ИСПОЛЬЗОВАНИЕ ЕСТЕСТВЕННО–НАУЧНЫХ МЕТОДОВ В АРХЕОЛОГИЧЕСКИХ ИССЛЕДОВАНИЯХ

USE OF NATURAL–SCIENTIFIC METHODS IN ARCHAEOLOGICAL RESEARCH

Research Article / Научная статья
УДК 903.05(470.55/.58)"638":902.654
https://doi.org/10.14258/tpai(2024)36(3).-05
EDN: LKJWRO

CHEMICAL AND METALLOGRAPHIC ANALYSIS OF FERROUS AND NON-FERROUS METALLURGY PRODUCTS FROM THE ARCHAEOLOGICAL SITES OF THE EARLY IRON AGE AND THE MIDDLE AGES OF THE SOUTHERN URALS

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Abstract. The archaeometallurgical direction in archaeology is defined as an interdisciplinary field that studies all aspects related to the reconstruction of the processes of production and use of metals by human groups. Being a complex interdisciplinary discipline, archaeometallurgy is capable of demonstrating expressive results provided that the methods of archaeology, geochemistry, materials science, mineralogy, geophysics, physical chemistry and a number of other disciplines are integrated. Recently, the constant increase in new archaeological information obtained during excavations of settlement and burial sites of the Early Iron Age and the Middle Ages in the territory of the Southern Urals requires the formulation of new research tasks. The proposed article examines in detail six metal products (fragments of boilers, a bow, a bit, a spearhead and a mirror), which were analyzed using modern metallography methods. For the first time, an analysis of the chemical composition of the objects was performed and the features of the alloys used were established. Based on the analogies involved, the question was raised about the methods of producing cast iron utensils at the Yabalakly-1 settlement of the late Middle Ages, as well as non-ferrous metal products from monuments of the Early Iron Age, in addition, with regard to ferrous metallurgy.

Keywords: Southern Urals, metallurgy, late Middle Ages, Chiyalik culture, settlement, burial ground, early Iron Age, metallographic analysis

Acknowledgments: this study was supported by the RSF grant №23-78-10057 "Dynamics of Cultural Progress and Development of the Southern Urals from the Ancient Times until it Became a Part of Russia (the 4th century BC — 14th century AD): an Interdisciplinary Archaeological Study" and carried out on the basis of the Center for Collective Use of the IMSP RAS "Structural and Physics-Mechanical Research of Materials".

For citation: Krymskiy S.V., Ruslanov E.V., Protsenko A.S., Safuanov F.F. Chemical and Metallographic Analysis of Ferrous and Non-Ferrous Metallurgy Products from the Archaeological Sites of the Early Iron Age and the Middle Ages of the Southern Urals. *Teoriya i praktika arheologicheskih issledovanij = Theory and Practice of Archaeological Research.* 2024;36(3):78–94. (In English). https://doi. org/10.14258/tpai(2024)36(3).-05

ХИМИЧЕСКИЙ И МЕТАЛЛОГРАФИЧЕСКИЙ АНАЛИЗ ИЗДЕЛИЙ ЧЕРНОЙ И ЦВЕТНОЙ МЕТАЛЛУРГИИ С ПАМЯТНИКОВ РАННЕГО ЖЕЛЕЗНОГО ВЕКА И СРЕДНЕВЕКОВЬЯ ЮЖНОГО УРАЛА

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Резюме. Археометаллургическое направление в археологии определяется как междисциплинарная область, изучающая все аспекты, связанные с реконструкцией процессов производства и использования металлов человеческими коллективами. Являясь сложнейшей междисциплинарной дисциплиной, археометаллургия способна демонстрировать выразительные результаты при условии интеграции методов археологии, геохимии, материаловедения, минералогии, геофизики, физической химии и еще целого ряда дисциплин. В последнее время постоянное приращение новой археологической информации, полученной в ходе раскопок поселенческих и погребальных памятников эпохи раннего железа и средневековья на территории Южного Урала, требует постановки новых исследовательских задач. В предлагаемой статье детально рассматриваются шесть металлических изделий (фрагменты котлов, дужка, долото, наконечник копья и зеркало), которые были проанализированы методами современной металлографии. Впервые выполнен анализ химического состава предметов и установлены особенности использованных сплавов. На основе привлеченных аналогий поставлен вопрос о способах производства чугунной посуды на селище Ябалаклы-1 эпохи позднего средневековья, а также изделий из цветных металлов с памятников эпохи раннего железа.

