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# Examination of Metal Finds from the 10<sup>th</sup> Century Cemetery of Kiskunfélegyháza (Hungary)

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#### ABSTRACT

This case study presents the results and conclusions of chemical and metallographic analyses carried out on metal finds (gilded silver mounts, jewelry made of silver- and copper-based alloys, and iron horse fittings) found in nine graves excavated at the 10<sup>th</sup> century site of the Terjék-tanya at Kiskunfélegyháza (Hungary). The examinations were performed with portable handheld X-ray fluorescence spectrometer (ED-XRF), optical microscopy (OM) and scanning electron microscopy equipped with an energy dispersive spectroscope (SEM-EDS). Beside the determination of the chemical composition of the non-ferrous artefacts and the inclusions of the iron samples, the aim of the study was to detect traces and characteristics of different manufacturing methods such as fire gilding, forging, *etc*.

#### 1. Introduction - archaeological background

The excavation of the 10th century cemetery identified on the Kiskunfélegyháza-Terjék-tanya site was performed by the Kiskun Museum and the Research Centre for Archaeology of the Institute for Hungarian Studies within the framework of a joint project in April 2020 (Gallina et al., 2021). The excavation showed that the cemetery had been heavily disturbed by sand quarrying and metal detecting. When the burial site was excavated, the connection between certain graves and some scattered finds could be reconstructed only partially and it remained often hypothetical. The archaeological excavation revealed that the cemetery consists of a number of graves arranged along one line. The site can be dated to the mid-10<sup>th</sup> century AD. Coins of Hugo of Provence and Lotar II of Pavia were found in one of the graves, giving thus the year 931 as the terminus post quem of the burial (Gallina et al., 2021). In the conquest period this cemetery must have been the burial site of an elite Hungarian

community – or possibly of a family –because, besides the silver jewellery, a gold hair band and harnesses were recovered from the graves. As we know from excavations on other sites dated to the period of the Hungarian conquest (9<sup>th</sup>–10<sup>th</sup> centuries) that surround Kiskunfélegyháza, the region was densely populated and was quite an important area at that time (Tóth, 1974; Somogyvári, 1992; Balogh, 2003; Varga, 2011). Important archaeological sites, cemeteries of the conquering Hungarians, and a map of the excavation site of Terjék-tanya can be seen in the Figure 1. The location of grave 1 could not be precisely determined, but it could be between grave 2 and grave 3. Most of the finds belonging to grave 1 were found with a metal detector.

#### 2. Materials and methods

Some objects made of precious metals and copper-based alloys have been analysed by energy dispersive X-ray fluorescence (henceforth ED-XRF). An Oxford Instrument X-MET8000 portable ED-XRF spectrometer (50 kV, Rh anode, Silicon

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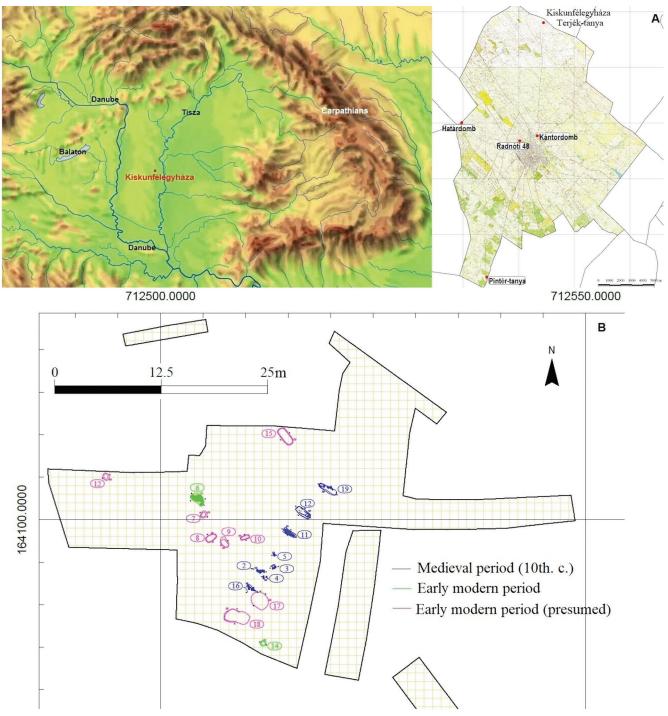


Figure 1. A: 10th-century archaeological sites (marked with red dots) of the area surrounding Kiskunfélegyháza (Hungary) B: excavation map of the site of Terjék-tanya.

Drift Detector) has been used. Three calibration methods were employed for the measurements. In most cases we used the Precious FP mode, developed for the analysis of elements found in alloys and in particular precious metals (Au, Ag). Further, we used the mode Alloy FP, developed for the analysis of the most common elements found in alloys, and the Alloy LE FP, which is similar to Alloy FP but also includes light elements, such as, for example, Mg, Al and Si. The concentration range for each element goes from 0% to

100% in all three cases. Fundamental parameter (FP) methods use a complex mathematical analysis of X-ray fluorescence to calculate the concentration of elements. For metals with inherently unknown composition, such as historical finds, this method is highly suitable and recommended. In the case of these metal objects only a non-destructive examination was possible. The general aim of the analyses was that of determining the chemical composition of the alloys and of the possible coatings. The measurements were carried out



under a radiation shield (dome) whenever it was possible – *i.e.*, when the size of the object allowed it – or by hand using a collimator and a camera pointed at specific locations, here marked with numbers in the figures. In the latter case, when the object was too small or narrow or when the measurement was carried out at the edge of the object, the Fe level resulted higher than usual. The Ti content might have been caused by the paper placed under the object. In all cases, the tests were performed with a measurement time of 30 seconds. The analysis results are reported below in tabular form for each object. The data are expressed as weight percent.

