SPECIALIZED IRON PRODUCTION IN ROMAN TIMES – METALLOGRAPHIC EXAMINATION OF FORGING PRE-PRODUCT BARS

ABSTRACT

In Roman times, according to sources, mining and metallurgy were organized and managed centrally. The area of Pannonia was supplied with iron ore from the Majdan Mountains from the early Iron Age onward, which was then largely processed according to needs by local blacksmiths. Particularly interesting questions are raised by the square column bars found at several sites in Southern Transdanubia. Similar finds were found in 1880 near Hrvatska Dubica in Croatia. Aleksandar Durman hypothesized Syrian influences in the production of iron bars, defined as Roman and about 20 cm long, forged to a weight of 11–15 libra, or 3.6–4.91 kg, based on finds found in the vicinity of the aforementioned site. In Pannonia, square column-shaped iron bars are known near the Danube (Intercisa – Dunaújváros) and the Kapos Valley (Dombóvár, Regöly), but they are heavier than the finds from Croatia, weighing 5.4–10 kg. Iron metallurgy is highly technology- and raw material-dependent, and is traditional in every detail, even from compulsion to ensure quality. Therefore, metallographic examinations play a particularly important role in research. A recent analysis of Pannonian square-bar bars shows that several pieces of bloom were forged during their production. Their microstructure is heterogeneous, typically ferrite-perlitic in which ferrite is found with Widmanstätten morphology. All indications are that the raw material blocks provided a standard good quality raw material for the production of different tools, weapons, etc.

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INTRODUCTION

Traces of an iron processing workshop on both sides of a creek were observed in the 1970s during excavation (Szabó 1966) of the pioneer railway in front of the Roman gate of the Alsóhetény fortress, north of Dombóvár (Tóth 2009; Tóth 2003). One rail construction worker reported that he had observed a number of red-burned spots on the shore-side at the time, where he also found several pieces of iron, similar to half-bricks bisected in length, as well as blooms, all but only two of which are now lost. Presumably, this scattered assemblage of artefacts may also have included the two iron blocks that were placed in the metallography laboratory of the University of Miskolc. The test results for these two objects are described below.

The size and dimension proportions of the two blocks are slightly different. The width of block 1 is 10 cm. It has a rectangular cross-section, and the length of the block is 26 cm. Block 1 differs from the previously known, often square-shaped so-called iron bricks in its shape. Iron block 2 has a standard shape: it is 35 cm long and 9 cm wide. The objects examined fit well into the procedure known from Roman sources. The essential element was that mining and metallurgy were centrally organized and managed (Rebrik 1987). The area of Pannonia was supplied with iron raw material processed from ore from the Majdan Mountains from the early Iron Age onward (Simič 1951), which was then processed according to the needs of local blacksmiths (Gömöri 2003). One of the best known archaeological remains of this raw material supply system, and of particular interest to us, is the ensemble of finds found in 1880 near Hrvatska Dubica (Crew, Crew 1977). Nintety-seven square bar-shaped iron bars for commercial use formed this find assemblage (Durman 2002). Aleksandar Durman assumes Syrian influences in the production of the 28 still existing Roman iron bars, about 20 cm long and forged to a weight of 11–15 libra, ie 3.6 to 4.91 kg, based on finds found in the vicinity of the site (Durman 1999). In Pannonia, a similar raw material is known from the vicinity of Intercisa (Gömöri 2000), and Endre Tóth from Alsóhetény in the Kapos Valley mentions three pieces from the north-western part of the fortress (Szabó 2012), although János Gömöri mentions four in his site register. He determined their weight at 5.6 kg and even published the results of one of his metallographic examinations. According to these, the material of the tested sample consists of ferritic layers of different grain sizes with a 0.25 mm thick bark of lamellar perlite. It is in a cold-worked state. The analysed chemical composition was: C: 0.05 wt%, Si: 0.05 wt%, Mn:0.09 wt%, S:0.003 wt%, Cr:0,0002 wt%, P:0.01 wt%, Ni:0 wt%. In the Kapos Valley at Regöly, Viktor Cziráki collected about a dozen square bars. There were once many more in the village, but these were used as heavy door supports, and the blacksmiths used them as raw materials, making an anvil from two for example (Sperl 1999). The 6–10 kg weight of the iron blocks is also significantly higher than that of the Croatian bars. Compared to the bars from Alsóhetény, the sample of Regöly contains a higher amount of additives based on microprobe analysis, for example, C:0.15–2.41 wt%, Si:0.19–17.71 wt%, Mn:1.34–12.26 wt%, S:0.18–0.83 wt%.

