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ED-XRF analysis of silver and gold jewelry found in Medieval necropolis Crkvina, Donja Zgošća (Kakanj municipality), BiH

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Abstract: During archaeological campaigns in 2010, 2012 and 2015 at the Medieval necropolis Crkvina, in central Bosnia, beside skeletal remains, pieces of jewelry, ceramic, construction material, fabric and small artefacts from different periods were also found. The most important finding was a signet ring, with the lily motif, believing to belong to Bosnian king or some high class nobility from Medieval period. Beside the ring, other richly decorated silver and gold jewelry was also found. The goal of this study was, for the first time, to provide information about composition of the most interesting artefacts and also to enhance knowledge about Medieval jewelry industry in Bosnian Kingdom. Results showed that the majority of jewelry was made from silver, of highest quality, with gold being used for surface gilding. Higher than usual copper content in some of the rings (more than 3% in weight percentage) was possibly added intentionally, to enhance properties of the silver. One of the rings, without any decorations was proved to be brass made, with zinc content as high as almost 20%. Results of this study contribute to the better understanding of the otherwise previously scarce knowledge of jewelry tradition in Medieval Bosnian Kingdom.

Keywords: ED-XRF, chemical analysis, Medieval jewelry, silver rings, gold, Medieval Bosnia

Introduction

Throughout the history, gold and silver were always considered a precious materials and were symbol of high status, almost exclusively reserved for nobility and rich merchants. Archaeological findings from prehistoric times up to most recent past show that these metals were used for making a variety of objects, from rings, crowns, earrings, necklaces, up to death masks. Use of gold or gold/silver combination for craftsmanship of death masks is recorded throughout world, with specimens from Philippines, Near East, Greece, central Balkans, Hungary and even pre-colonial South America societies¹. Most famous one is

probably death mask of Tutankhamun. A symbolism of gold and silver in burial contexts is linked to their properties, as being resistant to corrosion and degradation and thus they were perfect gift for the eternal life.

Small objects made from gold and silver were also placed with the deceased, and mostly included rings, earrings or small tokens. Findings of such objects points out to a person being a wealthy one during lifetime, often from the high class. In Medieval Bosnia and Herzegovina (BiH) people were buried under the impressive stone monuments called *stećci*, which came in different shapes and sizes, often richly decorated with scenes from everyday life, hunting or profound inscriptions describing life and after-life. Individual *stećci* and small- or large-scale necropolis can be found throughout whole BiH,

¹ Taylour 1994, 168-189; Shimada et. al. 2000, 28-61; Whitaker 2006, 284; Ilijeva / Penkova 2009, 195-199; Estrella 2014, 230; Fodor 2014, 119.

but also in Montenegro, Serbia and Croatia, in areas bordering present day BiH and which in the Middle Ages were part of then Bosnian Kingdom.

The most famous stećak is probably Zgoščanski stećak (Stećak from Zgošća), found in village Zgošća, Kakanj municipality, in Central Bosnia, just few kilometers from the medieval capital of BiH, Bobovac. It was discovered in 1891, during construction works and in 1913 it was relocated to the botanical garden of National Museum of BiH in Sarajevo, where it still stays. During archaeological campaigns in 2010 and 2012 at the Zgošća, new stećci were discovered, with skeletal remains underneath, some of them with grave goods such as small hoops, rings, buttons and golden brocade threads. All of this suggested that this place was used for inhumation of nobility and possibly king itself and his family.

Medieval necropolis Crkvina

The exact local toponym of the location is Crkvina (in most of the Slavic languages it means *church* or *place* where church was before), just where the river Zgošća exists its canyon (coordinates: 34TBP7047891551). Nearby archaeological locations include Stjepangrad or Crnač bronze age hillfort (3 km north of the Crkvina; coordinates: 34TBP7099993765) and another medieval necropolis, Zagrađe (some 4 km north-east from Crkvina; 34TBP7214493966). Location of Crkvina and surrounding archaeological locations is shown on Google Image capture (Figure 1). Both of these locations are on prominent ground elevation, with great visibility towards river canyon and access roads in the valley, making them great strategic points in the past.

