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The Galena Objects from Neolithic Ulucak: The Earliest Metallic Finds in Western Turkey

ÖZLEM ÇEVİK – MURAT DİRİCAN – AYDIN ULUBEY – OSMAN VURUŞKAN*

Abstract

The earliest metal finds in central and eastern Anatolia are small copper and malachite beads dating from the 9th millennium BC onwards. However, the presence of metallic finds in Neolithic contexts from western Anatolia are rarely known. An analysis of metallic finds from Ulucak Höyük shows that galena was used at the site from the early 7th millennium BC to the early 6th millennium BC. Objects made of galena from initial phases at the site are considered personal ornaments, while an increasing number of galena lumps in relation to ovens were found in later phases. Thus, galena finds from Ulucak Höyük suggest that at first this raw material seemed to have been perceived as an exotic "stone", while a full understanding of its properties may have been developed later.

Keywords: galena, Ulucak Höyük, Neolithic, personal ornaments

Öz

En erken metal buluntuları temsil eden bakır ve malahit yapımı küçük boncuklar, Orta ve Doğu Anadolu'da MÖ 9. binyıldan itibaren görülmektedir. Bununla birlikte Batı Anadolu'da Neolitik döneme tarihlenen metal buluntu seyrektir. Ulucak Höyük'te ele geçen metal buluntuların analizi, galenin erken MÖ 7. binyıldan MÖ 6. binyılın başına kadar yerleşimde kullanıldığını göstermektedir. En erken evrede bulunan galen yapımı nesneleri kişisel süs eşyaları temsil ederken, geç evrelerde artan sayıda galen topağının fırınlarla ilişkili olarak ele geçtiği kaydedilmiştir. Bu nedenle Ulucak galen buluntuları, olasılıkla başlangıçta bu hammaddenin egzotik bir "taş" olarak algılandığını, kimyasal özelliklerine dair tam bir anlayışın ise daha sonra geliştirildiğini işaret etmektedir.

Anahtar Kelimeler: galen, Ulucak Höyük, Neolitik, kişisel süs eşyaları

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Introduction

Archaeological evidence suggests a series of developmental sequences can be traced between the first appearance of metal objects and the onset of extractive metallurgies. The earliest metal finds found in Anatolia date from the 9th millennium BC and are small copper and malachite beads.[1] The use of native copper then became widespread during the late 7th/early 6th millennium BC, with examples found from Iran to Europe.[2] During the Neolithic, most of the copper objects were shaped by cold-hammering. Some, however, like those from Çayönü and Aşıklı dating to the 8th millennium BC, were clearly made from annealed native copper.[3]

Galena (a lead sulfide ore, PbS) is one natural metal found in Neolithic contexts from the Near East. When compared to the abundance of copper and malachite artefacts, galena objects have only been recorded at two Neolithic settlements (fig. 1). Three galena balls with textile impressions are known from Tell-Halula in northern Syria.[4] These balls, found in burial contexts, are dated to the latest PPNB phases - the end of the 8th millennium BC. In Çatalhöyük thirteen galena beads, first mistakenly identified as lead, were found in Level VIB, dating to around the mid-7th millennium BC.[5] Moreover, a piece of galena found next to a limestone figurine in a special deposit from the upper levels of Çatalhöyük (Level III) indicates long-term use of this raw material at the site.[6]

Unlike the situation observed in eastern and central Anatolia, the occurrence of metallic finds in Neolithic contexts from western Anatolia is rare. Thus, the recent discovery of metallic objects from Ulucak and a malachite bead from Uğurlu[7] on the island of Gökçeada (Imbros) represent the only known finds from western Anatolia in the 7th millennium BC. Until now, the earliest known metallurgical activities in western Anatolia have been traced to the late 4th and the early 3rd millennium BC, with lines of evidence from Limantepe, Baklatepe, Troy, Çukuriçi and Ilıpınar.[8]

In this article, we present an analysis of the metallic finds found at Ulucak Höyük, dating from the early 7th millennium BC through to the early 6th millennium BC. XRF, XRD and SEM-EDX analyses were applied in order to determine the mineralogical and chemical properties of the archaeological finds and to compare them with modern samples taken from a nearby lead mine. The Ulucak metallic objects are also considered symbolic media. As a result, this article will also discuss the dynamics which may have stimulated the initial use of metallic ores.

Metallic Finds: Contextual and Chronological Setting

Ulucak Höyük lies 25 km east of İzmir in west-central Turkey (fig. 2). The mound is located in the western part of the Kemalpaşa plain, which is surrounded by the Nif and Spil mountains in its southern and northern ends respectively. Ulucak is a small mound covering an area of

1 Birch et al. 2013; Lehner and Yener 2014; Yalçın 2016; Erdoğu 2017.

2 Roberts et al. 2009, 1013-14.

3 Maddin et al. 1999; Özdoğan and Özdoğan 1999; Esin 1995, 1999.

4 Molist et al. 2010, 37-41.

5 Radivojevic et al. 2017, 105-6; Sperl 1990.

6 Meskell et al. 2016.

7 Erdoğu 2017, 36.

8 Şahoğlu and Tuncer 2014, 71; Kaptan 2008; Erkanal 2008; Horejs and Mehofer 2015; Begemann et al. 1994.

ca. 1 ha with 11 m of stratigraphic sequence.[9] The Neolithic occupation at the site, which is designated by Levels VI through IV, is dated from 6850 to 5670 cal BC. The analysis of the cereals and the animal bones prove that the subsistence at the site was based on a fully-fledged agricultural system, starting from the basal layers onwards.[10]

Metallic finds have been found in Level VI (6850-6500 cal BC) and Level IV (6000-5670 cal BC). Their absence in between (Level V) may be the result of excavation bias. There are five worked metallic finds (fig. 3), with the remaining objects (n=25) considered metallic lumps (fig. 4 and table 1). The total weight for the metallic lumps is about 1.5 kgs. The measurement of these lumps is highly varied, from small (0.65 x 0.29 cm) to large (7.31 x 3.78 cm).

