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Analysis of the Technical Aspects and Microstructure of Copper Alloys in Ancient Iron Age Metallurgy in Sagzabad

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Abstract

The settlements of the Qazvin Plain, particularly at Tepe Qabrestan and Sagzabad, demonstrate a continuous sequence of metallurgical development in the prehistoric period of this region. The aim of the present study is to examine the functional aspects, manufacturing technology, chemical composition, microstructure, and alloy phases of metal artifacts recovered from the Iron Age levels of the Sagzabad site. In this context, the study aims to address key questions regarding the production processes and manufacturing techniques of the artifacts, the alloying practices employed, as well as the chemical composition and formation conditions of the identified corrosion products. To achieve these objectives, various analytical techniques were employed, including XRF for determining the chemical composition. The results indicated the presence of various alloys, including copper–arsenic, copper–antimony, tin bronze, and nearly pure copper. Metallographic analysis of two samples with preserved metallic cores revealed a cast structure with a distinct core, providing important information about the manufacturing techniques. In addition, SEM-EDS observations confirmed the presence of α-phase dendrites and the formation of γ-phases associated with copper–arsenic and copper–antimony alloys. Elemental analysis results indicate varied patterns in the use of copper–arsenic and copper–antimony alloys, suggesting that ancient metalworkers possessed considerable knowledge of how to achieve desirable mechanical properties in their metallic products. In addition, the identification of silver inclusions and sulfide intermetallic compounds within the microstructure allows hypotheses to be proposed regarding the types of ore sources used in the metal extraction process. The identified manufacturing technologies ranged from two-part mold casting to the production of chisels with both spiral and simple cross-sections. The analyzed artifacts comprise decorative, ornamental, and Functional objects. This typological and technological diversity provides a comprehensive view of Iron Age metallurgical practices at Tepe Sagzabad.

Keywords: Copper–Arsenic, Copper–Antimony, Bronze, Sagzabad Iron Age, Ancient Metallurgy.

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Introduction

The Qazvin Plain is one of the principal and long-lasting centers of human settlement on the Iranian Plateau, with continuous evidence of occupation from the sixth to the early first millennium BCE, as documented at sites such as Tepe Zagheh, Tepe Qabrestan, and Tepe Sagzabad. The first scientific archaeological excavation in this region was conducted in 1968 by the Department of Culture and Arts of Qazvin. From 1970 onward, Tepe Sagzabad became one of the main research centers under the educational and scientific excavations of the Department of Archaeology at the University of Tehran (Negahban, 1972). Tepe Sagzabad is a large mound covering approximately 125,000 square meters, rising about 5 meters above the surrounding plain. The first excavation trench, designated as Trench A (5×5 m), was opened in the southeastern part of the mound. This trench reached virgin soil at a depth of 517 cm, revealing nineteen stratigraphic layers. Following the discovery of valuable ceramics from the main trench (A), additional test trenches B, C, D, and E were excavated successively in the northwest, west, south, and southeast sectors. Altogether, thirty-six well-defined archaeological layers were documented, extending from the natural soil to the remaining mound surface. These excavations, particularly in Trench A in the southeastern area of the mound, exposed a sequence of 36 archaeological layers representing continuous occupation from the late fourth to the mid-first millennium BCE (Majidzadeh, 1977: p. 53; Shahmirzadi, 1977: pp. 81–98). Stratigraphic studies and the material culture recovered from Tepe Sagzabad especially during the renewed excavations conducted between 1997 and 1999 (Talaei, 2014: p. 51). clearly illustrate the sequence of settlement and population movements from Tepe Zagheh to Tepe Qabrestan and subsequently to Tepe Sagzabad. Among these, the site locally known as Tepe Qabrestan, located about 300 meters west of Tepe Sagzabad, has been identified as the cemetery associated with the Iron Age settlement at the main mound. Although many graves suffered significant damage due to illicit digging, the metallic artifacts recovered from the site still provide valuable evidence concerning the metallurgical technologies of the Iron Age (Negahban, 1972: pp. 12, 14). The archaeological program in the region pursued dual objectives regional survey and excavation of the main mounds and also included test trenching, surface surveys, artifact classification, and the preparation of archaeological maps (Negahban, 1972: pp. 1–25).

In this context, one of the overlooked aspects has been the systematic and detailed study of the metallic artifacts recovered, particularly from the cemetery site; these objects, encompassing various weapons, tools, and copper-alloy ornaments, provide a unique opportunity to investigate alloying technologies, sourcing of raw materials, and post-burial corrosion mechanisms. Despite compelling evidence for the presence and evolution of metallurgical technologies in the Iron Age of the Qazvin Plain, few targeted and interdisciplinary archaeometric, metallurgical, and deterioration studies have focused on these artifacts. Therefore, the present study, employing analytical and technical methods, aims to examine the structure, composition, manufacturing techniques, and corrosion states of metallic samples from the Sagzabad cemetery, thereby contributing to the reconstruction of Iron Age metallurgical traditions in this region and enhancing our understanding of past human interaction with technology and the environment. The primary objective of this research is to identify the uses and nature of these objects, the manufacturing technologies, and the alloying practices of metallurgical products in the Early Iron Age at this site. The level of technological sophistication and functional applications of these artifacts is analyzed to shed light on the skill and knowledge of ancient metalworkers in production, fabrication, and shaping techniques.

Aims of the study: This study seeks to conduct a detailed investigation of the microstructure, manufacturing methods, and chemical composition of copper alloys from the Iron Age in the Sagzabad cultural context. Considering the long-standing tradition of metallurgy in the Qazvin Plain, which began in the sixth millennium BCE and continued intermittently until around 800 BCE at archaeological sites such as Tepe Zagheh, Qabrestan, and Sagzabad, examining the technical and structural characteristics of these artifacts is of particular importance. The historical continuity of settlement and metallurgical activities in this region provides an ideal context for research aimed at understanding production techniques, technical skills, and material characteristics used in the fabrication of metal objects. Accordingly, this study focuses on the technological and structural aspects of Iron Age copper alloys, seeking to offer a clearer perspective on the technical knowledge and production methods of this cultural period.

Research Questions and Hypotheses: Considering the long-standing and prominent tradition of metallurgy in the Qazvin Plain, particularly at Tepe Zagheh, Qabrestan, and Sagzabad, it is expected that Iron Age metallurgical practices had reached a notable level of technological maturity and sophistication. During this period, skills in metal extraction, alloying, and the production of copper-based objects were likely developed in a complex and systematic manner.

Accordingly, this study seeks to address the following questions: 1. How were the production processes and manufacturing techniques of metallic artifacts executed in the Iron Age culture of Sagzabad? 2. What methods were employed in alloying and combining metals in these artifacts? 3. What were the compositions and formation conditions of the corrosion products present in the artifacts?

The underlying hypothesis is that the Iron Age metallurgists of Sagzabad possessed advanced technical knowledge, enabling them to produce a variety of copper-based alloys with controlled compositions and tailored properties, and that the observed corrosion products reflect specific post-depositional chemical and environmental processes. The research hypothesis is based on the premise that the experiences and technological knowledge of earlier periods at Sagzabad contributed to the development of more advanced techniques in alloying, casting, and shaping metal objects. It is further anticipated that microstructural and chemical composition analyses will provide evidence of a systematic approach in the selection of raw materials, alloy types, and manufacturing processes.

