

INVESTIGATION OF ARCHAEOMETALLURGICAL FINDINGS FROM FELIX ROMULIANA LOCALITY

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Abstract

Remains of metallurgical activities have been discovered recently at the locality Felix Romuliana near Zaječar (Eastern Serbia), with a lot of slag occurrences and metal findings. Samples, taken from this locality, have been investigated using different characterization methods – chemical analysis, XRD and SEM-EDX, in order to clarify the Early Byzantine metallurgical activities at Felix Romuliana.

Key words: Archaeometallurgy; Characterization; Felix Romuliana locality

1. Introduction

Felix Romuliana (Zaječar, Eastern Serbia), well known as imperial fortress and palace of the Roman emperor Galerius, is dated to the end of 3rd and the first two decades of the 4th century A.D. [1,2] It is

also known as a church estate and shelter towards the end of the classical world (later half of the 4th and the first half of the 5th century; as an Early Byzantine settlement (from the middle of the 5th to the early decades of the 7th century); and as mediaeval town [2,3].

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Recently, after 50 years of systematic archaeological exploration, this famous locality showed one more aspect – as a potential archaeometallurgical locality, dated to early Byzantine period – the end of the fifth and first half of sixth century. Namely, large metallurgical object - a kind of smelting furnace (Fig.1), has been discovered in 2004 during the archaeological investigations at Felix Romuliana, together with a lot of slag occurrences and different metal findings. It is already archaeologically described [4,5] and preliminary archaeometallurgical investigations have been done last year [6,7]. In order to contribute to the better knowledge of Felix Romuliana history from the point of view of metallurgical activities in early Byzantine period, further physico-chemical characterization and investigation of archaeometallurgical findings from this locality were done and presented in this paper.

2. Experimental

Samples Numerous samples found in and near the furnace at Felix Romuliana, were used for the experimental investigation. The results of physical-chemical characterization of three characteristic samples - slag (S5), clay (S7) and coal (S10), are presented in this paper. The slag fragments were irregularly shaped, with numerous traces of the ground remnants. Photographs of typical samples are given in Fig.2.

Techniques For the experimental investigations presented in this paper, following experimental techniques were used: chemical analysis, XRD analysis and SEM/EDX analysis:

- Chemical analysis was done using standard procedure and optical emission spectrograph apparatus Jarrell-Ash with microphotometer (model 70.000)).
- X-ray diffraction analysis was



Fig.1. The smelting furnace discovered at Felix Romuliana

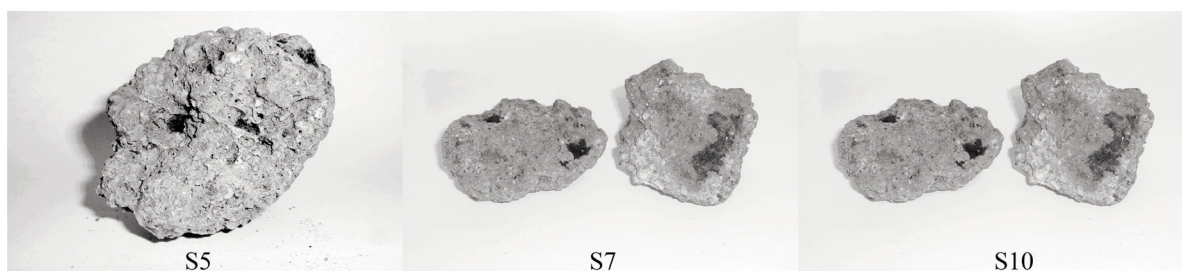


Fig.2. Photographs of some investigated samples from the Felix Romuliana locality

performed at Siemens apparatus with Cu-anticathode and Ni-filters, with 40kV and 20mA.

3. Results

Results of the chemical analysis - main components, done by standard procedure and optical emission spectrography for the investigated samples, are given in Table 1.

the sample of slag (S5), and the presence of plagioclase and quartz in the sample of clay (S7) - Fig. 3.

