The Development of Ferrous Metallurgy in Ryzan Principality (Ancient Rus’)

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Keywords

Ryazan Principality, metallurgical complex, ore occurrence, archaeometallurgy, technological schemes

Abstract

Medieval metallurgical complexes in Ancient Rus’ that have been studied are far from numerous. Three such complexes have been discovered on the territory of the former Principality of Ryazan (now Ryazan region of the Russian Federation). We have traced the full metallurgical cycle – from the exploitation of the ore occurrence to the manufacturing of the finished product – at the Istye 2 settlement. Over the course of archaeological and archaeometallurgical study of the settlement, we localized a rural metallurgical complex that supported the handicraft production of a large city with its products.

Introduction

Ferrous metallurgy is a complex, multifaceted system of production. The first step in the reconstruction of ancient metallurgical processes is the search for ore occurrences and metallurgical facilities related to them. Unfortunately, such facilities in Ancient Rus’ have not been studied extensively. This can be explained, first and foremost, by the fact that metallurgical complexes, which are located at a significant distance from settlements, have rarely come to archaeologists’ attention. At the same time, without careful study and mapping of such complexes, it is impossible to understand the level of development of the local population’s iron production, the nature of the initial raw materials for blacksmithing, and the relationship of the metallurgical complex to specific craft centers.

The wide distribution of iron ore is well known. However, it was not easy to find suitable iron ore deposits for the production of high quality iron and steel products. For example, Eastern European blacksmiths avoided phosphorus iron ores to the middle of the 1st millennium AD, because phosphoric iron cracks during cold-working due to its reduced ductility (cold shortness) (Thile and Hošek, 2015, p.114; Zavyalov, Rozanova and Terekhova, 2012, p.31). The difficulty of smelting certain types of iron ores is evidenced by the experiments of Crew (2013, p.39) with hard rock ores. The difficulty of finding suitable ores for the bloomery process was demonstrated by Norwegian naturalist Ole Evenstad (1995, pp.54-55) as early as at the end of the 18th century.

Taking the aforesaid into account, we developed a research project “Sources of raw materials of Ancient Rus craft centers”. The project aims was to summarize the results of analytical and experimental studies of ferrous metallurgy and ironworking in the Ryazan principality. One of the objectives of the investigation was searching for archaeological sites related to ore occurrences. It was also necessary to investigate whether these ore deposits were exploited in medieval times and thus formed the basis for the development of ferrous metallurgy and metalworking in ancient Ryzan. The task of the project was also to determine where the produced bloomery iron was delivered.

Over the course of the Pereyaslavl-Ryazan archaeological expedition’s work, which took place in Ryazan region, three such ore occurrences were recorded: on the banks of the Istya, Loknya, and Pronya rivers (Figure 1). Of them, only the ore occurrence on the Istya River could be directly linked to an archaeological site that had traces of metallurgical production. Geological data indicates that the origins of the Istye ore can be traced to the mineralization of limestones of the Carboniferous and Devonian Periods. The average ore bed thickness here is 0,36 m, in some places more than 2 m (Dvorov, 1965, p.191).

In the words of mining engineer M. Buynevich (beginning of the 20th century), in this ore occurrence, “the ore is limonite and sphaerosiderite, with mostly limonite

