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PHYSICO-CHEMICAL CHARACTERIZATION OF SLAG OCCURRENCES AT TILVA NJAGRA LOCALITY (BOR REGION, EASTERN SERBIA)

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Abstract

Physico-chemical characterization of slag occurrences at Tilva Njagra locality (Bor region, Eastern Serbia) was done and the results of the research activities are presented in this paper. The samples were investigated using chemical analysis, XRD, DTA and EDXRF.

Keywords: archaeometallurgy, physico-chemical characterization, Tilva Njagra locality

1. Introduction

Bor region (Eastern Serbia) is well known for mining and metallurgical activities from ancient times to nowadays [1-3]. Recognostic investigation of the terrain at Tilva Njagra locality, about 7-8km from village Zlot and 4km from Brestovačka Spa (Bor region,) was done recently. Some tumulus have

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been discovered at both sides of the old road from Brestovačka Spa to Zlot (see Fig.1), and also at the locality Tilva Njagra, where a lot of different findings were found – ceramic, animal bones and also, a certain amount of slag. Near this site, a mining hole of about 20m length was found, as well as numerous slag occurrences.

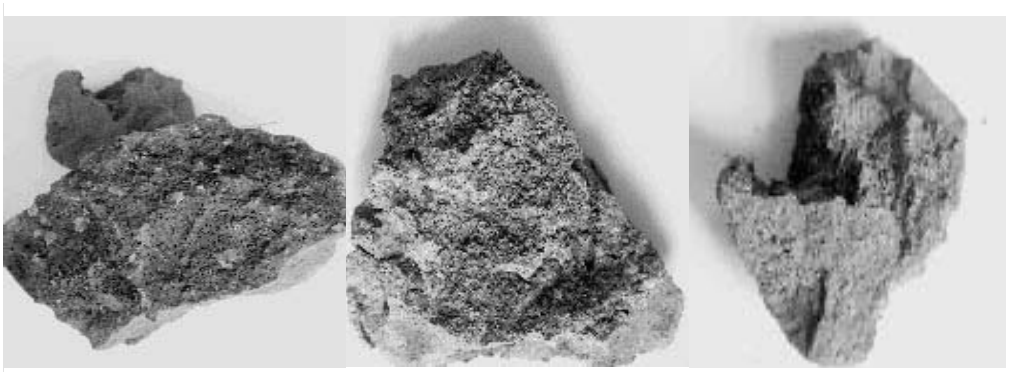
Fig.1. A view on one of a tumulus found at Tilva Njagra

Since physico-chemical characterization of slag occurrences may give very important information about the type of metallurgical activities and used technology [4], the samples from Tilva Njagra locality were examined and the results of the research activities are presented in this paper.

2. Experimental

Samples Three typical samples (TNJ-1, TNJ-2, TNJ-3) of slag origin from Tilva Njagra site were used for the experimental investigation. Photographs of investigated samples are given in Fig.1.

Slag samples could be macroscopically described as monolithic, possessing compact structure, with not so expressed porosity. Gray, metallic-gray, with red and yellow color details, was noticed at the fractures of samples, as well as slight brightness. The slag fragments were of about 5-10cm in size, with traces of the ground and no remnants of charcoal and flux.



TNJ-1 TNJ-2 TNJ-3

Fig.2. Photographs of investigated samples (TNJ-1, TNJ-2, TNJ-3) from Tilva Njagra site

Techniques For the experimental investigations presented in this paper, following experimental techniques were used: chemical analysis, XRD analysis, EDXRF spectroscopy, thermal analysis and optical microscopy.

Chemical analysis was done using optical emission spectrograph apparatus Jarrell-Ash with microphotometer, model 70.000).

X-ray diffraction analysis was performed at Siemens apparatus with Cu-anticathode and Ni-filters, with 40kV and 20mA.

Energy dispersive X-ray fluorescence spectroscopy was done at Canberra apparatus, using Cd-109 (22.1 keV, activity 740 MBq) radio isotope for the excitation.

DTA-TG-DTG analysis was performed at Derivatograph MOM (Budapest, Hungary) with heating rate of 10^{o}/min up to the maximum temperature of 1000^{o}C.

3. Results and discussion

Results of the chemical analysis, done by optical emission spectrography for the investigated samples (TNJ-1 – TNJ-3) from Tilva Njagra site, are given in Table 1.

Table 1. Results of chemical analysis - samples TNJ-1 – TNJ-3(in wt%)*

Sample	Ti	Mn	Ca	V	Pb	Mg	Cu	Na
TNJ-1	0.300	0.013	0.074	0.015	0.010	0.070	<0.1	identified
TNJ-2	0.200	0.010	0.100	0.045	0.0055	0.080	<0.1	"
TNJ-3	0.400	0.015	0.078	0.010	0.0022	0.078	0.007	"

** Presence of Fe, Si, Al determined as macrocomponents*

Typical X-ray diffraction diagram for the sample TNJ-1 is shown in Fig.3, while DTA- DTG curves for the same sample are given in Fig.4.