Ключевые слова: Южный Урал, металлургия, позднее средневековье, чияликская культура, селище, могильник, ранний железный век, металлографический анализ

Благодарности: работа выполнена при поддержке гранта РНФ №23-78-10057 «Динамика культурного развития и освоения Южного Урала с древности и до вхождения в состав России (IV в. до н.э. — XVI в. н.э.): междисциплинарное археологическое исследование» и проведена на базе Центра коллективного пользования ИПСМ РАН «Структурные и физико-механические исследования материалов».

Для цитирования: Крымский С.В., Русланов Е.В., Проценко А.С., Сафуанов Ф.Ф. Химический и металлографический анализ изделий черной и цветной металлургии с памятников раннего железного века и средневековья Южного Урала // Теория и практика археологических исследований. 2024. Т. 36, №3. С. 78–94. https://doi.org/: 10.14258/tpai(2024)36(3).-05

Introduction

In recent years, scientists have paid much attention to the study of medieval archaeological sites in the Southern Urals (Kuzminykh, 1983; Rudenko, 2000; Perevoshchikov, 2002; Borzunov et al., 2023). Through the efforts of archaeologists and historians, extensive material on ferrous metallurgy has been accumulated and analyzed (Ryazanov, 1997, 2003, 2011). At the same time, the attribution of finds to specific cultures was carried out using traditional archaeological methods, namely, by comparing the design and artistic features of objects. However, the study and reconstruction of the methods and technology of manufacturing objects seems to be an extremely important scientific task, since the constant improvement of technologies and their mutual borrowing constitute no less a layer of development of the material culture of peoples than the complication, for example, of the level of artistic design.

Thus, the practice of identifying specific cultures that has developed in archaeology is often associated with the use of typical household items in the everyday life of the population, for example, pottery (linear-band ceramics and spherical amphorae culture) or stone products (boat-axe culture) (Dardeniz, 2024). In connection with this, a pressing scientific task is the wide use of modern metallographic methods for analyzing the chemical composition and structure of metal products found during archaeological excavations (Wayman, 2000; Stepanov et al., 2021; Dolfini, 2024; Daragan, Polin, Gleba, 2024). This analysis will provide new data on both the cultural-historical and technological development, and the mutual influence of different cultures and peoples. Of considerable interest in this regard is the study of medieval metallurgy, in connection with the development at this stage of metallurgical production of a number of metals and alloys, the main ones of which are copper and bronze, and also, undoubtedly, cast iron and steel (Yuminov et al., 2013; Shishlina et al., 2020; Ankusheva et al., 2022). Unfortunately, using of methods of natural sciences in archaeological research is still sporadic (Liangren et al., 2024; Vertman, Pletneva, 2020).

The proposed publication is the first of a planned series of articles conceived as continuation of the large-scale work begun by S.V. Ryazanov on introducing into scientific circulation materials from many years of archaeological research on ferrous and non-ferrous metallurgy monuments of the early Iron Age and the Middle Ages, within the framework of their technological and historical interpretation.

Materials and Methods

The selection of ferrous and non-ferrous metallurgy objects presented in the article, consisting of cast iron and iron products from the late medieval settlement of Yabalakly-1 and a bronze mirror from the Yurmash-1 early Iron Age burial ground, was obtained during excavations in 2019 and 2021 (Ruslanov, 2023, pp. 118–130; Safuanov et al., 2023. p. 87; Ruslanov, Krymskiy, Protsenko, 2024, pp. 181–188) (Fig. 1).