Three metallic pendants belong to the earliest occupation, Level VI (fig. 3a, b and e). These personal ornaments are of particular significance as they represent the earliest portable symbolic media at Ulucak. Level VI is represented by two adjacent buildings (Buildings 42 and 43) flanked by open spaces with fire installations.[11] Building 42, and the adjacent fire installations, were rebuilt three times while the earliest phase was contemporary with Building 43. Scattered animal bones surrounding the fire installations suggest that they were used for cooking. These buildings, with lime-plastered and red-painted floors and walls, are thought to have been of communal character. Both buildings seem to have been deliberately left clean and covered with a green and sterile layer. No pottery or other clay objects were attested in this earliest phase at the site. One of the pendants has a triangular shape (fig. 3a) and was found in an ashy deposit around the hearth in an open space located at the southern end of Building 43. Two of the pendants are stylized human (figs. 3b and 5) and lozenge shaped (fig. 3e) and were uncovered in a thin fill lying between Building 40 in Level Ve and the wall debris of Building 42 in Level VI. These pendants are considered within the context of Building 42, as Building 40 was directly built on the wall debris of the former building. Archaeological evidence found in relation to Buildings 42 and 43 suggest that they were ritually abandoned. This includes the deliberate placement of objects as part of ritual abandonment of Building 42 and 43 including grinding stones and specific animal bones such as scapulae and mandibles. Additionally, stone beads, grinding stones, and animal bones found in a special deposit above Building 54 in Ulucak Vd provide further evidence that personal ornaments were part of building closure deposits.

Two metallic objects together with twenty-five lumps were found in Ulucak IV (figs. 3c-d and 4). Level IVb (5840-5700 cal BC) has been investigated in a relatively large area, covering ca. 1000 m^2. This phase is characterized by adjacent mudbrick dwellings which were arranged along the narrow streets. The earlier phase of this horizon, IVc (6005-5840 cal BC), is only known from a specialized pottery production workshop.[12] This workshop, consisting of six post-framed structures, revealed a large number of clay loaves, unfinished coil vessels, red hematite lumps, and the remains of pigmented grinding stones used for powdering hematite.

One of the metallic objects (fig. 3d) is reminiscent of the abbreviated human clay figurines from the same horizon (fig. 6) in Ulucak IV. This metallic figurine was found beneath fallen wall debris immediate outside Building 13, which caused great conflagration in Level IVb, and possibly belongs to the same building. Another object looks like a chisel (fig. 3c) when

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9 Çilingiroğlu et al. 2004, 3-5.

10 Çakırlar 2012, 26.

11 Çevik 2019, 221-26; Çevik and Abay 2016, 187-93.

12 Çevik 2016.

compared with stone examples (fig. 7). However, it seems that it was not used for the same purposes. The context of a chisel-like object is not clear as it was found in a fill between Levels IVa and IVb which was partly disturbed by Late Roman building activities.

The metallic lumps (fig. 4) were only attested in Ulucak IV. About half of these lumps were found in buildings with substantial ovens. Most of these *in situ* finds came from Buildings 61 and 66 where pottery production in specialized and domestic contexts took place. The ovens in these buildings, however, appear to have been primarily used for pigment production seen by traces of red pigment on oven bases and a heavy concentration of red hematite lumps surrounding the ovens. The sudden increase in quantity of metallic lumps after 6000 BC, and their contextual relationship with ovens, is significant given the connection between metallic lumps and pottery production, but their connection is yet unknown.

Metallic Ore Sources and Chemical Analysis of the Ulucak Samples

Evidence for mining and metallurgical activities in Anatolia dates back to prehistoric periods.[13] However, metallic sources are particularly rich in the eastern and northeastern regions of Anatolia where such activities are more intensely observed both in the past and present. Nevertheless, the sources and exploitation of copper, gold, silver, lead and zinc have also been reported in western Anatolia.[14]

The shiny appearance of metallic ore lumps could have attracted the Neolithic community of Ulucak. The Neolithic inhabitants at the site must have had easy access to the rich metallic ore sources on the slopes of Nif Mountain, which lies immediately south of the site. In fact, a modern lead mine located about 4 km southeast of Ulucak Höyük is still actively exploited (fig. 2).

It is necessary to determine the mineralogical and chemical properties of both archaeological samples and modern samples by analytical methods. The aim of this analysis is to determine the properties of the material and to conduct a provenance analysis. In order to determine the mineralogical and chemical properties of the metallic finds (Ulucak OVG, OTC, RUO, LOP) (fig. 5), XRD, XRF and SEM-EDX analyses were performed on the metallic figurine from Phase IV (fig. 3d) and on two modern metallic ore samples (Modern 1 and Modern 2) from the above-mentioned lead mine. Since we were not allowed to take samples from the modern mine site, the modern raw material samples were provided by miners.