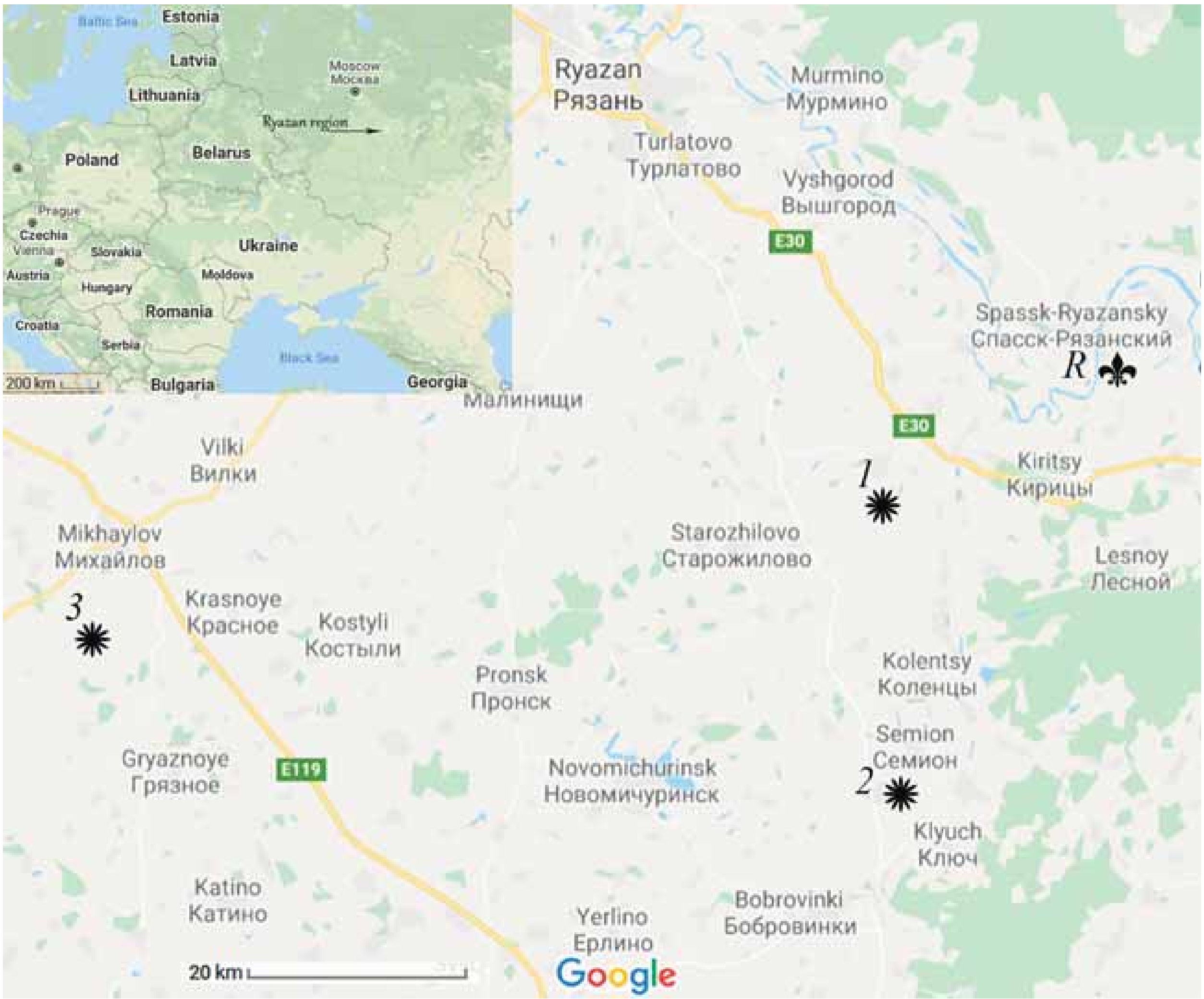


Figure 1. Map of ore occurrences in Ryazan region: 1 – Istye; 2 – Pronya (near hill-fortress Tolpino); 3 – Loknya. R – the capital of Ryazan principality (Staraya Ryazan).

on the outside, occurs at a depth from 9 arshines to 10 sazhens[1] at the watershed, depending on the terrain. The ore occurs in a virtually pure layer of varying thickness" (GARO. F. R-903).

Materials and Methods

The iron objects as well as the ore and slags were analyzed by X-ray fluorescence and metallographic investigations. The specimens for metallographic analyzes were prepared using standard techniques. The examined samples were cut out from cutting-edges of knives or from other functional parts of the objects. The samples were then mounted into Wood' alloy (Sn-12.5 %, Pb-25 %, Cd-12.5 %, Bi-50 %), grinded and then polished with chromium oxide. The microstructures of iron objects were determined with an MMR-2R optical microscope, after etching a polished sample with nital reagent (3 % solution of HNO_3 in ethyl alcohol) at magnifications of 150 x and 490 x. The size of the grains was evaluated after the Russian state standard (GOST 5639-82). Microhardness was measured on a microhardness machine PMT-3 with a diamond pyramidal indenter with 100 g load.