Research Methodology: In line with the practical objectives of this study, a combination of library research, examination of study samples, and instrumental analyses was employed to investigate and analyze the manufacturing techniques and corrosion mechanisms of the artifacts. Metallographic analysis was conducted to observe the microstructure using a metallographic microscope under both bright-field light (BFL) and polarized light (PL) conditions. Identification of manufacturing techniques, microstructures, and phases was carried out through scanning electron microscopy (SEM). Energy-dispersive X-ray spectroscopy (EDS) was used for semi-quantitative measurement of the chemical composition of the phases. Additionally, Micro-X-ray fluorescence (Micro-XRF) was utilized to identify the elemental composition relevant to the manufacturing technology of the artifacts.

Research Background

Negahban and Talaei consider three major sites in the Qazvin Plain Tepe Zagheh, Tepe Qabrestan, and Tepe Sagzabad as forming a continuous sequence of occupation from the Late Neolithic to the Early Iron Age (Negahban, 1977; Talai, 1984; Mazaheri, 2004). Previous archaeological investigations of ancient metallurgical finds in the Qazvin Plain have resulted in typological classifications of these objects (Malekzadeh, 1977: p. 77). The growing use of modern instrumental techniques has enabled more precise studies aimed at understanding metallurgical technologies at Tepe Sagzabad, shedding light on the emergence of various metals in prehistoric Iranian societies and their metallurgical processes in north-central Iran. Laboratory elemental analyses of seven metal artifacts recovered from the fifth (two artifacts) and sixth (five artifacts) occupational layers during 1997–1998, corresponding to the Early and Middle second millennium BCE, were carried out at the Iranian Atomic Energy Organization using the PIXE4 method. The results indicate that the artifacts were made and processed from relatively pure oxidized copper ores containing minor amounts of arsenic (0.1–1.22 wt%) and tin (0.23–1.83 wt%). In some samples, traces of copper ores with malachite and chalcopyrite were also present. Metalworkers at Sagzabad used relatively pure copper (97.97%) as well as copper–arsenic alloys and bronze (Talaei, 2002: pp. 547–564). Subsequent studies employing instrumental analyses in Iran have focused on understanding manufacturing techniques and identifying the elemental composition of copper-alloy artifacts from

Qazvin Plain sites. Based on these studies, the earliest metallurgical evidence indicates the use of native copper. Metallographic analyses confirm the use of hammering for shaping objects (Talaei, 2014: pp. 44–45). According to Pigott, archaeological evidence shows the distribution of copper deposits and their significance in Iran as a central metallurgical region in the Iranian Plateau, compared to other parts of Southwest Asia, highlighting Tepe Qabrestan as one of the best examples of an early copper smelting workshop in the region, deserving detailed study to describe technological styles (Pigott, 2004). Research on metallic samples from Iron Age graves at Sagzabad employed ICP-MS to determine chemical composition and provenance, alongside metallography and SEM-EDS to examine microstructure, manufacturing techniques, and chemical composition. The results indicate the use of copper alloys with high nickel, cobalt, and iron content, suggesting uncontrolled metallurgical processes followed by cold and hot hammering as well as annealing. The presence of minor elements also points to a shared provenance for most of the materials. Notably, there is evidence for the use of two distinct metallurgical technologies and ore sources during the late second and early first millennium BCE (Iron II) in the Qazvin Plain (Ghodousian *et al.,* 2017: pp. 167–187).

Several archaeological studies have compared metallurgical data from contemporary Qazvin Plain sites. For example, a 2002 study on copper-alloy pins and daggers from Tepe Yahya (circa 800 BCE) using ICP-MS provided an important comparison for technological and cultural exchange with the Qazvin Plain. Evidence shows that the introduction of new metals and alloys, such as arsenical copper, did not completely replace earlier metals; native copper continued to be produced for about 300 years before arsenical copper gradually became dominant. Arsenical copper was initially used for inexpensive local ornaments. Northern tools from the site mainly contained low-arsenic copper (1–2%), whereas ornaments had higher arsenic content (3–7%) to achieve a silvery sheen and improved aesthetic qualities (Thornton, 2002). A critical research issue in the study of native and bronze copper is the sourcing of arsenical copper in the Iranian Plateau. Pigott suggests that preliminary geological studies should provide new information on copper sulfo-arsenide deposits. When archaeological copper artifacts are analyzed comprehensively in terms of stratigraphy, typology, and scientific analysis, they can provide new insights into the production sites and exchange of arsenical copper in the Iranian Plateau (Pigott, 2004). One extensive study on a wide range of archaeological finds including metal objects, metal lumps, and smelting slags was conducted at Tepe Zagheh and Tepe Qabrestan (Pigott *et al.,* 2003). According to these results, Zagheh metalworkers used locally available native copper to produce simple tools, such as daggers, nails, and ornaments, through cold hammering and annealing (Oudbashi *et al.,* 2022). Additionally, sulfide compounds were linked to covellite and chalcopyrite in carbonate ores. Analyses of the high residual copper beads in the slags from Tepe Qabrestan indicate that the primary copper extraction process had low efficiency. Most artifacts were identified as copper–arsenic or copper–arsenic–antimony alloys, with lead content deriving from minerals such as arsenopyrite, argentotennantite, or tennantite. The low sulfur content in the studied samples is attributed to the combined use of oxide and sulfide ores (Zhao *et al.,* 2021).

Materials and Methods

Study Objects

The artifacts examined in this study belong to the Archaeology Institute of the University of Tehran and were recovered from Iron Age graves located on the western margin of Tepe Qabrestan. These graves are culturally and historically closely associated with the neighboring site, Tepe Sagzabad, which belongs to the Iron Age cultural context.

The studied collection includes:

- An earring (TG-24) with an approximately dumbbell-shaped form.
- A bird-shaped head (TG-28), likely part of a larger object.
- Two rod-like objects (TG-32 and TG-33) with relatively narrow tips at one end.

Based on typology and morphology, TG-24 and TG-28 can be classified as decorative or ornamental objects, while the two rod-like items (TG-32 and TG-33) are likely utilitarian. The latter possess thickened ends but are completely mineralized and do not retain any intact metallic cores, which may be related to burial conditions or environmental moisture (Fig. 1).

Fig. 1: Image of the studied objects (Authors, 2024).

Precise classification of the objects is important, as their chemical composition varies depending on type and function an observation also noted in metallurgical studies of various sites across the Iranian Plateau, reflecting technological and functional diversity in different historical periods. In this context, sampling was carried out from areas with the highest likelihood of containing a metallic core, and to examine manufacturing details, detect possible joints, and assess the extent of corrosion, the objects were radiographed using a Gilardoni X-ray device (Italy) at 70 kV for one minute from a distance of 50 cm with a current of 4.5 mA at the Faculty of Conservation and Restoration, Art University of Isfahan. Subsequently, samples were taken, mounted, and polished. After preparation, observations were conducted using a Field Emission Scanning Electron Microscope (FESEM) QUANTA FEG-450 (FEI, USA) in BSE mode at higher magnifications to observe phases in the microstructures, coupled with Energy Dispersive X-ray Spectroscopy (EDX) using the TEAM™ system (UK) at the Central Laboratory of Isfahan University of Technology. For metallographic studies, samples were etched in an alcoholic ferric chloride solution for 3–5 seconds. Metallographic examinations under polarized light (PL) were performed using a D/A POL Primotech microscope (Zeiss, Germany) at the Central Laboratory of the University of Art, Isfahan. Quantitative chemical composition analyses were conducted at the Central Laboratory of the Art University of Isfahan using a Micro-XRF device (Unisantis xmf-104, Switzerland) with the smart XRF software, a wavelength of 1.07 Å, equipped with a molybdenum tube and a Si-PIN detector (Tallomi), with measurements taken at 35 kV and 0.5 mA for 300 seconds.