Stratimirović² in his paper from 1891 was the first one to present scientific and literature data about Zgoščanski stećak where he described its ornaments and possible interpretation of motifs carved in stone, together with photos and geographical area where it was found. He also proposed origin of the stone material used for the stećak, and that was limestone, possibly from one of the two neighboring quarries, used in the Middle Ages. Same author proposed more detailed clarification of motifs, 35 years after initial discovery. Based on the two letters still

visible, topography of place and richness of motifs, he concluded that beneath stećak member of Middle Age Bosnian nobility was buried, most probably ban Stephen II (died in 1353)³. From its initial discovery and until today, location went through major changes, which almost completely destroyed it, mostly due to the buildings being erected⁴. During excavations in 2010 and 2012, a total of 28 stone monuments and accompanying graves underneath them were found.

It is important to note that during previous and excavations in 2010 and 2012, beside medieval layer, prehistoric and antiquity material was also found. Prehistoric material includes fragment of ceramic and bronze needle⁵. Antique material included tombstones (dating to III CE)⁶ and red fragments of ceramic and bricks⁷. A possibility of church existence was proven before, from the excavations in 1948, when walls and foundations of building were found, dating to the antiquity period⁸.

Samples and chemical analysis

For this paper, a total of eight samples, of different types of jewelry were chosen for analysis by non-destructive technique, energy dispersive fluorescence of X-rays (ED-XRF). All samples were generously conceded by Kakanj Museum, where they are publicly displayed. They were recovered during archaeological campaigns in 2010, 2012 and 2015 at the Crkvina location. During archaeological campaigns in 2010 and 2012, beside material that was analysed in this paper, additional jewelry material was also found. It included few silver earrings, two more silver buttons and another metallic ring, probably made of silver. For testing we have chosen the material that was of the best quality, in the good state of preservation and that was most interesting for analysis, for the scientific community and the broader public. It is worth mentioning that finding of these artifacts, especially signet ring with the lily motif, was a major news for days in local and state newspapers and online.

³ Stratimirović 1926, 45-46.

⁴ Fekeža-Martinović 2014, 28.

⁵ Ibid, 36.

⁶ Sergejevski 1948, 171-172.

⁷ Fekeža-Martinović 2014, 36.

⁸ Čremoški 1949/50, 411-416.

² Stratimirović 1891, 122-141.