The X-ray fluorescence analyzer NITON XL3t GOLDD+ was used to determine the chemical composition of ore and slag iron conglomerate or slag cakes (mechanical mixture of slag, wüstite and iron particles – by-products of the smelting process).

Archaeological investigations

On the edge of the ore field, we discovered an Ancient Rus settlement, designated as Istye 2 (Figure 2). The site is located on the right (eastern) slope of the valley of the River Istya (right inflow of the Oka River). The settlement is situated on a relatively gentle, completely plowed plot. The slope from the contour of arable land to the riverside of the Istya River is steep, well sodden; everywhere

Figure 2. Settlement Istye 2. View from north. Photo: V. Zavyalov.

limestone outcrops with ironing are observed. It was established that the highest concentration of artifacts is observed in the southern part of the settlement on an area about 200 x 200 m in size. Based on ceramic materials, the settlement can be dated to the 12^{th}-13^{th} century.

Traces of metallurgical production were discovered at the site – remains of bloomery furnaces, numerous fragments of iron-slag conglomerate, bloomery iron and fragments of ceramic tuyeres (Figure 3) (Bulankin, Zavyalov and Ivanov, 2012). It is important to emphasize that all of the metallurgical artifacts were concentrated in the southwestern edge of the settlement, where metallurgical complexes were likely located. The large scale of these finds indicates that production volumes were significant.

The remains of the bloomery furnaces practically did not survive as a result of many years of plowing of the site's area. Only in one case it was possible to fix the base of the furnace. It consisted of limestone laying of a basement shape measuring about 1.6 x 1.0 m. The base of the furnace had a concave profile, at the bottom of which a thin layer of charcoal with a thickness of 1-2 cm was traced over the entire area. The filling of the structure is represented by numerous pieces of slag-iron conglomerate, total weight of about 60 kg and fragments of clay tuyeres (425 fragments). Rare pieces of clay coatings, calcined limestone, iron ore and animal bones were also found during the clearance.

The documented remains of the furnace did not provide any evidence for the reconstruction of a specific furnace type. However, numerous fragments of the slag-iron conglomerate - just a few hundred kilograms of the conglomerate were collected - suggest that the metallurgists used shaft furnaces without a slag outlet.

Results of analyzes

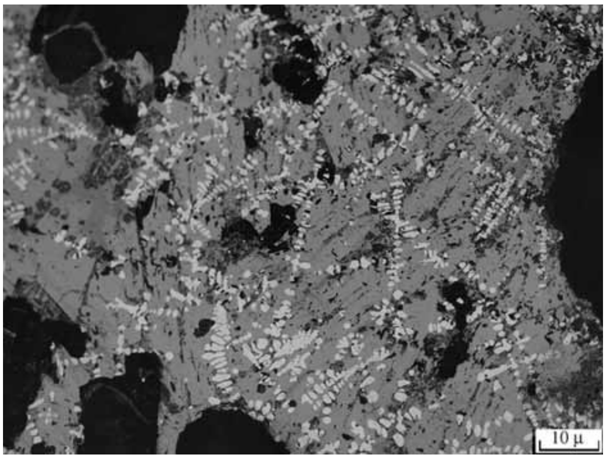
A total of 24 samples of metallurgical objects (10 samples of ore and 14 specimens of slag-iron conglomerate) from Istye 2 were analyzed. A comparative analysis of the data obtained uncovered a substantial similarity in their distribution. Microstructure of slag-iron conglomerate is presented by wüstite dendrites in large fayalite crystals and glassy phase (Figure 4: 1).

It has been determined that the Istye ore contained more than 45 wt. % iron (FeO – 52.1-57.1 wt. %). The content of silica in the ore is about 26.0-33.3 wt. % SiO_2, alumina – 7.3-9.2 wt. % Al_2O_3 (Table 1). A single sample had a very high iron content of 75.7 wt. % FeO, and in this sample magnesium (4.1 wt. % MgO) was determined too.

