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Original Research Article

Laboratory Studies on the Alloy Composition and Microstructural Features of Metal Objects from the Taj Amir (Dehnow) Cemetery, Yasuj, Dating to the Second Millennium BCE*

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Abstract

The study and understanding of ancient technologies used for producing and utilizing metals in various objects across different regions of Iran are of great importance. With the invention of copper-based alloys, such as tin bronze and arsenical bronze, ancient craftsmen achieved a remarkable level of technical knowledge and metallurgical skill. The archaeological site of Taj Amir Cemetery is located on the southern slopes of the Dena mountain range, in the eastern part of Yasuj (Kohgiluyeh and Boyer-Ahmad Province, southwestern Iran). This cemetery was identified in 2009 CE during construction activities related to the new library of Yasuj University of Medical Sciences. Excavations at the site yielded a collection of metal artifacts, particularly bronze objects, from burial contexts. In the present study, a selection of the better-preserved bronze artifacts—chosen for their diversity in form and function and belonging to contemporaneous burial layers of the Taj Amir (Dehnow) cemetery were subjected to laboratory analyses, including metallography, X-ray fluorescence (XRF), and scanning electron microscopy equipped with energy-dispersive X-ray spectroscopy (SEM-EDS). The objective was to obtain a clearer understanding of alloying technology and manufacturing techniques during the mid-second millennium BCE. For this purpose, microstructural analysis, scanning electron microscopy with EDS, and XRF elemental analysis were employed to determine the compositional and structural characteristics of the samples. The results revealed that two of the analyzed specimens were made of tin bronze alloys with relatively consistent tin contents, indicating that the ancient metallurgists had considerable knowledge of tin control in the bronze-making process. Moreover, one sample exhibited a high silver content, confirming its silvery appearance. Structural evidence suggests that these objects were produced by smelting, casting, and subsequent hammering, involving repeated cycles of cold working and annealing during the shaping process.

Keywords: *Taj Amir Cemetery, Bronze, Microstructural Analysis, XRF, SEM-EDS.*

Introduction

Metallurgy has always been regarded as one of the primary indicators of human development and cultural advancement and is considered a major milestone in the evolution of civilizations from ancient to modern times. The use of metals not only signifies technological progress and the

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** Corresponding Author: hr.bakhshan@aui.ac.ir, 0989133089406 specialization of human activities but also represents a fundamental factor in the transformation of industry and social life. The utilization of metals in the production of tools and implements was among the most significant accelerators of progress in prehistoric societies. Over the past decades, Iran has attracted considerable scholarly attention as one of the principal centers of metalworking in the ancient world. Numerous field and laboratory studies have been conducted on metallurgical remains unearthed from archaeological excavations across the Iranian Plateau (Oudbashi et al., 2012, 55–74). These investigations have included the examination of metal artifacts, molds, slags, and tools associated with metalworking processes, providing evidence for the remarkable technological achievements of ancient Iranian metallurgists. The Taj Amir Cemetery is located approximately 200 meters from the Bashar River, on a slope with an inclination of about 30 degrees, in the eastern part of Yasuj, southwestern Iran. The site was discovered in the autumn of 2009 CE during earthmoving operations for the construction of the library of Yasuj University of Medical Sciences (Rahimi & Vahdati, 2016, 101–120). According to the preliminary excavation reports, more than 7 hectares of the site were destroyed, and only about 3 hectares have remained intact (Figs. 1 & 2). During the first excavation season, 16 closely spaced graves were investigated, and geophysical studies helped delineate the approximate extent of the cemetery at around 2 hectares. The excavations revealed notable structural features such as stone-lined graves, large stones placed at the southern ends of graves as symbolic markers, and the construction of entrances to burial chambers. Grave goods, including pottery vessels, bronze artifacts, and stone tools, were discovered alongside the human remains. Architectural and archaeological evidence suggest that the cemetery was used by semi-nomadic populations and dates to around 2000 BCE. In 2011 CE, the site was officially registered on the National Heritage List of Iran under the name Dehnow Cemetery, derived from the nearby village of the same name (Ghezelbash et al., 2016, 171–190).

The high density of graves within a limited area indicates that the cemetery covered a relatively extensive and intensively used burial zone. Significance of the Site The scientific and historical value of the Dehnow Cemetery lies in the fact that it preserves vital information about the life, technology, and belief systems of the populations inhabiting the Boyer-Ahmad region approximately 3,500 years ago. Based on archaeological evidence obtained through systematic excavations, the Taj Amir Cemetery can be attributed to Elamite-related nomadic or semi-nomadic communities active during the first half of the second millennium BCE.

Fig. 1. Location of the Taj Amir Cemetery within the campus of Yasuj University of Medical Sciences, satellite view showing the cemetery adjacent to the university mosque. Source: https://www.google.com/maps/search

Fig. 2. Excavation trenches and test pits. Source: Ghezelbash et al., 2016, 177–178.

In this study, a selection of well-preserved and morphologically diverse bronze artifacts from contemporaneous burial layers of the Taj Amir (Dehnow) Cemetery in Yasuj were subjected to laboratory analyses. The aim was to identify technological characteristics and manufacturing techniques from the mid-second millennium BCE. The analytical procedures included elemental composition analysis by X-ray fluorescence (XRF), microstructural and microscopic examination using scanning electron microscopy with energy-dispersive spectroscopy (SEM-EDS), and optical metallography. Each of these methods provided complementary data regarding alloy composition, production stages, and possible thermal treatments applied during manufacturing.

The primary objective of this research is to reconstruct aspects of ancient metallurgical technology in southern Iran and to assess the extent of technological awareness among ancient metalworkers in controlling the composition and mechanical properties of copper-based alloys. The central research questions can be summarized as follows:

1) What is the chemical composition and elemental ratio of the alloying components in the metal artifacts from Taj Amir Cemetery?

2) Can differences in manufacturing techniques or levels of technical proficiency be identified based on microstructural and compositional characteristics?

3) What clues do these data provide regarding the provenance of raw materials and the level of metallurgical knowledge in the mid-second millennium BCE?

The results of these analyses are expected to offer a clearer understanding of alloying practices, manufacturing sequences, and possibly the existence of local workshops or raw material exchange networks in the Yasuj region. Thus, the present study not only serves as a case analysis of a specific group of bronze artifacts but also contributes significantly to reconstructing the technological patterns of metalworking across the Iranian Plateau during the mid-second millennium BCE.