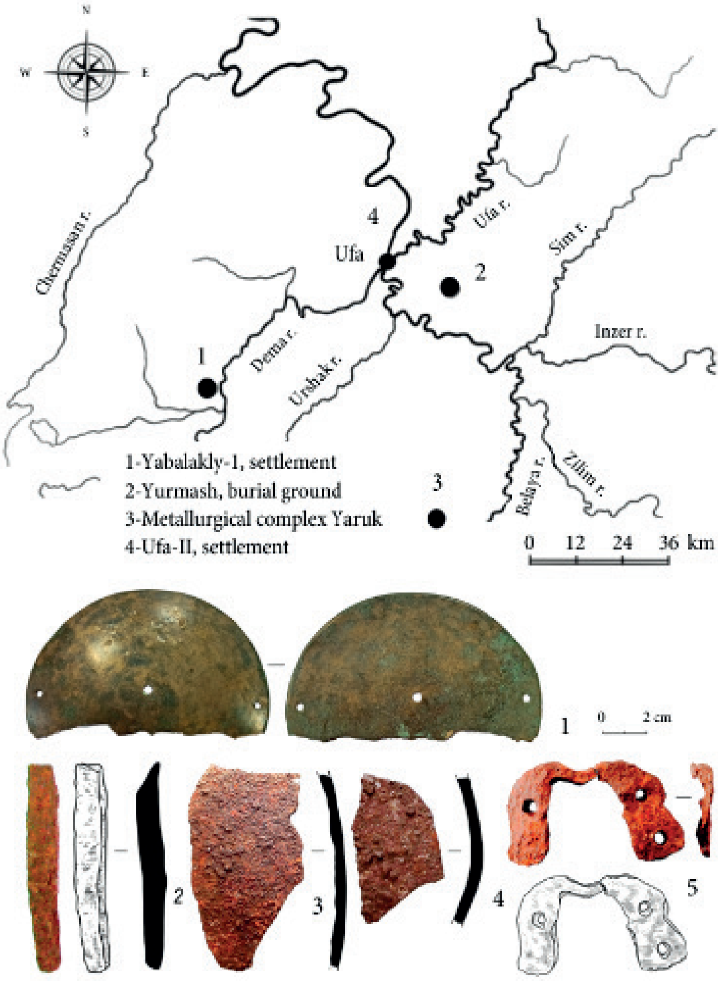

Fig. 1. Geographical layout of archaeological sites and researched items: 1 — mirror; 2 — chisel; 3, 4 — boiler fragments; 5 — boiler hoop

Рис. 1. Географическое расположение археологических объектов и исследованные предметы: 1 — зеркало; 2 — долото; 3, 4 — фрагменты котлов; 5 — дужка котла

The bronze mirror comes from the Yurmash-1 early Iron Age ground burial ground. The necropolis was discovered in 2012 by a team from the Department of Archaeological Research of the Institute of Linguistics and Astronomy of the Ufa Scientific Center of the Russian Academy of Sciences (now the Institute of Linguistics and Astronomy of the Ufa Federal Research Center of the Russian Academy of Sciences) under the leadership of V.V. Ovsyannikov. In 2019, the research was continued by I.M. Babin. It is located 1 km north of the village of Turbasly, the Iglinsky District, Republic of Bashkortostan, and occupies a high cape of the native terrace of the right bank of the Yurmash River (right tributary of the Ufa River). Based on the materials obtained, the necropolis is attributed to the late stage of the Kara-Abaz culture (1^{st}–3^{rd} centuries AD) (Safuanov et al., 2023, p. 87).

Fragments of cast-iron boilers, a bow and an iron chisel come from the Yabalakly-1 settlement, discovered by E.V. Ruslanov in 2021 during exploration work to search for late medieval monuments in the Dema River valley. The Yabalakly-1 settlement was discovered in 2021 during reconnaissance work to search for late medieval monuments in the Dema River valley. The site is located 1.17 km north of the northern outskirts of the village of Yabalakly, Chishminsky District, Republic of Bashkortostan, on the left steep bank of the Dema River. The site is located on a subtriangular cape formed by the modern riverbed and the oxbow lake Doga-kule. The site is flat, 2–3 m above the water's edge, covered with meadow vegetation. To the north, the site drops sharply by 1.5–2 m, which, apparently, is due to the fact that during the meandering, the bed of the Dema River changed its position, shifting to the east and leaving behind the oxbow lake Doga-kule. The area of the site is more than 10 hectares. The stratigraphy of the site based on the results of excavations in 2023 is as follows: turf — 5 cm, loose light-gray sandy humus (cultural layer) — 5–35 cm, light-gray alluvium — 35–40 cm, humified sandy loam with loam inclusions (transition layer to the continental base) — 45–75 cm, mainland — light-brown dense loam (deeper than 75 cm). The thickness of the cultural layer was 45 cm. In 2023 and 2024, excavations were carried out at the settlement, the total area studied was 190 sq. m. Thus, the obtained archaeological material (fragments of jugs, porridge dishes, stirrups, parts of cast-iron boilers) allows us to determine the time of existence of the monument within the 14^{th} century.