Figure 3. Ceramic tuyere and metallurgical slags from Istye 2. Photos: V. Zavyalov.

Table 1. The chemical composition of iron ore from Istye ore occurrence.

No.	FeO	SiO_2	Al_2O_3	P_2O_5	K_2O	CaO	MnO	MgO
Istye_ore-1	56.7	27.6	7.6	1.8	1.4	2.9	1.7	nd
Istye_ore-2	52.2	30.8	8.2	0.8	3.2	2.5	2.1	nd
Istye_ore-3	53.1	31.3	8.9	0.9	1.5	2.5	1.3	nd
Istye_ore-4	53.8	32.5	7.6	1.3	1.7	2.4	0.6	nd
Istye_ore-5	51.5	31.3	8.1	1.8	1.9	3.9	1.4	nd
Istye_ore-6	57.1	23.1	8.0	0.9	3.3	3.9	2.5	nd
Istye_ore-7	55.6	31.4	7.3	0.9	1.2	1.8	1.5	nd
Istye_ore-8	52.1	33.3	7.9	1.1	1.6	2.3	0.9	nd
Istye_ore-9	59.6	26.0	9.2	1.6	0.9	2.0	0.5	nd
Istye_ore-10	75.7	12.3	3.2	0.3	0.8	1.5	1.9	4.1



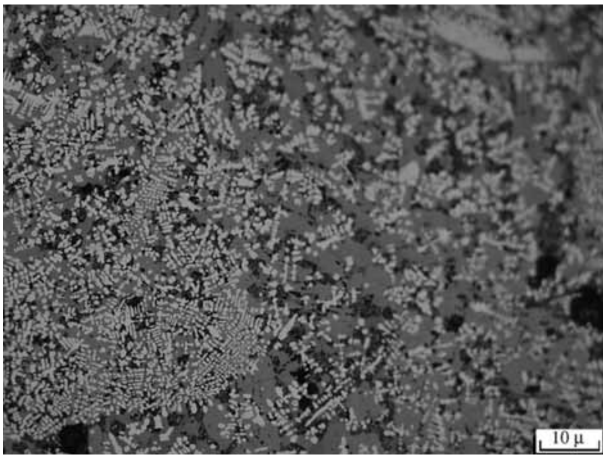


Figure 4. Microstructure of slags: Wüstite dendrites (white) in large fayalite crystals (grey). 1: Slag from Istye 2 (left); 2: Experimental slag (right). Photos: V. Zavyalov.

It should be noted that there is a higher iron content in the slag-iron conglomerate compared to the ore. The highest iron content was shown by samples in which magnesium oxide was present (Table 2).

The phosphorus content in both ore and slag-iron conglomerate, with rare exceptions, did not exceed 2 wt. % P_2O_5. In the course of experimental work, it was found that iron extracted from the ores of the Istye ore deposit contained practically no phosphorus (Zavyalov and Terekhova, 2013, p.55). This conclusion correlates with the results of mass archeometallographic analyses of iron artifacts from medieval sites on the territory of the Ryazan Principality: phosphorous (high-hard iron) is fixed on single and, as a rule, imported items (Zavyalov and Terekhova, 2013, p.147).

The results of the X-ray fluorescent analysis allowed us to identify trace elements that are characteristic of both slag-iron conglomerate and ore. They include calcium, potassium and manganese. Sulfur and phosphorus present in small quantities. One of the main elements for characterizing ore and slag is calcium. As one can see from the tables, all samples show a relatively high content of lime (2.5-5 wt. % CaO in ore and 0.4-5.3 wt. % CaO in slag). The presence of calcium is probably due to the occurrence of ore in limestone rocks.

Unfortunately, due to the fact that the metallurgical facilities were largely destroyed during the many years over which the site was plowed up, it does not seem possible to reconstruct the type of furnace or the nature of the process. Therefore, we decided to conduct experimental smelting of ore from Istye ore occurrence.