Results

As mentioned, the objects were examined by X-ray imaging to identify manufacturing details, possible decorations, cracks and microcracks, and structural density and compactness. According to these images, the structures of samples TG-24, TG-28, and TG-33 appeared as bright white. Sample TG-24, initially identified as an earring, contained a cavity for attachment via a pin to the ear, which was filled with burial soil deposits. This cavity was located at the terminal part of the earring. In sample TG-28, the joint line of a two-part mold under the bird's head was detectable, indicating casting as the manufacturing method for this object.

Additionally, a cavity representing the bird's eye is present, which unfortunately is not visible in the X-ray image due to the altered orientation of object TG-28. In sample TG-32, the structure appears as alternating bright and gray layers. Considering the rod-like shape of TG-32, it is likely that the object was either formed by rolling or hammering a sheet around its longitudinal axis, or that the observed layers resulted from

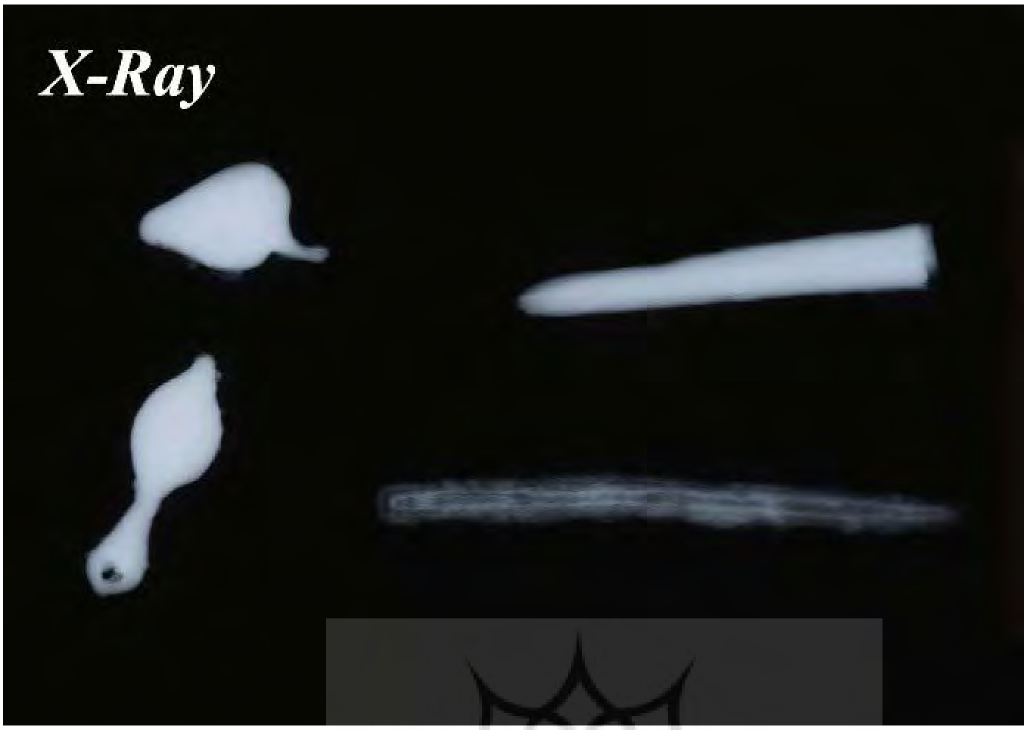

Fig. 2: Imaging objects with X-Rays (Authors, 2024).

periodic corrosion over time. Sample TG-33 exhibits a dense and massive structure, suggesting a higher probability of a metallic core being present (Fig. 2).

Microscopic Observations

Sample TG-24 exhibits a structure characterized by yellow dendrites with inter-dendritic regions appearing gray under bright-field illumination. Additionally, scattered round bright inclusions are present within the matrix. Dark-colored phases are observable surrounding the gray matrix phase. In sample TG-28, similar scattered dark and bright inclusions can be observed within the microstructure. More detailed differences between the microstructure and composition of TG-24 and TG-28 were distinguishable through metallographic examination and semi-quantitative and quantitative analyses. As shown in the image, the cross-section of sample TG-32 is square and somewhat helical. This volume appears to have formed by twisting a sheet around a longitudinal axis. The center of this object is hollow, and except for limited regions, its metallic core has largely mineralized (Fig. 3).

Scanning Electron Microscopy

BSE images of the cross-sections revealed samples containing substantial regions of metallic core. The phases observed consisted of dark-colored, dark gray, light gray, and bright white areas (Fig. 4). In two samples, a dendritic microstructure was identifiable. In some regions, a dark gray margin surrounded the dendrites. According to Table 1,

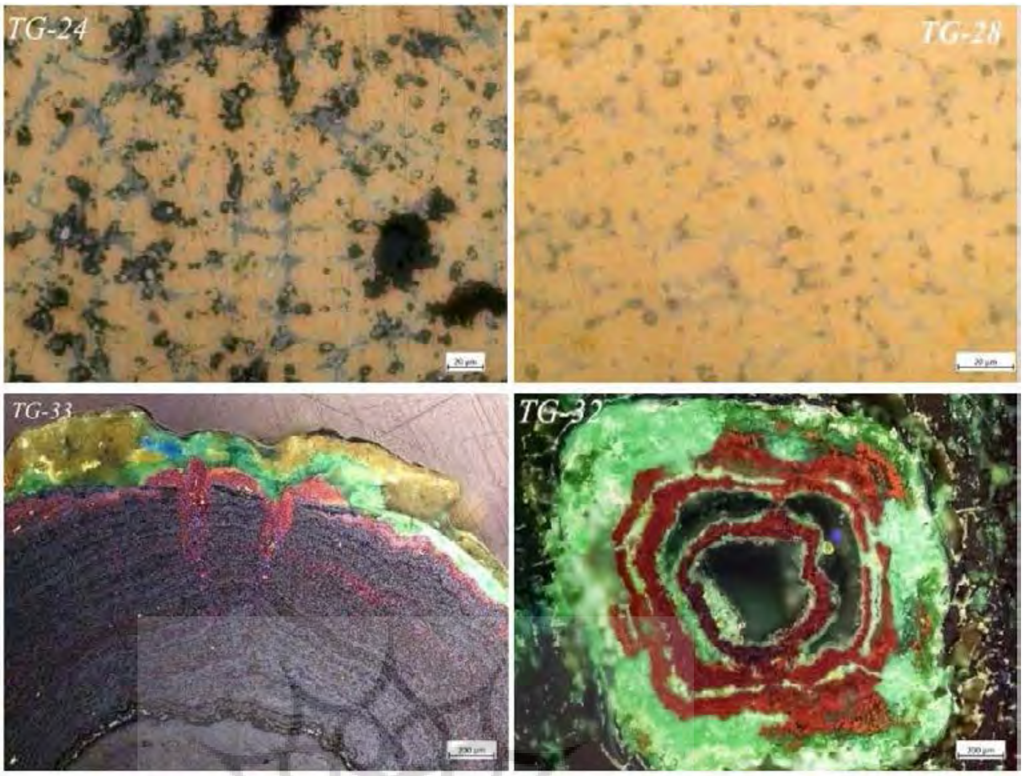

Fig. 3: Dendritic microstructure in BFL indicative of the casting process of TG-24 and TG-28 objects. Also, the predominantly mineralized structure of TG-32 and TG-33 in PL and scattered metallic regions in their microstructure (Authors, 2024).