X-ray Diffraction (XRD) analyses were carried out at the MAM (Marmara Research Center) in TÜBİTAK (The Scientific and Technological Research Council of Turkey), using an diffractometer XRD-6000 Shimadzu (CuKα source, l = 1.5405 Å). The X-ray patterns were collected at an interval of 0.01° and 6° width. The diffraction peaks observed are defined according to Hanawalt Search Manual, Inorganic Phases, Powder Diffraction Files.

As a result of XRD analysis (table 2), except for one of the modern samples (Modern 2), the main mineral components are galena, anglesite and cerusite.[15] The main identification of galena, as well as the presence of other lead mineral phases (anglesite, cerusite) (table 2), clearly confirms the mineral configurations of archaeological lump findings and one of the

[13] Tylecote 1976; de Jesus 1980.

[14] de Jesus 1978, 1980.

[15] Lafuente et al. 2015.

modern samples.[16] Anglesite and cerussite minerals are alteration products formed as a result of the galena mineral.[17]

The main mineral components of the other modern sample, Modern 2, were determined as pyrite, barite, smithsonite, quartz and calcite. These are not lead-containing minerals. However, these minerals are associated with the galena mineral. Galena is a common sulfide in hydrothermal veins in association with sphalerite, pyrite, chalcopyrite, marcasite, calcite, quartz, barite, fluorite, smithsonite and silver minerals.[18] In hydrothermal veins it is formed under a wide range of temperatures and in contact with metamorphic deposits in pegmatites. Limestones and dolostones are common host rocks.[19] Nif Mountain and its environs south of Ulucak, where the modern samples were taken, was a suitable geological resource area. Jurassic-Cretaceous-aged Neritic limestone units and Upper Senonian-aged clastics and carbonate units can provide suitable environments for this type of ore formation.[20]

X-ray fluorescence spectroscopy (XRF) analyses was also carried out at the MAM in TÜBİTAK. XRF analyses were performed on the same three samples in order to determine the main element concentrations. X-ray fluorescence spectroscopy analysis was performed using a Philips PW-2404 system equipped with 4 kW Rh x-ray source, 6 analyzer crystals (LiF 220, LiF 200, Ge 111, PE 002, PX-1 and PX-4), 3 detectors (argon flow proportional and scintillation detectors, sealed xenon detector) and Super Q 4.0 software. The samples were directly analyzed without sample preparation.

The results of analysis are presented in table 3. One of the modern samples (Modern 1) shows close similarity to the archaeological lumps, especially with regards to its lead content. Ulucak OTC provides the closest similairity. The second modern sample (Modern 2) has high zinc and iron concentrations in parallel to XRD results and does not contain lead.

In addition to archaeological lumps, the Phase IV metallic figurine (fig. 3d) was subjected to non-destructive SEM-EDX analysis. SEM-EDX analysis were carried out at the TÜTAGEM (Technology Research Development Application and Research Center in Trakya University) using a ZEISS-EVO® LS 10 scanning electron microscope system equipped with thermionic emission (W, LaB6), 3 nm @ 30 kV, 20 nm @ 1kV resolution, energy dispersive spectrometer (EDS) and backscattered electron detector (4QBSD). During the analysis, backscattered electron mode was also used, therefore, elemental density in the area where the analysis was applied was determined and mapped in different colors (fig. 8 and table 4). Areas with high lead (Pb) density are shown in pink. These results support the previous results of XRF analysis performed on metallic ore lumps.

Native lead is rarely encountered. The principal ore of lead is galena (lead sulphide), which, when it occurs in hydro-thermal veins, is frequently associated with silver ore minerals. Cerussite (lead carbonate) is an important, widely distributed secondary ore mineral of lead formed by the action of carbonated waters on galena.[21] Galena has a distinct silver color and a bright metallic luster, while it is relatively soft, heavy mineral.[22] The manufacturing techniques

16 Moore and Reynolds 1997.

17 Keim and Markl 2015.

18 Klein and Philpotts 2013.

19 Anthony et al. 1990.

20 MTA 1972.

21 Moorey 1994.

22 Austin et al. 2000, 123.

of metal objects from Ulucak are unknown. However, it has been suggested that galena can easily be shaped with stone-working techniques.[23]

Conclusion

The results of XRD and XRF analyses on the metallic lumps, together with the result of the SEM-EDX analysis on one of the archaeological objects, shows that galena was exploited throughout the Neolithic period at Ulucak Höyük. The close similarity between the galena lumps and the modern samples from the nearest lead mine indicates the possible provenance of the archaeological finds. Lead isotope analysis is one reliable methodology that can be used to identify the origin of metal artefacts.[24] Thus, in the next stage of the our study, lead isotope analysis will be conducted to accurately determine the provenance of the archaeological samples.