Site Description

The Taj Amir Cemetery, located near the village of Dehnow and approximately 200 meters from the Bashar River, lies on a gently sloping, forested hillside with an inclination of about 30 degrees. This natural setting has resulted in minimal sediment accumulation over the burials, leaving most graves relatively close to the surface (Ghezelbash et al., 2016, 177–178).

In 2012 CE, a new phase of archaeological excavations was conducted, leading to the discovery of a wide range of metal, ceramic, and stone artifacts. During this season, sixteen graves were excavated and documented in the northern part of the site. Geophysical surveys, covering nearly two hectares, were also carried out using a cesium–rubidium gradiometer. The resulting magnetic data revealed that the cemetery was extensive and densely packed with graves. Among the sixteen investigated graves, only five were found intact and in situ, while the rest had likely been looted or deliberately disturbed in antiquity. Sediment infiltration and the collapse of grave coverings had also contributed to the destruction of pottery and skeletal remains.

Despite these damages, the excavations yielded 156 artifacts, including three complete vessels, seventeen fragmentary ones, over 1,885 pottery sherds, and a valuable collection of stone and metal beads. Particularly noteworthy are the discoveries of 58 stone beads, 19 silver beads, and one bronze bead, mostly from grave no. 8 and jar burial no. 16, indicating the symbolic and decorative significance of personal adornments in the funerary practices of this community. Specific grave goods such as ten stone arrowheads from grave no. 4, a bronze dagger, and a painted pottery flask reflect both the functional diversity and the ritual symbolism of the objects interred with the deceased.

The considerable variety and quantity of pottery and metal artifacts suggest complex funerary customs and the elevated social status of the deceased within their community. The presence of metal items such as spearheads, bracelets, rings, and daggers indicates a well-developed mastery of metallurgical techniques and access to bronze and silver resources. Furthermore, the discovery of silver jewelry points to long-distance trade connections, as silver sources were limited locally and likely obtained through interregional exchange networks.

Examination of artifacts previously recovered during the 2009 construction-related disturbances—now stored in the Yasuj Museum—revealed an additional assemblage comprising thirteen bronze vessels, a carved steatite vessel, and several metal and ceramic fragments. Comparison between these finds and those from official excavations provides a more comprehensive understanding of the functional and typological diversity of the burial assemblages.

Overall, the data obtained from the Taj Amir Cemetery are of great importance for studying the social and cultural organization of ancient communities in southwestern Iran. The concentration of numerous metal artifacts among the grave goods underscores the economic and symbolic significance of metal in the lives of these people. Moreover, the available evidence indicates that this region was part of broader Elamite cultural and economic networks involving mobile pastoral groups during the first half of the second millennium BCE.

Therefore, the Taj Amir Cemetery should be regarded not merely as a burial site but as a key archaeological resource for understanding social interaction, economic exchange, and technological development in the central Zagros and southwestern Iran. The laboratory analyses of selected metal artifacts from this site provide valuable insights into ancient metallurgical technology, alloy composition, and manufacturing processes, offering a solid foundation for future archaeometallurgical research in the region.

Research Background

Elemental and microstructural analyses of metal artifacts across the Iranian Plateau, particularly within the Central Zagros and southwestern regions, have revealed significant technological progress in metalworking from the second millennium BCE. These studies have primarily focused on identifying alloy compositions, production techniques, and manufacturing processes of metal artifacts, thereby offering crucial insights into ancient mineral resources and the cultural and economic exchange networks of that period. Numerous investigations have been carried out in related sites throughout this region.

A focused archaeological and analytical study at this specific site was conducted by Ghezelbash et al. (2016). They reported that the excavation and documentation of sixteen graves in the northern section of the Taj Amir Cemetery and the geophysical survey covering two hectares constituted the main results of the first season of research. Their work also involved detailed descriptions of structural components of the graves, including the cobblestone coverings, stone-lined burial pits, grave entrances, and associated forecourts. The funerary assemblages consisted of pottery vessels, metal and stone objects, lithic tools, ornaments, and bronze and stone weapons. Based on the archaeological evidence, the authors concluded that the Taj Amir Cemetery likely belonged to an Elamite nomadic or semi-nomadic community active during the first half of the second millennium BCE.

Despite the importance of these archaeological findings, previous research has primarily concentrated on typological classification and descriptive analysis, while laboratory-based investigations such as detailed microstructural and elemental analyses of metal artifacts have received far less attention in this region. Comparable studies conducted in neighboring areas of the Central Zagros, however, such as at Daya Ardizi Morani, Babajilan, and Birgan, have applied advanced methods including SEM-EDS, metallography, and X-ray radiography, producing valuable information about alloy composition and manufacturing techniques.

For example, Palizvan et al. (2021) carried out archaeometallurgical analyses on seven Bronze Age hairpins from Daya Ardizi Morani in Lorestan. Their SEM-EDS results revealed that, except for one arsenical copper specimen, all hairpins were made of tin bronze with varying tin content, suggesting a lack of precise control over alloying, similar to findings from other Iron Age sites in Iran (Helwing, 2021). Microstructural studies also identified sulfide inclusions, indicative of the use of sulfidic copper ores, possibly mixed with oxidic ores. The observed deformed and recrystallized grains with twinning and slip lines pointed to the use of hammering, annealing, and cold-working cycles during production. These findings, along with other Central Zagros studies, provide a comprehensive picture of Iron Age metalworking skill and local resource exploitation.

Similarly, Oudbashi & Hasanpour (2016) examined Bronze Age artifacts from the Babajilan site in Lorestan using metallography and SEM-EDS. Their results showed that most objects were made of tin bronze with variable tin concentrations, again reflecting limited control during alloy production. The microstructural features revealed sulfide inclusions and dispersed lead globules, while metallographic analysis indicated alternating cycles of cold-working and annealing during shaping. These findings illustrate the metallurgical proficiency and production practices of Iron Age craftspeople in western Iran.

At the Zagheh site (Karun 4 Dam area), numerous metal objects dating to the Middle Elamite period were discovered. Four earrings were selected for technical, microstructural, and deterioration analyses (Pornak et al., 2024). SEM-EDS analysis identified alloys of brass, pure copper, bronze, and silver. Metallographic and X-ray radiographic studies showed that the brass earring was shaped through cycles of cold-working and annealing (ending with a cold-working stage), while copper and silver examples were produced by alternating hammering and annealing. The bronze specimen displayed dendritic structures, confirming casting as the primary manufacturing method. These results collectively provided a comprehensive understanding of production sequences, metallurgical techniques, and corrosion behavior in Middle Elamite metalwork, underscoring the importance of laboratory analysis in ancient metallurgy.