the EDS results for sample TG-24 were reported from 9 points. Point 1 analysis shows the elemental composition of the TG-24 matrix. The dendritic phase contains over 95% copper, and interestingly, a relatively higher lead content is present in the matrix, considering lead's low solubility compared to arsenic. In image TG-24a, asignificant intermixing of bright white phases with dendrites is evident. The percentage of arsenic (1.44%) in dendrites is lower than in bulk analysis (5.86%). This difference may be related to arsenic acting as an alloying element in the formation of the α + γ (alpha + gamma) phase. According to the Cu-As phase diagram, the α + γ phase begins to form at approximately 77.6% and around 685°C (Fig. 5). Typically, in eutectic phases, the α component of the eutectic may sometimes separate and join the α dendritic phase

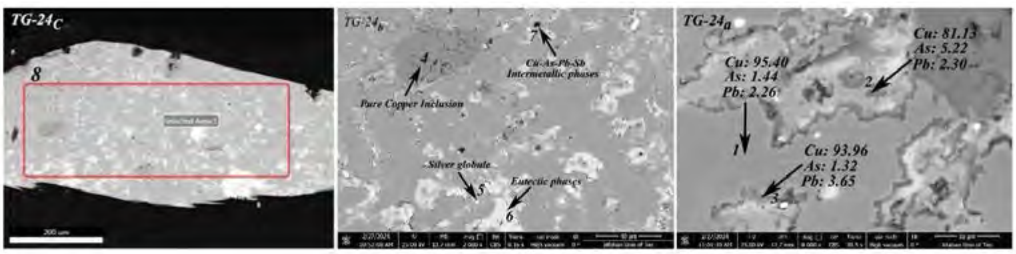

Fig. 4: EDS analysis points of sample TG-24. TG-24a: Composition of dendrites, intermetallic phase and marginal phase around dendrites. TG-24b: Dark inclusions, bright white phases and bright white globules. TG-24c: Selected bulk region (Authors, 2024).

(Scott, 1991). Additionally, point 2 represents an intermetallic accumulative phase with a composition of copper (81.13%), arsenic (5.22%), lead (2.30%), antimony (1.67%), and sulfur (8.86%). Similar compositions are observed at other points within the microstructure. What is seen at point 3 resembles the observations described above. As previously mentioned, the γ-phase growth in the microstructure of the object appears to be associated with the light gray regions, which could be more precisely evaluated through detailed metallographic studies. Point 4 shows an almost pure copper piece in the microstructure, which is not combined with other alloying elements. In the BSE images of TG-24, identifying the typical α-phase dendrite shapes in the microstructure is difficult. Predominantly oval globules, such as those at point 5, contain over 84% silver. At this point, antimony is reported at 4.40%, and the elements present are silver, copper, antimony, and lead. Point 6 analysis indicates a composition with over 51% lead, 31% copper, and more than 16% arsenic.

Table 1. EDS results of metallic core regions in sample TG-24 (Authors, 2024).

Sample	Area (%)	Cu (%)	As (%)	Pb (%)	Bi (%)	Sb (%)	Ag (%)	Fe (%)	S (%)	Sn (%)
1	04.95	44.1	26.2	0.50	0.12	0.17	0.28	0.20	-	-
2	13.81	22.5	30.2	0.42	0.67	0.10	0.18	8.68	12.0	-
3	96.93	32.1	65.3	0.43	0.21	0.04	0.22	0.14	0.02	-
4	40.99	-	0.02	0.09	0.07	-	0.20	0.18	0.04	-
5	54.6	66.0	1.96	0.42	4.40	20.84	0.29	0.38	1.16	-
6	09.31	16.26	51.18	0.31	0.70	-	0.44	-	-	-
7	56.73	12.58	9.38	0.33	2.47	0.66	0.58	0.33	0.10	-
8 (Bulk)	84.86	86.5	4.35	0.12	0.90	1.48	0.13	0.32	-	-

Contrary to common assumptions, the appearance of dark-colored phases in the microstructure in BSE images indicates the presence of light elements such as sulfur. At point 7, which also exhibits a circular morphology, an intermetallic phase composed of Cu–As–Pb–Sb was identified. Point 8 represents the bulk analysis of the object as shown in image TG-24C. Accordingly, the chemical composition of TG-24 consists of major elements including copper, arsenic, lead, silver, and antimony, as well as minor elements such as sulfur, iron, bismuth, and tin. The presence of these minor elements in the alloy composition can likely be related to the use of ore sources during the smelting and extraction process. Although a definitive conclusion on this matter would require more precise quantitative analyses, object TG-28 exhibits a BSE microstructure similar to that of TG-24. The EDS results for this sample are reported in Table 2. As shown in image TG-24a, selected area 1 represents the bulk region of the sample (Fig. 4). The major elements identified in this area include copper (88.27%), antimony (7.37%), silver (1.16%), and lead (1.47%), while other elements were detected at concentrations below 1%. In image TG-24b, major elements at point 2, corresponding to the α-phase in the microstructure, consist of copper (96.85%) and antimony (1.47%), with all other elements below 1%. Point 3 represents the matrix phase, which is also observable in regions between adjacent dendrites.

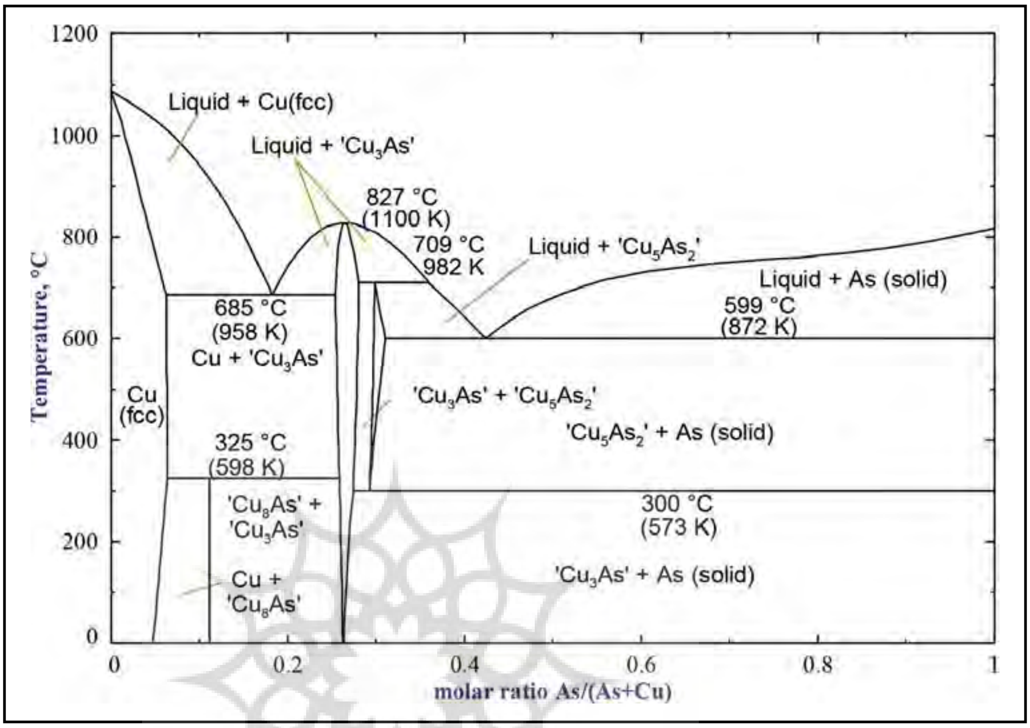

Fig. 5: Cu-As phase equilibrium diagram showing the formation of beta phase along with alpha phase of copper from 685 to 325 °C. (Shishin *et al.*, 2019).