A socketed spearhead with a feather in the form of two spikes was discovered during archaeological excavations of the Ufa-II settlement in 2023. Similar spears were widespread among many peoples of the Volga region in the 8^{th}–11^{th} centuries (Danich, 2010, p. 25; Izmailov, 1997, p. 73). The spearhead and the end of one spike are broken off. Without these elements, the dimensions of the tip are: total length 15 cm, feather length 5.6 cm, its greatest width 2.3 cm, sleeve diameter 1.8 cm.

The items included in the sample set originate from: boiler №1 — lifting material; boiler №2 — pit №2, seam №2; chisel and drill bit — pit №8, seam №2.

The samples for the studies were cut using a disc cutting machine. The sample surface preparation for the study consisted of mechanical grinding on abrasive paper with a transition to increasingly fine-grained abrasive. After that, grinding was performed with diamond pastes 5/3 and 3/2 with a gradual decrease in the size of the abrasive particles. The chemical composition of the samples was analyzed using a portable optical emission spectrometer Hitachi PMI-MASTER Smart (Germany) and a scanning electron microscope TESCAN MIRA 3

LMH (Czech Republic). Before testing, the samples were cleaned using an abrasive wheel and sandpaper. Due to the fact that the samples were archaeological artifacts subjected to long-term oxidation in the soil layer in the open air, the oxidized layer was characterized by depth and significant heterogeneity, penetration into the metal layer along the grain boundaries and pores with their filling. Therefore, mechanical removal of the oxide layer was not carried out completely, which could affect the accuracy of the study results. In addition, since some samples were fragments of boiler walls, the presence of microscopic organic residues could affect the accuracy of carbon content determination. Unless otherwise stated, the accuracy of the measured parameter did not exceed 5%.

Results and discussion

Iron objects

The analysis showed that the samples of the walls of boilers 1 and 2 (Fig. 1.-*3, 4*) are practically indistinguishable from each other in composition and, in fact, structurally represent hypereutectic cast iron, since the carbon content in them exceeds 4.3% (Table 1) (Strangwood, 2024). The sample of the iron bow (Fig. 1.-*5*) differs significantly in chemical composition from the samples of the boiler walls and is high-carbon steel (carbon content over 0.6%). These samples also differ significantly in the content of other elements. Thus, the silicon content in the material of the bow is significantly lower, and nickel, magnesium, titanium and lead — more than in the material of the boiler walls.

Tab. 1

Iron Items Chemical Composition

Таблица 1

Химический состав изделий черной металлургии

Sample	Chemical element, wt. %										
	Fe	C	Si	P	Mn	Cr	Ni	Al	Mg	Ti	Pb
Boiler wall 1	94.1	>4.50	1.02	–	0.05	<0.01	0.01	0.10	0.09	0.02	0.04
Boiler wall 2	94.3	>4.50	0.92	–	0.02	<0.01	<0.01	0.09	0.06	0.02	0.02
Boiler bow	97.6	1.55	0.55	–	0.02	<0.01	0.03	0.09	0.03	0.04	0.07
Bit	98.9	0.49	0.09	–	0.2	0.10	0.01	0.05	<0.01	<0.01	<0.01
Spearhead	99.12	–	0.71	0.17	–	–	–	–	–	–	–

The difference in chemical composition can be explained by both a different manufacturing method and a difference in the composition of the original ore and the place of its mining. Due to the fact that the boiler walls and the bow have different chemical compositions, it can be assumed that their manufacturing method was different. Thus, most likely, the bow was made separately, as a part of the boiler subjected to higher static and dynamic loads, however, destructive microstructural studies are required to determine the detailed method of steel manufacturing.

The chemical composition of the bit (Fig. 1.-*2*) allows us to conclude that it is made of medium-carbon steel (0.3–0.55% C). The low silicon content, while having a high proportion of manganese and chromium, is noteworthy. Obviously, this sample was made from an ore different from that from which the other studied samples were smelted. Thus, all four studied objects are products of medieval ferrous metallurgy, namely steel and cast iron. A fairly extensive analysis of medieval metallurgy monuments in the Southern Urals is given in the monograph by S.V. Ryazanov (Ryazanov, 2011).