Figs. 4 and 5 show micrographs of the slag. The investigated piece of slag has elliptical shape in the cross-section (actually it is ellipsoid) with axes of about 15 and 10 – 12 cm, and it is 4 – 5 cm thick in the centre. Its mass is 1.8 kg. The lower part is homogeneous, the upper one uneven and

Table 1. Results of chemical analysis

a) standard chemical analysis

Component (%)	S5 sample (slag)	S7 sample (clay)	S10 sample (coal)
SiO ₂	33.20	61.94	10.15
Fe _{tot}	35.26	11.53	0.77
FeO	31.90	7.19	-
Al ₂ O ₃	4.86	5.27	2.02
CaO	5.69	7.05	50.26
MgO; TiO ₂ ; P ₂ O ₅	/	/	1.38; 0.045; 0.71
Cu; Zn; Bi; Ni; Pb; Mn	/	/	0.12; 0.037; 0.30; 0.12; 0.12; 0.062

b) optical emission spectrography

Component(%)	Cu	V	Ti	Cr	Pb	Mg	Mn
S5 sample (slag)	0.016	0.010	0.046	<0.003	0.0015	0.16	0.026
S7 sample (clay)	0.005	0.006	0.037	<0.003	0.0013	0.12	0.028

The X-ray diffraction analysis was used for the identification of the mineralogical composition of investigated samples, which showed the presence of quartz and fayalite in

partially porous. Lamellar phase in Fig. 4 represents fayalite $2(\text{FeO} \cdot \text{CaO}) \cdot \text{SiO}_2$ with the composition of 64 to 70 mass % FeO, 1 to 5 mass % CaO, 25 to 30 mass % SiO₂.

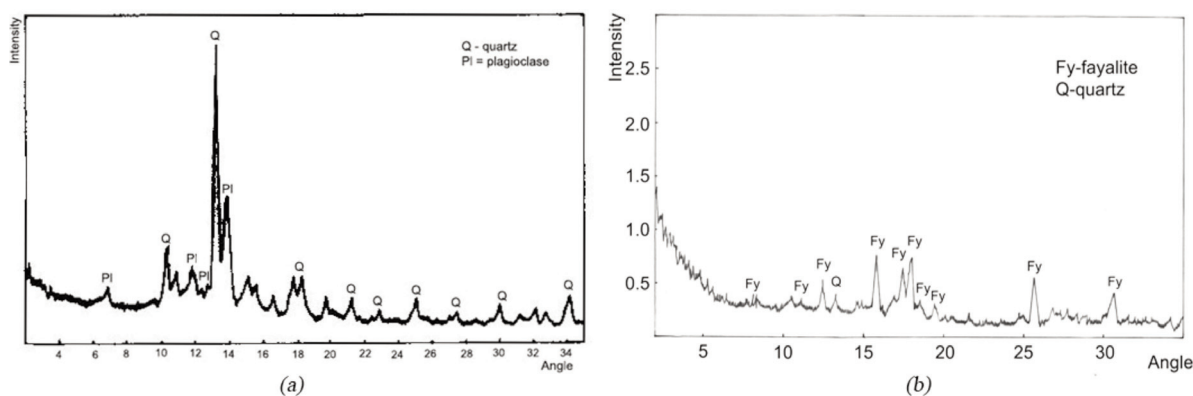


Fig.3. The X-ray diagram for the samples S5 (a) and S7(b)

Eutectic with glassy solidified phase, containing SiO_2 , Al_2O_3 , K_2O , FeO , etc, is found among the fayalite lamellae. Fig. 5 shows that there is also a great amount of wustite, FeO , next to fayalite. Wustite represents the primary phase of solidification that is followed by fayalite and eutectic. Melting point is in the interval of 1140 to 1200°C. This sample (as well as the other ones) contains only a very small amount of metallic iron. On single spots in the slag there are droplets with diameters up to 0.5 mm. The figure presents still another

independent phase (a darker one) that has composition of 55 to 60 mass % SiO_2 , 8 to 15 mass % Al_2O_3 , 25 to 25 mass % K_2O and 2 to 5 mass % potassium aluminosilicate [8, 9].