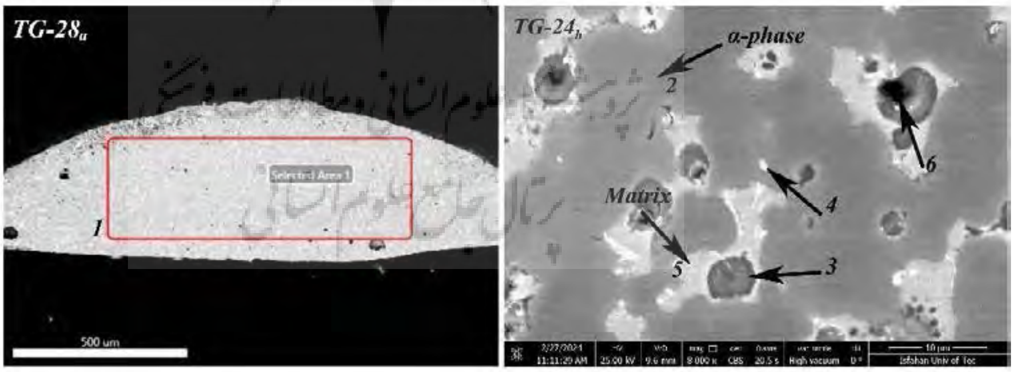

Fig. 6: EDS analysis of the metal core areas of sample TG-28 (Authors, 2024).

The major elements at these points include copper (80.57–84.02%), antimony (2.75–4.88%), sulfur (~11%), and lead (~2%). These observations suggest the presence of copper–antimony sulfide phases, likely including Cu_3SbS_4, $CuSbS_2$, Cu_3SbS_3, $Cu_{12}Sb_4S_{13}$, and $Cu_{14}Sb_4S_{13}$. In the BSE image, point 4 corresponds to a bright white globule. The elemental composition at this point. The main elements at this point are copper, antimony, silver, lead, and arsenic. Among all the analyzed points in TG-28,

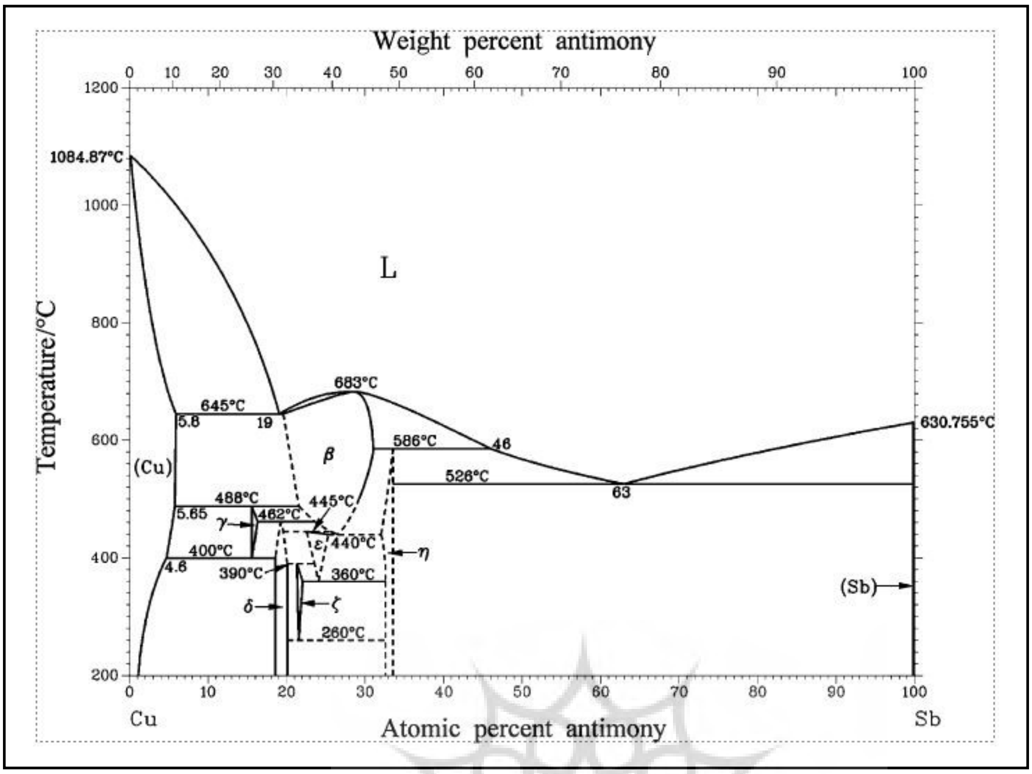

Fig. 7: Cu-Sb phase equilibrium diagram with beta phase formation between 5.8 and 19% from 645 to 488 °C. (Fürtauer & Flandorfer, 2012).

arsenic exceeds 1% only at this location. Point 5 corresponds to the matrix phase of the microstructure. The antimony content (19.91%) represents the highest concentration among all analyzed microstructural points. According to phase diagrams .The copper–antimony content falls within the α + γ phase range according to the phase diagrams. Point 6 appears to be an intermetallic phase composed of copper, antimony, sulfur, and lead. Given the dark color of this phase, it may include copper or lead–antimony sulfide compounds. Intermetallic phases such as Cu_2Sb, ternary Cu–Sb–Pb, or quaternary Cu–Sb–Pb–S compounds can also be inferred. Sample TG-32 is a rod-shaped object, with the distal end slightly reduced in thickness, and possessing an approximately square cross-section. As observed in images TG-32a and TG-32b of its cross-section, it was likely formed by rolling a copper sheet around its longitudinal axis (Fig. 8). This is evident from its layered circular structure, making the presence of a hollow center plausible, which may have later been filled with corrosion products .EDS analysis of TG-32 was conducted on the differences between dark gray and light gray phases, sampled from the outermost layers. The results of the EDS analyses are reported element-wise in Table 3.

The bulk EDS results of this sample can be categorized into several groups: the first includes copper, zinc, lead, and nickel; the second comprises iron and sulfur, likely originating from the ores used in the extraction process; the third consists of elements associated with corrosion processes, including oxygen and chlorine; and the fourth

Table 2: EDS analysis of the metal core areas of sample (TG-28 (Authors, 2024).

Sample	Area (%)	Cu (%)	As (%)	Pb (%)	Bi (%)	Sb (%)	Ag (%)	Fe (%)	S (%)	Sn (%)
1 (Bulk)	27.88	0.68	1.47	0.04	7.37	1.16	0.16	0.72	0.13	-
2	96.85	0.15	0.45	0.12	1.47	0.37	0.19	0.31	0.10	-
3	80.57	0.59	1.97	0.11	4.88	0.30	0.19	11.02	0.37	-
4	87.99	1.01	2.19	0.09	5.14	2.98	0.16	0.29	0.15	-
5	71.62	0.92	1.61	0.04	19.91	0.98	0.16	4.33	0.44	-
6	82.43	0.77	2.51	0.08	4.69	0.22	0.28	8.70	0.33	-

includes calcium, silicon, and magnesium, which may relate to long-term interaction with burial soil.