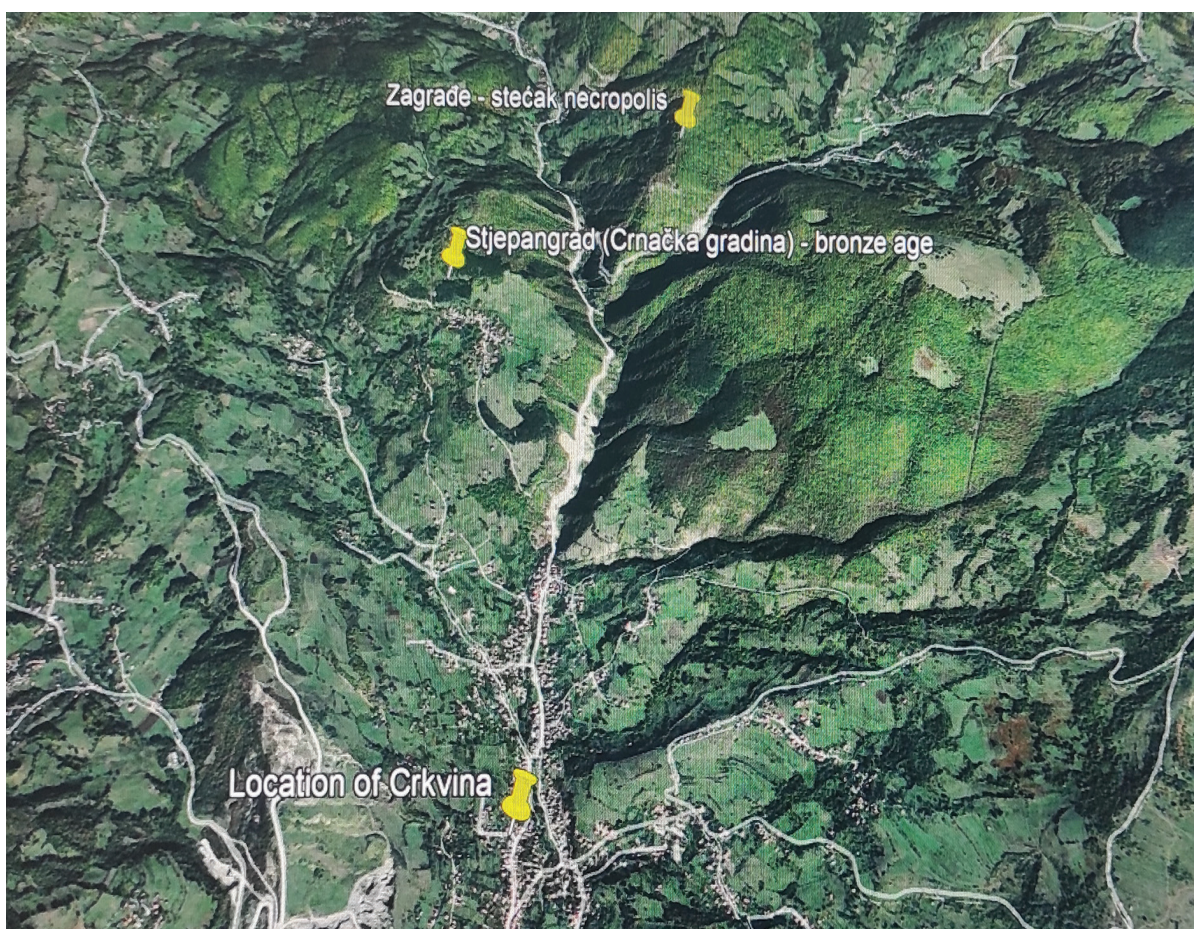


Fig. 1. Google Earth image showing location of Crkvina (original location of Zgošća stećak) and neighbouring archaeological locations

Table 1. shows short description of type of jewelry and where it was found at the location during excavation. Figures 2. and 3. show jewelry samples that was used for the analysis. Samples were analysed by energy dispersive X-ray fluorescence spectrometry (ED-XRF) technique, which is non-destructive technique, commonly employed for the analysis of precious artefacts, especially ones containing gold and silver⁹. ED-XRF only uses information gathered from the surface of material, without causing any damage to the sample. XRF radiation occurs when high-energy photons emitted from an x-ray source (primary x-rays) come into contact with a specific material (analyte), after which the photons cause excitation in the inner shells of the electron. The consequence of this excitation is the appearance of secondary radiation - fluorescent radiation. The elements present in the sample will emit

fluorescent radiation of discrete energies that are characteristic of these elements. This technique is fast, accurate and usually requires very little sample preparation before analysis.

In that way, ED-XRF has become a standard technique in archaeometry due to its non-invasive and non-destructive nature and the data collected with it can be used for studying manufacturing processes, provenance of raw materials, and geographical distribution of ancient mints¹⁰.

Since all the samples were immediately cleaned and conserved upon excavation by expert museum conservator and restorer, it was not possible to do any additional treatment and preparation for the ED-XRF analysis. Only thing that was done prior the analysis was to gently clean the surface with the cotton cloth, to remove any potential dust and minor particles, that were accumulated during exposure in museum glass showcases.

⁹ Brown / Schweizer 1973, 175-192; Del Hoyo Meléndez et al. 2015, 6-16; Mozgai et. al. 2021, 1-20.

¹⁰ Del Hoyo Meléndez et al. 2015, 6.

Table 1. *Instrument type used in the analysis and conditions employed during the analysis*

Instrument type	Oxford instruments X-Strata 920
X-ray tube	Coolidge tube with a voltage of 50 kV and current strength of 1 mA.
Detector	Multichannel proportional counter filled with inert gas (Xe) and is based on counts per second (CPS)
Element range	Titanium 22 to Uranium 92

Table 2. *Artefacts found during excavations in 2010, 2012 and 2015 that were analysed by XRF technique and their short description (according to Fekeža-Martinović, 2014)¹¹*