In contrast to the exploitation of native copper and malachite during the Neolithic period, artefacts made of galena have so far only been attested at Tell Halula, Çatal Höyük, and now Ulucak. There seems little evidence, if any, to suggest that the knowledge of exploitation of this raw material at Ulucak was transferred from the East, as local sources are close to the site. Furthermore, neither the subsistence economy nor the lithic technology at Ulucak suggests any similarities with the sites in central Anatolia.[25] Thus, the Ulucak Neolithic community may well have been innately impressed by the shiny appearance of galena.

It is also worth noting that objects made of galena from Ulucak represent symbolic media, such as personal ornaments and a figurine. The chisel-like object may also have been considered symbolically significant, as galena is a soft material for tool manufacture. It has been generally argued that practical technologies were stimulated by aesthetic curiosity and specific socio-cultural desires rather than economic or technical necessities.[26] At Çatal Höyük, for instance, a piece of galena found next to the limestone figurine in a special deposit is thought to have been associated with the manufacturing process of the figurine because of the abraded edges of the piece.[27] The abbreviated galena figurine from Ulucak and the use of galena as a tool in making figurines at Çatal Höyük may show us a particular significance that cross-culturally attributed to this raw material.

Hayden also placed prestige technologies as the first stage of technical achievements which later evolved into more practical applications.[28] Three galena pendants found in the earliest level at Ulucak can be considered prestige items. The percentage of the galena pendants is indeed rare, less than one percent, when they are compared with the total number of the personal ornaments made of stone, bone and shell from the site. Visibility and distinctiveness are considered important aspects of prestigious items. In a wider sense it has been stressed that prestige acts simultaneously as a mechanism of social distinction.[29] Personal ornaments are considered to be one of the body's paraphernelia which played an active role in the

23 Pernicka 2014, 449.

24 de Jesus and Dardeniz 2015.

25 Guilbeau et al. 2019, 15; Arbuckle et al. 2014.

26 Smith 1977, 146; Roberts et al. 2009, 1012; Clark 2015.

27 Meskell et al. 2016, 141 and fig. 7.

28 Hayden 1998, 33-34.

29 Bagley and Schumann 2013, 125-26.

consititution of past identities.[30] Accordingly, the recovery of galena ornaments from communal buildings at Ulucak may be further evidence to indicate certain individuals with affiliation to these buildings may have gained their social status by wearing these potentially prestigious ornaments.

The increase of galena lumps in Ulucak IV, and their close contextual relation to ovens, lead us to believe that galena may have been fired after 6000 BC. In two cases (Buildings 61 and 66) where a high number of galena lumps were found, the function of ovens was clearly related to pottery-making, and particularly for red pigment production. Thus, it is yet unknown whether these ovens were used for pigment production and galena firing, or whether galena had some role in pigment production itself. Exploitation of galena for pigment material is known from later periods.[31] However, the pigment colors originating from galena are black, gray and white. Therefore, the use of galena in pigment production can hardly be assumed as the surface color (slip) of Ulucak Neolithic pottery is mainly red. Nonetheless, galena was most likely perceived as an exotic "stone" initally to those at Ulucak, as a full understanding of its natural properties developed over time.

[30] Joyce 2005, 142-43.

[31] Austin et al. 2000, 123.

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FIG. 1 The map showing the sites mentioned in the text.

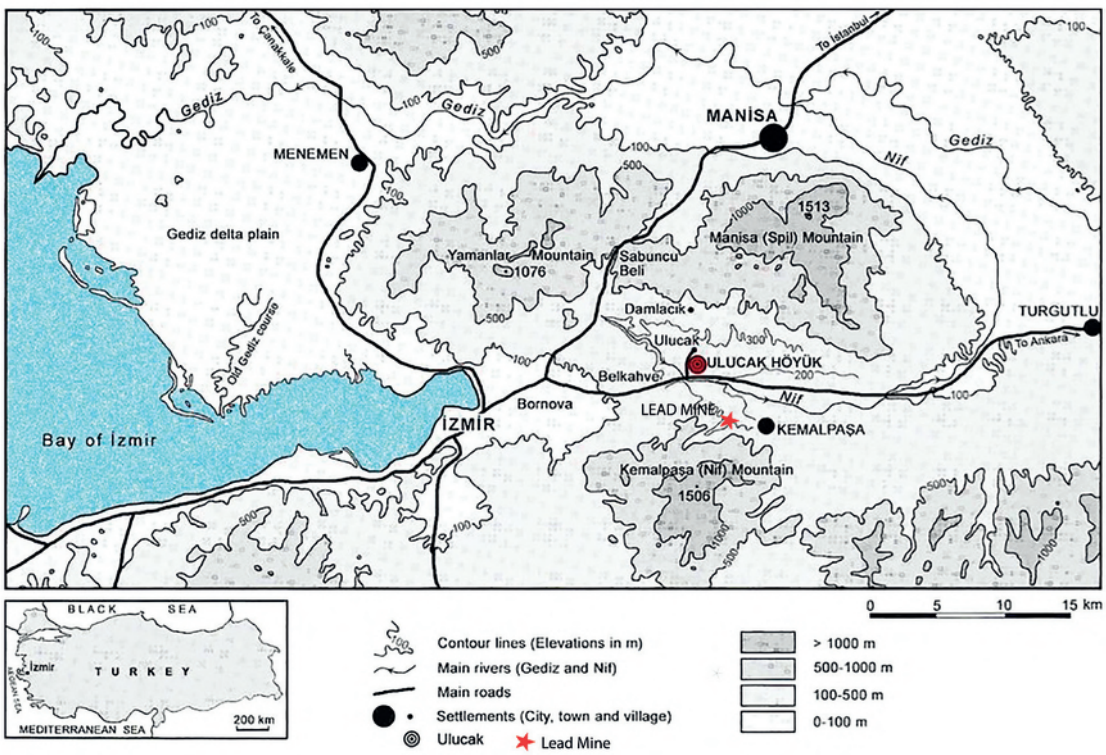


FIG. 2 The map showing the locations of Ulucak Höyük and lead mine.