Table 3: EDS test results of TG-32 sample, weight percentage of elements at different points (Authors, 2024).

Sample	Area (%)	Cu (%)	Zn (%)	Pb (%)	Fe (%)	Ni (%)	S (%)	O (%)	Cl (%)	Ca (%)	Si (%)	Mg (%)	C (%)
1 (Bulk)	50.68	1.03	2.84	0.65	0.53	2.77	7.98	6.28	1.01	4.95	3.45	-	-
2	93.79	0.90	0.49	0.52	0.43	13.09	0.66	0.02	0.16	1.72	2.11	-	-
3	18.86	1.00	2.45	0.52	0.52	1.29	0.96	0.40	0.33	1.87	2.49	-	-
4	40.83	-	-	-	-	5.16	-	0.03	-	2.67	-	8.75	-

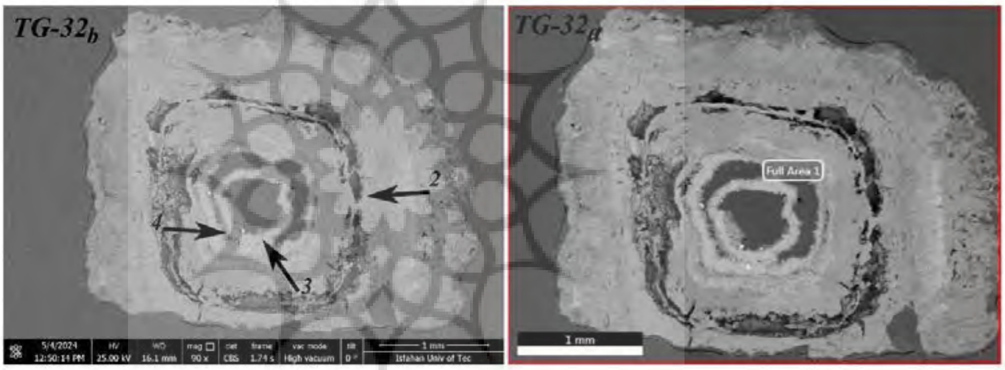

Fig. 8: EDS examination of bulk and selected areas of sample TG-32 (Authors, 2024).

Point 1 represents the bulk analysis of the sample. The bulk EDS analysis identifies lead (2.84%) as a significant constituent of the alloy composition. Given the multi-step and complex shaping process of this object, lead was likely added to improve casting and working processes. The presence of oxygen and chlorine confirms the penetration of corrosion into the object. Furthermore, due to the advanced corrosion observed, the detection of soil-derived elements in the cross-section is not unexpected. The formation of copper sulfide mineral phases at points 2 and 4, indicated by increased sulfur content, is predictable. The phases in this area could possibly be chalcocite. (Cu_2S) and covellite (CuS), which could have been present as effective minerals in the smelting and extraction processes of the ores used. Interestingly, carbon (8.75%) was detected only at this point among all analyzed locations, possibly originating from residual polishing paste penetrating the object's structure during finishing processes. Point 3 consists of copper, lead, zinc, and sulfur. Sample TG-33 is a rod-shaped object with a circular cross-section. Five EDS points were analyzed on the cross-section of TG-33. Most of the object has mineralized, with metallic remnants remaining only in very limited areas. The results of these analyses are reported in Table 5, and the analyzed points are shown in Fig. 9. These points include bulk, light and dark gray phases, and bright white globules. The identified elements can be classified into four categories: copper, zinc, and tin as metallic elements; iron and sulfur as elements originating from the ores used in the molten material extraction process; silicon and magnesium as soil-derived elements resulting from long-term burial, which is consistent with the mineralization of the object; and oxygen as the only element related to corrosion processes. EDS analysis of TG-33 identified a copper–tin alloy. In the bulk region, 1% zinc was detected, which does not appear to have been added intentionally for alloying or to enhance the technical properties of the object (Fig. 9). Further interpretation and discussion regarding this will require complementary analyses. Regarding sulfur and iron, it can be hypothesized that either a combination of sulfide- and iron-containing ores or ores with higher sulfur content were exploited. Another possibility is that the extraction and refining methods employed by the ancient metallurgist were more effective in removing iron than sulfur as impurities from the ore. As observed in the table, across all points, the sulfur content was detected in higher amounts than iron, although, considering the intrinsic properties of iron such as its atomic number 26 and density of 7.874 g/cm³ it would generally be expected to be more readily detected than sulfur (atomic number 16, density 2.07 g/cm³).

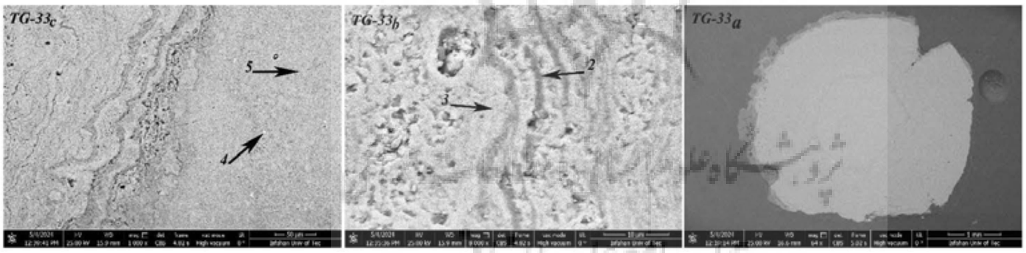

Fig. 9: EDS analysis of bulk and selected points and regions of sample TG-33 (Authors, 2024).

Table 4: EDS results of TG-33 microstructural regions (weight % of elements), (Authors, 2024).

TG-33 Point	Cu (%)	Sn (%)	Zn (%)	Fe (%)	S (%)	Si (%)	Mg (%)	O (%)
1 (Bulk)	16.72	9.38	1.00	0.61	8.45	3.08	2.08	3.23
2	50.65	19.32	1.32	0.90	8.42	1.58	1.27	1.70
3	31.50	31.01	1.03	0.88	8.77	1.94	2.02	4.05
4	17.46	26.19	2.42	0.68	16.81	2.93	2.16	2.64
5	7.65	18.37	1.16	1.05	7.96	1.74	1.86	2.78

Metallography

Metallographic studies were conducted with the aim of revealing the microstructure of the artifacts. It should also be noted that the metallic core in TG-32 and TG-33 was insufficient to allow direct metallographic analysis. Therefore, the cross-sections of TG-24 and TG-28, which contained a metallic core, were subjected to metallography. The samples were prepared, polished, and etched using a ferric chloride ($FeCl_3$) solution. Images of the cross-sections were captured at 50× magnification to better reveal comparative aspects of microstructural dimensions among the samples. As shown in Fig. 10, TG-24 and TG-28 exhibit well-defined microstructures. In TG-24, the matrix phases appear gray, interspersed between dendrites that have reddish-brown central regions with margins ranging from light orange to light brown.