Sample number/type	Place inside grave
1 – Signet ring	Inside grave 9. Ring was made by techniques of casting, engraving and repoussage. With the simplified motif of lily, used as a signet ring
2 – Silver ring	Within grave 13, containing remains of child, just off the tibia (2010 excavation)
3 – Gilded silver ring	Inside disturbed grave (grave 9) without cover stone plate, with bones scattered. Ring was made by techniques of casting, engraving and repoussage
4 – Silver ring with glass paste	Found during excavation of Crkvina in 2015 campaign*
5 – Bronze circlet	Bronze circlet, found inside grave 21, which was close to grave 9
6 – Silver button	A pair of buttons, found inside grave 9. Made with casting method
7 – Silver earring	Above the profile of the grave 13 (2010 excavation)
8 – Yellowish earring	Grave 36, which was partially destroyed. Female person, roughly 25 years old (2010 excavation)

*This data was confirmed by mr. Ajla Sejfuli, from Travnik Museum, who was involved in 2015 excavation

Instrument type and measurement conditions

The instrument was calibrated by the manufacturer xcalibur (<https://xcaliburxrf.com/precious-metal-alloys-nist-traceable/>) using certified standards for Au, Cu, Ag, Zn and Ni.

Figures 2 and 3 show jewelry that was analysed. All photos were taken with permission and the courtesy of Kakanj Museum.

Results and discussion

Results of the analysis of archaeological material are presented in the Table 3.

Interpretation of results was aggravated by the fact that this type of analysis was never performed in BiH, so comparison with other results was not possible. Only general conclusions could be made, which still can provide a lot of useful information about goldsmith tradition in Medieval BiH.

Since the most abundant precious metal in the samples was silver, it is worth mentioning a

couple of things regarding its origin and use in Medieval BiH. Whole region of Bosnia is rich with argentiferous ores, in large quantities¹². Region of Eastern Bosnia, around Srebrenica, bordered with rivers Drina and Jadar and mountains Kvarc, Staroglavica and Ludmer is known for especially rich ore deposits, where one ton of ore gives roughly 8% zinc, 6% lead and over 100 grams of silver¹³.

The results for the sample 1, a signet ring with the lily motif is probably most interesting for the broader public and of archaeological and historical importance. Results showed that the ring is made of silver, with gold plating. Since no copper was detected in this sample, the most likely assumption for the origin of the original alloy used in the making of the ring is that the alloy itself is of natural Au-Ag origin¹⁴.

Sample 2 is most probably sterling silver type of jewelry, since it contains almost 94% of silver and the rest is copper, added to enhance mechanical properties of the ring¹⁵. Copper

¹² Kovačević 1961, 142.

¹³ Škegro 1998, 92.

¹⁴ Magnavita / Krauss 2019, 422.

¹⁵ Marchand et al. 2014, 1; Wołoszyn et al. 2019, 458.

¹¹ 11 Fekeža-Martinović 2014.