Regarding the XRF analysis of both the silver- and the copper-based objects we have to keep in mind that this kind of surface analysis can only be considered indicative and semiquantitative at best, because the composition of the surface is in most ancient objects very different from that of the core metal. We know that in the period of this site a range of unusual alloys were employed over the entire European territory, and this was due to the recycling of metals. In particular, precious metals were melted down and re-used just as they were (without any refining) to produce ornaments more pleasing to the owners. Thus, the analytical data must be discussed in every single case and all possibilities must be kept in mind, paying attention to the instrumental error as well. Due to centuries of corrosion, tin might have redeposited on the surface, so the average values of this element inside the alloy may actually be lower. Iron always appears as a contaminant, but its measured value can be influenced by external factors as well. Therefore, the analyst must be very careful in the evaluation of the data.

Two samples from iron finds were examined by optical and scanning electron microscopy (OM and SEM-EDS). For the examination the samples were cut and embedded in epoxyresin. The surface of the examined sections was mechanically polished and etched with a 2% nital solution. A Zeiss Axio Imager M1m microscope was employed for optical imaging. The instrument is equipped with a computer-controlled stage featuring composite imaging for the examination of the whole surface. The SEM-EDS examinations were performed on a Hitachi S4300 CFE electron microscope, equipped with a Bruker energy dispersive spectroscope. The main objectives of the examination were to determine the characteristics of the microstructure and to characterise the nature of the hypothesised manufacturing process. The production technology can be deduced from the grain size, the fine or rough microstructure and the distribution of carbon. The inclusions were also examined and evaluated.

#### 3. Results and discussion

#### 3.1 Owl-shaped mount (grave 1)

The various measurements carried out on different areas of this small object have shown (Figure 2) that the material employed was silver, alloyed with the so-called gunmetal – *i.e.*, with the quaternary alloy of copper, tin, lead and zinc that was the most common alloy in use in this period. Gunmetal



Figure 2. Owl-shaped mount.

was mainly the result of the mixing of scrap with some fresh additions of suitable metals, when needed. We know that for a long stretch of time after the 3rd century AD throughout European territories only very little mining activity existed, and the surviving metallurgical production was mainly based on the recycling of scrap metal of all kinds. Only a few larger mines were still being exploited, and there was very little freshly-smelted metal in circulation. This situation lasted almost unchanged for many centuries and only by the 12th-13th century AD there was a sort of revival of deep mining and extractive metallurgy, aided by the invention of various hydraulic machines (see, for example, Le Goff, 1983, pp.222–223; Gille, 1993, pp.660–661). Because of this situation in vast areas of Europe, the addition of gunmetal or the addition of scrap to silver to render it harder and more resistant to wear does not represent a surprise and was a very common occurrence. After casting, the object had to be properly finished by cutting off pouring channels, vents and casting fins and removing the casting skin.

After the polishing process, the surface had to be carefully degreased, for example with strong vinegar or alum, as preparation for the gilding. The analysis data indicate clearly that the surface was amalgam gilded because of the regular presence of mercury all over the object. The amalgam gilding method is also called mercury gilding or fire gilding and has been known in Europe since the Hellenistic period, around the third century BC (Craddock, 1977, pp.109-110; Martinon Torres and Ladra, 2011; Perea et al., 2008; Giumlia-Mair, 2020, pp.5-7) - and even earlier in China, i.e., in the 5th century BC (Jett and Chase, 2000). It is even possible that it spread from China to the West; for the moment, however, there exists no study that might confirm this hypothesis (Giumlia-Mair, 2020, p.5). The method of amalgam gilding involves mixing gold fragments, filings, and broken wire pieces with mercury to form a greyish amalgam by grinding it in a mortar. The excess mercury can be removed from the amalgam by squeezing the mixture in a leather- or cloth bag so that the liquid mercury can run out of it. The ready amalgam (i.e., Au, Hg with around 10–20% Au) can be spread on the degreased surface of the object to be gilded, for example with a hard brush. The object is then slowly and prudently heated to a temperature that must remain under the boiling point of mercury (356.73°C)



**Table 1.** Composition of the owl-shaped mount (wt%).

No.	Calibration	Location	Mg	Al	Si	Ti	Fe	Cu	Zn	As	Ag	Sn	Au	Hg	Pb
1	Alloy_LE_FP	front	5.12	0.54	0.72		0.26	2.83	0.21	0.08	74.81	0.94	12.11	1.68	0.50
2	Alloy_FP	front					0.40	3.04	0.19	0.00	77.12	1.32	14.24	2.19	0.71
3	Precious_FP	front					0.37	3.23	0.23		79.42	0.00	13.65	2.21	0.79
4	Precious_FP	back					0.33	3.62	0.34		92.05	0.00	2.27	0.00	1.39
5	Alloy_FP	back					0.35	3.32	0.32	0.00	90.71	1.23	2.20	0.00	1.27
6	Alloy_LE_FP	back	10.84	0.83	0.48		0.20	2.56	0.26	0.11	81.72	0.80	1.42	0.00	0.75
7	Alloy_FP	front (coll.)				0.90	0.49	2.36	0.17	0.00	83.80	1.42	8.56	1.25	0.77
8	Alloy_FP	front (coll.)				0.39	0.30	3.49	0.21	0.00	72.12	1.29	18.13	2.81	0.71
9	Alloy_FP	front (coll.)				2.52	1.27	2.26	0.22	0.00	84.14	1.63	6.27	0.77	0.80
10	Precious_FP	front					0.21	2.91	0.21		79.28	0.00	14.17	2.27	0.83
11	Precious_FP	back					0.33	3.28	0.32		92.62	0.00	2.09	0.00	1.25
12	Precious_FP	front (coll.)				3.88	1.30	2.90	0.27		77.64	0.00	11.42	1.75	0.85
13	Precious_FP	front (coll.)				1.90	0.79	2.44	0.19		83.63	0.00	8.86	1.24	0.95
14	Precious_FP	front					0.00	3.53	0.20		72.04	0.00	20.10	3.34	0.69
15	Precious_FP	front					0.27	1.77	0.16		88.73	0.00	7.20	0.97	0.81