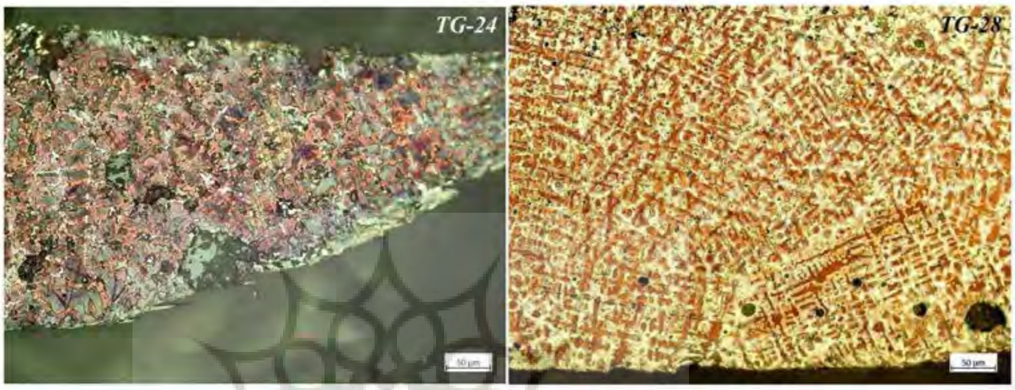

Fig. 10: Metallography of samples TG-24 and TG-28 shows dendritic microstructures indicative of casting (Authors, 2024).

In certain areas, bright white phases attached to the dendrites are visible in contrast to the gray matrix. This appears to be an intermetallic phase that has separated from the matrix. In some gray matrix phases, narrow light and dark bands can be observed in the microstructure. Such bands in the microstructure of ancient copper alloys are recognized as eutectic fillers between α-phase grains or dendrites. The condition of the dendrites and the microstructure shows no evidence of additional work carried out to shape or further manipulate TG-24. In TG-28, the matrix appears very pale white-yellow, while the dendrites are copper–brown. The margins of the dendrites are distinguishable in light brown. This feature usually results from a compositional concentration gradient of the constituent elements during the growth of α-dendrites. The elongated and regular shape of the dendrites suggests that casting was the sole method of production for TG-28, with no further shaping operations applied. The presence of some black voids in the images indicates gas evolution generated by the molten metal during casting, which has been preserved in the solidified structure.

X-ray Fluorescence Spectroscopy (XRF)

The results of this analysis are presented in Table 5. Based on the findings, all objects contain copper alloys, with nickel and lead present as consistent minor elements. The highest lead contents were found in TG-24 and TG-28 (1.75% and 2.84%, respectively), while the nickel content in all objects remained below 1%. It appears that nickel and lead can be considered trace elements useful for identifying the source of the ores used (Shanks, 2013). Zinc was detected only in TG-24 and TG-28, with amounts of 0.33–0.47%, and silver in TG-24 and TG-28 was 2.58% and 3.65%, respectively. In TG-33, silver was present at 0.67%. Arsenic was detected in TG-24 and TG-28 at 6.61% and 2.68%, respectively, and a significant amount of antimony (11.82%) was found in TG-28. These results suggest that alloying elements deliberately added by the ancient metallurgist into the molten metal include tin (TG-33), antimony (TG-28), arsenic (TG-24, TG-28), and silver (TG-24, TG-28). Furthermore, lead, nickel, iron, zinc, and tin in TG-24 and TG-28 can be considered residual elements from the ores used in the production process. Accordingly, TG-24 can be classified as an arsenical copper alloy, while TG-28 can be considered an antimonial–arsenical copper alloy. The addition of lead in these two objects likely served to improve casting properties. The relatively high silver content may be interpreted as an unintentional impurity derived from the ore. Among the studied objects, TG-33 is a bronze alloy. Its high tin content indicates deliberate addition to the molten metal. Finally, TG-32 can be considered almost pure copper. Given the manufacturing technique, which appears to involve rolling followed by rotation around the longitudinal axis, it is reasonable to infer that the ancient metallurgist exploited the superior ductility of pure copper compared to copper alloys.

Table 5: Micro-XRF quantitative analysis of the chemical composition of the studied objects (Authors, 2024).

Total (%)	Zn (%)	Ag (%)	Ni (%)	Fe (%)	Pb (%)	As (%)	Sb (%)	Sn (%)	Cu (%)	Sample No.
96.99	0.33	2.58	0.07	0.17	1.75	6.61	0.92	0.39	87.31	TG-24
96.99	0.47	3.65	0.06	0.08	2.84	2.68	11.82	0.13	78.31	TG-28
98.01	—	—	0.10	0.18	0.09	—	—	—	97.01	TG-32
96.18	—	0.67	0.09	0.26	0.09	—	—	20.53	74.53	TG-33

Notes: "—" indicates that the element was below the detection limit. Total (%) represents the sum of detected elements in each sample.

Discussion

As observed in the Cu–As phase equilibrium diagram (Fig. 3), (Shishin, Chen, Hidayat, & Jak, 2019), the α + γ phase begins to form at 83.6 wt.% arsenic at 685 °C and continues until 5.6 wt.% arsenic at 325 °C. Based on the copper and arsenic contents determined by Micro-XRF, sample TG-24 can be classified as a copper–arsenic alloy. The use of copper–arsenic alloys (19.4 wt.% As) in Iron Age Sagzabad artifacts has also been documented in previous studies (Mortazavi *et al.,* 2011). According to Micro-XRF analysis, TG-24 contains silver (2.58 wt.%) and lead (1.75 wt.%). Some studies have reported a direct correlation between arsenic content and the presence of antimony and silver (Key, 1964). Metallographic analysis revealed α dendrites in reddish-brown color and gray α + γ matrix. The metallized core shows no evidence of additional mechanical shaping, indicating that the object was primarily cast. EDS analysis identified α dendrites composed of 95 wt.% copper, 1.44 wt.% arsenic, and 2.26 wt.% lead. eutectic patterns, visible as alternating dark and light bands in the matrix (Fig. 8), represent α + γ phases in the inter-dendritic regions, a common feature in ancient copper alloys (Scott & Schwab, 2019). The γ phase corresponds to Cu_3As according to recent thermodynamic modeling of Cu–As systems (Shishin *et al.,* 2019). Bright white micro-spheres observed in BSE images (point 5) were identified as silver inclusions (20.84 wt.%) via EDS (Oudbashi *et al.,* 2019). Points 6 and 8 were characterized as Cu–Pb–As intermetallic compounds (Fig. 2, Table 1), while dark inclusions correspond to multicomponent Cu–As–Pb–Sb phases, consistent with previous studies on multiphase inclusions in copper alloys (Garbacz-Klempka, *et al.,* 2015). According to the Cu–Sb phase diagram (Fig. 7), the β phase begins forming at 645 °C with 11 wt.% antimony and continues to form α + β mixtures down to 488 °C (Scott, 1991). Based on Micro-XRF, TG-28 is a Cu–Sb–As alloy containing lead (2.84 wt.%) and silver (3.65 wt.%). Some sources classify alloys with less than 2 wt.% lead as incidental lead-rich alloys (Begemann *et al.,* 2008). EDS analysis of TG-28 bulk identified copper (27.88 wt.%), antimony (37.7 wt.%), lead (47.1 wt.%), silver (16.1 wt.%), with other elements below 1 wt.%. Metallography revealed straight, elongated α dendrites of copper-brown color within a pale yellow-white (α + β) matrix, with no evidence of mechanical or thermal post-processing. The object appears to have been produced by double-mold casting, as indicated by the mold line on its surface. EDS of the α phase within core dendrites showed 96.85 wt.% Cu and 1.47 wt.% Sb, with other elements below 1 wt.%. The α + β eutectic filler in inter-dendritic regions contains 62.71 wt.% Cu and 91.19 wt.% Sb. The β phase corresponds to Cu_3Sb in the equilibrium phase diagram (Fürtauer & Flandorfer, 2012). Bright white micro-spheres within the matrix may represent intermetallic compounds rather than lead or silver inclusions (Fig. 4, Table 2). Points 3 and 11 within the eutectic matrix appear to have formed under the influence of α phase and the surrounding eutectic, potentially forming intermetallics such as Cu–Pb–Sb–S or Cu–Sb–S, which may also produce two-phase compounds like Cu_4Sb and Cu_2S, contributing to alloy hardness. Generally, copper–arsenic alloys are more common than copper–antimony alloys. Copper–antimony alloys form a variety of intermetallic compounds characterized by phase separation and refinement (Scott & Schwab, 2019). The deliberate use of antimony and arsenic may have compensated for limited tin availability in the Sagzabad region, as seen in neighboring areas such as the Caucasus (Pike, 2002). Sample TG-32 is a small rod-like object with potential functional use. Radiographic imaging revealed alternating dark and light layers. Cross-sectional sampling confirmed a spiral internal structure. The majority of the object is mineralized, with corrosion products in red and green visible throughout the cross-section (Fig. 2). Micro-XRF of the remaining metallic core indicates almost pure copper with minor lead (0.09 wt.%) and negligible nickel (Giumlia-Mair *et al.*, 2002). EDS analysis of bulk regions confirmed the presence of copper, lead, and ~1 wt.% zinc, consistent with Micro-XRF results (Table 5). The chosen composition aligns with the manufacturing technique, allowing ease of shaping from nearly pure copper (Safaei-Ghalati *et al.*, 2018). BSE images showed no sulfide or iron inclusions within TG-32.Sample TG-33 is a rod-like object likely intended for functional use. Radiography revealed a compact structure. Contrary to expectations based on X-ray brightness, TG-33 lacks a fully preserved metallic core. Cross-sectional analysis shows predominantly mineralized structure, with only small residual metallic areas used for Micro-XRF analysis. TG-33 was identified as a copper–tin alloy (Tămaș & Andrii, 2020), with minor silver, iron, nickel, and lead (<1 wt.%), (Table 5). EDS of TG-33 bulk confirmed the presence of copper, tin, and zinc (Table 4), corroborating the Micro-XRF results (Valério *et al.*, 2012). Iron and sulfur were also detected, likely originating from sulfide-rich copper ores (chalcopyrite and bornite) used in the smelting process (Bakhshandehfard, 2010).

