1693	1818
Mo	31	26	21	15	24
Na	7203	11864	7906	7777	8708
Ni	88	33	211	207	1492
P	1055	802	1070	321	814
Pb	43673	387	738	231	82
S	4524	3053	3830	1348	1814
Sb	3343	35	21	38	35
Sc	9	6	5	6	8
Se	21	12	11	11	9
Sr	1546	1925	1611	1045	1377
Ti	1980	1953	1429	1293	1963
V	72	57	49	64	60
Y	25	16	19	41	29
Zn	1273	46	148	174	170
Zr	114	134	96	100	112

Pyroxene slag from Shahdad has been demonstrated after Ca-rich melt and recrystallization in some reaction zones, e.g., around the furnace wall or within the furnace wall where oxygen fugacity is higher than the core of the melt. Thereafter black dots of iron oxide in different charges can be observed on the pyroxene laths (**Fig. 4B**). Consequently, the Fe-rich slag presents iron constituents in the form of spinel crystals with magnetite compositions (**Fig. 4C**). Melilite is the by-product of copper slag smelting (Emami et al. 2016). The higher Ca- and Si-rich melt within the slag's texture is the best requirement for building melilite. Melilite cannot be observed within the olivine slag (fayalitic slag) because the Ca-Si composition and temperature are not sufficient for crystallizing the mineral. Within the pyroxene-type slag (Group II), however, they are mostly visible with a zone-effect color of bluish-white (**Fig. 4D**). Normally, Mg-rich melilites present a pinkish-orange color within the core of the crystal. Due to the lack of enough Mg within the melt, however, melilite does not represent an orange color in these samples, and it will be classified as Fe-rich akermanite crystals (**Fig. 4D**) (Hezarkhani & Keesmann 1996).

ICP-MS of ten slag and three ores are given in **Table 2**. According to the trace element composition, the studied slag samples seem to be similar to each other. The only contrast can be observed in sample 057 and the presence of lead within it, which is higher than the others. Except for all major elements such as Fe, Cu, Al, Ca, Na, K, and Mg, the other trace elements varied or remained stable within the fabrication of the slags that have been used within this periphery. The higher variation among Cu, Fe, As, Ag, Bi, and Pb can be related to the usage of porphyry-bearing ores (a Fahlerz) from ophiolitic regions, which are well known in southeastern Zagros Orogeny.

Mineralogical composition and texture of the slag

Following microscopic observation of the slag through the polished section, the texture of the slag will be classified by different sulfide, oxide, and metallic constituents. Indeed, the formation of the slag can be determined both through the smelting condition and the location of the formation within or outside the kiln. Slag compositions viewed with reflected light microscopy are described as copper-rich. Metallic copper can be determined as tiny, isolated droplets of different sizes within the glassy matrix (**Fig. 5A**). The color hue of metallic copper (reddish to white with higher reflection) has changed by the presence of the diverse amount of As, Fe, Bi, and S within the metal droplets (Bachmann 1982). The droplets made of metallic copper are formed via rapid cooling within the high-viscosity character of silica melts. Thereafter,

droplets scatter and remain stable as rounded shapes within the cooled melt. In this stage of formation, copper droplets can be formed in association with metallic iron or iron oxide in the composition of magnetite (creamy bright color) and covellite (CuS) with reflected bluish-white color (**Fig. 5B**).

The sulfides can be characterized by higher or lower temperature reactions via smelting. Sulfide composition has formed, and it is seen in various stages within the slag texture as follows.

Crystallization products from sulfide-rich melt

In this case, the typical crystallization character will be observed through the droplets which have been surrounded by sulfide composition (Cu, S and Fe) in blue/grey color (**Fig. 5C**). Nevertheless, another complex crystallization can be allocated via different solubility factors of sulfides (in different sulfide compositions) in the form of iron-rich spinel (dendritic structure) around the sulfide phases within a fayalite rich matrix (prismatic structures in grey) (**Fig. 5D, E**). In smelting processes for primary copper production, the main composition through out of the smelting is the presence of typical fayalite slag with high FeO content, especially near the bottom. The formation of a solid magnetite structure above fayalite is the reason for oxidation reaction under higher oxygen fugacity (fO_2).

Decomposition and conversion of the primary sulfides

The primary sulfides from the ore deposit have a high tendency to form segregation due to their temperature and composition. In this case, the use of Fahlerz as primary sulfide can be modified by the observation of yellow pyrrhotine-chalcocopyrite paragenesis within the slag texture. Ex-solution of sulfides can be seen mainly in form of bornite-digenite lamella within covellite as from the copper-rich sulfide phases (**Fig. 5F**).