Fig. 2. Socketed spearhead

Рис. 2. Втульчатый наконечник копья

The spearhead sample, unlike the four studied samples, was not steel or cast iron, but iron with silicon and phosphorus impurities (Fig. 2, 3, Table 1). These impurities are of metallurgical origin due to the difficulty of removing these elements from the metal during smelting, due to uniform distribution of alloying elements (Fig. 4). It is nessesary to note, that probably this item was cast due to presence of pores (Fig. 3).

According to his data, forty-three archaeological sites associated with ferrous metallurgy have been recorded in the territory of the Southern Urals. Fourteen sites have been reliably identified by the author as metallurgical sites, but without the possibility of their precise interpretation and dating at present. As is known, the basis of the bloomery process is the direct reduction of iron ore into metallic iron (Strangwood, 2024). A specially prepared charge was loaded into a low bloomery furnace — a mixture of ore, charcoal and, often, so-called fluxes (usually limestone). Combustion of coal with the supply of raw (i.e. not preheated, as in the modern blast furnace process, but cold) air (hence the bloomery process) created a high temperature and a reducing atmosphere with a predominance of carbon monoxide (CO) in the working space of the furnace. The iron ore, which was mainly composed of iron oxides, silica (SiO_2), alumina (Al_2O_3) and other oxides, was subjected to chemical changes under such conditions. Under the influence of chemical reactions, one part of the iron oxides was reduced to metallic iron. The reduced microscopic particles of iron gradually descended together with the column of charge down to the nozzle, into the high-temperature zone, heated up and stuck together into a spongy mass impregnated with liquid slag — bloom. Another part of the iron oxides, having been reduced to ferrous oxide (FeO), together with the oxides obtained from the gangue ore and fluxes, formed a low-melting slag (Baikov, 1948, pp. 356–381).

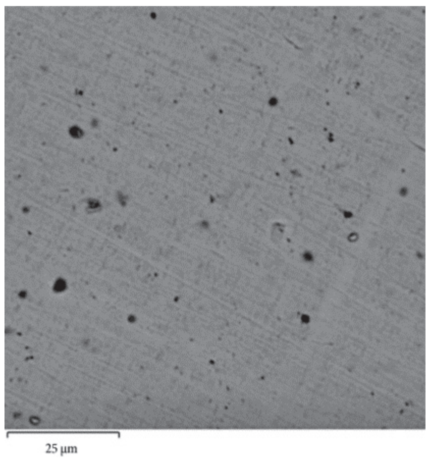

Fig. 3. Electron microscopic image of the sample structure (spearhead)

Рис. 3. Электронно-микроскопическое изображение структуры образца (наконечник копья)

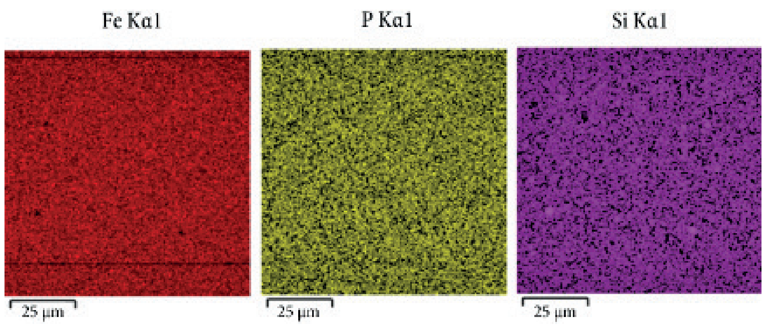

Fig. 4. Maps of spatial distribution of chemical elements (spearhead)

Рис. 4. Карты пространственного распределения химических элементов (наконечник копья)

To increase the volume of the bloomery furnace, the length and width (or diameter) of the working chamber were also increased. To ensure a uniform combustion process at all levels and a high working temperature per unit volume of the furnace shaft, a certain number of air-blowing nozzles are required. Consequently, changing the parameters of the length and width of the furnace entailed an increase in the number of air-blowing nozzles. This path was acceptable for increasing the productivity of furnaces, but due to the limited capabilities of the technology of that time, its limit was soon reached (Dolfini, 2024).