In the Birgan site (KR385) of Kouhrang, Chaharmahal and Bakhtiari Province, also dated to the second millennium BCE, five metal samples were selected for laboratory study (Khodabakhshi et al., 2019). Using metallography, SEM-EDS, Micro-PIXE, and X-ray radiography, the researchers determined that the artifacts were composed of tin bronze (Cu–Sn) with different tin levels and arsenical copper. Microstructural observations revealed evidence of hammering and casting cycles, demonstrating advanced metalworking skills of the period.

Two additional key sites, Tepe Forudgah and Sangtarashan in Khorramabad, yielded finely crafted bronze artifacts. Two bronze samples from each site were analyzed and compared due to their close chronology and geographic proximity. The applied methods included metallography, SEM-EDS, X-ray radiography, and CT tomography. Results indicated that all studied objects were made of tin bronze and exhibited microstructural evidence of hammering and annealing during production (Gravand et al., 2019).

In a more recent study, Bakhshandehfard et al. (2025) analyzed six metallic samples excavated from two Elamite sites Tepe Sanjar in the northern Susiana Plain and Tepe Qalagap in the Azna Plain of Lorestan. The goal was to determine chemical compositions and microstructural characteristics. The results revealed that all samples bore evidence of cold- and hot-working as well as annealing, though most were severely corroded. The alloy compositions included tin bronze, leaded bronze, and arsenical copper. Comparison with findings from northwestern Iran showed remarkable similarities in materials, elemental ratios, and metallurgical techniques, suggesting shared ore-processing methods and technological traditions across regions.

The innovation of recent research in the Taj Amir area and surrounding sites lies not only in presenting archaeological data but also in integrating advanced laboratory techniques for analyzing alloy composition, microstructure, and manufacturing methods of metal artifacts. This approach enables the reconstruction of ancient production processes and provides deeper insights into economic and cultural exchange networks of the second millennium BCE, effectively bridging gaps in earlier scholarship. The examination of these artifacts, alongside comparable archaeometallurgical studies, allows for a comprehensive understanding of metalworking technologies, alloy compositions, and production methods, emphasizing the significance of the Taj Amir Cemetery as a key reference site for exploring cultural, economic, and technological interactions across the Central Zagros region.

Research Method

This study was conducted based on laboratory analyses, technological examinations, and corrosion layer investigations. Three metallic samples were purposefully selected from the metal artifacts excavated at the Taj Amir cemetery site, as follows: Sample No. 1 (TA124-1): a metallic vessel with a thickness of 2 mm and a height of 5 cm; Sample No. 2 (TA251-2): a metallic vessel with a thickness of 0.5 mm and a height of 4.9 cm; and Sample No. 3 (TA167-3): a metallic ring with two perforations at its ends, a thickness of 0.2 mm, and a diameter of 1.3 cm (Fig. 3). After preparation, these samples were subjected to a series of laboratory tests to determine their elemental composition, microstructure, and technological characteristics. The resulting data made it possible to identify the type of alloy,

A B C

TA124-1 TA-251-2 TA167-3

Fig. 3. Metal artifacts from the Taj Amir cemetery in Yasuj selected for analysis. Source: Authors.

manufacturing technology and to gain a better understanding of ancient metallurgical practices at this site.

From a typological perspective, the first two samples belong to the category of vessels, while the third falls into the class of ring-shaped objects. To examine their composition and manufacturing technology, the following analytical methods were applied:

1) Metallography using optical microscopy: For studying the metal structure, examining grain morphology, identifying alloy phases, and recognizing evidence of mechanical deformation.

2) SEM-EDS: To observe microstructural features of cross-sections, semi-quantitatively determine the elemental composition, and closely investigate corrosion layers and related products.

3) X-ray fluorescence (XRF): For determining the weight percentages of major and minor elements and achieving a more accurate understanding of the alloy composition.

4) Pathological (corrosion) studies: To examine corrosion forms, intensity of degradation, and burial-induced alterations.

The selection of these methods followed the standard approaches commonly applied in archaeometallurgical research. The results were fundamental in identifying alloy types, differentiating between various bronze groups, and analyzing manufacturing technologies. Furthermore, the integration of metallographic data with elemental analyses enabled a partial reconstruction of corrosion processes and the environmental conditions that affected the objects during burial. Ultimately, the combined use of these analytical methods provided a comprehensive framework for understanding the production technology, alloy composition, and conservation state of the metal artifacts from the Taj Amir cemetery.

Findings

• Microstructural analysis

To investigate the microstructure of the alloys, metallographic cross-sections were prepared from the three selected metallic samples from the Taj Amir cemetery. For this purpose, the sections were first mounted in a two-component epoxy resin (resin+hardener) and then polished using silicon carbide abrasive papers, progressing from coarse to fine grades. Final polishing was performed with diamond pastes of 6 µm, 3 µm, and 0.1 µm to achieve a smooth, scratch-free, and reflective surface suitable for phase and microstructural observation. This meticulous preparation enabled clear visualization of the alloy phases and structural features.

For microstructural examination, a polarized microscope (BK-POL/BK-POLR model) equipped with a Canon EOS Kiss X4 CCD camera and Breeze System software was used. Observations were made both before and after etching to reveal differences between phases and grain boundaries. The etching solution for each sample was chosen according to its alloy composition. The two copper-based samples were etched with the standard copper alloy reagent (Ferric chloride blue solution), while the silver-rich sample was etched with an acidic potassium dichromate solution, following the procedure described by Scott (1991, 72).

In addition to microstructural analysis, a ZSM-1001-3E stereomicroscope was employed to study the macroscopic surface features and overall condition of the samples. This step helped identify surface defects, corrosion traces, and technological markers such as hammering or casting traces. Analytically, the integration of these methods illustrates the connection between archaeometric and archaeometallurgical pathology studies. The polarized microscope allowed precise observation of metallographic phases, deformation patterns, and possible inclusions, while the stereomicroscope provided preliminary information on manufacturing techniques and surface degradation.