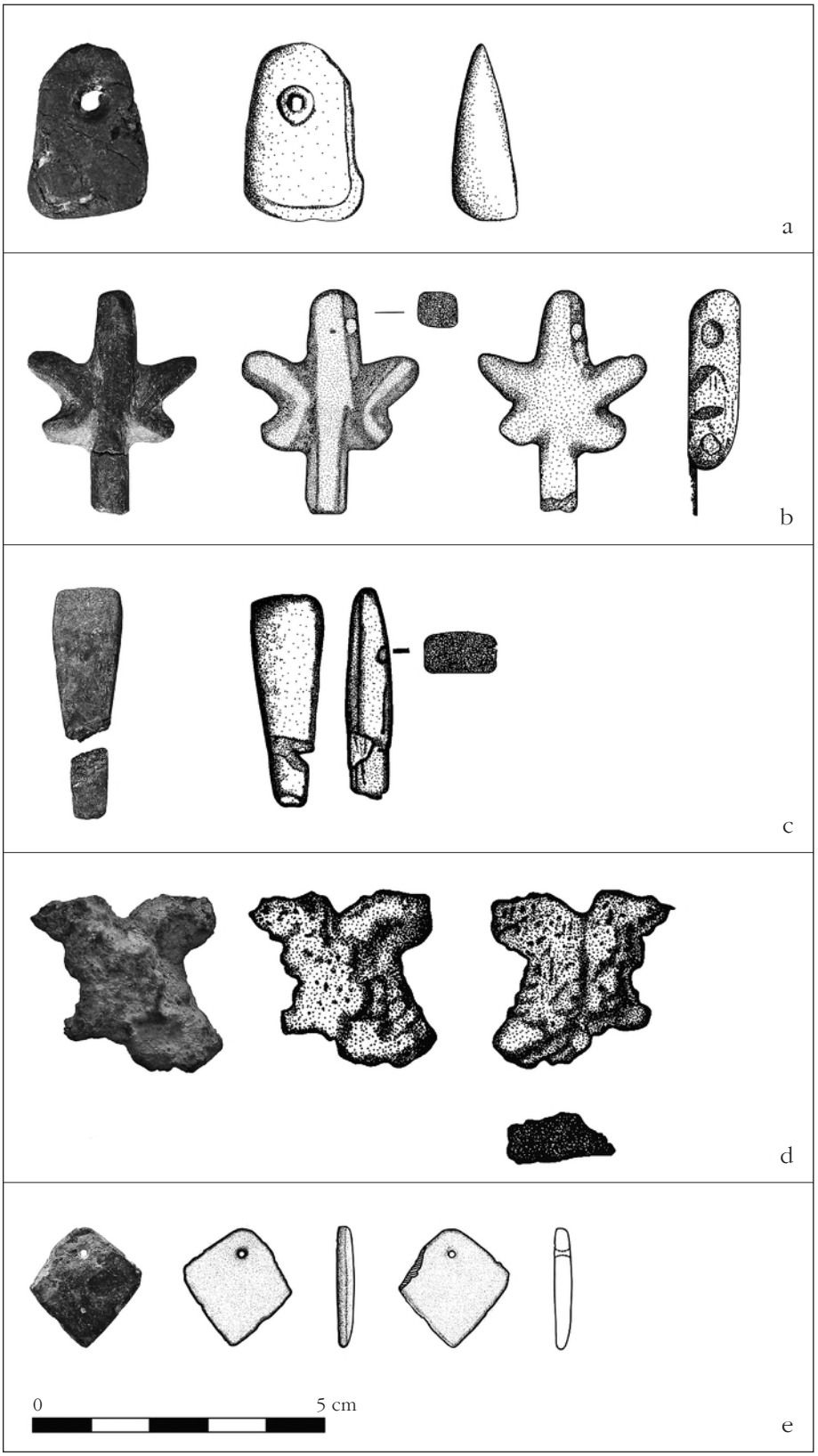


FIG. 3
Photos and drawings of galena objects.