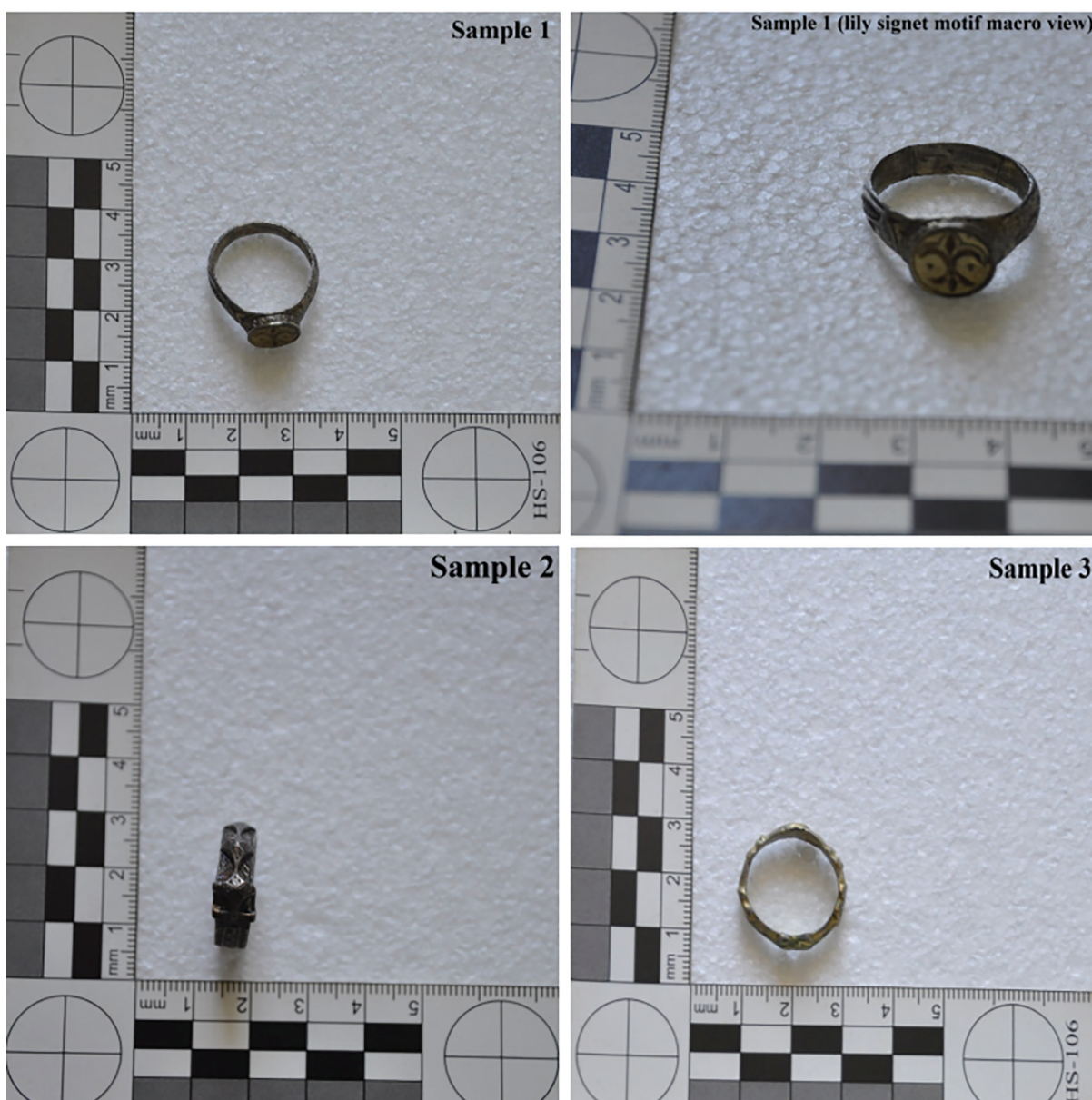


Fig. 2. Photographs of samples 1, 2 and 3, with scale (photos taken by Almira Kevilj-Olovčić)

addition is common goldsmith and metallurgical way to decrease melting point of silver and it is also used as a joining material or filler in silver products¹⁶. Amount of copper above 2.6% suggests intentional addition of this metal to the silver products¹⁷.

As for the sample 3, it is similar to sample 1, when it comes to precious metal composition on the surface of the sample. However, for the sample 3 we additionally measured part that was damaged with a scratch, so we could analyze

deeper into the sample, i.e., to exactly quantify the silver content in the ring. The results showed that it is composed almost completely from the silver, as high as almost 95%. Low copper, zinc and gold content indicate that silversmithing techniques employed in the Medieval Bosnia were at their peak and craftsmen were able to make jewelry of the finest possible quality.

Sample 4 is ring found during 2015 archaeological campaign, without any details or motifs, but with the inserted yellow glass paste. Although this ring lacks details that adorn other rings from this location, it is nevertheless valuable specimen

¹⁶ Magnavita / Krauss 2019, 420.

¹⁷ Ibid.

