HUGE AMOUNTS OF IRON RAW MATERIAL FROM THE EARLY IRON AGE SETTLEMENT OF DÉDESTAPOLCSÁNY-VEREBCE (N-HUNGARY) – A PRELIMINARY ARCHAEOMETALLURGICAL STUDY

KIEMELKEDŐEN NAGY MENNYISÉGŰ VAS NYERSANYAG DÉDESTAPOLCSÁNY-VEREBCE-BÉRC KORA VASKORI TELEPÜLÉSÉRŐL - BEVEZETŐ ARCHEOMETALLURGIAI TANULMÁNY •

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Abstract

The team of the Institute of Archaeological Sciences of the Eötvös Loránd University has been investigating the Early Iron Age hillfort at Dédestapolcsány-Verebce-bérc (Northeast Hungary) since 2020. The settlement was destroyed by siege in the late 7th century BC, as evidenced by hundreds of early Scythian bronze arrowheads and burnt buildings. Based on the recovered metal and pottery findings the settlement dated to the Early Iron Age in the Carpathian Basin (end of the 7th century – beginning of the 6th century BC).

The quantity of the Early and Middle Iron Age iron and bronze artefacts and pieces of iron raw material on the site is exceptionally high. More than 30 depots were unearthed which include pieces of iron raw material. In the whole territory, the number of these finds is more than 600. The average weight of the pieces was 1.54 kg. A few selected objects were sampled and subjected to archaeometric analysis (OM and SEM-EDS). The main aim of the examinations carried out by the experts of the Archaeometallurgical Research Group of the University of Miskolc (ARGUM) was the material characterisation of the samples to figure out what kind of processing has been applied and reveal how the iron raw materials can be connected in any way to the other iron objects found at the site.

Based on the results, it can be concluded that the iron pieces are compacted with a slightly heterogeneous structure. Each one is a part of a single bloom, not several pieces of different blooms assembled together. Numerous pores and cavities were observed in the microstructure of the samples. Their basic character is similar, although, they differ from each other, mainly in terms of carbon content and degree of forming. These pieces are not typical semi-finished products; they can be identified somewhere halfway between primary bloom and compacted bar.

Kivonat

Az Eötvös Loránd Tudományegyetem Régészeti Intézetének kutatói 2020 óta vizsgálják a Dédestapolcsány-Verebce-bércen (Északkelet-Magyarország) feltárt kora vaskori erődített települést. A települést a Kr. e. 7. század végén egy ostrom pusztította el, amit az itt előkerült több száz, bronzból öntött, korai szkíta nyílhegy és a leégett épületek maradványai bizonyítanak. A megtalált fém- és kerámia leletek alapján a település a Kárpát-medence korai vaskorára (Kr. e. 7. század vége – 6. század eleje) keltezhető.

A lelőhelyen előkerült vas- és bronz tárgyak, illetve nyersanyagtömbök mennyisége kiemelkedően magas. Több mint 30 olyan gödröt tártak fel, amelyben vas alapanyagok darabjai voltak. A teljes területen több mint 600 ilyen darab került elő. A vasdarabok átlagos súlya 1,54 kg. A leletek közül három kiválasztott darabon a Miskolci Egyetem Archeometallurgiai Kutatócsoportjának munkatársai archeometriai vizsgálatokat végeztek (OM és SEM-EDS). A vizsgálat fő célja a minták anyagszerkezeti sajátosságainak feltérképezése, feldolgozásuk lehetséges módszereinek feltárása. A kutatás során kérdés volt továbbá az is, hogy ezek az alapanyagok a

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vasfeldolgozási munkafolyamat melyik részéhez tartoznak és egyáltalán kapcsolatba hozhatóak-e a lelőhelyen előkerült többi vastárggyal.

Az eredményekből kiderült, hogy a vasdarabok tömörítettek és enyhén heterogén szövetszerkezetük van. Mindegyik önmagában egy darab kohósított buca része, nem több bucadarabot dolgoztak össze. A minták szövetszerkezetében számos pórus és üreg volt megfigyelhető. A vizsgált minták alapvető jellege hasonló, karbontartalmuk és alakítottságuk mértéke által mégis különböznek egymástól. A darabok nem tekinthetők tipikus félkész termékeknek, valahol a primer vasbuca és a tömörített tuskó között azonosíthatók.