Experimental works

We have conducted several experiments on the smelting of Istye ore in order to understand how the bloomery process went (Zavyalov and Ratkin, 2009). The used ore was a solid concretion of irregular shape, with the color of ochre. The preliminary operation of the bloomery

Table 2. The chemical composition of slag-iron conglomerate from the settlement of Istye 2.

No.	FeO	SiO_2	Al_2O_3	P_2O_5	K_2O	CaO	MnO	MgO
Istye-1	64.7	18.0	7.3	1.1	2.5	5.4	1.1	nd
Istye-2	71.3	15.2	7.1	1.1	0.9	0.6	0.8	2.8
Istye-3	71.9	13.7	6.8	1.5	1.1	1.6	2.2	1.0
Istye-4	69.0	15.0	5.7	4.2	0.5	5.0	0.6	nd
Istye-5	84.6	7.1	3.6	0.4	0.3	0.4	nd	3.6
Istye-6	75.0	16.7	5.0	0.9	0.3	0.5	0.1	nd
Istye-7	83.2	11.2	4.4	0.4	0.2	0.4	0.1	nd
Istye-8	83.9	10.6	4.3	0.4	0.2	0.4	0.0	nd
Istye-9	66.8	18.8	7.3	0.9	2.1	3.2	0.9	nd
Istye-10	69.5	17.7	6.0	1.0	2.3	2.6	0.8	nd
Istye-11	70.5	16.9	6.5	2.7	0.5	1.9	0.9	nd
Istye-12	70.8	15.9	7.9	1.9	0.6	1.9	0.9	nd
Istye-13	69.9	15.9	7.9	2.4	0.8	2.0	0.9	nd
Istye-14	69.2	15.5	6.4	0.6	1.8	4.7	1.3	nd

Figure 5. The experimental bloomery furnace. Photo: V. Zavyalov.

process was to enrich the ore by the roasting process. A layer of ore was placed on the row of wooden piles, which in turn was covered by piles. The roasting of the ore lasted 1-1.5 hours, until all the wood was burned off. Finally, the ore lost 7-10 wt. % of its original weight.

Our investigations have shown that the separation of gangue (silica and alumina) occurs at this stage. The chemical composition of the ore varied: if the raw ore contained about 30 wt. % SiO_2, then the burnt ore contained less than 25 wt. % SiO_2. The alumina (Al_2O_3) content shows a similar picture. This observation confirms the necessity of conducting such a preliminary operation as roasting ore (Kolchin and Krug, 1965, p.202), during which not only organic impurities were burned out and moisture was removed, but also a partial purification of the ore from silica and alumina took place.

Guided by the type of Ancient Rus furnace known from archaeological and ethnographic data (Kolchin, 1953, pp.26-31; Naumov, 2008), we built up an experimental model[2] on which we conducted several smelting processes using Istye ore (Figure 5). An electronic motor with the speed of 260 l/min was used as blower support. The ore/charcoal proportion was 1:1. The smelting processes lasted 2-2.5 hours and for the process 10-12 kg of ore were used. Unfortunately, no more than 1 kg of bloomery iron could be produced during the most successful experimental smelting processes.

The metallographic analysis of metal detected a structure of ferrite with disproportionate (big and medium) grains and a large amount of big slag inclusions. The measured microhardness of ferrite is 170-181 HV0,1. Metallographic analysis of the slag-iron conglomerate obtained during the experiments showed similarities with the composition of the archaeological artifact (Figure 4: 2). The chemical composition of experimental and archaeological slag-iron conglomerate was also similar. This is especially noticeable for such micro-impurities as phosphorus, calcium, potassium, and manganese. The experiments demonstrated that ore from the Istye ore deposit is suitable for the bloomery process.

Archaeometallographic investigations

It should also be mentioned that a considerable number of forged products were discovered in the settle-

Table 3. Summary of the metallographic information.