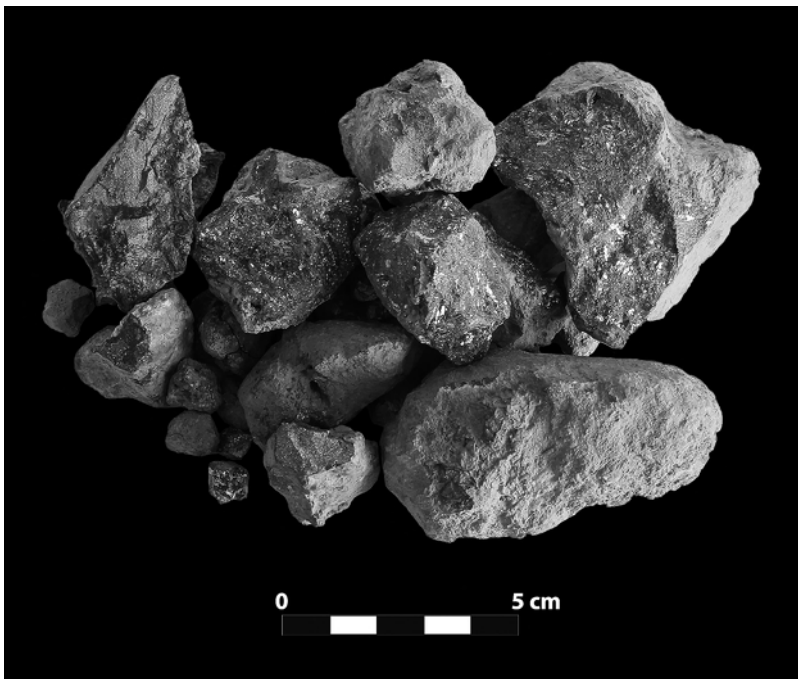


FIG. 4
Galena lumps
from Ulucak IV.

FIG. 5
Galena pendant
(level VI) and a chisel
like object (level IV).

FIG. 6 Galena figurine and abbreviated clay figurines from Ulucak IV.

FIG. 7
Chisel like galena object and stone chisels.

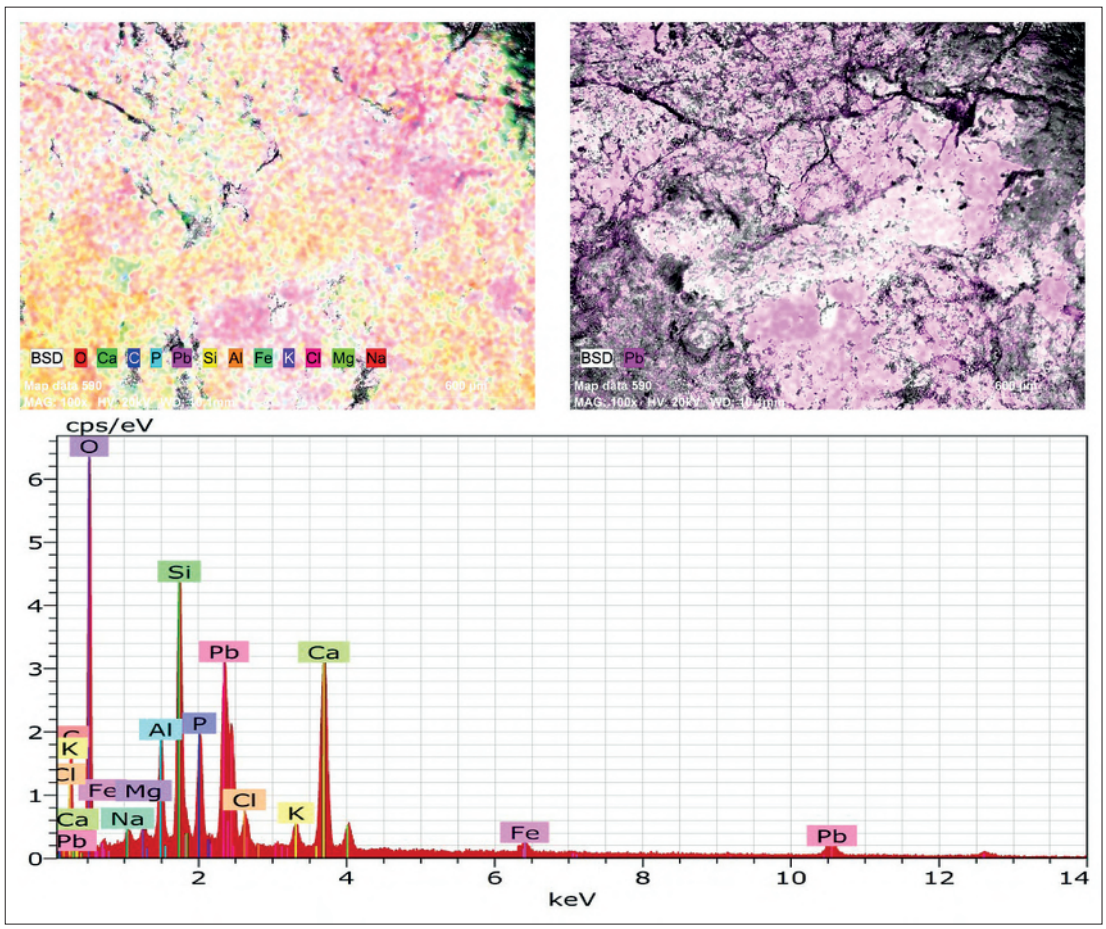
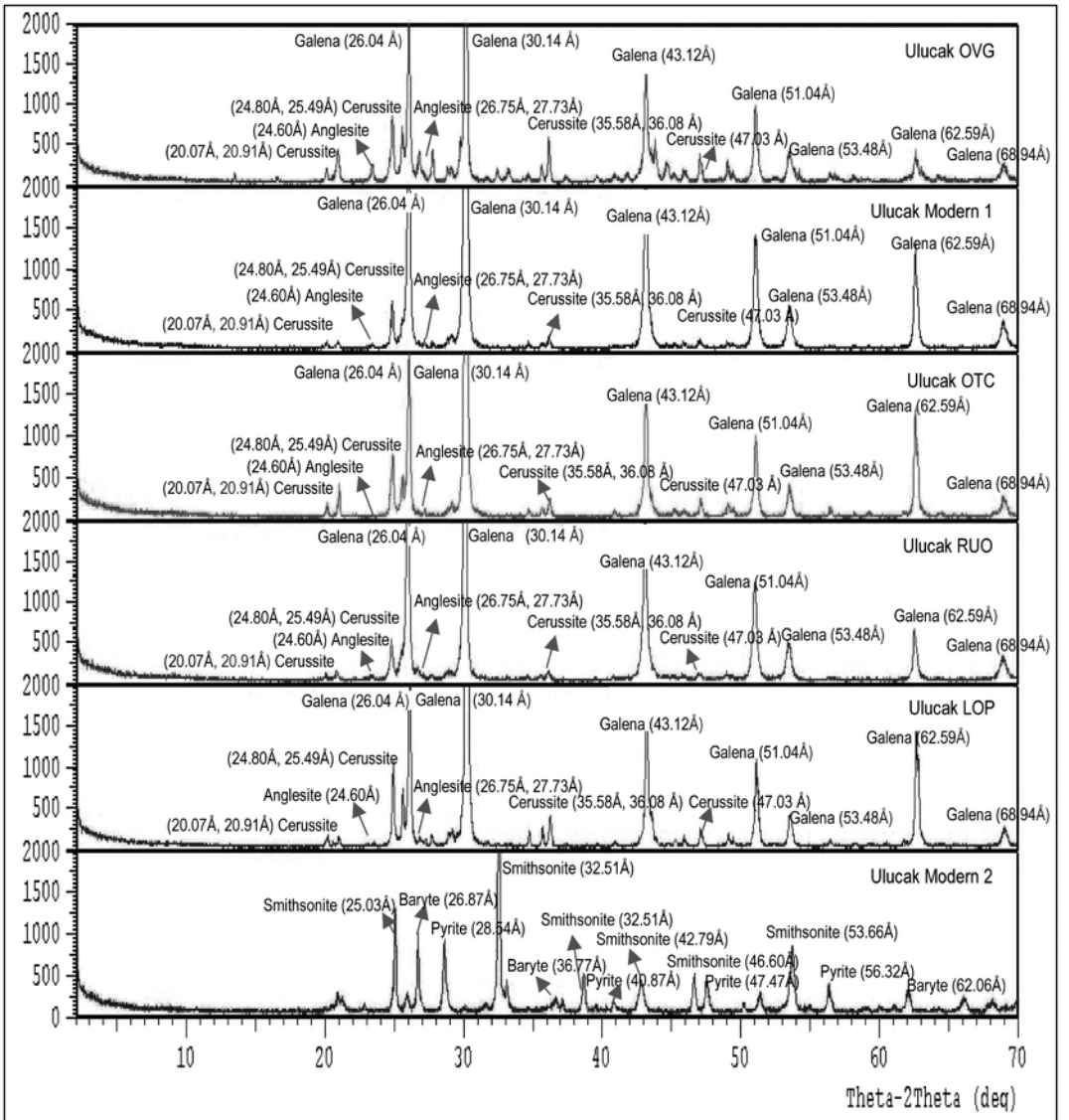