In regard to the sample shape it can be concluded that slag was collected on the bottom or it flew from the metallurgical reactor. A high percentage of potassium oxide and the glassy phase meant that melting point of slag was reduced by additions of fluxes. Slags compositions suggested that slag was formed during the reduction of iron ore or it was liquated

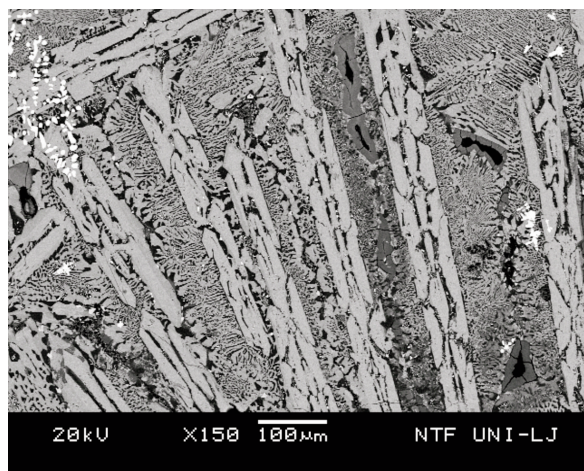


Fig.4. Micrographs of the slag with fayalite and eutectic

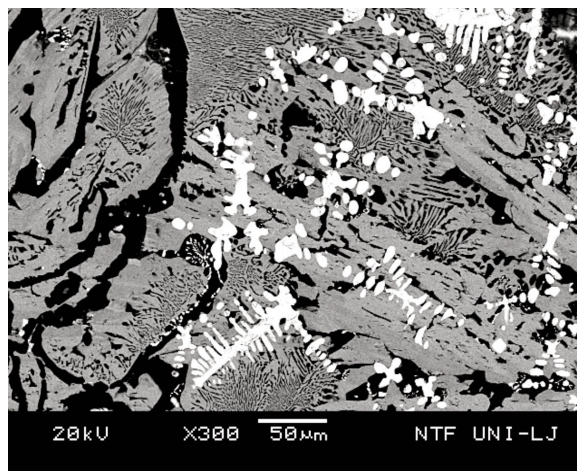


Fig.5. Micrographs of the slag with wustite, fayalite and potassium aluminosilicate

during the heating of loup in the forge hearth. Similar slag compositions are found also in forge welding. There was also a piece of flat iron among the slag samples that was completely corroded (rusty). Its oxide layer was composed of goethite and magnetite.

4. Discussion

Obtained results of the physico-chemical characterization of the archaeometallurgical findings from Felix Romuliana locality, point to the iron metallurgy.

The metallurgical treatment of an iron ore is basically reduction. At lower temperatures (800°-1300°C), the reduction process is possible in the semi solid state. It produces a piece of semi solid iron and fayalite slag. But reduce iron is poor in carbon, which must be consolidated by hammering, but can be immediately used. That is the “direct method”, which has been the normal way to produce iron in Europe until the medieval period [10], and most probably typical for the metal workshop in Felix Romuliana.

Several types of furnaces were used to produce iron by the direct method during the past [10, 11]. A major difference is the way the iron is separated from the slag during the operation. The slag can be tapped outside of the furnace or allowed to flow in the lower part of the furnace. More, some furnaces can be used only once and others are reusable several times, while the air supply can be provided by natural draught or using below. Also, the size and shape of the furnace can vary widely. In the case of smelting furnace discovered at Felix Romuliana, it may be supposed that the slag was tapped outside of the furnace and that mentioned furnace was reusable.