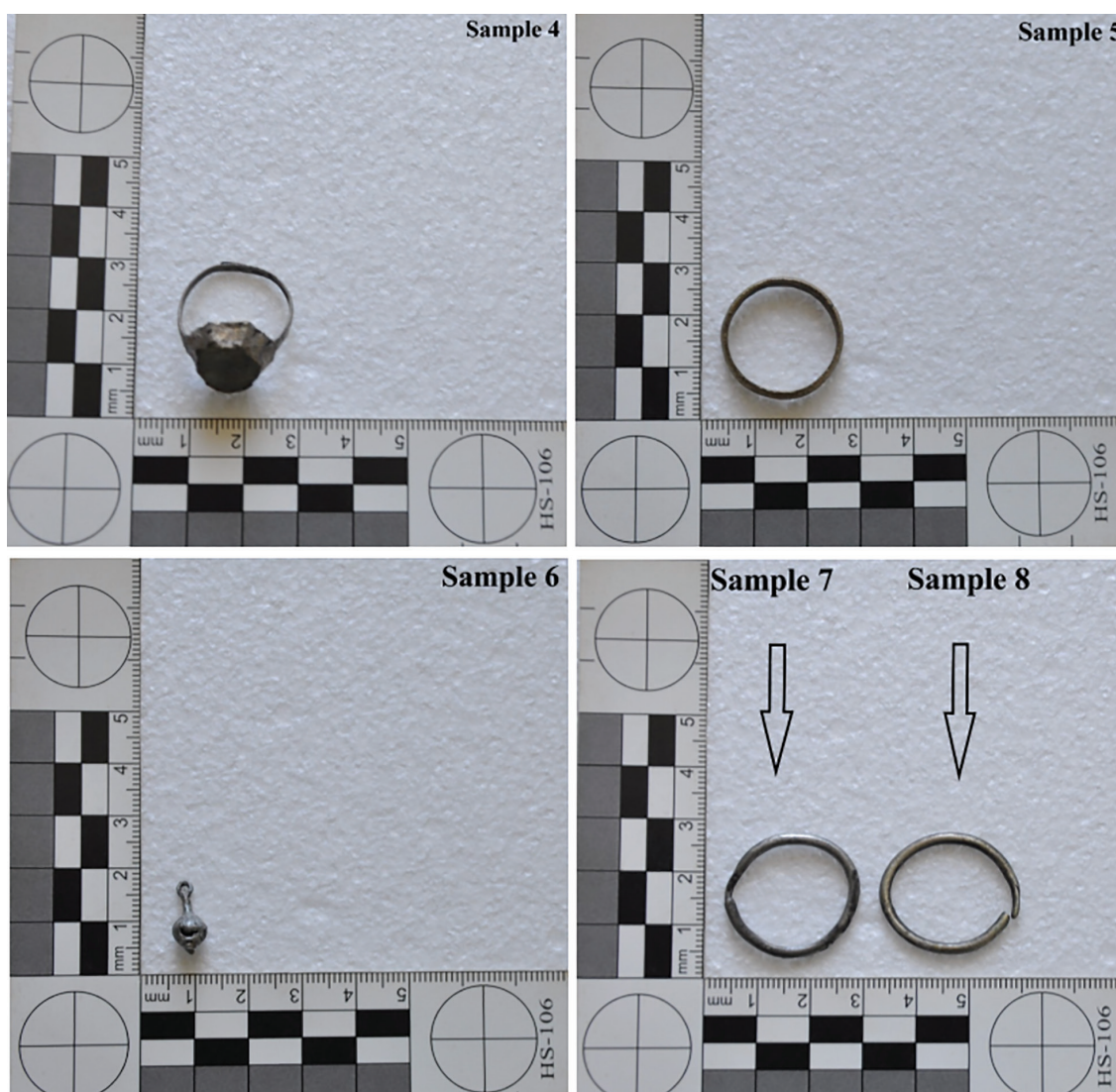


Fig. 3. Photographs of samples 4, 5, 6, 7 and 8, with scale (photos taken by Almira Kevilj-Olovčić)

since its silver content is highest of all analysed items and goes up to 95.39%.

Although sample 5 was considered to be made of bronze, during archaeological excavation, analysis showed different results. Visual inspection before analysis assumed it was perhaps some other copper based alloy, possibly brass, which contains zinc, unlike bronze that contains tin. Both of these alloys have different visual appearance, with brass being yellow and bronze having distinctive green or greenish patina. Another difference between these two alloys is brass being more corrosion resistant and will, if other environmental conditions are favourable, keep his distinctive golden colouring. Results confirmed that the material for ring is brass indeed, with high content of zinc, 19.16%. When it

comes to archaeological typology of copper alloys, they are classified by the content of alloying metals (tin, zinc and lead), with brass being the one having more than 8% of zinc¹⁸

During excavations in 2012, two pairs of silver buttons were also found. One of those buttons was chosen for analysis (sample 6). Results showed it was composed from combination of gold and silver, with high silver content of 77.23%. As it is case with other samples with copper content around 3%, it is clear that this metal was added on purpose to enhance mechanical properties of buttons during production.

Samples 7 and 8 were earrings, found in different graves, during 2010 archaeological campaign. Visually, they showed different characteristics, in

¹⁸ Pollard et al. 2015, 698.