KEYWORDS: IRON AGE; SCYTHIAN; RAW MATERIAL; ARCHAEOMETRY; METALLOGRAPHY

KULCSSZAVAK: VASKOR; SZKÍTA; NYERSANYAG; ARCHEOMETRIA, METALLOGRÁFIA

Introduction – archaeological background

The hillfort of Dédestapolcsány-Verebce is located on the north-western edge of the Bükk Mountains. This fortified settlement is divided into residential areas which covers ca. 150 hectares. The settlement was founded in the Early Iron Age, and it was destroyed by the siege in the late 7th century BC. An early Scythian military venture from the east, from the territory of the steppe horse nomads, besieged and occupied the flourishing center, as evidenced by hundreds of early Scythian bronze arrowheads, burnt buildings and melted bronze objects (V. Szabó 2023; V. Szabó & Bakos 2022, 337-343).

The last research work in this area started in 2020 and hundreds of iron, bronze and gold artefacts (jewellery, tools, daggers, sickles, etc.) and huge amounts of iron raw material were unearthed during the excavation. Interestingly, more than 30 depots, including pieces of iron objects (bloom or bar?), were found in this area. In the whole area, the number of these finds is more than 600 (V. Szabó et al. 2022; 2023). One of the most outstanding depots is the no. 2022/9. which contained altogether 96 pieces of such kind iron find (**Fig. 1**). The assemblage was discovered in the western parts of the examined territory in the summer of 2022. The average weight of the pieces was 1.54 kg, and the depot weight was ca. 145 kg in total. Based on the characteristic pottery found in the pit in question, the depot was dated to the Early Iron Age in the Carpathian Basin (end of the 7th century – beginning of the 6th century BC) (V. Szabó et al. 2022, 218).

Regarding the iron pieces some questions have arisen: what kind of semi-finished production are these pieces? Which manufacturing phase of the ironworking do they belong to?

Fig. 1.: Depot No. 2022/9 found at the hillfort of Dédestapolcsény-Verebce-bérc.

1. ábra: Dédestapolcsány-Verebce-bérc földvárának 2022/9. sz. depója.

The examined finds and the methods of analysis

Three iron finds (No. 1; 27; 46) were chosen for metallographic analysis. To examine the whole cross-section, 1 cm wide samples were taken from each object (**Fig. 2**). Considering the size of the artefacts, the sampling process was carried out by an industrial water jet cutter which ensured that the material did not heat up during the operation and avoided the changes in the microstructure of the samples. However, considering the technical possibilities of the microscopy, the samples were also cut into smaller pieces (**Fig. 2**), thus, none of them were longer than 6 cm so the samples were easily applicable for the examinations.

Before microscopic investigations, the samples were ground, polished, and etched with 2% Nital solution.

After that, the microstructure of the samples was examined with an optical microscope (Zeiss Stereo Axio Imager) equipped with a computer-controlled stage featuring mosaic imaging for the examination of the whole surface. Besides this, with the help of optical microscopy, it was possible to characterize the general phases and inclusions of the objects.

To identify the different phases in higher magnification and to perform elemental analysis, SEM-EDS measurements (Zeiss EVO MA10 scanning electron microscope equipped with EDAX energy dispersive spectroscopy) were taken. This method allows us the observe the phases, morphology, and structures in higher magnification. The examinations were carried out by the experts of the Archaeometallurgical Research Group of the University of Miskolc (ARGUM).

Although there is an abundant and growing literature on the early archaeometallurgy of iron, there are relatively fewer studies of primary iron blooms and the intermediate products of iron bars. In the comprehensive studies by Pleiner (2000; 2003) and Buchwald (2005), the basic characteristics of ancient iron blooms and bars are well-defined. However, there are few examples of detailed metallographic studies of iron blooms and even fewer studies in which the results of metallographic studies of primary blooms and the intermediate product made from them are discussed. Examples of the former include the study by Strobl and colleagues (2010) on the structure of medieval blooms with a diameter of 18–19 cm, and the latter is the article by Saage and colleagues (2017) on metallographic studies of iron blooms and bars found in 14th–17th century smithy sites. The medieval bloom from Styria was not forged and different microstructures of hypo-eutectoid and hyper-eutectoid materials were observed (Strobl et al. 2010). The metallographic analysis of iron blooms and bars from the smithy site of Käku (Estonia) has provided evidence for different steps in iron processing. Little or no marks of forging indicate that primary forging was done, and varying levels of quality could be detected among the iron bars (Saage et al. 2017). The study of Navasaitis and Selskienė (2007) should be mentioned for its unique topic. In this study, they report on the structural analysis of a small lump composed of separate cast iron trickles.