• Sample TA124-1 (metal vessel No. 1)

In the examined historical sample, the presence of flattened and elongated grains, slip lines, annealing traces, and stress cracks indicates the systematic application of mechanical and thermal processes during manufacture. The elongated grains resulted from cold or semi-hot hammering during the forming stages, whereas the presence of annealing structures suggests that the craftsman intentionally applied heat treatments at specific intervals to relieve internal stresses and restore ductility. The observed slip lines within grains are evidence of plastic deformation due to hammering, while the stress cracks may have formed as a result of excessive mechanical work without adequate annealing or rapid cooling after heating. Furthermore, both intergranular and intragranular corrosion observed in the metallic cross-section may indicate heterogeneity in alloy composition and selective corrosion activity along grain boundaries. Altogether, these metallographic features demonstrate that the object was produced through a well-controlled sequence of hammering and annealing cycles, reflecting a high level of technical knowledge and metallurgical skill. Such processes enhanced both the mechanical strength and ductility of the metal, allowing the production of fine and complex objects (Artioli, 2010). When compared with metallographic findings from other archaeological sites in Iran, the Taj Amir results reveal strong technological parallels. For example, bronze artifacts from Marlik in Gilan exhibit similar microstructural features, including evidence of hammering and annealing (Oudbashi & Hessari, 2017). Likewise, in southeastern Iranian sites such as Jiroft (Weeks, 2004), Espidej (Bakhshandehfard et al., 2024), and Shahr-e Sokhta (Bakhshandehfard et al., 2025), metal sections show that craftsmen employed comparable thermo-mechanical cycles for the precise shaping of vessels and tools.

These comparisons indicate that metalworking techniques across the Iranian Plateau, while locally adapted, were based on shared metallurgical principles and comparable technical approaches. Thus, the microstructural evidence from the Taj Amir samples not only clarifies their production processes but also situates them within a broader technological tradition of ancient Iranian metalworking.

- Metal Vessel No. 2 (2-251TA)

The microstructure of this sample exhibits numerous stress cracks and slip lines, which can be attributed to the hammering stages during the object's manufacture (Caron et al., 2004, 775–788). The etched microstructure consists of fine recrystallized grains containing distinct twin lines (Fig. 4). Crystal twinning occurs when two separate crystals share some points of the crystal lattice symmetrically, resulting in specific and orderly crystal growth. The interface at which the lattice points are shared between the twinned crystals is called a twin plane, and it is a characteristic feature of worked metallic structures. Intergranular and intragranular corrosion is also observed (Fig. 5).

According to the results, the tin and copper contents of the two bronze samples are nearly identical, and the uniform composition of the alloy indicates a high level of precision in alloying. This finding demonstrates the advanced technical knowledge and metallurgical control of ancient craftsmen. Moreover, the strong microstructural similarity between the samples not only confirms the homogeneity of the alloy but also suggests the use of similar manufacturing methods. For more detailed data, see Table 1.

- Metal vessel No. 3 (3-167TA)

Examination of the microstructure reveals that the alloy consists of two main solid-solution phases: α-phase, a copper solid solution rich in silver and β-phase, a silver solid solution rich in copper.

The β-phase mainly appears as dispersed particles along the grain boundaries of the α-phase. The presence of silver in solid solution within the α-phase enhances the mechanical strength of the alloy through the solid-solution strengthening mechanism. It also reduces strain-induced lattice energy, thereby raising the alloy's recrystallization temperature. Silver-copper alloys are classical examples of eutectic systems. In the Ag–Cu phase diagram, the eutectic temperature is approximately 779 °C, with a composition of about 72 wt % silver. Upon cooling

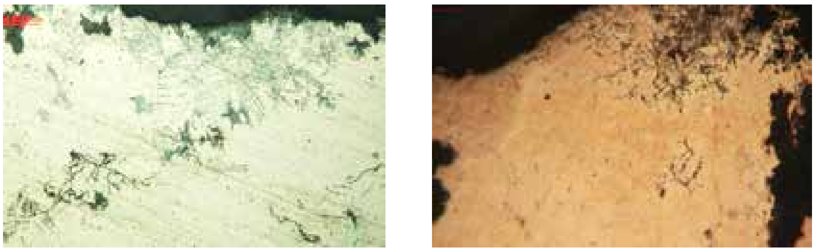

Fig. 4. In the microstructure of the metal vessel sample No. 1 (1-124TA), numerous slip lines are visible, indicating cold hammering after annealing. Intergranular corrosion along grain boundaries is also evident (magnification ×200). Source: Author's archive.

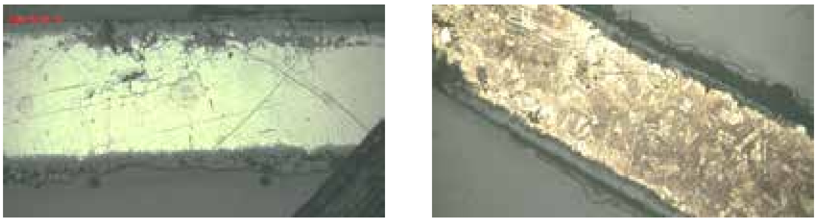

Fig. 5. The microstructure of metal vessel No. 2 (2-251TA) shows numerous slip lines indicative of hammering, and intergranular corrosion is visible (magnification ×200). Source: Author's archive.

from the molten state, both α and β phases precipitate simultaneously, forming the two-phase α + β region. At lower temperatures, the mutual solubility of silver in α and copper in β decreases, leading to the formation of discrete particles, a hallmark of eutectic microstructures. This phase configuration and final microstructure directly influence the mechanical behavior and response of the alloy to thermal and mechanical treatments, reflecting precise control of alloy composition and cooling rate during the production of silver–copper alloys for decorative or functional purposes (Scott, 1991, 12–13). The examined microstructures show elongated bright and dark regions representing the separation of silver and copper phases. These features provide direct evidence of plastic deformation and hammering during object manufacture (Fig. 6). The elongated grains and observed phase patterns suggest that the craftsman optimized the metal's properties through controlled mechanical and thermal cycles to achieve the desired form. Overall, the microstructures correspond to a typical high-quality silver–copper alloy. The α-phase grains indicate purer silver regions, while the eutectic zones consist of a mixture of silver and copper. According to X-ray fluorescence (XRF) analysis, the chemical composition of the sample includes approximately 75.65 % silver and 21.63 % copper.