During the reduction of iron ore, a slag,

containing the impurities from the ore and the unreduced iron oxides, is formed. Also, the slag is formed during smithery process [10], by accumulation of different kinds of materials inside the hearth during the process or sand and clay, which may be put on the metal to prevent oxidation and decarburization or to clean the surfaces before welding. Finally, the charcoal and the other fuels give ashes that can be partly incorporated into the slag. Very large accumulations of slag are found in the areas of intensive production, which is true in the case of Felix Romuliana metal workshop, also.

As furnace or metal workshop remains are usually badly damaged and difficult to investigate without heavy archaeological work on the field, slag is the most accessible material to study ancient metallurgy. It is very important to state that the furnace remains exist, too, in the case of Felix Romuliana. This could be very significant for further detailed exploration and explanation of technological processes done at this site during the early Byzantine period.

5. Conclusions

According to obtained experimental results of the physico-chemical investigations, following conclusions about the metallurgical activities at the Felix Romuliana in early Byzantine period could be made:

- Significant remains of the smithy shop or smelting furnace as a part of a large metallurgical object have been discovered, recently. Numerous slag fragments and metal objects were found at investigated site.

- The conditions for metallurgical activities at this locality were good, due to

the vicinity of water and presence of woods.

- Iron metallurgy is proven based on present content metallic iron, magnetite and wüstite and low content of non-ferrous metals (Cu, Ag, Pb) in the investigated samples.

- If reduction process was done at lower temperatures.

- It could be supposed that smelting process was done with presence of different fluxes, using wood and charcoal as the fuel.

- Great porosity of the investigated slag samples may be due to the foaming of primary slag during the reduction.

- According to the fact that phosphorus and sulfur have not been determined in the samples, it can be supposed that rich iron oxide ore and very clean, related to impurities. For direct reduction the reach iron ore was used as the raw material.

- Having in mind that there are occurrences of oxide iron ores, especially limonite, in the vicinity of this locality, one may conclude that local ores were used for the production, as was suggested for some other archaeometallurgical sites in this region [12]. Also, there are references in literature [13] about processing of limonite ores in Majdanpek, in antique period, which confirm that conclusion.

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References

1. Roman imperial towns and palaces in Serbia, Ed. by D. Srejšović, Serbian Academy of Sciences and Art, Belgrade, 1993. *(in Serbian)*
2. D. Srejšović, A. Lalović, Felix Romuliana, Center for archaeological research (Faculty of Philosophy, University of Belgrade), Belgrade, 1995.
3. M. Živić, Felix Romuliana – 50 years of solving, National Museum Zaječar, 2003.
4. S. Petković, M. Živić, Early Byzantine ironworking object at the site Gamzigrad-Romuliana in Eastern Serbia, International Symposium - Metallurgy of the Southeast Europe from ancient times till the end of XIX century, Sozopol (Bulgaria), 2005, Reports, p.101.
5. S. Petković, M. Živić, Razvitak, XLV (221-222) (2005) 38. *(in Serbian)*
6. D. Živković, N. Štrbac, M. Cocić, M. Šteharik, Z. Stević, B. Marjanović, International Symposium - Metallurgy of the Southeast Europe from ancient times till the end of XIX century, Sozopol (Bulgaria), 2005, Reports, p.125.
7. D. Živković, N. Štrbac, Razvitak, XLV (221-222) (2005) 43. *(in Serbian)*
8. Slag Atlas Verlag Stahleisen GmbH, Düsseldorf (1995)
9. J. Lamut, Unpublished papers
10. R.F. Tylecote, Metallurgy in archaeology, Edward Arnold Publishers Ltd., London, 1962.
11. P. Crew, P., Historical Metallurgy, 25 (1) (1991) 21.
12. R.F. Tylecote, Metallurgy in archaeology, Edward Arnold Publishers Ltd., London, 1962.
13. V. Simić, Historical development of our mining, Scientific Book, Belgrade, 1951. *(in Serbian)*