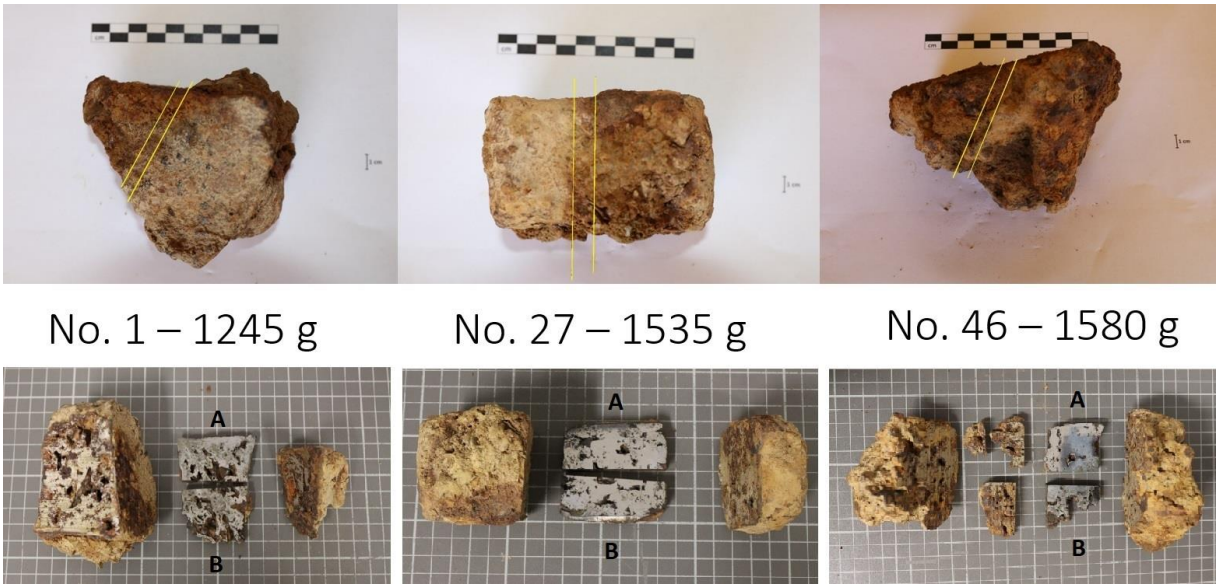

Fig. 2.: The iron objects examined. The yellow lines show the places where the samples were taken from.
2. ábra: A vizsgált vasleletek. A mintavételi helyeket sárga vonal jelzi.

Several metallographic studies have been carried out by our research group on samples of iron blooms, amongst which early medieval pieces weighing around 10 kg (Török et al. 2018) and iron blooms of extraordinary size from a Pannonian Late Roman fortress (Török & Barkóczy 2023) could be found. Regarding the Early Iron Age, metallographic analysis of an iron fragment from the tumulus of Regöly (Hungary) revealed a very specific microstructure, indicating that the supposed bloom fragment is not a direct product that came directly from the bloomery furnace; it could be a secondary (intermediate) product instead (Török et al. 2022).

Results and discussion

Sample No. 1/A and 1/B

Sample1/A shows a heterogeneous structure with a huge number of pores and cavities. The mosaic image in **Fig. 3.** is a good illustration of this diverse microstructure where areas with cementite, ferrite-pearlite and ferrite can also be distinguished. In a higher magnification, pearlite and secondary cementite can be identified beside the ferritic-pearlitic areas.

Because of this, the carbon content of the sample is relatively high (~ 0.7–0.8 wt%). Secondary cementite was also found in the microstructure of Sample No. 1/B. Areas with this structure were more common near the fragmented part of the sample (**Fig. 4**).

Inclusions were found in small quantities in the microstructure of Sample No. 1/A. They are located mostly near cavities and pores. These inclusions consist mainly of iron-oxide, but in a few instances, traces of Al-silicate grains were also observed which can originate from the lining of the bloomery furnace or forge.

In the case of Sample No. 1/B, extended inclusions with slaggy structures were detected in several places in the microstructure. SEM analysis revealed that such slag inclusions have heterogeneous structures. They typically originate from the smelting process. Wüstite dendrites (**Fig. 5A, 1**) and complex oxides of light elements (**Fig. 5A, 3**) were found between the fayalitic parts (**Fig. 5A, 2**), which are commonly present in the smelting slags and inclusions of ancient iron artefacts as well (Buchwald 2005, 96-104). Mn-, and P-contents indicate a smelting origin as well. Moreover, in some parts higher K-, and Na-contents were detected which may be derived from charcoal ash residues (**Fig. 5B, 4**). In certain areas, the metal formed islands with ferritic structures that are interspersed with thin pearlite bands. Between the ferrite grains, bridge-like slag-melt can be seen which is a kind of agglomerate (**Fig. 6**). This phenomenon does not mean metallic bounding, albeit the solid grains were coagulated together, and the formation of grain boundaries can already be observed in some places. In this process, the molten slag is a kind of accelerating passive medium (liquid phase sintering for rapid diffusion). It is a kind of conservation of a certain phase of the direct reduction process of the smelting.