In the monograph by S.V. Ryazanov examined in detail the Yaruk metallurgical complex and similar ones in Bashkiria, which were called “Yaruk-type complexes” (Ryazanov, 2011, pp. 7–19). The metallurgists of Yaruk and similar monuments increased the number of air-blowing tubes to six to ensure uniform heating of the furnace shaft space and, accordingly, to obtain a larger bloom. At the metallurgical complexes of the Southern Urals, shapeless metal ingots are also present in the lifting material (in the Yaruk ravine — in large quantities in the layer and in accumulations of metallurgical waste). Some ingots are iron, almost pure in carbon content, while others have the structure of high-carbon steel or cast iron. At the Ural sites, such ingots are sometimes very large — from 500 to 1000 grams. The author proposed a reconstruction of the method for producing molds for casting cast iron boilers (Ryazanov, 2011, p. 101, fig. 29). The assumption about the possibility of using nickel ores of the Southern Urals as alloying additives in the Bulgar ferrous metallurgy to obtain high-quality steel deserves attention (Korolev, Khlebnikova, 1961, p. 160; Semykin, 2015, p. 35). Thus, based on the results of chemical analysis of iron objects and literature data, it can be assumed with a high degree of probability that all analyzed objects could have been made directly at the Yabalakly-1 settlement, which is indirectly indicated by the found fragment of metallurgical slag. However, the absence of finds of remains of furnaces and slag in large quantities allows us to assert that further studies of the settlement are necessary. At the same time, the objects could have been made at the already identified nearby metallurgical complexes of the Yaruk type, located 50–60 km to the east and southeast of the settlement.

Bronze mirror

A metal mirror was examined, which was preserved in fragments, however, the general symmetry of the object allows us to draw a conclusion about its original geometry and dimensions (Fig. 1.-*1*). The mirror is missing a side part of approximately one third. Initially, it had the shape of a concave disk with a diameter of 12.5 cm. At a distance of 5 mm from the edge of the disk, there are two symmetrical holes with a diameter of 3 mm. In the center of the disk there is a hole with a diameter of 4.5 mm. The thickness of the disk is 2 mm, thinning to 1 mm towards the edges. On the side of the lost part of the object, the chip is brittle, without significant traces of patina. On the front side, the disk has a smooth surface. On the surface, swellings of corrosive origin are observed. Traces of patina are observed mainly on the back side of the disk. On the back side, the surface of the disk is uneven, with a characteristic relief in the form of small craters, which indicates the foundry origin of the product. Results of the analysis of the chemical composition mirrors showed that it was made of tin bronze with a high nickel content (Table 2).

It should be noted that the composition of this bronze differs significantly from the composition of modern tin bronzes (Freudenberger, 2024). The high nickel content is probably an impurity circulation, and is due to the use of copper-nickel ore. However, the question of the purposefulness of alloying with nickel remains open. This assumption is supported by the fact that the addition of nickel as an alloying additive increases the strength and corrosion resistance of bronze. On the other hand, the high nickel content in this product is significantly higher than modern nickel bronzes, which may indicate that the nickel content in the product was not consciously controlled.

Tab. 2

Chemical composition of the mirror from the Yurmash-1 burial ground in a comparative context

Таблица 2

Химический состав зеркала с могильника Юрмаш-1 в сравнительном контексте

Sample	Chemical element, wt. %									
	Cu	Pb	Sn	As	Sb	Fe	Ni	Al	Si	Bi
Yurmash-1 burial ground	60,98	0,826	18,0	–	–	5,89	13,3	0,194	0,4	0,160
Besoba burial ground, barrow 9	base	0.5	1.0	0.2	0.01	–	–	–	–	0.001
Besoba burial ground, barrow 11	base	0.005	5.0	0.04	0.005	–	–	–	–	0.001
Abatskiy 3 (barrow 6, burial 10)	base	0.12	>30.3	0.15	–	0.21	0.02	–	–	0.06
Abatskiy 3 (barrow 2, burial 17)	base	0.2	21.86	0.15	–	0.05	0.05	–	–	0.04
Chepkul' 9 (barrow 7, burial 3)	base	0.11	23.57	0.11	–	0.42	0.02	–	–	0.08
Tutrinskiy burial ground	base	0.17	20.7	0.1	–	0.24	0.07	–	–	0.02
Fomintsevo	base	<0.5	22.13	0.03	–	0.33	–	–	–	0.03