The results obtained from the microstructural examination of the sample show a remarkable consistency with experimental evidence and previous findings from similar studies. As reported in earlier research, silver–copper alloys exhibit a eutectic microstructure of α+β phases, with the β phase appearing as dispersed particles along the grain boundaries of the α phase. The presence of silver, both as a solid solution in the α phase and as precipitated β-phase particles, contributes to enhancing the mechanical strength of the alloy while also influencing the thermodynamic behavior of the system, including the recrystallization temperature and the mutual solubility of the constituent elements (Zuo et al., 2015, 69–72).

- **Scanning electron microscopy (SEM)**

To determine the elemental composition of the samples, a Scanning Electron Microscope (SEM) equipped with an Energy Dispersive X-ray Spectrometer (EDS) was employed at the Razi Foundation for Applied Science, Tehran. In addition, an Energy Dispersive X-ray Fluorescence Spectrometer (XRF) made in France was used to determine the weight percentages of the elements present in the samples. The combination of these two analytical techniques enabled precise identification of the alloying elements and facilitated the study of phase differentiation and alloy composition in the metal artifacts.

Table 1. SEM-EDS analysis results of samples (weight percent, %wt). Source: Authors.

Samples	C	O	Si	S	Cl	Fe	Cu	Zn	As	Ag	Sn	Sb	Pb
TA124-1	7.98	2.25	0.15	0.16	0.19	0.27	75.1	0.94	0.54	–	10.99	0.79	0.76
TA251-2	7.47	2.15	0.18	0.19	0.18	0.13	74.89	0.50	0.55	–	12.27	0.81	0.68
TA167-3	7.52	–	0.38	0.31	–	0.10	1.80	0.49	0.73	18.87	0.76	–	0.73

- Sample 1 (1-124TA)

SEM-EDS analysis was conducted on seven designated points (A to G) and the metal matrix of this sample to investigate the elemental composition, impurities, and overall structure. The results indicated that the metallic matrix contains significant amounts of copper and tin, characteristic of a tin-bronze alloy. Impurity levels in the matrix were very low, and the surface exhibited minimal corrosion. Slight variations in elemental composition across different regions were observed, which may be attributed to the heterogeneous distribution of copper and tin or the diffusion of other elements related to manufacturing processes and burial conditions. In summary:

At points A and B, copper and tin were dominant, while small amounts of carbon, oxygen, and antimony indicated minor impurities or possible surface corrosion effects.

At points C and E, in addition to copper and tin, small quantities of lead and silicon were detected, which might result from alloying additives or environmental contamination.

Point F showed a higher oxygen content along with a specific Cu–Sn composition, suggesting localized copper oxide formation.

At point G, the relative proportions of copper, tin, and oxygen reflect minor surface alterations and phase dispersion.

Overall, the SEM-EDS data indicate that the sample was made of relatively pure tin bronze, with some evidence of surface oxidation and corrosion. The distribution of metallic elements and impurities can be interpreted in light of the production and shaping processes (hammering and annealing). The findings suggest that the craftsman maintained good control over the alloy composition (Fig. 7).

The highest lead content in this sample is 1.98%, which is lower than the amount usually added intentionally to facilitate casting. Therefore, it is unlikely that lead was deliberately added for casting purposes (Petersen, 2010, 277–278). The relatively high copper content is notable and may reflect the metallurgist's high proficiency in extracting copper from ore. The analysis results of all samples are presented in Tables 1 & 2. In these tables, SEM-EDS data are divided into the matrix and specific areas (Table 1), while XRF results are shown as weight percentages in Table 2.

- Sample 2 (2-251TA)

SEM-EDS analysis was conducted at four designated points (A to D) and on the matrix to identify elemental composition, impurities, and corrosion effects. The results show variations in metal composition and element distribution across different areas, likely caused by manufacturing processes, surface alterations, and reactions with the burial environment:

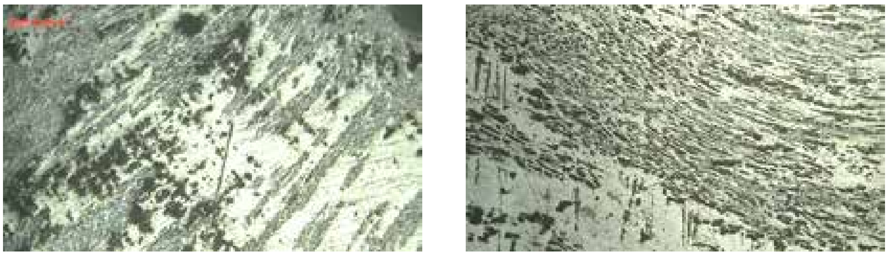

Fig. 6. In the microstructure of the metallic vessel sample No. 3 (3-167-TA), at 200× magnification, elongated bright and dark bands are visible, indicating phase separation between copper and silver within the alloy. This elongated pattern provides strong evidence of hammering and plastic deformation during the manufacturing process, demonstrating that the craftsman employed mechanical techniques to shape and control the metal's properties. Source: Author's archive.

Region A: The dominant composition includes copper with lower tin content. The reduction of tin indicates dezincification, a phenomenon that typically occurs during prolonged exposure to corrosive environments such as moist soil, leading to decreased mechanical strength, color changes, and surface structural modifications (Robbiola et al., 1998; Scott, 2002). The presence of oxygen confirms surface oxidation in this area.

Region B: A relative increase in tin with a decrease in copper and higher oxygen content suggests internal corrosion. Tin corrosion leads to the accumulation of insoluble compounds, such as tin oxides, which can limit the progression of dezincification. Parallel straight lines in the grain sections indicate hammering and plastic deformation.

Region C: In addition to copper and tin, significant amounts of sulfur and iron were observed, possibly resulting from the formation of copper sulfides such as chalcopyrite and pyrite (Klein, Hurlbut & Cornelius, 1993). These findings indicate advanced corrosion in this region and the influence of burial conditions on the metal surface.

Region D: Increased tin, decreased copper, and high oxygen levels indicate surface oxidation and corrosion, reflecting chemical and phase changes over time.

Overall, SEM-EDS results show that the sample is made of tin bronze, and the elemental composition in various areas has been affected by environmental processes and surface corrosion. Local variations in copper-to-tin ratios and the presence of secondary elements indicate phase heterogeneity and diverse environmental effects (Fig. 8).

SEM-EDS analysis of the Taj Amir Cemetery samples was conducted on the matrix and four designated regions (A–D) to determine elemental composition, impurities, and environmental effects. The data show that the sample is made of tin bronze with deliberate proportions of copper and tin. The matrix contains high copper and approximately 12–13% tin, reflecting precise control over element ratios by the craftsman.