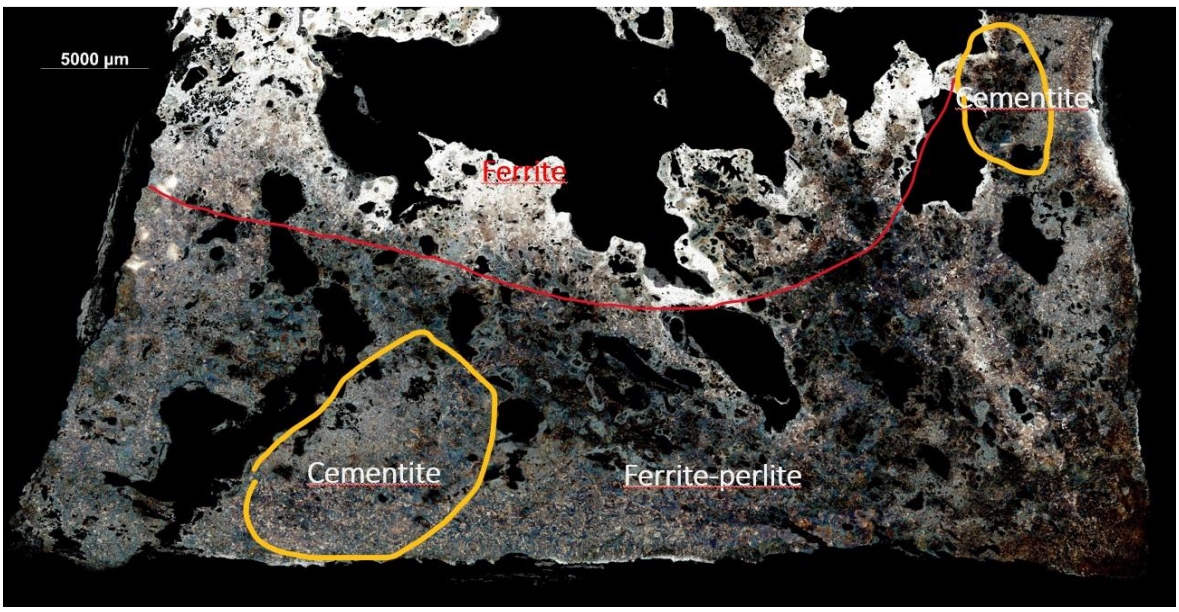

Fig. 3. SEM-BSE (back scattered electron) mosaic image of sample No. 1/A.

3. ábra: Az 1/A minta SEM-BSE (visszaszórt-elektron) mozaikfelvétele.

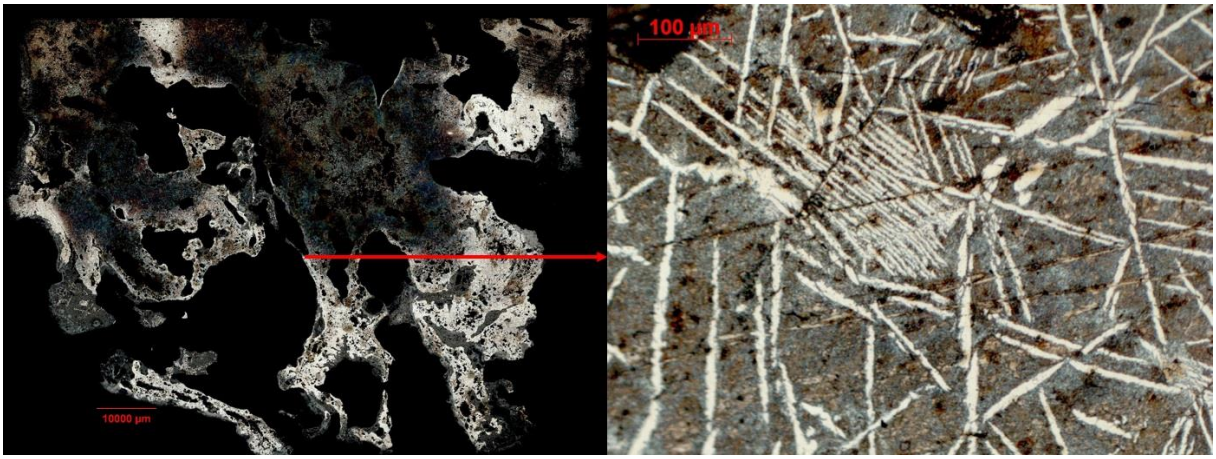

Fig. 4.: Secondary cementite (right) in the fragmented part of sample No. 1/B (left: SEM-BSE, left: OM image).

4. ábra: Szekunder cementit (jobbra) az 1/B minta töredékes részében (bal: SEM-BSE, jobb: optikai mikroszkópos felvétel).