A comparative analysis of the data on the chemical composition of similar cultural products is given in Table 2. It is noteworthy that the mirror in question differs significantly from the mirrors found in the Besoba (Degtiareva, Kuz'minykh, 2018), Abatskiy (Tigeeva, Belonogova, 2018a, b), Chepkul (Tigeeva, Belonogova, 2018a, b), Tutrinskiy and Fomintsevo (Tigeeva, Belonogova, 2018b) burial grounds. Thus, the content of the main alloying element, tin, is comparable to that in one of the mirrors from Abatskiy, and in the mirrors from Chepkul', Tutrinskiy and Fomintsevo. This fact testifies to the similarity of the manufacturing technology. However, the content of lead and bismuth in the mirror in present paper sample is slightly higher than in all the other compared samples. It is noteworthy that such elements as arsenic and antimony are completely absent in the sample that was the subject of analysis in this article. An analysis of the mirrors in the article (Tigeeva, Belonogova, 2018b) revealed that all of them were made only of tin bronze with a fairly high tin content in the alloy — up to 31%, and a conclusion was made that, apparently, these items were among the prestigious items, the casting of which required a certain unification in compliance with the specified alloy recipe. According to the authors, such a high concentration of tin in the items is not accidental. An increase in the tin concentration to 20–23% affected the color of the item, giving it a golden-yellow color, and up to 24–28% — gray steel. When lightly striking the mirror, a melodic sound is noted, probably also associated with the high content of this element in the alloy. Thus, the tradition of making mirrors from high-tin bronze is not associated with the local metal production of the Sargatka tribes, and the decorations are probably imported. The authors of the study note one of the main centers of mirror production associated with the Volga region and the activities of the workshops of the Savromat and Sarmatian archaeological cultures. However, it is difficult to determine which center the Sargatka mirrors considered in (Tigeeva, Belonogova, 2018b) are related to due to the identity of the chemical composition of the products, as well as a single production stereotype, implying a specific method of quenching hot-forged mirrors in cold water. Traditional connections with the Volga region, as well as proximity to the area of the Sargatka tribes of Tobol-Ishim region, allow us to assume the leading role of the first production center. The authors of (Degtiareva, Kuz'minykh, 2018) came to similar conclusions. They found that all sacredly significant objects were made of tin and tin-arsenic bronzes. At the same time, the tin admixture was often unreasonably high — up to 31%, which led to the fragility of the metal despite special heat treatment modes. The production centers of Rudny Altai and Central Kazakhstan, from where tin and tin-arsenic alloys came, have been established as the main vectors of historical and metallurgical contacts of the Savromat tribes, in particular, in the manufacture of bronze mirrors, and the authors consider their northern neighbors — miners and metallurgists of the Itkul culture — to be the main suppliers of copper for the early nomads of the Southern Urals and Western Kazakhstan. At the same time, the mirror in present paper stands out significantly from similar products with a very high nickel content, which raises the question of the nature of such a chemical composition. One of the reasons may be the nature of the original raw material, presumably contamination of the original ore or crucible with residues of ore used to manufacture ferrous metallurgy products. Another possible reason may be the use of complex ore, but this issue undoubtedly requires further research and clarification. Thus, in (Tigeeva, Belonogova, 2018a; Tishkin, Seregin, 2011) it is reasonably asserted that the bronze mirrors of the Eurasian nomads were partly made in China according to fairly high production standards using technological methods that were advanced for their time, while some were local imitations or counterfeits, which raises the question of the reasons for the significant deviation of the chemical composition of the mirror under study from similar samples.

Conclusions

1. As a result of the metallographic analysis, the features of the used alloys of ferrous and non-ferrous metallurgy items from the Yabalakly-1 settlement and the Yurmash-1 burial ground were established.

2. It was shown that the studied products have significant differences in chemical composition, which is due to different purposes and production technology. Samples of boiler walls 1 and 2 are made of hypereutectic cast iron, the boiler bow is made of high-carbon steel, the spearhead was produced from quite clear iron with metallurgical impurities and the bit is made of medium-carbon steel. The bronze mirror is made of tin bronze with a high nickel content.

3. Comparison with previously obtained data allows us to conclude that the cast iron and steel objects could have been made both at metallurgical complexes such as Yaruk and directly at the site itself.

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There is no conflict of interest / Конфликт интересов отсутствует.

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Статья поступила в редакцию 06.09.2024;
одобрена после рецензирования 20.09.2024;
принята к публикации 24.09.2024.
The article was submitted 06.09.2024;
approved after reviewing 20.09.2024;
accepted for publication 24.09.2024.