Table 3. Results of the analysis by ED-XRF technique (values presented in weight percent, %)

Sample	Gold (%)		Silver (%)		Copper (%)		Zinc (%)
1		42.99		54.43		0.05	2.52
		37.18		59.91		0.00	2.90
		46.33		50.79		0.00	2.88
	$\bar{x}$	42.16	$\bar{x}$	55.04	$\bar{x}$	/ $\bar{x}$	2.77
2		0.30		94.69		5.01	n.d.
		0.08		92.78		7.14	n.d.
		0.23		94.05		5.72	n.d.
	$\bar{x}$	0.20	$\bar{x}$	93.84	$\bar{x}$	5.96	$\bar{x}$ /
3		63.16 (2.11)		34.75 (94.53)		0.37 (3.36)	1.72
		54.62 (2.14)		41.47 (94.30)		2.46 (3.56)	1.46
		68.94 (2.04)		28.81 (94.59)		0.69 (3.37)	1.56
	$\bar{x}$	62.24 (2.10)	$\bar{x}$	35.01 (94.47)	$\bar{x}$	1.17 (3.43)	$\bar{x}$ 1.59
4		0.64		96.70		2.66	n.d.
		0.78		94.81		4.41	n.d.
		0.84		94.65		4.51	n.d.
	$\bar{x}$	0.75	$\bar{x}$	95.39	$\bar{x}$	3.86	$\bar{x}$ /
5		0.00		0.19		81.87	17.94
		0.00		0.18		79.96	19.86
		0.00		0.20		80.12	19.68
	$\bar{x}$	/ $\bar{x}$		0.19	$\bar{x}$	80.65	$\bar{x}$ 19.16
6		20.61		76.22		3.17	n.d.
		19.36		77.43		3.21	n.d.
		18.79		78.03		3.17	n.d.
	$\bar{x}$	19.59	$\bar{x}$	77.23	$\bar{x}$	3.18	$\bar{x}$ /
7		2.07		93.20		4.74	n.d.
		2.11		93.19		4.70	n.d.
		2.11		93.22		4.67	n.d.
	$\bar{x}$	2.10	$\bar{x}$	93.20	$\bar{x}$	4.70	$\bar{x}$ /
8		1.90		89.09		9.01	n.d.
		1.88		89.10		9.02	n.d.
		1.83		89.23		8.94	n.d.
	$\bar{x}$	1.87	$\bar{x}$	89.14	$\bar{x}$	8.99	$\bar{x}$ /

n.d. analysed element not detected in the sample; $\bar{x}$ represents mean arithmetic value for three measurement repeats and this value was used in the discussion of results; sample 3, bold italic values in bracket represent results for analysis of the scratched part of the ring

terms of coloration. This difference in coloration could possibly be explained by the difference between content of silver and copper in earrings. Earring that was sample 7 had silverish appearance, with the silver and gold content more than the yellow earring (sample 8). Also, copper content in yellow earring was almost twice as much as in silver one. In both of the earrings no zinc

was detected. Since no data for exact chemical composition of medieval jewelry from BiH is not available at time, we tried to find analogies in neighbouring countries. Only available data were for the silver earrings from Medieval Serbian capital, Ras or Trgovište, near Novi Pazar. Although earrings were of different type, the crescent one, with more pronounced decorations, analogy is

important since these earrings had silver content over 90%; one earring had silver content ranging from 94.9 to 96.7% and other one had silver content ranging 95.8 to 97.2¹⁹.

As for the zinc, it was found in three out of eight samples. If we put aside possible deliberate addition of zinc in sample 5, in the other two samples zinc was admixture, that occurred in the original material. This isn't anything unusual, since ores of zinc, silver and copper very often come together, in the same ore veins.

Another important fact regarding archaeological metal objects is the occurrence of the surface enrichment effect, which generally means that the content of certain materials on surface and inside differs. Possible explanation of this is the oxidation of silver and copper, with the extrication of sulphide components²⁰. In those cases, surface layer can be removed with careful cleaning and polishing. This effect is studied broadly by different authors and described in papers²¹. But what if we have a precious artefact, one of a kind, where removing and polishing of outer surface could cause damage to the artefact itself? The only possible solution would be to analyse parts where there are cracks or damages, that were caused by environmental factors, prior the excavation. Of course, that is not always the possibility, but in our case, we had one artefact (sample number 3), where there was a damage of gilded surface part and we could analyse deeper into the bulk of the ring. As we have already discussed this sample where it was clear that the silver content of the bulk of the ring is almost 95%, the results are close enough to the content of silver in other artefacts in this paper. So, in this particular sample possibility of surface enrichment could be excluded.