No.	Artifact	Carbon content (% C)	Phases present	Microhardness (DPH) (kg/mm²)	Technique
12173	Knife		Martensite	322-383	Forged from cemented steel, quenching
12174	Knife		Ferrite	135-151	Slant welding, hardened and tempered
			Sorbite	350-383	
12175	Knife	0.2	Ferrite	160-206	Carburization at the blade, hardened and tempered
			Ferrite and Perlite	160-193	
			Sorbite	383-420	
12176	Knife		Ferrite	193	V-shaped welding, quenching
			Martensite	514	
12177	Knife		Ferrite	160-170	Three-fold welding, hardened and tempered
			Sorbite	254-274	
12178	Knife	0.2-0.3	Ferrite	122-160	Forged from bloomery steel
			Ferrite and Perlite	160-170	
12179	Knife	0.2-0.3	Ferrite	122-151	Forged from bloomery steel
			Ferrite and Perlite	135-160	
12180	Knife	0.1-0.8	Ferrite and Perlite	151-221	Carburization at the blade, quenching
			Martensite	383-350	
12181	Knife	0.4	Ferrite	160-181	Carburization at the blade
			Ferrite and Perlite	236-254	
12182	Knife	0.2-0.4	Ferrite	143-151	Carburization at the blade, hardened and tempered
			Ferrite and Perlite	160-206	
			Sorbite	350-383	
12183	Knife		Ferrite	170-193	Forged from iron
12184	Knife		Ferrite	160-181	Forged from iron
12185	Knife	0.5-0.6	Ferrite	116-221	Carburization at the blade
			Ferrite and Perlite	128-193	
12186	Knife	0.4-0.5	Ferrite	128	Carburization at the blade
			Ferrite and Perlite	135-221	
12187	Knife		Martensite	322-350	Forged from cemented steel, quenching
12188	Knife		Ferrite	143-181	Forged from iron
12189	Knife		Ferrite	110-135	Slant welding, hardened and tempered
			Sorbite	350	
12190	Knife	0.3-0.4	Ferrite	170-193	Carburization at the blade, quenching
			Ferrite and Perlite	206-254	
			Martensite	383-514	
12191	Knife	0.2-0.8	Ferrite	143	Carburization at the blade, quenching
			Ferrite and Perlite	193-206	
			Martensite	236-274	
12192	Knife		Ferrite	221-254	Butt-welding, quenching
			Martensite	383-642	
12193	Knife	0.4-0.5	Ferrite	181-206	Slant welding
			Ferrite and Perlite	236	
12194	Knife		Ferrite	128-170	Forged from iron

No.	Artifact	Carbon content (% C)	Phases present	Microhardness (DPH) (kg/mm^2)	Technique
12195	Knife		Ferrite	151-206	Slant welding, quenching
			Martensite	274-383	
12196	Knife	0.2	Ferrite and Perlite	221	Forged from bloomery steel, quenching
			Martensite	274-350	
12197	Knife	0.2	Martensite	322-572	Forged from cemented steel, quenching
			Ferrite and Perlite	193-221	
12198	Knife		Sorbite	181-221	Forged from cemented steel, hardened and tempered
12199	Knife		Sorbite	274-322	Forged from cemented steel, hardened and tempered
12200	Knife		Ferrite	122-143	V-shaped welding, quenching
			Martensite	322-464	
12201	Axe	0.5-0.6	Ferrite	181-221	Carburization at the blade, quenching
			Ferrite and Perlite	160-181	
			Martensite	322-350	
12202	Fire-steel		Ferrite	181-193	Butt-welding, quenching
			Martensite	514-946	
12203	Fire-steel		Ferrite	128-160	Butt-welding, quenching
			Martensite	572-642	
12332	Knife		Ferrite	193-221	Butt-welding, quenching
			Martensite	572-946	
12333	Knife	0.2-0.4	Ferrite Ferrite and Perlite	181-221	Forged from bloomery steel
12334	Knife	0.1	Ferrite Ferrite and Perlite	135-170	Forged from bloomery steel
12335	Knife		Martensite	350-383	Forged from cemented steel, quenching
12336	Knife		Ferrite	135-151	Carburization at the blade, quenching
			Martensite	274-322	
12337	Knife	0.3-0.6	Ferrite,	128-160	Forged from bloomery steel
			Ferrite and Perlite	160-181	
12338	Knife	0.3-0.4	Ferrite	181-206	Carburization at the blade
			Ferrite and Perlite	193-221	
12339	Knife	0.3-0.4	Ferrite	122-151	Slant welding
			Ferrite and Perlite	206	
12340	Knife		Ferrite	181	Forged from iron
12341	Knife	0.2	Ferrite	128-151	Slant welding
			Ferrite and Perlite	151-160	
12342	Knife	0.3-0.4	Ferrite	221	Slant welding, quenching
			Ferrite and Perlite	236	
			Martensite	236-420	
12343	Knife		Ferrite	116-143	Forged from bloomery steel, quenching
			Martensite	274-350	
12344	Knife		Ferrite	181-206	Forged from iron
12345	Knife		Ferrite	170-181	Carburization at the blade, quenching
			Martensite	514	
12346	Knife		Ferrite	181-274	Forged from iron