because too much heat would spoil the gilding. The ideal temperature would be 280-300° C. When the grey amalgam turns to yellow the mercury volatilises and leaves a thin, but durable layer of gold on the surface. The gilding is first rather dull and porous and has to be burnished, but after the polishing with special tools or even simple polished stones like carnelian or hematite, the gilded area looks like shiny solid gold. Some mercury always remains in the gold layer and can be easily detected (Giumlia-Mair, 2020, p.7). A very similar technique was described by Theophilus Presbyter in his work Schedula diversarum artium (early 12th century AD) (Theobald, 1933; Dodwell, 1961; Takács, 1986). As our analysis data indicate, this gilding technique was employed on several other objects from this site. The amount of gold left on the back of this object was only around 2%, while on the front 12-20% of gold was measured (Table 1). Obviously, the quantity of mercury decreased proportionally to the decreased quantity of gold, (as it did here on the more damaged parts of the front). This is perhaps the reason why mercury could not be detected with this method of analysis in the gilding on the reverse of the owl mount, but it is possible that originally it was present in the gold layer. As we determined some mercury on the ornamented side, however, and none on the reverse, where there was only 1 to 3% Au, there is still the possibility that some gold was present in the silver, which might have been recycled from gilded silver objects. A further explanation for the presence of the gold might be a "technical error" during the gilding process: the artisan might inadvertently have touched the reverse.

#### 3.2 Gilded silver rosette (grave 1)

The base metal of this decorative rosette (Figure 3) contains a rather high amount of copper, with some tin, lead and zinc (Table 2). Apparently also in this case the silver was alloyed (and diluted) with some gunmetal. Even a relatively small amount of copper can greatly increase the strength of silver alloy, whilst even an equal-parts silver-copper alloy retains the brilliant white colour of silver. In this case, it is worth noting that the eutectic point of the Ag-Cu diagram is 28.1% copper in silver, *i.e.*, the alloy theoretically has the best castability with this composition.

Some areas of the mount (the petals of the flower-like pattern) have been gilded by using the fire gilding process described above, but the gilding has partially worn off and is essentially the cause of the differences in composition determined on the various areas of the rosette. The analysis results of the measurement carried out on the reverse of this object shows again a low amount of gold, for which we can hypothesise the same origin as in the previous object.

#### 3.3. Five silver plaquettes (grave 1)

Both sides of the plaquettes (Figure 4) appear to be similar and without any gilding. We call the sides from which the holes were pierced the "front", as they were visible when in use. All



Figure 3. Gilded silver rosette.



Table 2. Composition of the gilded silver rosette (wt%).

No.	Calibration	Location	Mg	Al	Si	Ti	Fe	Cu	Zn	As	Ag	Sn	Au	Hg	Pb	Bi
1	Alloy_LE_FP	front (coll.)	12.04	0.62	0.23		0.15	31.16	0.49	0.08	52.23	0.22	1.97	0.24	0.44	0.00
2	Alloy_LE_FP	front (coll.)	8.79	0.61	0.51		0.17	16.96	0.21	0.07	48.22	0.25	20.16	3.57	0.21	0.00
3	Alloy_LE_FP	back	10.57	0.37	0.24		0.00	22.42	0.31	0.14	63.91	0.24	0.99	0.00	0.60	0.11
5	Alloy_LE_FP	back	14.14	0.37	0.19		0.00	28.61	0.39	0.12	54.37	0.21	0.87	0.07	0.57	0.00
6	Alloy_LE_FP	front	6.70	0.00	0.28		0.00	22.00	0.23	0.11	51.13	0.33	14.31	4.14	0.19	0.00
7	Alloy_FP	front					0.14	31.78	0.43	0.10	58.18	0.16	6.93	1.21	0.48	0.09
8	Alloy_FP	front					0.16	16.34	0.14	0.00	50.74	0.32	26.05	5.15	0.35	0.05
9	Alloy_FP	back					0.13	30.49	0.44	0.17	66.03	0.13	1.30	0.00	0.67	0.12
10	Precious_FP	front (coll.)					0.00	17.08	0.12		50.26	0.00	26.56	5.21	0.36	0.00
11	Precious_FP	front (coll.)					0.00	29.78	0.33		57.73	0.00	9.77	1.74	0.65	0.00
12	Precious_FP	back					0.00	33.06	0.50		64.14	0.00	1.25	0.00	0.95	0.12
13	Precious_FP	front (coll.)				0.34	0.18	29.93	0.36		57.98	0.00	8.93	1.64	0.65	0.00
14	Precious_FP	front (coll.)					0.00	16.57	0.15		52.36	0.00	24.78	5.33	0.40	0.00
15	Precious_FP	front (coll.)					0.00	18.76	0.14		52.19	0.00	23.12	5.04	0.42	0.00
16	Precious_FP	back					0.00	32.48	0.44		64.77	0.00	1.26	0.00	0.94	0.11
17	Precious_FP	front (coll.)					0.00	39.15	0.62		56.91	0.00	2.20	0.28	0.76	0.10
18	Precious_FP	front (coll.)					0.00	15.09	0.14		50.17	0.00	27.95	5.87	0.37	0.00
19	Precious_FP	front (coll.)					0.00	23.07	0.14		51.99	0.00	19.83	4.18	0.45	0.00

measurements were carried out under the radiation shield. As with the previous objects the plaquettes are made of silver, alloyed with a high amount of a quaternary copper alloy, and show the same type of composition on both sides (Table 3). The low gold admixture determined by the measurements seems to come from the recycling of gilded silver objects (or perhaps, less probably, of a gilded quaternary alloy) for the manufacture of this ornament. Plaquette D is more damaged than the rest, and at first sight it seems to contain more silver than the other pieces; however, the slight differences

in composition among this group of items is probably simply due to the different degree of corrosion of the pieces. Obviously, oxidation first attacked the less noble metals in the alloy, dissolving them and leaving the silver. There is no trace of the gilding process on the surfaces of the plaquettes.

## 3.4 Gilded silver mount (found with a metal detector) and silver studs (grave 1)

When we calculate the composition of the silver mount (Figure 5) by subtracting the gold of the gilding layer, we

Figure 4. Silver plaquettes.