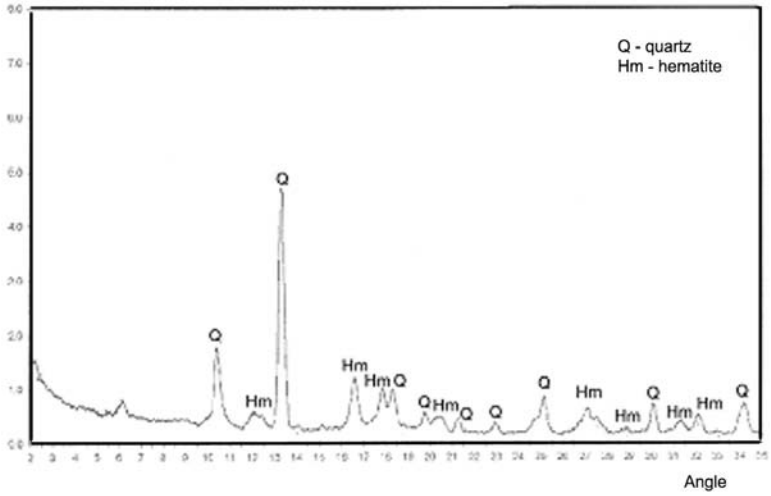


Fig.3. X-ray diffraction diagram (sample TNJ-1)

According to obtained XRD recordings, identification of mineralogical phase composition was done and main presence of hematite (Fe_2O_3) and quartz (SiO_2) was determined in investigated sample TNJ-1, as well as for the other samples.

Results of thermal analysis, given for the sample TNJ-1, show one endothermic peak at 245^{o}C, which occurs without mass change at TG curve and probably correspond to the phase transition of the present Ti_2O_3 [5].
EDXRF spectra obtained for the samples TNJ-1 is presented in Fig.5, while the results of the semi-quantitative analysis are given in Table 2.

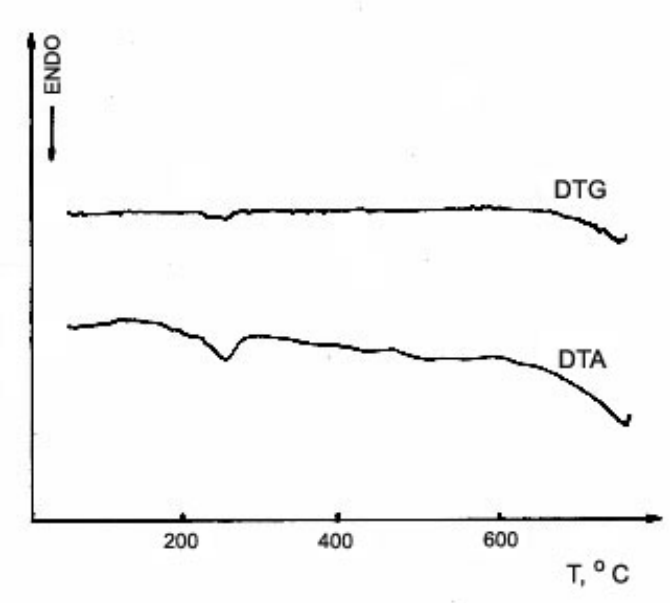


Fig.4. DTA -DTG curves at 10°/min (sample TNJ-1)

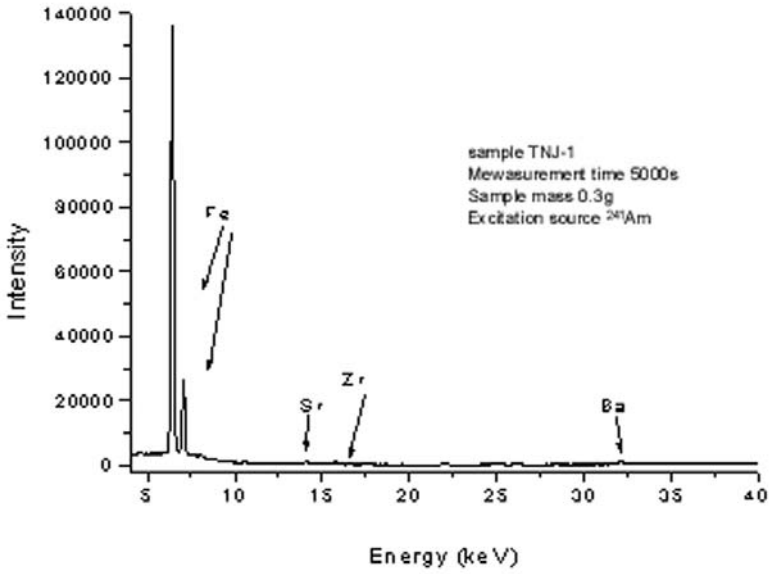


Fig.5. EDXRF spectra of the sample TNJ-1

Table 2. Results of the semi-quantitative analysis

Element	Sample TNJ-1	Sample TNJ-2	Sample TNJ-3
Fe	34,46 % wt	37,42 % wt	6,14 % wt
Ti	0.82 % wt	0,85 % wt	0,52 % wt
Cu	102 mg/kg	208 mg/kg	105 mg/kg
Sr	167 mg/kg	156 mg/kg	83 mg/kg
Ba	133 mg/kg	191 mg/kg	161 mg/kg

4. Concluion

According to the results of the experimental investigations obtained by presented techniques, following conclusions could be made:

- iron metallurgy activities could be proven based on the high content of iron (in the form of hematite) and low content of non-ferrous metals (Cu and Pb) in the investigated samples;
- it could be supposed that smelting process was done with presence of SiO_2 and, eventually, CaO as fluxes;
- the reduction process was done at lower temperatures, since presence of fayalite has not been determined in the samples;
- porosity noticed in the investigated slag samples may be due to the separation of gas, formed by combustion of wood and charcoal from the melt; and
- according to the remnants of the ground in slag samples, the slag was probably poundered from furnace to the ground directly.

Presented results and conclusions, obtained for the site Tilva Njagra (Bor region), may present a contribution to a better knowledge of early metallurgical activities in Eastern Serbia, from the archaeometallurgical point of view.

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