Remains of primary sulfides

The relic and remains of primary ore have been detected in all slag in the form of the covellite composition. Covellite used as primary ore for copper extraction had orange inner-reflect, in comparison to chalcocite (Keesmann et al. 1991).

Crystallization products from silica melt

Crystallization from the melt has typically formed a diverse composition between the metallic droplets and sulfide composition within the glassy slag (**Fig. 5C**). These characters that some phases will be recrystallized from the contact area between silica melt and different sulfide-rich phases have been described in many German texts as "Zwickelfüllungen" (Keesman 1993).

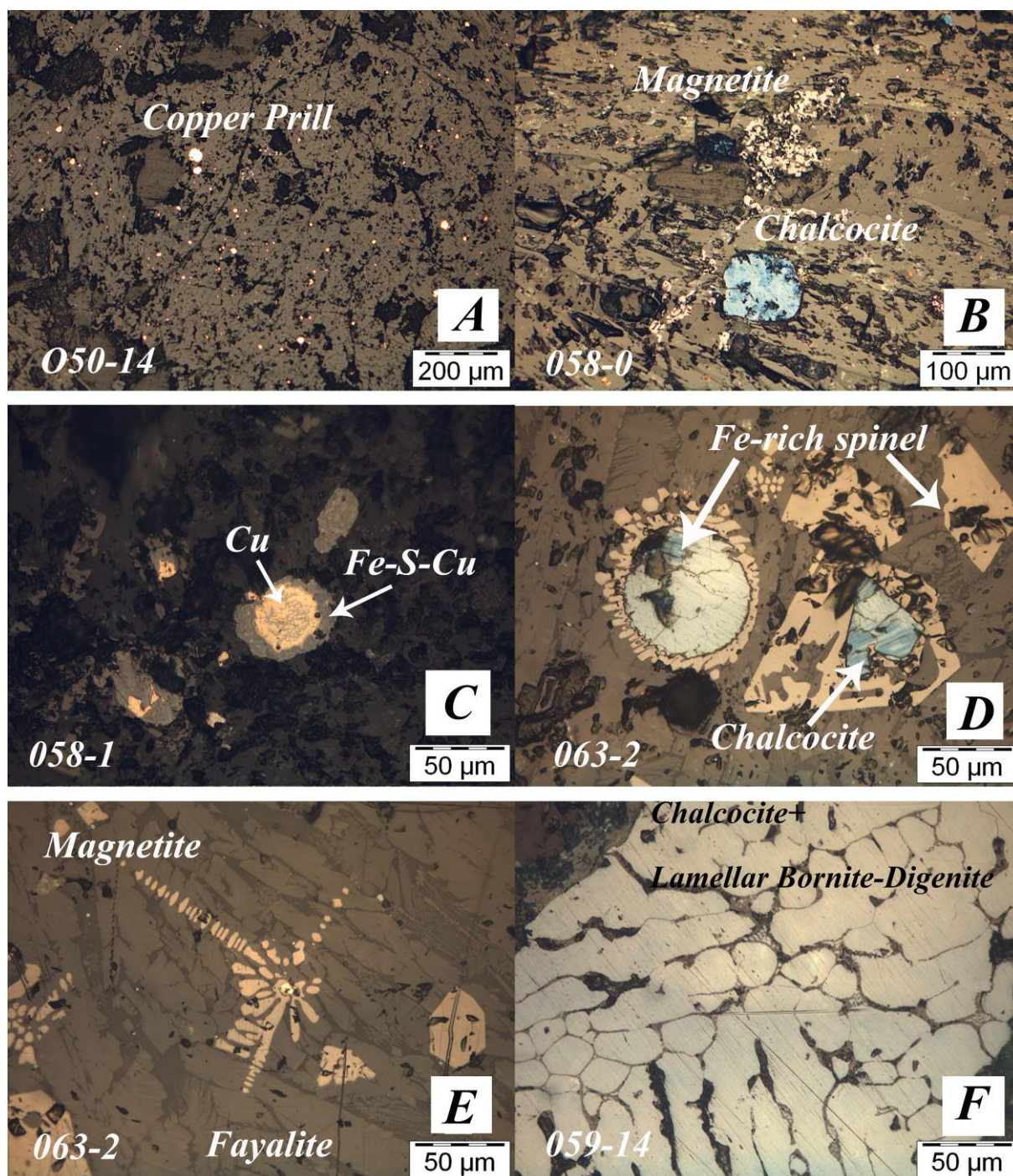


Fig. 5.: Microscopic investigation of the slag and their mineralogical constituents and textures.