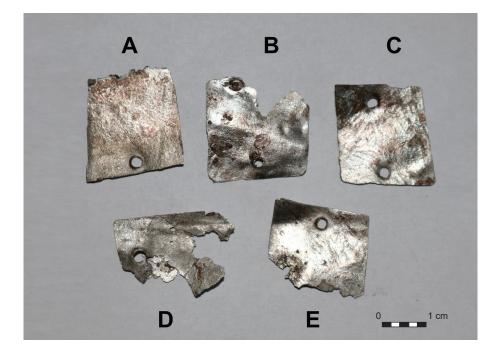




Table 3. Composition of silver plaquettes (wt%).

No.	Calibration	Location	Mg	Al	Si	Fe	Cu	Zn	As	Ag	Sn	Au	Pb
1 A	Alloy_LE_FP	front	13.01	0.36	0.09	0.00	43.09	4.32	0.21	36.98	0.47	0.64	0.76
2 A	Alloy_LE_FP	back	15.55	0.00	0.09	0.00	42.17	4.19	0.22	36.49	0.40	0.00	0.74
3 A	Alloy_FP	front				0.15	48.21	5.16	0.25	43.59	0.43	0.86	0.88
4 A	Precious_FP	front				0.00	50.02	4.48		43.39	0.00	0.81	1.29
5 C	Precious_FP	front				0.00	37.57	2.37		57.30	0.00	1.13	1.63
6 C	Precious_FP	back				0.16	34.38	2.52		59.92	0.00	1.18	1.84
7 B	Precious_FP	front				0.00	43.48	1.69		52.51	0.00	0.94	1.38
8 B	Precious_FP	back				0.00	50.46	1.74		45.76	0.00	0.80	1.24
9 D	Precious_FP	front				0.00	33.00	2.47		61.74	0.00	1.11	1.68
10 D	Precious_FP	back				0.00	25.68	1.87		69.21	0.00	1.37	1.87
11 C	Precious_FP	back				0.16	49.54	1.78		46.29	0.00	0.87	1.35
12 C	Precious_FP	front				0.00	45.35	2.95		49.19	0.00	0.96	1.56
13 A	Precious_FP	back				0.00	49.32	4.32		44.19	0.00	0.82	1.35
14 A	Precious_FP	front				0.19	49.95	4.71		43.03	0.00	0.79	1.33
15 A	Precious_FP	front				0.00	48.65	4.58		44.57	0.00	0.85	1.35
16 E	Precious_FP	back				0.00	37.74	1.63		58.10	0.00	0.99	1.53
17 E	Precious_FP	front				0.00	39.45	1.58		56.38	0.00	1.00	1.59
18 E	Precious_FP	front				0.15	37.69	1.54		58.22	0.00	0.95	1.44
19 E	Precious_FP	front				0.00	39.82	1.83		55.76	0.00	0.99	1.60

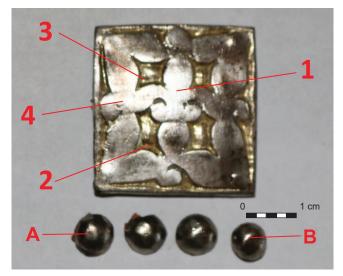


Figure 5. Gilded silver mount and studs for boots.

come to an alloy containing 24–26% of copper, which is almost the eutectic composition that gives the best castability. The small number of contaminants due to recycling comprises 1–2% of lead, and, apparently, around 0.5% of bismuth (Table 4). This latter element would greatly disturb any kind of hammering process, both in copper and in silver. It is known that as little as 0.02 % of bismuth in a copper-based alloy would cause cracking when the metal is intensively worked (Giumlia-Mair, 1992). In a cast piece, however, this element is not as damaging as in an alloy that has to be hammered, and it might come from the addition of unrefined copper to the melt. The noticeable values of



Figure 6. Pendant ornament.

mercury that are without doubt correlated with the amount of gold clearly indicate the use of fire gilding for this item as well. The residue of this decorative process is well detectable



Table 4. Composition of gilded silver mount and silver studs (wt%).

No.	Calibration	Location	Fe	Cu	Zn	Ag	Au	Hg	Pb	Bi
1	Precious_FP	(coll.)	0.17	22.77	0.00	65.15	7.82	2.45	1.07	0.57
2	Precious_FP	(coll.)	0.16	17.91	0.00	60.83	15.04	4.56	0.82	0.42
3	Precious_FP	(coll.)	0.00	18.63	0.00	60.09	14.80	4.91	0.86	0.44
4	Precious_FP	(coll.)	0.00	24.64	0.00	64.30	7.12	2.50	0.94	0.49
5	Precious_FP	back	0.00	27.92	0.00	69.54	0.94	0.00	1.03	0.56
6	Precious_FP	back	0.00	32.16	0.10	65.17	0.98	0.00	1.03	0.56
A	Precious_FP	(coll)	0.47	21.78	0.19	74.49	1.17	0.00	1.42	0.49
В	Precious_FP	(coll)	0.49	23.43	0.18	72.65	1.21	0.00	1.53	0.51

Table 5. Composition of the pendant ornament (wt%).