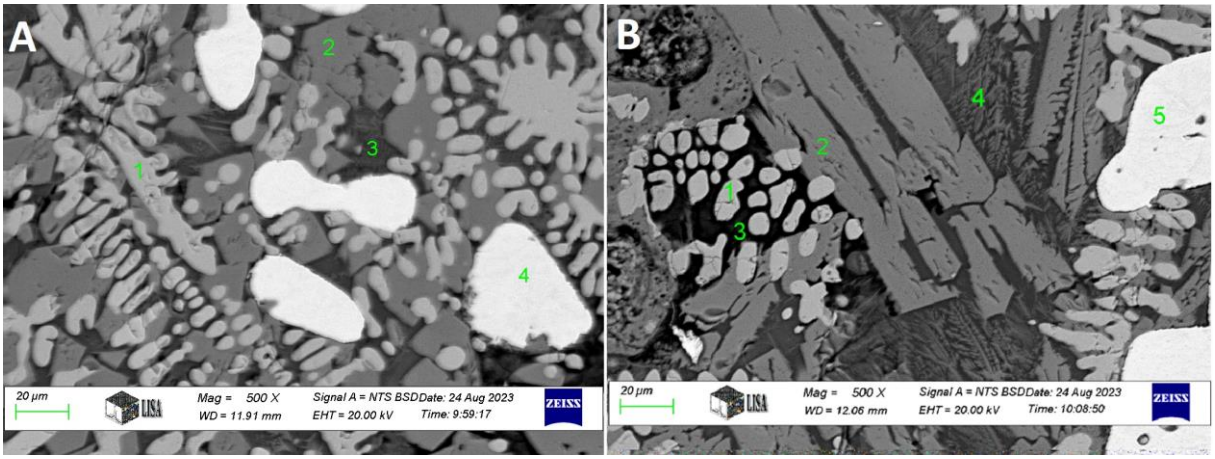

Fig. 5.: SEM-BSE images of inclusions in Sample 1/B.

Chemical compositions in wt%: A/1: O: 15.50; Al: 0.46, Fe: 84.04; A/2: O: 24.52,Al: 0.25, Si: 17.56,Ca: 2.05,Mn: 1.62, Fe: 54.00;A/3: O: 26.57, Na: 1.18, Mg: 0.25, Al: 7.70, Si: 21.05, P: 1.01, K: 4.78, Ca: 10.53, Ti: 0.38, Mn: 0.62, Fe: 25.94; A/4: Fe: 100; B/1: O: 15.45, Fe: 84.55; B/2: O: 23.18, Mg: 1.39, Si: 17.03, Ca: 2.61, Mn: 1.71, Fe: 54.08; B/3: O: 23.37, Mg: 0.22, Al: 10.59, Si: 11.92, P: 1.70, Ca: 4.11, Fe: 48.09; B/4: O: 25.49, Na: 1.05, Mg: 0.16, Al: 5.57, Si: 19.76, P: 0.80, K: 3.84, Ca: 11.53, Mn: 0.69, Fe: 31.12; B/5: Fe: 100.

5. ábra: Az 1/B minta zárványainak SEM-BSE felvételei.

Kémiai összetételek tömeg%-ban: A/1: O: 15,50; Al: 0,46, Fe: 84,04; A/2: O: 24,52, Al: 0,25, Si: 17,56, Ca: 2,05, Mn: 1,62, Fe: 54,00; A/3: O: 26,57, Na: 1,18, Mg: 0,25, Al: 7,70, Si: 21,05, P: 1,01, K: 4,78, Ca: 10,53, Ti: 0,38, Mn: 0,62, Fe: 25,94; A/4: Fe: 100; B/1: O: 15,45, Fe: 84,55; B/2: O: 23,18, Mg: 1,39, Si: 17,03, Ca: 2,61, Mn: 1,71, Fe: 54,08; B/3: O: 23,37, Mg: 0,22, Al: 10,59, Si: 11,92, P: 1,70, Ca: 4,11, Fe: 48,09; B/4: O: 25,49, Na: 1,05, Mg: 0,16, Al: 5,57, Si: 19,76, P: 0,80, K: 3,84, Ca: 11,53, Mn: 0,69, Fe: 31,12; B/5: Fe: 100.