Region A: Relative tin depletion, increased oxygen, and trace amounts of other elements (chlorine and lead) indicate dezincification and surface oxidation due to prolonged contact with moist soil, leading to discoloration, reduced mechanical strength, and surface changes (Robbiola et al., 1998; Scott, 2002).

Region B: Evidence of tin depletion and increased oxygen suggests internal corrosion and tin leaching. Accumulation of tin oxides may have restricted corrosion progression in some areas. Parallel straight lines in the grains indicate hammering and plastic deformation.

Region C: Significant sulfur and iron content indicates the possible formation of sulfide compounds such as chalcopyrite and pyrite, representing advanced corrosion and burial effects (Klein et al., 1993).

Region D: Increased tin, reduced copper, and high oxygen content signify surface oxidation and corrosion. The presence of minor elements like silicon and magnesium suggests environmental element infiltration from surrounding soil and rocks.

In summary, SEM-EDS analysis shows that the sample

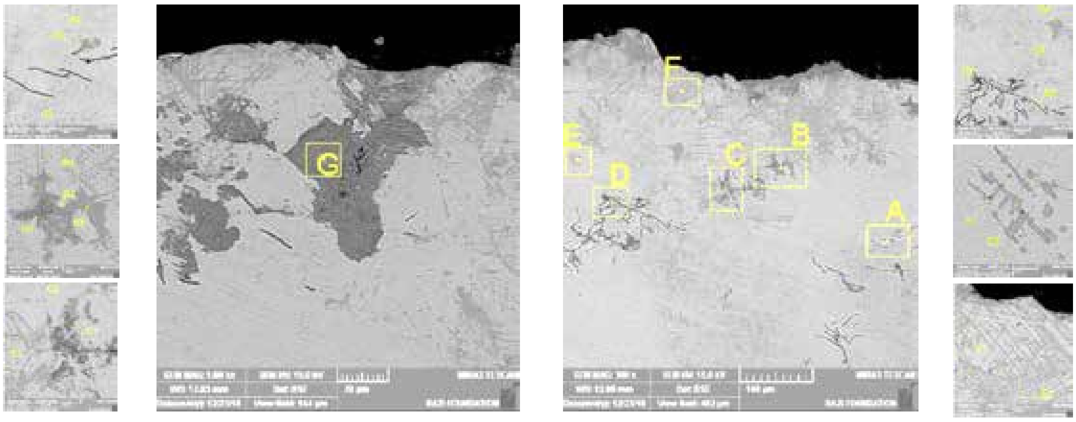

Fig. 7. Examination of the metallic structure, corrosion products, and analysis of Sample 1 (1-124-TA) at specific points using a Backscattered Electron (BSE) mode SEM at 300× magnification. Source: Authors.

Table 2. XRF analysis results of samples (weight percent, %wt). Source: Authors.

Samples	Ni	Pb	As	Au	Ag	Sb	Sn	Zn	Cu
251	0.38	0.03	0.24	0.01	0.08	0.03	10.85	0.33	88.01
167	0.06	0.38	0.33	1.57	65.75	0.08	0.07	0.16	21.63
124	0.58	0.02	0.38	0.15	0.04	0.01	9.51	0.28	88.77

is a relatively pure and controlled tin bronze alloy but has been influenced by environmental processes, including dezincification, internal corrosion, surface oxidation, and secondary element penetration. These results allow comparison with similar samples from other Iranian sites, indicating that bronze production techniques in the period involved precise control of element ratios, hammering, and annealing cycles (Scott, 2002, 225).

- Sample 3 (3-167TA)

SEM-EDS analysis was performed at five designated points (A–F) and on the matrix to assess elemental composition, impurities, and metal condition. Results indicate that the sample is a silver–copper alloy, and variations in elemental ratios across different areas reflect phase heterogeneity and environmental effects:

Region A: Dominated by silver with minor copper and tin. Low oxygen and carbon indicate minimal corrosion and a nearly pure metal surface.

Region B: In addition to silver and copper, notable calcium content was detected, likely due to soil influence and formation of calcium bicarbonate (Huang et al., 2021), indicating chemical reactions with the burial environment.

Regions C and D: Elemental composition reflects a eutectic silver–copper alloy. The two-phase structure includes fine α-phase plates with dispersed β-phase. Primary α-phase appears as dendritic cores or solid solution grains, while grain boundary fillers consist of eutectic α+β phases. These features indicate that cooling rates and temperature control during production determined the alloy's phase structure and properties (Scott, 1991).

Region E: High silver content, absence of oxygen, and no corrosion indicate an almost pure metallic surface.

Region F and matrix: High silver and significant copper content suggest that this section was produced as a silver object. The metal matrix shows a uniform silver–copper composition with limited environmental or corrosion effects (Fig. 9).

Overall, SEM-EDS analysis demonstrates that these samples are silver–copper alloys with carefully controlled element ratios and eutectic phase characteristics. The findings indicate that ancient metallurgists achieved a simultaneously hard, durable, and aesthetically pleasing alloy. Pure silver alone was too soft for functional or decorative objects, while adding copper enhanced hardness and durability, facilitated casting, and produced fine-grained, bright surfaces. This evidence demonstrates that artisans not only had access to raw materials but also empirically knew the optimal ratios to produce suitable alloys for jewelry and artistic objects. In the studied samples, as previously mentioned, one object is silver and two are bronze. In the two bronze samples, the tin and copper contents are nearly equal, and alloying was uniform and controlled, reflecting advanced metallurgical knowledge and technology. These findings indicate mastery over alloying principles and chemical composition control, as well as the existence of technical knowledge networks among Elamite communities and southwestern Iranian nomads engaged in active cultural and economic exchanges. The presence of repeated cycles of cold working and annealing in the metallic sections indicates that artisans understood the mechanical behavior of metals and used thermal treatments to enhance ductility and final strength. Combined with archaeological data, these results allow reconstruction of manufacturing techniques and provide a deeper understanding of skill level and organization in prehistoric Iranian metallurgy. Furthermore, the data enable evaluation and comparison of chemical and microstructural features with contemporaneous samples from other Iranian sites.