No.	Calibration	Location	Ti	Fe	Cu	Zn	As	Ag	Sn	Au	Hg	Pb
1	Precious_FP	(coll.)	0.00	0.18	41.93	0.23		45.32	0.00	9.17	2.26	0.91
2	Precious_FP	(coll.)	0.37	0.26	42.71	0.18		49.43	0.00	4.84	0.95	1.25
3	Precious_FP	(coll.)	0.00	0.16	42.96	0.24		46.44	0.00	7.47	1.79	0.94
4	Precious_FP	(coll.)	0.00	0.15	28.89	0.16		58.67	0.00	9.38	1.48	1.26
5	Precious_FP	round part back	0.00	0.00	58.49	0.25		38.94	0.00	0.95	0.00	1.37
6	Precious_FP	hanging part back	0.00	0.19	42.88	0.23		53.79	0.00	1.31	0.00	1.60
7	Alloy_FP	round part front	0.00	0.19	40.72	0.20	0.12	44.82	0.72	9.89	2.26	0.66
8	Alloy_FP	(coll.)	0.53	0.27	42.23	0.16	0.18	51.56	0.73	2.73	0.44	0.90
9	Alloy_FP	(coll.)	6.30	2.21	30.71	0.24	0.20	49.74	0.87	7.45	1.24	0.86
11	Precious_FP	round part front	0.00	0.19	42.51	0.22		47.41	0.00	7.11	1.57	0.98
12	Precious_FP	round part back	0.00	0.00	58.14	0.24		39.37	0.00	0.92	0.00	1.33
13	Precious_FP	hanging part front	0.00	0.16	27.40	0.14		60.08	0.00	9.46	1.44	1.32
14	Precious_FP	hanging part back	0.00	0.28	46.43	0.26		50.23	0.00	1.27	0.00	1.52
15	Precious_FP	(coll.)	0.00	0.20	44.29	0.26		43.43	0.00	8.69	2.15	0.97
16	Precious_FP	(coll.)	0.00	0.20	40.95	0.19		51.39	0.00	5.02	1.01	1.25

even on the worn surfaces. In the deeper recesses of this mount a still significant gold content was detected. The analysis of the reverse side of the mount shows that the silver alloy also contains around 1% gold, and the same amount of lead, both elements probably coming from recycling and from the use of unrefined copper.

The material of the two studs – possibly originally employed as decoration for boots or perhaps for leather straps belonging to harnesses, bridles or a saddle – is silver with a slightly lower copper content than in the case of the previously-discussed plaquettes.

#### 3.5 Pendant (found with a metal detector)

The material of this piece (Figure 6) is essentially the same as in the previous objects: an alloy of silver and a quaternary alloy. The difference in composition (Table 5) of the various parts of the object are due to the corrosion of the less noble metals present in the alloy and to segregation phenomena. Nevertheless, we should keep in mind the possibility that these pieces with a rather high copper content might have been also surface-treated, if the colour of the silver resulted to be too pink for the taste of the customer. The practice of

oxidising the baser metals from the surface of debased silver is very ancient. This was done by placing the debased silver objects in a strong organic acid to remove the copper and any other base metals from the surface. The object was then burnished and looked like solid silver or, at least, like a better quality and more "silvery" kind of metal. Early examples of treated rings from a burial in the Nahal Qana Cave are dated to the 5<sup>th</sup> millennium BC, and they were surface-enriched (Shalev, 1993); several other early examples of this practice exist (Giumlia-Mair, 2020, pp.8–9). The silver artefacts belonging to this group might have been at least partly surface-treated before the gilding was applied.

The copper content of the round part of this pendant is higher than that of the hanging part, and some measurements (*e.g.*, on the back of the round part) gave a copper content that was higher than silver. This is not strange, when we think that debasing silver was a very common practice in Roman times after the 2<sup>nd</sup> century AD and even more so later, in the Middle Ages. We know that in the third century the Roman "silver" coinage only contained around 4% of silver in copper (Cope, 1972; Giumlia-Mair, 2001, p.770; Zwicker *et al.*, 1993). The traces of fire gilding on the front of the



Figure 7. Pendant ornament.

object are clearly identifiable. Originally the entire surface of the small circular area in the centre of the round part was gilded.

#### 3.6 Pendant ornament (grave 16)

This ornament (Figure 7) has the same shape and design of the previous one. The composition (Table 6) of the material is also very similar and this suggests that the two objects belonged to the same set of ornaments and represent a pair. In the case of this particular piece, the gold layer, applied by fire gilding, seems to be thicker than on the other objects or it is simply much less worn and better preserved. It is again



Figure 8. Fragments of a silver ring.

amalgam gilding, because when the measurements indicated a decrease in the gold content on the surface, there was a decrease of mercury as well.

#### 3.7 Fragments of a silver ring (grave 11)

This object (Figure 8) is very fragile and thin; therefore, only one fragment (marked with an X) belonging to it has been analysed. The item has a rather homogeneous aspect, and there was no reason to think that there might be differences in the composition of the various fragments. The piece was examined under a radiation shield dome, as it exhibits a very thin cross-section. This thin strap-like object is made of silver with a gold content of less than 2% and with a little copper and lead (Table 7). Lead is a typical contaminant of medieval silverware because lead and silver are usually present in the same ore. The amount of lead in these objects is relatively high to be a natural impurity of the silver coming from the ore; however, we have to note that medieval silver typically has a lead content in the range between 0.05 to 1.0%, (Merkel, 2016, p.25). The composition of this ring might also suggest that the intensive hammering and annealing that must have been carried out in the manufacture of this piece caused the

Table 6. Composition of the pendant ornament (wt%).

No.	Calibration	Location	Fe	Cu	Zn	Ag	Au	Hg	Pb
1	Precious_FP	round part front	0.16	28.60	0.16	47.18	17.67	5.11	0.79
2	Precious_FP	round part back	0.00	59.05	0.22	38.54	1.05	0.00	1.14
3	Precious_FP	hanging part front	0.19	42.90	0.19	49.62	5.43	0.48	1.19
4	Precious_FP	hanging part back	0.00	40.50	0.16	56.54	1.40	0.00	1.40
5	Precious_FP	(coll.)	0.00	31.95	0.18	48.12	14.46	4.10	0.93
6	Precious_FP	(coll.)	0.00	29.48	0.15	55.90	10.46	2.86	0.98

Table 7. Composition of silver ring (wt%).

No.	Calibration	Location	Fe	Cu	Ag	Au	Pb	Bi
1	Precious_FP	front	0.00	0.34	97.48	1.87	0.18	0.00
2	Alloy_FP	front	0.20	0.37	96.46	1.89	0.18	0.10
3	Precious_FP	back	0.00	0.55	97.54	1.78	0.13	0.00



Table 8. Composition of the bracelet (wt%).