5. ábra: A salakminták ásványi komponenseinek és szerkezetének mikroszkópos vizsgálata.

Reaction products (decomposition)

As mentioned above all the slags have to be classified as iron and copper-rich composition with chalcopyrite and chalcocite relics within the silica melts. However, metallic droplets are iron-poor and completely rich in Cu or Cu-S composition; this can be seen as an ex-solution effect on the iron-rich phases (**Fig. 5F**).

Petrography and texture of the pottery from Shahdad

Pottery from Shahdad has been studied petrologically and petrographically because they might have preserved some aspect of metallurgy within their fabric. The pottery sherds from Shahdad have typically been characterized as porous sherds. According to the additives applied within the potter, they are classified as coarse-grained sherds with different color in thickness with

coarse-grained black sand and flint as additives. The sherds were studied through different magnifications for better clarifying their textural and fabrication features (**Fig. 6**). Observation via loop shows the visible coarse-grained additives and cracks (**Fig. 6A**). Cracks appeared normally within the inner part of the fabric and might have been produced via temperature shock through shrinkage. Cracks are not visible in the surface area. Petrographically, the matrix contained crushed quartz of almost the same size concerning similar processing approaches through preparation and separation (**Fig. 6B**). Muscovite minerals appear orangish red because the temperature did not exceed 900°C (Emami 2020). The matrix has two colors because two different clayey raw materials were used (Anaya et al. 2024).

Internal reflection light microscopy in the dark-field shows the green light which may pass through the polished surface of a mineral and be reflected from below and specify the presence of copper-bearing phases within the matrix of the sherds (**Fig. 6C**). Many cracks are caused due to temperature shock via rapid cooling within the fabric. The observation through reflected light mode in the bright field shows exactly how many copper droplets are within the sherds (**Fig. 6D**). The droplets are surrounded with sulphide ores as the relics from silica melt as explained above. The sulphides are in the same composition and crystal stage as have been determined within the slag, and they show similar use in these objects. The pottery sherds thus have been used as technical pottery throughout copper extraction. The microtexture of the Shahdad pottery shows that they have been used for metal extraction and are the pieces from chimneys.