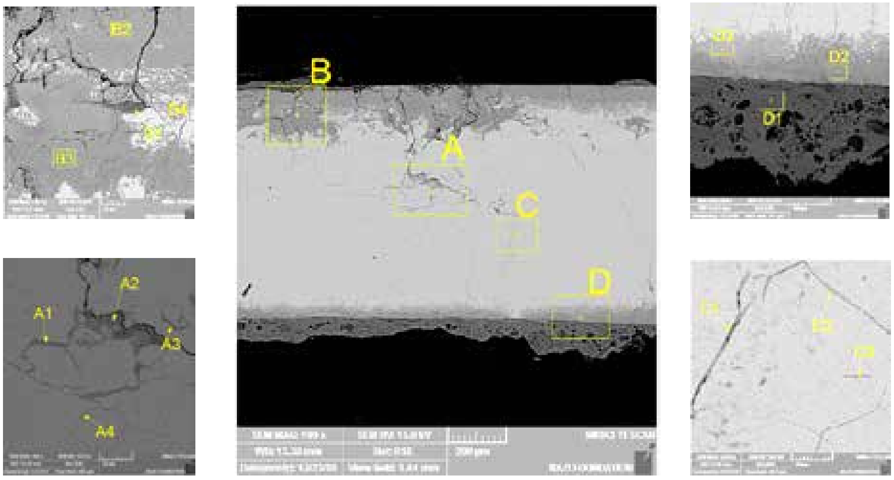

Fig. 8. Examination of the metallic structure, corrosion products, and analysis of Sample 2 (2-251-TA) using BSE-mode SEM at 100× magnification. Source: Authors.

- X-ray fluorescence (XRF) analysis

X-ray fluorescence (XRF) spectroscopy is a common non-destructive method for determining the qualitative and quantitative elemental composition of materials. It can accurately identify the percentage of elements in samples and applies to rocks, minerals, sediments, and metallic objects. XRF works by measuring secondary fluorescence emitted when a sample is irradiated with primary X-rays. Each element has a unique fluorescence spectrum, known as its “fingerprint.” In this study, μ-XRF analysis was performed using an Unisantis S.A. XMF-104 instrument. Samples were irradiated at 35 kV and 400 μA for 300 seconds under atmospheric conditions. Data processing and elemental quantification were conducted using Smart XRF software.

Advantages of μ-XRF for archaeological materials include: Non-destructive analysis, leaving samples intact; Ability to measure light and heavy elements and map elemental distribution across the sample surface; Results are comparable with similar archaeological samples, providing insights into production technology and material selection.

Limitations include: Reduced sensitivity to very light elements (e.g., H, Li); Limited penetration depth in metallic samples, giving surface-biased results; Surface corrosion, soil deposits, or oxide layers can affect measurement accuracy. With proper surface preparation and selection of multiple points, μ-XRF remains a reliable and widely used tool for elemental analysis in studies of ancient metallurgy.

XRF results indicate higher copper content in samples 124 and 251 compared to SEM-EDS, while silver content in sample 167 is lower than the SEM data. The 1.57% gold detected in sample 167 likely reflects surface gold, either from gilding, residual particles in corrosion layers, or contamination from the burial environment, rather than the bulk alloy composition. SEM-EDS, due to shallow penetration depth (a few microns), primarily reflects surface features, while XRF provides more comprehensive quantitative elemental data, enabling precise assessment of main alloy ratios and technological attributes.

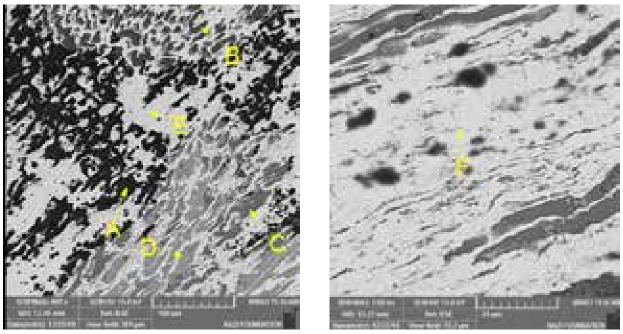

Fig. 9. Examination of the metallic structure, corrosion products, and analysis of Sample 3 (3-167TA) using BSE-mode SEM at 400× magnification. Source: Authors.

Discussion

Microstructural observations in samples 124 and 251 reveal clear evidence of controlled mechanical and thermal processing during artifact production. Flattened and elongated grains, slip lines, annealing marks, and stress-induced cracks indicate sequential cycles of cold or semi-hot hammering and intermittent annealing. Flattened grains result from plastic deformation, while slip lines reflect direct hammering. Annealing marks suggest awareness of internal stress control and metal workability, whereas stress cracks likely result from excessive mechanical work without adequate annealing or rapid cooling. Intergranular and intragranular corrosion observed indicates relative alloy heterogeneity and selective corrosion along grain boundaries. Sample 167 exhibits a two-phase silver–copper alloy: α-phase (copper-rich solid solution containing silver) and β-phase (silver-rich solid solution). The β-phase appears as dispersed particles along α-phase grain boundaries. Silver in the α-phase strengthens the alloy via solid-solution hardening and reduces lattice strain energy, particularly with uniform distribution, increasing the alloy's recrystallization temperature.

The studied samples display classical eutectic α+β microstructure. According to the Ag–Cu phase diagram, the eutectic temperature is ~779 °C with ~72% silver. During solidification, α and β phases co-precipitate, forming a two-phase α + β structure. At lower temperatures, mutual solubility decreases, leading to copper-rich α and silver-rich β, forming dispersed β particles along α grain boundaries—a hallmark eutectic structure. Phase distribution and grain size directly influence mechanical behavior, including hardness, bending strength, and response to thermal and mechanical treatment. Elongated features and phase separation lines indicate effects of hammering, bending, and annealing on plastic deformation and mechanical optimization. SEM-EDS and XRF results corroborate these observations. In sample 167, silver is ~65.75%, copper 21.63%, and gold 1.57%, likely from surface coating or dispersed particles, not bulk alloy. Differences between XRF and SEM-EDS reflect method limitations: XRF measures bulk matrix composition, while SEM-EDS is surface-sensitive. XRF confirms uniformity and precise control of main alloy elements in bronze samples 124 and 251, with tin around 10–13%, demonstrating high metallurgical skill. Microstructure also reveals uniform grains, well-dispersed β-phase, and optimized mechanical properties. Elongated light and dark areas, α–β phase separation, and eutectic patterns provide direct evidence of controlled hammering, bending, and annealing. These findings align with previous studies on silver–copper alloys, showing that mastery over melting, solidification, and thermodynamic behavior was critical for producing high-quality, durable objects (Scott, 1991, 12–13). In Table 3, the chemical composition (average of major elements), alloy type, microstructural characteristics, and technological and historical considerations obtained from several historical sites including Haft-tappeh, Sangtarashan, Qalagap, and Sanjar are summarized and compared.