No	. Calibration	Fe	Cu	Zn	Ag	Au	Pb	Bi
1	Precious_FP	0.00	27.58	0.10	69.91	1.00	1.41	0.00
2	Precious_FP	0.18	12.01	0.10	84.66	1.22	1.71	0.11
3	Precious_FP	0.18	15.25	0.13	81.28	1.26	1.78	0.11
4	Precious_FP	0.00	19.31	0.00	78.70	1.11	0.88	0.00
5	Precious_FP	0.20	10.48	0.00	86.25	1.21	1.74	0.12

Table 9. Composition of the torques (wt%).

No.	Calibration	Ti	Fe	Cu	Zn	As	Ag	Sn	Au	Pb
1	Precious_FP	13.41	5.47	28.95	0.39		47.71	0.00	1.21	1.30
2	Precious_FP	16.81	7.47	28.98	0.52		40.15	0.00	1.16	1.49
3	Alloy_FP	12.64	5.39	31.27	0.51	0.21	45.62	0.97	1.13	0.96
4	Alloy_FP	18.28	7.96	24.34	0.60	0.20	43.03	1.02	1.19	1.23

loss of the baser elements that had been introduced into the alloy by the addition of gunmetal.

#### 3.8 Bracelet (grave 11)

In accordance with the wearing custom of bracelets in this period, this silver bracelet with widened rounded ends was found on the middle part of the forearm of the deceased. However, the ends were turned downwards, so that the ribbed decorations at the ends were hidden (Gallina *et al.*, 2021, p.357).

All measurements on the bracelet (Figure 9) were carried out free hand (*i.e.*, with camera and collimator). This ornament must have been worn for a long time and the contact with the skin might have oxidised out of the surface the baser elements originally present in the material. This is also suggested by the rather consistent amount of gold determined all over the piece, while copper, lead and zinc show more fluctuation (Table 8). This indicates that, with all probability, the alloy contained more base metals. The presence of relatively high bismuth is puzzling and is possibly due to instrumental error. This element renders copper and silver very fragile under the hammer, and, as the bracelet is made by hammering the presence of bismuth in the alloy is doubtful.

#### 3.9 Torque (grave 11)

In grave 11, the torque, twisted from double-layered bent silver wire, had been found closed with the clasp below the cervical vertebrae. The appearance of silver necklaces made of similar thin wires can be dated from the second quarter of the 10<sup>th</sup> century (Révész, 1996, p.92), however, it is necessary to distinguish them from the similar artefacts made by folding a single wire into two and by folding and twisting a single wire into three layers. The former are the rarer and probably earlier versions of this type of artefact, although their use may have been parallel.

The examination of a complete torque (Figure 10) with a portable ED-XRF spectrometer is not an easy task because of the awkward shape of the object that cannot fit under the dome. Collimator and camera were used for all measurements; as the torque has a thin cross section; however, the measurement results were strongly influenced by the paper under the object as reflected in the high Ti and Fe determined by the system (Table 9). Nevertheless, the determined values can be considered informative in terms of the proportions of the components. The torque is made of silver with a relatively high copper content, and the baser elements in the alloy are better preserved than, for instance, in the thin ring discussed above.





Figure 9. Bracelet.



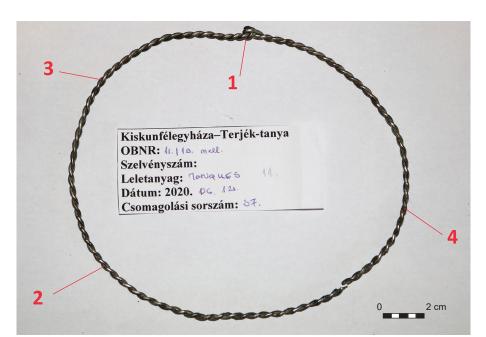


Figure 10. Torques.

### 3.10 Fragments of a copper-based torques (found with a metal detector)

The fragments of a torques (Figure 11) are made of a brass containing 18–24% zinc. It is worth noting that only low tin and lead amounts have been detected in the alloy. Higher Sn- and Pb-percentages (2–4%) are more common in brass objects (Török *et al.*, 2016; Szentpétery and Török, 2022) found at 10<sup>th</sup>–11<sup>th</sup>-century sites in Hungary; however, we also have to remember that, rather often, tin tends to redeposit on the surface due to the long period of time in the soil, during which the corrosion acted on the alloy.

The reddish colour of unalloyed copper becomes paler with an addition of up to 7% Zn, but it is still red. The alloy becomes yellowish-red when the amount of zinc increases to up to 14–15%. With a Zn content of 15–17%, the alloy becomes clearly yellow. The colour of brass is most similar to that of gold with a Zn content of 20–30%. The addition of



Figure 11. Fragments of bronze torques.

Table 10. Composition of the fragments of bronze torques (wt%).

No.	Calibration	Location	Ti	Fe	Cu	Zn	As	Ag	Sn	Au	Pb
1	Alloy_FP	A (coll.)	1.93	0.72	72.31	24.33	0.13	0.00	0.00	0.00	0.00
2	Alloy_FP	C (coll.)	2.74	1.08	72.91	21.96	0.22	0.13	0.17	0.00	0.00
3	Precious_FP	A (coll.)	3.59	1.30	71.76	21.45		0.00	0.00	0.15	0.27
4	Precious_FP	C (coll.)	3.40	1.28	72.36	20.99		0.00	0.17	0.15	0.31
5	Precious_FP	B (coll.)	0.31	0.29	76.07	22.92		0.12	0.20	0.00	0.10
6	Precious_FP	A	0.00	0.00	75.92	24.08		0.00	0.00	0.00	0.00
7	Precious_FP	A	0.00	0.00	75.96	24.04		0.00	0.00	0.00	0.00
8	Precious_FP	A	0.00	0.00	76.36	23.64		0.00	0.00	0.00	0.00
9	Precious_FP	В	0.00	0.30	80.42	18.88		0.17	0.22	0.00	0.00
10	Precious_FP	В	0.00	0.27	81.49	17.77		0.15	0.21	0.00	0.11
11	Precious_FP	C	0.00	0.00	76.27	23.42		0.13	0.19	0.00	0.00
12	Precious_FP	C	0.00	0.00	75.84	23.82		0.14	0.20	0.00	0.00
13	Precious_FP	B (coll.)	1.87	0.80	73.98	22.08		0.07	0.21	0.00	0.22