ment. Their archaeometallurgical examination allows us to get an idea of the peculiarities of the development of local blacksmithing. Using a light microscope, 43 knives, an axe blade and two fire steels were examined (Table 3). All knives had ledges at the transition from blade to tang. The back of the knife is straight or slightly curved, the edge slightly curved towards the tip. The length of the blades are 6-10 cm, the length of the tangs are 3-5 cm.

As the metallographic investigation shows, the most common technique in the manufacturing of knives from the settlement of Istye 2 was carburization at the blade (11 artifacts) (Figure 6: 1). Most blades of this group (7 artifacts) are heat-treated (usually hardened and tempered). The carbon content in non-hardened specimens was 0.3-0.6 % (carbon content in quenched samples could not be determined). It should also be noted that carburization at the blade was rarely used in urban handicraft, which is evidenced by the analytical data that has accumulated up to today.

Six knives were forged from high-quality cemented steel (homogeneous cementation of the billet). All objects are heat-treated: three are hardened, and three are hardened and tempered.

Seven knives are forged from bloomery steel. The carbon content reaches 0.6 wt. % in some areas but in most cases it is 0.2-0.3 wt. %. Two blades in this group were heat treated (Figure 6: 2).

Seven knives are forged from bloomery iron (microhardness 170-181HV0,1). It should be noted that most of them were severely corroded or had a sharpened blade, which does not exclude the use of additional techniques to improve the working part in their manufacture.

One knife was produced at three-fold welding technology – steel core flanked by ferritic iron. This technology was typical for the 10th-11th century in Russian blacksmith craft. However, in the mid of the 12th century three-fold welding technology was changed to welding-on one. Moreover, the artifacts with three-fold welding blades were very rare among Russian implements after this time (Kolchin, 1953; Zavyalov, Rozanova and Terekhova, 2012).

The technology of welding a steel blade onto an iron base, which was predominant in the Ancient Russian art of forging, also took an important place in production. 11 investigated knives had welded-on blades. Eight blades have been heat treated.

Only a fragment of a blade represented the investigated axe, so it is impossible to say anything about the type of item. The axe was forged from bloomery iron with carburization at the blade and the axe edge was tempered (Figure 6: 3).