FIG. 8 Results of SEM-EDX analysis, backscattered electron mode.

TABLE 1 Context and dating of galena finds from Ulucak Höyük.

No	Code	Object	Trenc	Level	Context	Dating
1	JMC	Pendant (fig. 3a)	L13	VI	From the ashy fill (unit 269) around the hearths in open space	6850/6830-6500 cal BC
2	FRM	Pendant (fig. 3b)	L13	VI	Above the wall debris of Building 42.	6850/6830-6500 cal BC
3	CEB	Chisel	M12	IVa	From the fill possibly related to flimsy architectural remains of the latest phase of Level IV.	5700-5670 cal BC
4	KSE	Anthropomorphic figurine (fig. 3d)	013	IVb	Outside Building 12 but beneath the fallen mudbrick wall of the same building.	5840-5700 cal BC
5	FRL	Pendant (fig. 3e)	L13	VI	Above the wall debris of Building 42.	6850/6830-6500 cal BC
6	CFI	Lump	M13	IVb	From the courtyard of Building 19.	5840-5700 cal BC
7	JES	Lump	013	IVb	Fill	5840-5700 cal BC
8	JVB	Lump	013	IVb	Fill	5840-5700 cal BC
9	JZE	Lump	013	IVb	Fill	5840-5700 cal BC
10	KBD	Lump	013	IVb	Fill	5840-5700 cal BC
11	KHV	Lump	013	IVb	Fill	5840-5700 cal BC
12	KOI	Lump	013	IVb	Fill	5840-5700 cal BC
13	LCV	Lump	013	IVb	Fill	5840-5700 cal BC
14	LJS	Lump	013	IVb	Fill	5840-5700 cal BC
15	LOP	Lump	013	IVb	Fill	5840-5700 cal BC
16	MOT (2 pieces)	Lump	013	IVb	From fire debris of Building 52.	5840-5700 cal BC
17	MRE	Lump	013	IVb	From fire debris of Building 52.	5840-5700 cal BC
18	ONB	Lump	M12	IVb	From fire debris of Building 57.	5840-5700 cal BC
19	OTC	Lump	M12	IVc	From fire debris possibly belonging to Building 61 or 62 (pottery workshop).	6005-5840 cal BC
20	OVG	Lump	M12	IVc	Building 61 (pottery workshop)	6005-5840 cal BC
21	PIR	Lump	M12	IVc	From the small cache pit dug into the floor of Building 61 (pottery workshop).	6005-5840 cal BC
22	RUO	Lump	010	IVb	Building 66 around the oven	5840-5700 cal BC
23	RUP	Lump	010	IVb	Building 66 around the oven	5840-5700 cal BC
24	RUV	Lump	010	IVb	Building 66 around the oven	5840-5700 cal BC
25	RYH	Lump	010	IVb	Building 66 around the oven	5840-5700 cal BC
26	SDY	Lump	010	IVb	Building 66 around the oven	5840-5700 cal BC
27	SLD	Lump	010	IVb	Building 66 around the oven	5840-5700 cal BC