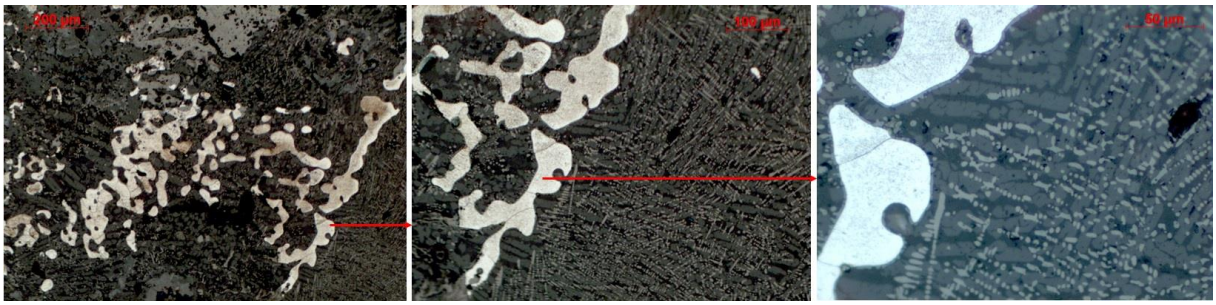

Fig. 6.: OM images of Sample 1/B

6. ábra: Az 1/B minta optikai mikroszkópos képe

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Sample No. 27/A and 27/B

The Sample No. 27 is different in character than the previous one. This piece of bloom (No. 27/a) is compacted, and its shape is smoother, and more brick-like whereas the shape of the Sample No. 1. is irregularly fragmented. Although larger pores and cavities were also found in this bloom (**Fig. 7**). The microstructure of the object is basically ferritic with ferrite-pearlitic areas. The carbon content in this case is significantly lower (~ 0.4–0.5 wt%) than in the first case. In the ferritic areas, line-like fractures or gaps were observed but these are not cracks. Along these gaps, inclusions were detected. However, the mentioned cavities were often filled with non-metallic material which is presumably a product of corrosion.

During the compression hammering of the iron, the parts of the heterogeneous carbon containing bloom with different microstructures were squeezed together, thus, a larger gap was formed between the parts (**Fig. 8**). A good example is the SEM image made by secondary electrons in the bottom left corner of **Fig. 8**, showing separated ferritic and ferritic-pearlitic areas at higher magnification.

Fig. 7.: OM mosaic image of Sample No. 27/A

7. ábra: A 27/A minta optikai mikroszkópos mozaikfelvétele

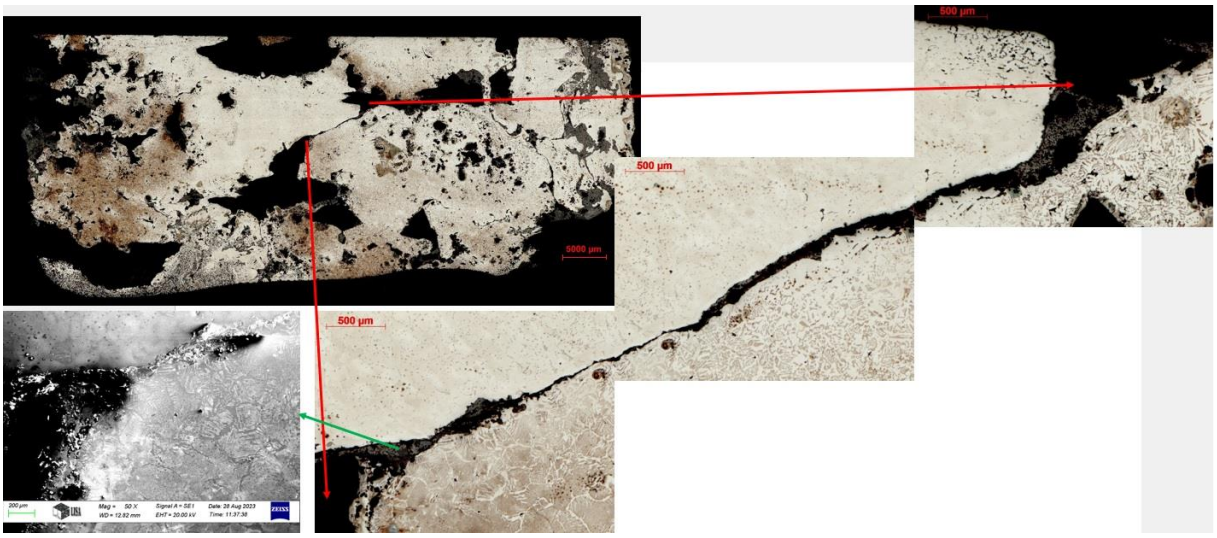

Fig. 8.: OM and SEM-SE (secondary electron) images of a gap in the Sample No. 27/B

8. ábra: OM- és SEM-SE (szekunder elektron) felvételek a 27/B minta egyik hézagjáról