As we have mentioned earlier in the text, it was a challenge to compare our results with same or similar findings, since literature data and research are either unavailable or non-existent. So, we tried to focus more on the inter-relationships between findings itself and metals they were made of. We showed that one of the rings is not bronze but made of brass. Also, we pointed out that difference between even few percent

of some component can make great impact on visual appearance and structure of the same type of artefact.

In the end, as a next step it would be good to provide analysis of similar material from other Medieval locations in BiH, coupled with analysis of metal content in original ores from the areas they were exploited from during Medieval times. Only in that way we can gain a better picture of goldsmithing and silversmithing tradition in BiH during Medieval period.

Acknowledgements: We would like to express our gratitude to the Institute for Metrology of BiH, for providing XRF instrument for analysis. Also, to Kakanj Museum and its director Mirza Mušija for opportunity to bring artefacts from Kakanj to Sarajevo and analyse them. Big thanks go to fellow archaeologist colleagues, Edin Bujak and Adisa Lepić for providing much needed literature for this paper. And thanks to mr. Ajla Sejfuli, from Travnik Museum for information about excavation in 2015.

Sažetak

ED-XRF analiza srebrenog i zlatnog nakita pronađenog u srednjovjekovnoj nekropoli Crkvina, Donja Zgošća (Općina Kakanj), Bosna i Hercegovina

Tokom arheoloških kampanja 2010, 2012, i 2015. godine na srednjovjekovnoj nekropoli Crkvina (naselje Donja Zgošća, opština Kakanj), u centralnoj Bosni, pored ostataka kostura, pronađeni su i komadi nakita, keramika, građevinski materijal, tkanine i sitni artefakti iz različitih vremenskih razdoblja. Najvažniji nalaz bio je srednjovjekovni prsten – pečatnjak s motivom ljiljana, za koji se vjerovalo da pripada bosanskom kralju ili nekom plemstvu visoke klase iz srednjeg vijeka. Osim tog prstena, pronađen je i drugi bogato ukrašen srebrni i zlatni nakit, kao naprimjer prstenje i naušnice. Ukupno je analizirano osam artefakata. Cilj ovog istraživanja bio je ptvi put dati informacije o sastavu najzanimljivijih artefakata, te unaprijediti znanje o srednjovjekovnoj industriji nakita u bosanskom kraljevstvu. Rezultati su pokazali da je većina nakita izrađena od srebra, najviše kvalitete,

¹⁹ Wołoszyn et al. 2019, 456-459.

²⁰ Sándor et al. 2003, 283; Marchand et al. 2014, 1.

²¹ Sándor et al. 2003, 283-288; Beck et al. 2004, 153-162; Mišta-Jakubowska et al. 2019, 6705-6723.

dok se zlato koristilo za površinsku pozlatu, također najviše kvalitete. Što se tiče prstena pečatnjaka, rezultati analize su pokazali da je materijal za prsten vjerovatno iz istog rudnog izvora, jer nisu pronađeni tragovi bakra ili drugog metala unutar prstena. Kod nekih uzoraka bakar je bio prisutan u nešto većim količinama nego što je uobičajeno (više od 3% u težinskom postotku), kako bi se najvjerovatnije poboljšala svojstva srebra. Pokazalo se da je jedan od prstena, bez ikakvih ukrasa, izrađen od mesinga, sa sadržajem cinka od čak 20%, čime se može objasniti njegova gotovo savršena očuvanost i vizuelni izgled. Naučnice koje su pronađene su iako jednostavnog izgleda (obične okrugle karičice), bile načinjene od najkvalitetnijeg srebra, s dodatkom bakra koji je doprinijeo boljim mehaničkim svojstvima predmeta. Rezultati ovog istraživanja doprinose boljem razumijevanju, do sada inače oskudnog znanja o tradiciji nakita u srednjovjekovnom bosanskom kraljevstvu.

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