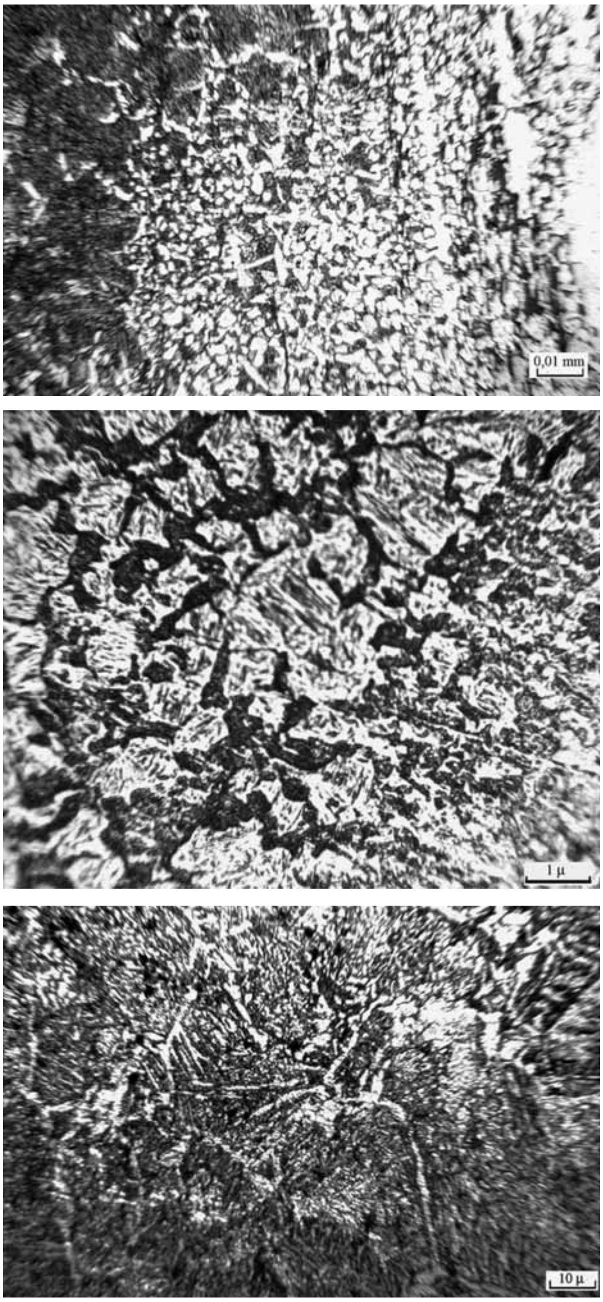


Figure 6. Microstructure photos of iron artifacts from Istye 2: 1 – an. 12191, knife, ferrite and perlite; 2– an. 12190, knife, martensite and troostite; 3– an. 12201, axe, widmannstätten lines. Photos: V. Zavyalov.

The fire-steels were made according to a uniform technological scheme: welding-on of the working part with subsequent quenching. One can note the high hardness of the hardened working part from up to 946 HV0,1.

Conclusion

Overall, the level of local iron production corresponded to a certain chronological stage in the development of Ancient Russian blacksmithery. However, nonetheless, the main form of production at the Istye 2 settlement was metallurgy, which, judging by production volumes, exceeded local needs. Thus, it follows to pose the question of the sale of the metallurgical products.

Considering that the capital of the Principality of Ryazan (Staraya Ryazan) was located in the immediate vicinity of the Istye 2 settlement (Figure 1), it was natural to expect that this was the most important destination for the iron products. Here were the consumers and traders of the iron produced in Istye, which was also easy to receive, and trade by the convenient connection to Staraya Ryzan from Istye 2 down the Istya and Oka rivers. Thus, over the course of our work on the basis of a specific Ancient Russian settlement, we gradually traced the metallurgical process from the processing and smelting of ore to the production of bloomery iron and its refinement by blacksmiths. In addition, we managed to localize a rural metallurgical complex that supported the handicraft production of a large city with its products.

Acknowledgement

Article was prepared with the financial support of the Russian Science Foundation, project 19-18-00144.

The authors would like to express their gratitude to dr. J. Hošek (the restoration laboratory of the Institute of Archaeology of the Academy of Sciences of the Czech Republic (Prague) for providing the opportunity to conduct analytical research using the X-ray fluorescence analyzer, and also express their sincere appreciation to experimental blacksmith M.A. Ratkin for his help and direct involvement in carrying out experiments to model the bloomery process.

Notes

1 The arshine and the sazhen are Ancient Russian measurements of distance. One arshine was equal to 0,7112 m, and one sazhen was equal to 2,1336 m.

2 The experimental furnace was composed of firebricks and had a height of 0,8 m and a diameter of 1,05 m, a shaft height of 0,65 m, lower diameter of 0,3 m and upper diameter of 0,2 m.

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