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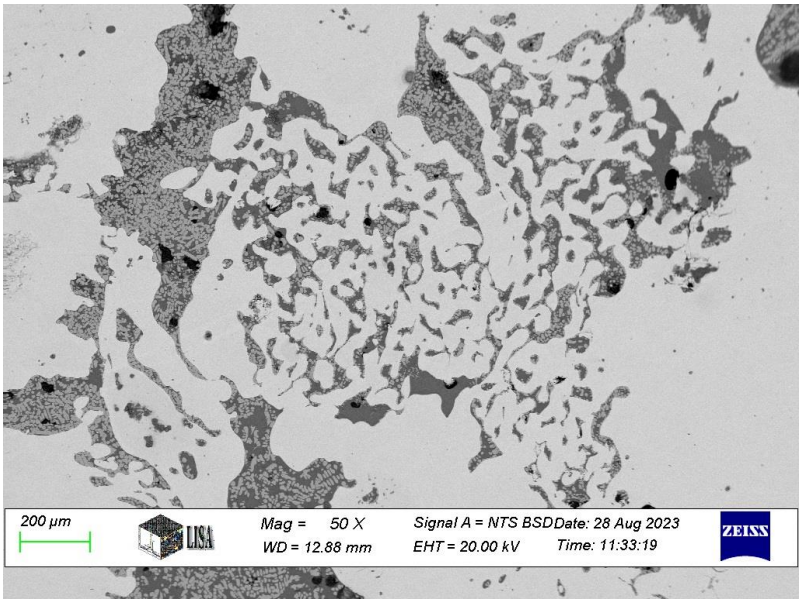