In summary, Taj Amir artifacts, with tin content of 10–13% and minor silver, demonstrate more advanced alloying than typical Elamite bronzes (2–8% tin or arsenical copper). The presence of silver increases hardness and surface aesthetics. Microstructural features, including eutectic α+β (Cu–Ag) and evidence of repeated hammering and annealing, indicate sophisticated control over mechanical processing. Taj Amir craftsmen employed precise thermal and mechanical cycles to optimize mechanical properties, exceeding contemporary techniques in southwestern Iran. Metallographic and chemical evidence indicate the artifacts reflect high technical knowledge, careful control of element ratios, mechanical and thermal processing, and stable, uniform microstructure, demonstrating an advanced understanding of chemical and mechanical properties for functional and decorative objects.

Conclusion

The results of the analysis of metal samples from the Taj Amir cemetery indicate that two samples from this collection were made of tin bronze alloys, with copper and tin as their primary elements. The tin content in these two samples ranges approximately between 12 and 13%, with no significant differences observed, suggesting precise and deliberate control by the metalworkers over the alloy composition during production. Such control would have been crucial in determining the mechanical quality, hardness, and formability of the alloy, reflecting a deep understanding of the physical and chemical properties of metals by the craftsmen of that period. In addition to the main elements, small amounts of secondary and trace elements, including zinc, sulfur, iron, antimony, lead, and arsenic, were detected in the samples. These likely originated from natural impurities in the ore or from the processes of metal extraction and smelting. Although present in minor quantities, these elements indicate metallurgical challenges of the time and limitations in the primary metal resources available in the region, providing valuable information about extraction techniques, processing, and selection of raw materials. Metallographic studies and microscopic observations of longitudinal and cross sections revealed that the samples exhibited single-phase recrystallized grains with twin lines and strain lines. These features indicate the use of advanced mechanical processes, such as cold working and annealing, for metal shaping. Metal vessels and sheets were formed through controlled hammering, which generated work-hardening and ensured uniform stress distribution across the crystal lattice. These microstructures, in addition to reflecting the high practical skill of the craftsmen, demonstrate their awareness of how microscopic variations influence mechanical properties such as bending resistance, hardness, and the durability of objects. Stretching and strain lines in the microstructure further confirm mechanical methods and may indicate efforts to enhance surface strength and prevent breakage during use or burial. The third sample, containing a high silver content (~18.87%), can therefore be classified as a silver object or silver-enriched alloy. Metallographic analysis of this sample revealed distinct light and dark elongations and clear segregation between copper and silver, reflecting precise control during hammering and informed selection of elemental composition. Its microstructure included α-phase silver grains and eutectic regions composed of a mixture of silver and copper, indicating a sophisticated understanding of alloy melting and solidification properties and demonstrating that producers consciously adjusted the composition and ratios of elements based on the intended application. X-ray fluorescence (XRF) results showed the eutectic contained approximately 65.75% silver and 21.63% copper, reflecting high precision in element ratio control and production of high-quality samples. The findings indicate that metalworkers of the period, aware of the effects of impurities and thermal and mechanical processes, were able to produce durable, corrosion-resistant objects that retained part of their original structure even after thousands of years of burial. The presence of secondary elements and the controlled tin content also suggests deliberate selection and processing of mineral sources, with access to metal resources in the region or surrounding areas. Furthermore, the results from the Taj Amir cemetery demonstrate that the studied objects were primarily made of copper–silver alloys with α+β eutectic microstructures. The chemical composition and the uniform dispersion of β-phase particles along α-phase grain boundaries indicate careful control of the alloy composition and mastery of smelting and forming techniques. Minor variations in microstructure, particle size, and β-phase distribution suggest limited variation in the skill and precision of the craftsmen during melting and shaping, yet the overall quality of the objects remains high and consistent. The study also shows that the metalworkers successfully produced alloys with optimal mechanical properties and stable microstructures, reflecting their deep understanding of how microscopic changes affect durability and performance. The presence of secondary elements and controlled tin content in the bronze samples offers limited clues about the provenance of raw materials and the selection of mineral sources, although more comprehensive archaeological and geochemical data would be required to fully confirm trade networks or metal supply routes. The examined metal objects were not only functional and decorative but also reflected technical knowledge, industrial skills, an understanding of metallurgical processes, and awareness of environmental

Table 3. Comparative summary of alloy composition, microstructure, and technological features of taj amir dehno metal artifacts vs. selected elamite sites. Source: Authors.

Site	Date	Microstructural features	Cu (%)	Sn (%)	As (%)	Ag (%)	Pb (%)	Ni (%)	Other elements	Alloy type
Haft-tappeh	Mid-2nd millennium BCE	–	95–98	0.2–4.6	–	–	0.6–1.9	≤0.7	Minor P	Low-tin bronze
Sangtarashan	Mid–late 2nd millennium BCE	Fine grains, slip lines, recrystallization; mechanical work evidence	86–91	9–13	0.03–0.7	≤1	0.6–0.8	≤0.4	Minor P, Zn, Sb	Tin bronze
Qalagap	Mid-2nd millennium BCE	Fine grains, slip lines, recrystallization; some without Cu–Sn structure	69–83	1.3–6.3	0–2	–	≤0.5	–	–	Tin bronze & arsenical copper
Sanjar	Mid–Late 2nd millennium BCE	Fine grains, recrystallized; some lacking δ-phase	74–95	0–18	0–2	–	–	–	–	Tin bronze & arsenical copper
Taj Amir Dehno, Yasuj	Late 2nd millennium BCE	Flattened and elongated grains, slip lines, annealing, eutectic α+β Ag–Cu, controlled recrystallization	75–99 (XRF vs SEM-EDS)	9–13	0.3–0.7	up to 65.75 (Ag–Cu)	0.02–0.68	0.06–0.58	Minor Sb, Zn, Fe, P	Tin bronze & Ag–Cu alloy

effects on metal durability. This study, beyond examining the chemical composition and microstructure of the alloys, contributes to a better understanding of advanced metallurgical techniques and the technical skills of the society that produced these objects, clearly illustrating the role of metalworking technology in both everyday and ritual contexts.

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Declaration of Conflicting Interests

The authors declare that they have no competing interests in conducting this research.

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