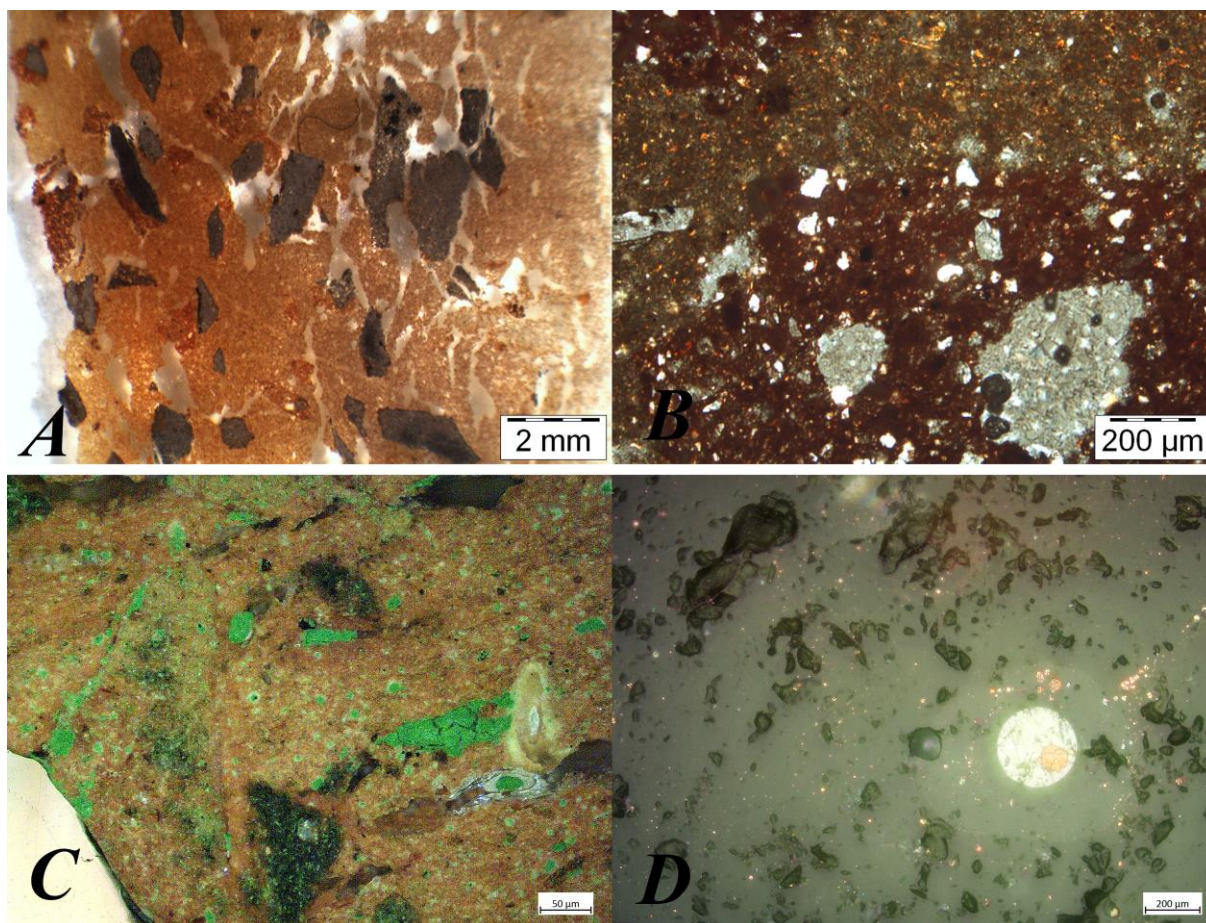


Fig. 6.: Microtexture of the Shahdad pottery types used for metal extraction through different types of observation: (a) viewed with a loop; (B) observation with a polarized microscope under cross-polarized light; (C) observation with a polarized microscope under reflected light and dark field; (D) microscopic view under reflected light mode.

6. ábra: Fém kinyerésére használt shahdadi kerámia (Sh.C_01) mikroszerkezetéről készült különböző felvételek: (A) nagyítóval készült felvétel; (B) polarizációs mikroszkóppal készült kép merőleges polarizátor állás esetén; (C) polarizációs mikroszkóppal készült visszaszórt felvétel sötét látóterű megvilágítás esetén; (D) mikroszkópos felvétel visszaszórt fénnel.

Conclusion

Shahdad is one of the essential localities for the study of the early beginning and use of copper in the Central Iranian Plateau. Shahdad is located close to the Lut Desert in the Central Iranian Plateau. Archaeometallurgical researchers have been interested in Shahdad due to the widespread metallurgical activities (and remains of such activities) during the Early Bronze Age. Field surveys have already confirmed the early use of copper through metallurgical remains, e.g., slags and pottery. Archaeological studies have shown that the periphery of Shahdad to be a permanent city dating back to the 3rd millennium BC. Shahdad was prosperous due to the presence of important evidence of ancient metallurgical remains related to copper extraction. In accordance with the landscape of Shahdad, consisting of many fragments of metal tools, copper ores, moulds, crucibles, and additionally remains of furnaces, scientific research still can be enforced.

Pottery sherds and slags have been observed macroscopically and microscopically to find particular traces of metallurgy from the heyday of this region.

Investigations were supplied with a new synopsis by re-tracing the copper metallurgy. It is revealed that they mainly used copper sulphide (covellite) as Cu-bearing ores. Two different types of slag were identified regarding their color, texture, and fabrication. Pottery sherds were also associated with copper metallurgy based on copper carbonate and copper oxide enrichments often occurring inside their porosity. Microscopic observation of slag and sherds indicate that the used copper ores mostly consist of covellite-chalcocite bearing ores. The smelting strategy is implemented under the control of accessible technological factors such as the level and type of technology at that time in the Early Bronze Age, accessible raw materials (ores), and technological circumstances, e.g., vegetation for producing the heat and clayey soil for earthen based constructions. Moreover, the complexity of metallurgy in Shahdad established a precise knowledge of innovation and/or adaptation of technological features, which were transferred to other areas such as Jiroft and Tal-e Iblis in the south and southeast of Kerman province.

Contribution of authors

Mohammadamin Emami Conceptualization, Resources, Writing – Original Draft, Writing – Review and Editing, Formal Analysis, Investigations, Supervision. **Soraya Elikay Dehno** Methodology, Formal Analysis. **Nasir Eskandari** Data Curation. **Christian Pritzel** Methodology, Formal Analysis, Visualization.

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