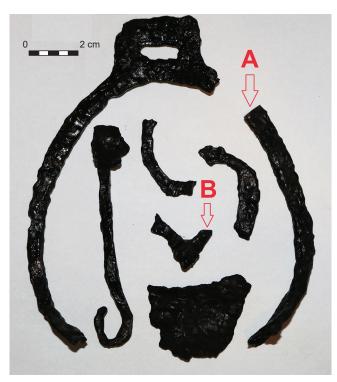


Figure 12. Fragments of an iron stirrup and a bit or buckle.

zinc renders harder and more wear resistant the copper alloy. Brass reaches its highest hardness when it contains 28.5% zinc. This percentage increases the tensile strength and castability, but it reduces the malleability. The most coldworkable brass alloy is the one containing 15-20% zinc, but it cannot be hot worked (Bokor, 1998). The presence of other metals in the gunmetal alloy further lowers the melting point, facilitating the casting, and improves the malleability as well. The only exception is lead that renders the alloy more fragile, when added in higher amounts (above 3-4%). In this case, however, only traces of lead were determined in the alloy (Table 10). The artisans that produced these torques wished to obtain a drawable and twistable alloy, easy to shape when cold and with a colour as similar to gold as possible. In this case they employed the best possible composition. This shows the skill and empirical knowledge they applied in their professional routine.

Figure 13. Composite image of the whole cross-section of the sample of the stirrup.



#### 3.11 Fragments of an iron stirrup and a bit (grave 1)

Two samples of fragments of different iron objects were examined with optical microscopy and SEM-EDS. Sample A comes from a stirrup, and sample B from a fragment of a bit (horse mouthpiece) or possibly a buckle (Figure 12).

#### 3.11.1 Sample A

The metallographic examination showed mostly a pearlitic area with ferrite network that can be observed on the left side of the composite image of the whole section of the stirrup sample (Figure 13). At higher magnifications, the ferrite grains are very angular. The structure becomes more ferritic toward the right side (Figure 14). Although toward the lower and upper edges of the cross-section (i.e., the surface of the object), the amount of pearlite decreases and thus the carbon content is reduced; this is not a folded material. The layered structure could rather have developed in this way as a result of the object being rotated during the initial forging operations (compacting). The ferrite grains and pearlite colonies are small, and the pearlite structure is so fine that it cannot be easily observed by optical microscopy. This implies a faster cooling than equilibrium and a relatively high carbon content. The stirrup is made from a single material and by simple hot forging (so called "bulk forging").

Based on the SEM-EDS examination, it can be assumed that the angularity of the ferrite is caused by the relatively rapid cooling and the so-called Widmanstätten effect, which is an oriented, needle-like growth of ferrite grains. The small black parts in the ferritic area (Figure 14/D) are most probably carbide grains.

It is worth mentioning that metallographic analyses of stirrups from the Avar period of the Carpathian Basin (6th-9th centuries AD), often showed a slightly higher proportion of pearlitic areas and the use of folding as a forging process (Török et al., 2017; Török and Barkóczy, 2022). This technology, however, did not give a significant difference in the quality of the stirrup as a final product.

Figures 15 and 16 show SEM images of the inclusions present in sample A, and the spots marked with green numbers indicate the points measured by EDS method (in wt%). The C content values given as composition are only indicative, as the carbon detection by EDS is highly uncertain.





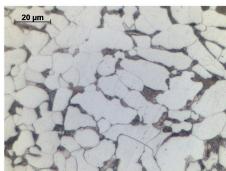




Figure 14. OM images of the samples of the stirrup.

A) Pearlitic area (left side of the composite image) B) Ferritic area (right side of the composite image)





C) Pearlitic area at higher magnification

D) Ferritic area at higher magnification

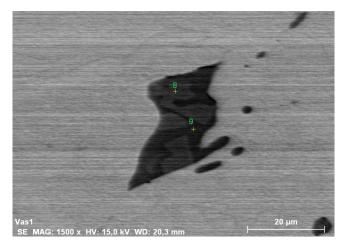


Figure 15. SEM image of an inclusion of the sample of the stirrup. 8: C:1.91, O:46.29, Mg:2.48, Al:28.35, Mn:3.27, Fe:20.25; 9: C:0.47, O:43.20, Na:2.23, A1:14.42, Si:18.47, K:1.46, Ca:6.47, Mn:3.42, Fe:9.86.

The inclusions we investigated may have originally been slag inclusions that had formed during smelting present in the bloom. They have a metallurgical origin: the composition indicates that they derived from a bog ore, charcoal ash (characterised by higher K, Na and Mg contents), and furnace linings (characterised by Al-silicate); no phosphorus was detected in any of the cases. Manganese, which is specific for this ore, was measured in all types of inclusions in the sample and in rather significant amounts.

The shape of the complex microstructured inclusions is strongly distorted by the effect of intensive forging. In Figure 16 an inclusion (42) is coated by a layer of almost pure iron oxide (41): hammerscale probably was formed secondarily during the shaping process. The relatively low values of silicon content are surprising.



Figure 16. SEM image of an inclusion of the sample of the stirrup. 40: Fe:100 (ferrite); 41: C:1.53, O:24.68, Al:1.31, Si:0.44, Fe:72.04; 42: C:1.36, O:45.71, Mg:2.96, Al:29.16, Si:0.62, Mn:2.54, Fe:17.62; 43: C:2.08, O:42.33, Al:33.92, K:3.27, Ca:0.93, Fe:17.47.

#### 3.11.2 *Sample B*

In the cross-section of sample B, there are metallic areas that are smaller than in the case of the stirrup. This sample is mostly ferritic, with a significantly higher fraction of almost pure ferrite, compared to the previous sample (Figure 17). Only a small amount of pearlite can be observed on the left side of the composite image (Figure 18/A). A large area of the sample consists of large grains, which are a consequence of the low carbon content. Tiny precipitates can be observed in the ferrite grains as black spots, which are presumably to be interpreted as tertiary iron-carbide (Figure 18/C).