TABLE 2 Results of XRD analysis.



	Ulucak OVG	Ulucak OTC	Ulucak RUO	Ulucak LOP	Ulucak Modern1	Ulucak Modern2
Galena / PbS	X	X	X	X	X	
Cerussite / $Pb(CO_3)$	X	X	X	X	X	
Anglesite / $Pb(SO_4)$	X	X	X	X	X	
Srebrodolskite / $Ca_2Fe^{3+}{}_2O_5$				X		
Barium Lead Sulfate / BaO_8PbS_2	X		X	X		
Lead Oxide Sulfate / Pb2O(SO4)		X		X	X	
Calcite / $CaCO_3$		X				
Celstine / SrSO4	X					
Quartz / SiO_2		X		X		X
Smithsonite / $Zn(CO_3)$						X
Pyrite / $Fe^{2+}S_2$						X
Baryte / $Ba(SO_4)$						X
Barium Iron Oxide / $BaFe_{12}O_{19}$						X

TABLE 3 Results of XRF analysis.

	Ulucak OVG (wt%)		Ulucak OTC (wt%)		Ulucak RUO (wt%)		Ulucak LOP (wt%)		Ulucak Modern1 (wt%)		Ulucak Modern2 (wt%)		Det.Limits
	unn. c	norm. c	unn. c	norm. c	unn. c	norm. c	unn. c	norm. c	unn. c	norm. c	unn. c	norm. c	
O	21.1	21.409	21.0	21.515	22.3	22.697	22.5	22.325	22.6	22.826	32.5	32.603	0.85267
Na	0.18	0.193	0.04	0.039					0.04	0.043			0.00244
Mg	0.22	0.242	1.5	0.144	0.03	0.025	0.1	0.091	0.07	0.069	0.1	0.103	0.00754
Al	0.86	0.853	0.57	0.525	0.25	0.278	0.4	0.450	0.22	0.214	0.3	0.313	0.00196
Si	1.6	1.504	1.2	1.111	0.9	0.810	1.2	1.332	0.31	0.322	6.9	6.844	0.00153
P	0.07	0.086	0.32	0.334	0.18	0.178	0.21	0.255			0.02	0.024	0.00140
S	8.6	8.573	8.7	8.977	10.1	10.515	8.9	9.049	11.5	11.415	5.6	5.716	0.00110
K	1.9	0.188	0.16	0.173	0.05	0.063	0.21	0.192	0.06	0.065	0.08	0.074	0.00138
Ca	0.68	0.600	1.12	1.138	0.7	0.699	1.65	1.697	0.14	0.153	0.3	0.327	0.01743
V			0.12	0.119									0.00131
Fe	0.4	0.347	0.3	0.289	0.3	0.269	1.03	1.046	0.09	0.096	16.4	16.332	0.00710
Ba	1.9	1.905	0.6	0.666	2.3	2.243	6.1	6.391			3.9	3.849	0.00374
Pb	62.7	63.145	65.3	64.969	61.3	61.838	56.5	56.306	64.8	64.797			0.00142
Cr											0.01	0.013	0.00216
Zn	0.25	0.235			0.4	0.353	0.82	0.867			33.2	33.500	0.00324
Cd											0.25	0.243	0.00873
Co											0.05	0.041	0.00987
Cu	0.76	0.719			0.03	0.032					0.01	0.016	0.00484

TABLE 4 Results of SEM-EDX analysis.

Elements	Unn. C [wt.%]	Norm. C[wt.%]	C Atom. [at.%]	C Error[wt.%]
O	33.89	41.67	59.13	4.32
Pb	14.92	18.35	2.01	0.56
Ca	12.05	14.82	8.40	0.39
C	8.50	10.46	19.77	1.40
Si	4.15	5.11	4.13	0.21
P	2.14	2.63	1.93	0.11
Al	1.84	2.26	1.90	0.12
Fe	1.59	1.95	0.79	0.08
K	0.98	1.21	0.70	0.06
Cl	0.61	0.75	0.48	0.05
Na	0.35	0.43	0.43	0.06
Mg	0.29	0.36	0.34	0.05