Fig. 9.:
SEM-BSE image of modified inclusions in sample No. 27/B

9. ábra:
A 27/B minta egyik módosult zárványának SEM-BSE felvétele

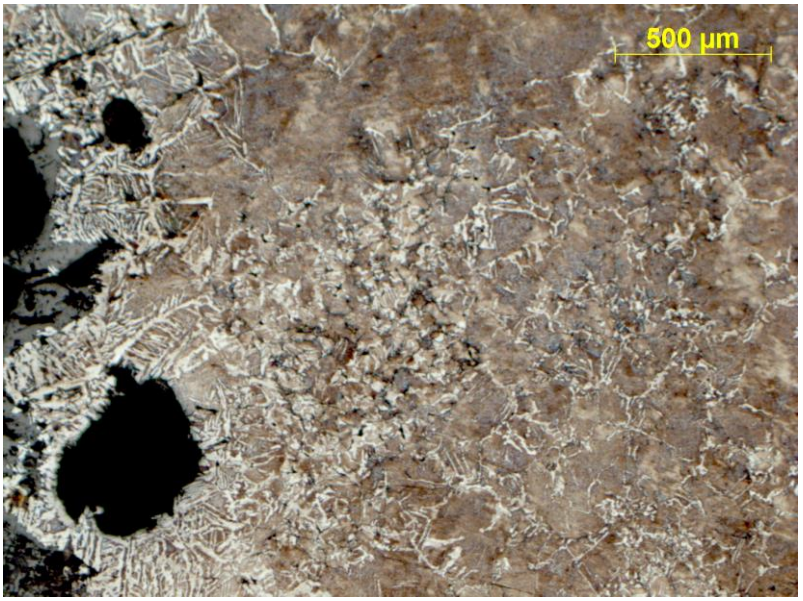

Fig. 10.:
OM image of sample No. 46/B

10. ábra:
A 46/B minta optikai mikroszkópos felvétele

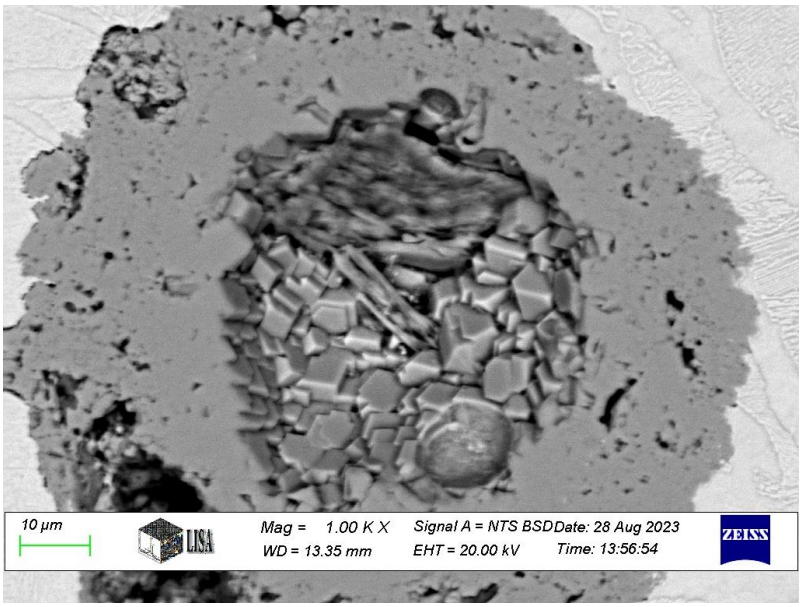

Fig. 11.:
SEM-BSE image of inclusion in sample No. 46/B

11. ábra:
A 46/B minta egyik zárványának SEM-BSE felvétele

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These different parts may even have different mechanical behaviour during hammering, but it is still the same bloom. If several pieces of different blooms had been hammered together, there would also be such a gap in the areas with identical microstructure. However, this case generates an interesting perception. If we are examining a finished, heavily corroded iron artefact and due to this corrosion only two different layers can be distinguished, this does not necessarily mean the application of the folding technique during the forging process. It could also be caused by the heterogeneity and structure of the raw material, such as in this example. This piece of bloom was thoroughly compacted and formed into a bar, but despite this, a lot of large cavities and numerous inclusions remained in the material which is typical in the case of ferritic structure (Buchwald & Wievel 1998). The SEM-EDS analysis of inclusions revealed that besides the typical three-phase metallurgical inclusions locked into the metal, there are also slag inclusions of smelting origin, whose structure (**Fig. 9.**) has already been modified by corrosion products or by the scale produced during compaction hammering.

Sample No. 46/A and 46/B

The microstructure of the Sample No. 46/A and B is mostly ferritic-pearlitic. Just like in the case of Sample No. 27, small gaps at the slightly different microstructure can also be detected. This may be the result of the fact that the reduced metallic grains did not fully weld together during the smelting, nor did they do so during the hammering process (**Fig. 10**).

The shape of this bloom is also brick-like, it is well visible that the piece was compacted. In the microstructure of the sample, no inclusions specifically originating from smelting were found. The non-metallic details that appear in the cavities or are observed as inclusions are iron oxide or iron silicate in various forms and compositions, respectively, some of them are Fe-Al silicates (**Fig. 11**). The latter may have remained from the clay wall of the furnace, but most of them may be mainly corrosion products.

Conclusion

Three samples were taken from iron objects found at the Early Iron Age settlement at Dédestapolcsány-Verebce-bérc. The slices of the samples were cut into smaller pieces. The OM and SEM-EDS examinations revealed that the blooms are compacted and more or less purified from slag. The essential characteristic of the objects is the heterogeneous microstructure caused by the different carbon content. In the microstructure, numerous pores and large cavities were observed, which is usual in the cases of historical blooms and bars (Pleiner 2000; Buchwald 2005; Strobl et al. 2010; Saage et al. 2017). Based on the results, it can be stated that each object examined is a part of a single bloom, which means it does not consist of several pieces of different blooms assembled together. Although their basic characteristics are similar, they still differ from each other, mainly in terms of carbon content and the degree of compacting and forming.

The most compacted object is No. 27, its shape is more brick-like and has a smoother surface, than the others. The carbon content in this object is not very high, just like in the case of No. 46. However, the microstructure of No. 1 showed a broad variety of Fe-C phases. Besides ferrite and ferrite-pearlite, secondary cementite (close to the surface) was also found in the microstructure but only in the case of No. 1. Several gaps were observed in the microstructure of objects No. 27 and 46, which were surely formed during already the smelting process.

Besides reoxidation-caused iron-oxide inclusions, slag inclusions specifically formed during the smelting process were found in samples No. 1. and 27. At the same time, no classic smelting slag inclusions were found in sample No. 46. In some inclusions of sample No. 1, a small amount of phosphorus (1.1–1.7 wt%) was detected, but this element was found only in the slag inclusion not in the metal, so phosphorus did not play a role in the later forging process.

Returning to the questions posed at the beginning of this study, it can be assumed, that these iron pieces are albeit well compacted, yet not typical semi-finished products. They are neither a bloom nor a bar, being somewhere halfway between them. Their differences from one another reflect the different technical conditions of the smelting (i.e. time, temperature, charging, etc.) and maybe the different characteristics and the works of the bloomeries.

Another interesting question would be the connection between the objects examined and the other iron artefacts found in the territory. Samples were taken from four iron axes found in the hillfort of the site, which had been analysed by OM and SEM-EDS. In the cases of metallographic examinations of socketed axes, it was possible to identify such types of raw material that were represented by the samples of this study. However, a detailed study of the axes found in the Dédestapolcsány-Verebce-bérc is still underway and the results will be reported in another paper.

Contribution of authors

Béla Török Conceptualization, Methodology, Validation, Formal analysis, Investigation, Writing – Original Draft, Writing – Review and Editing,

Visualization, Funding acquisition. **Péter Barkóczy** Validation, Investigation, Writing – Original Draft, Writing – Review and Editing. **Gábor V. Szabó** Investigation, Resources, Writing – Review and Editing, Funding acquisition.

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