Conclusion

The present study, focusing on the chemical composition, microstructure, and manufacturing techniques of metal objects recovered from Iron Age graves at the periphery of Tepe Qabrestan, represents a significant step toward expanding our understanding of ancient metallurgy in the Qazvin Plain. Unlike previous studies, which primarily offered general identifications of metals and cursory references to arsenical copper and copper-antimony alloys, this research employs precise analytical data and a techno-typological approach to reveal new dimensions of metallurgical diversity and object functionality in the Iron Age. Analytical results indicate that metalworkers of this period were adept in advanced casting techniques and the deliberate use of alloys to produce decorative items, jewelry, and utilitarian tools, demonstrating a profound understanding of the physical and aesthetic properties of metals. The use of arsenical copper and antimonial copper alloys with varying concentrations, the presence of dendritic, eutectic phases, and dispersed silver inclusions in metallographic sections, along with the identification of minerals such as chalcopyrite and bornite, reflect the high level of technical skill and resource knowledge among these ancient metallurgists. Microstructural analysis further reveals that, contrary to expectations, some objects show no evidence of secondary mechanical or thermal working, suggesting the use of simpler or function-specific manufacturing strategies in certain cases. Additionally, the substitution of tin with other alloying elements highlights the adaptability of metalworkers when faced with resource limitations. Overall, this study goes beyond mere compositional identification, integrating analytical data with archaeological insights such as functional classification, raw material sourcing, and indigenous knowledge to provide a novel perspective on Iron Age metalworking in the Qazvin Plain. The results contribute to a deeper understanding of the complexity of ancient metallurgical practices and cultural production mechanisms, offering a foundation for future research in archaeometallurgy and technological archaeology.

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This article is extracted from the research project conducted by first writer and second writer in Art university of Isfahan. Accordingly, The content is collected by the first and second writer and manuscipt is prepared under supervision and guidance of the first Writer.

Conflict of Interest
In adherence to ethical publication standards, the authors affirm that there are no conflicts of interest, either personal or financial, that could have influenced the content or conclusions presented in this research.

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چکیده

استقرارهای دشت قزوین در تپهٔ قبرستان و سگزآباد توالی شکوفایی در فلزگری پیش‌ازتاریخ دشت قزوین را نشان می‌دهند. هدف این مطالعه، بررسی جنبه‌های کاربردی، فناوری ساخت، ترکیب شیمیایی، ریزساختار و فازهای آلیاژی اشیاء فلزی به‌دست‌آمده از عصر آهن محوطهٔ سگزآباد است. در این راستا، تلاش شده است به این پرسش‌ها پاسخ داده شود که فرآیند تولید و روش ساخت این اشیاء چگونه بوده، از چه شیوه‌هایی برای آلیاژسازی در آن‌ها بهره گرفته شده و محصولات خوردگی تشکیل‌شده چه ترکیباتی داشته و تحت چه شرایطی به‌وجود آمده‌اند. به‌منظور دست‌یابی به این اهداف، از روش‌های آنالیز دستگاهی ازجمله میکروفلورسانس پرتو ایکس (XRF) برای تعیین ترکیب شیمیایی استفاده شد. نتایج این آنالیزها وجود آلیاژهای متنوعی نظیر مس-ارسنیک، مس-آنتیموان، مفرغ و نیز مس تقریباً خالص را نشان‌دادند. در دو نمونه دارای مغز فلزی، به کمک متالوگرافی، ریزساختار ریخته‌گری با مغزه‌دار مشخص شد که اطلاعات ارزشمندی دربارهٔ شیوه‌های ساخت در اختیار قرار داد؛ هم‌چنین، بررسی‌های انجام‌شده با میکروسکوپ الکترونی روبشی مجهز به طیف‌سنجی پراکندگی انرژی پرتو ایکس (SEM-EDS)، به شناسایی دندریت‌های فاز آلفا و نیز تشکیل فازهای گامای ارسنیکی و آنتیموانی مس منجر شد. نتایج آزمون‌های عنصری برروی این نمونه‌ها، بیانگر الگوهای متنوعی از کاربرد آلیاژهای مس-ارسنیک و مس-آنتیموان است؛ امری که نشان‌دهندهٔ آگاهی فلزگران باستانی از ایجاد خواص مکانیکی مطلوب در فرآورده‌های فلزی بوده است؛ علاوه‌بر این، شناسایی آخال‌های نقره‌ای و ترکیبات بین‌فلزی سولفیدی در ریزساختار، امکان ارائه فرضیاتی دربارهٔ نوع سنگ‌های معدنی مورداستفاده در فرآیند استحصال فلز را فراهم کرده است. فناوری ساخت اشیاء از ریخته‌گری و قالب‌گیری دو کفه‌ای تا شکل‌دهی اسکنه‌هایی با مقطع مارپیچ و ساده است. اشیاء پژوهش در طبقه‌بندی اشیاء تزئینی، زیورآلات و اشیاء کاربردی قرار می‌گیرند. این گونه‌شناختی مختلف در کنار تفاوت ترانشه‌های کاوش‌شده طرح کلی خوبی از تنوع فلزگری در عصر آهن تپهٔ سگزآباد را در اختیار می‌گذارد.

کلیدواژگان: مس ارسنیکی، مس آنتیموانی، مفرغ، عصر آهن، سگزآباد، فلزگری باستانی.

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