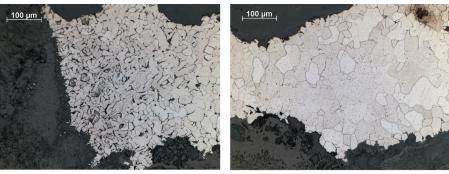
This material is slightly softer than the previous one. The object is made by simple "bulk forging" of a single material.



**Figure 17.** Composite image of the whole cross-section of sample B.

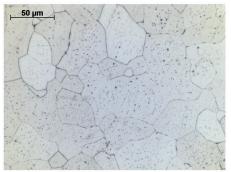


Figure 18. OM images of sample B.



A) A slighly pearlitic area (left side)

B) Ferritic area (central area of the composite image)



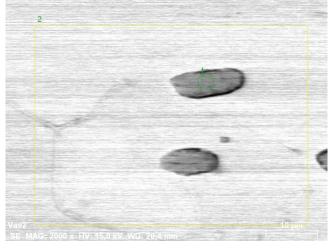
C) Tiny iron-carbide precipitates in the ferrite grains

No special forging, heat treatment or other special processes were used.

Fewer inclusions were found in this sample than in the case of the stirrup. The lens-shaped inclusions (Figure 19) were smaller and generally of the same type. They were arranged in a row in the soft, ferritic structure, according to the direction of the hammering. Their composition suggests that they are Fe-Al-silicate slag inclusions with high phosphorus content, and a legacy from smelting.

#### 4. Conclusions

Nine members of the local community were buried in the cemetery of the Kiskunfélegyháza–Terjék-tanya, and the graves were dug in a single row. The gilded silver artefacts we examined adorned the clothes of the buried persons. Most of the more impressive silver objects from the cemetery – *i.e.*, the owl mount, the rosette, the decorated silver mount, the studs and the plaquettes – come from grave 1. The exception are the pendants, one of which was found outside of context with the metal dectector, while the second comes from



**Figure 19.** SEM image of an inclusion of the sample B. 1: C:1.53, O:26.65, Al:1.09, Si:11.92, P:3.38, Fe:55.42.

grave 16. The workmanship of the various silver fittings, the gilding and the finishing of the objects all suggest that this group of items belonged together. We should not forget



that the graves had been disturbed and mixed up, when sand extraction had been carried out in the area, or by people looking for "treasures" with the metal detector. The two-piece pendant ornaments were typical Hungarian costume decorations in this period, however, in the cemeteries of the common people, pieces made of bronze are usually found. Particularly typical (Bálint, 1991, pp.123–126) is the two-piece ornament with a rhombus-shaped pendant found in grave 16. Five more examples similar to this piece have been found on the site with the help of metal detectors. The owl-shaped mounts are also not unknown in other 10<sup>th</sup> and 11<sup>th</sup> century cemeteries on the Hungarian Plain. A good example is grave 72 in the cemetery of Homokmégy-Székes, where the bird's head is more stylised and made of brass (Török *et al.*, 2016, pp.214 and 218).

The five silver plaquettes found in grave 11 were most likely decorations for clothes (Gallina et al., 2021), but similar pieces have also been observed on horse harnesses in burials of the period (Varga, 2016). The large, but thin and fragile silver ring, the bracelet and the silver torque that come from grave 11 could well represent the funerary set of a member of the same family or social circle. Their manufacture and finishing, however, is very different from that of the previous group of silver pieces: these are rather simple, hammered and very linear objects, without any decoration or gilding. The impression is that they were made by a different pair of hands, not by the same artisan that produced the gilded silver objects. The low but regularly appearing tin and lead percentages in the torque seem to suggest that this alloy also contains some scrap metal. In the cases of the gilded silver mount and studs (Figure 5) and silver bracelet (Figure 9), part of the bismuth content might be the result of an incorrect deconvolution of a lead energy peak (peak overlap). These items may contain some bismuth, but probably not in such quantity. The copperbased torque fragments have been recovered with the metal detector and they consist of double rods twisted to form a ring. A relatively high zinc content (more than 10%) could also be detected in some cases of ED-XRF and SEM-EDS examinations of brass finds from other 10th-11th century cemeteries and settlements of the Magyars (Török et al., 2016; Szentpétery and Török, 2022); however, the zinc content values in Table 10 appear to be extremely high. This high zinc content raises questions about the production of brass in this period. Were there smithsonite and sphalerite mines that could be exploited at the time? Would the artisans of the period be able to actually produce fresh brass? A possible answer could be that the raw material for the brass objects came mainly from the south or the southeast. Brass ingots with a zinc content of nearly 30% have been found at the 10th century metalwork centre near Nadarevo in Bulgaria. The extremely low levels of tin and lead suggest that this was fresh brass and not recycled metal (Doncheva et al., 2017). Regarding the metal art centres of medieval Bulgaria, it is worth noting that silver objects with a copper content of almost 50%, around 1% of tin, between 1% and 1.5% of lead and a gold content between 1% and 4% have been observed on several occasions (Doncheva et al., 2013). The results can be compared with the composition of the silver

objects we have studied, especially the silver plaquettes and the reverse of the gilded objects.

Neither the stirrup nor the other fragments of horse fittings differ from the general types of the period in shape, material, or manufacturing technique. If the silver plaquettes mentioned among the dress ornaments belonged to a horse harness, they would then have been sewn onto a leather component.

The XRF analysis of the finds from the Kiskunfélegyháza – Terjék-tanya site helped to illustrate the habits of the local metal artisans of the 10<sup>th</sup> century, with the recycling of both copper-based alloys and precious silver, still with some gilding remains recognisable inside the alloy, which testify to the re-melting of partially-gilded silver items. The traces of mercury identified in the partly worn gilded decoration also demonstrate the use of the amalgam gilding technique. The carefully forged stirrup and its metallographic structure show the skill and expertise of the local blacksmiths in the 10<sup>th</sup> century AD.

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