Iron metallurgy is highly technology- and raw material-dependent, and is traditional in every detail, even from compulsion to ensure quality. For example, the maximum weight of bloom produced in the same type of smelter can only be varied within relatively narrow limits. Thus, based on the Hungarian finds, which differ significantly in weight from the Croatian bars, and the Alsóhetény and Regöly bars, which differ in their material composition, it is still doubtful that these square-sectioned bars were made both in Roman times and around Siscia. Only in the southern areas of the Carpathian Basin does the square column dominate. The

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Noricum smelters, based on Celtic traditions, produced bars with double-conical ends. Their iron bars, manufactured from the 7th century, are mainly known from the northern areas (Tóth 2003). The spread of the semi-finished iron raw material preforms from the Balkans and along the Danube River also shows that the development of the two different forms of raw material blocks is due to the different historical and metallurgical traditions in Roman times. It is also extremely important to clarify to what extent of the tested iron blocks were of a standardized composition and quality, where and in how many places they could be made. Due to the relatively small number of bars and the extremely small number of reported analytical results, only some of the questions could be answered for a long time. With a new data set, we are also trying to address this issue by evaluating the results of the present analyses.

The examination of metal artefacts in both bronze and iron reveals important information about the raw material, the specifics of the manufacturing technique, and to a certain extent information about smelting can also be obtained (Barkóczy *et al.* 2012; Török *et al.* 2017a; Török *et al.* 2013). In the case of copper alloys, even a carefully performed compositional analysis can provide a lot of information (Benkő, Barkóczy 2017), but in the case of iron, due to the complexity of the phase transformations, metallographic analysis is always necessary (Török *et al.* 2017b). The metallographic analysis also assigns a state to the chemical composition of the material, and from the analysis of the microstructure, it can also deduce the conditions of the transformation processes taking place (Póliska *et al*. 2006). The results obtained provide valuable information to inform archaeological observations and investigations concerning the given area, age, or culture (Gyucha *et al.* 2015; Horváth *et al.* 2020).

EXAMINATIONS

Metallographic examinations were performed on samples taken from the iron bars shown in Fig. 1. Metallographic testing is typically a destructive material-testing method. Heritage conservation aspects must also be considered when applying metallographic analysis. As the object is presumably a forging pre-product bar and several non-public collections of objects can be found, it is possible to take several samples as well. Fig. 1 shows the sections, and slices of the bars from which the samples are taken. The slices were further cut to pieces, as shown in Fig. 2. Both iron bricks (block 1, block 2) and sections (K – centre, B – at the edge) are marked. In addition, sections from the slice were numbered. The identification of the samples is clearly shown in Figs 1 and 2. Cutting was needed due to the size of the slices of the bricks. It was not possible to prepare the whole slice in one for microscopic examination.

The samples were prepared mechanically (grinding, polishing) in the first step. For grinding fine-grained SiC (220, 320, 500, and 800) was used. Aqueous grinding was performed to avoid heating of the samples. Polishing was performed with 3μm diamond particles. After polishing, the cleaned, prepared surface was chemically etched with 2 v/v% Nital (98 v/v% ethanol and 2 v/v% nitric acid (HNO_3)). The prepared surface was examined with an optical microscope (Zeiss AxioImager M1m) and a scanning electron microscope (Zeiss EvoMa10). The electron microscope was equipped with an energy-dispersive microprobe (Bruker) for local analysis of chemical composition. The optical microscope is equipped with a computer-controlled stage so that mosaic images can be taken of the whole prepared surfaces (Fig. 3).

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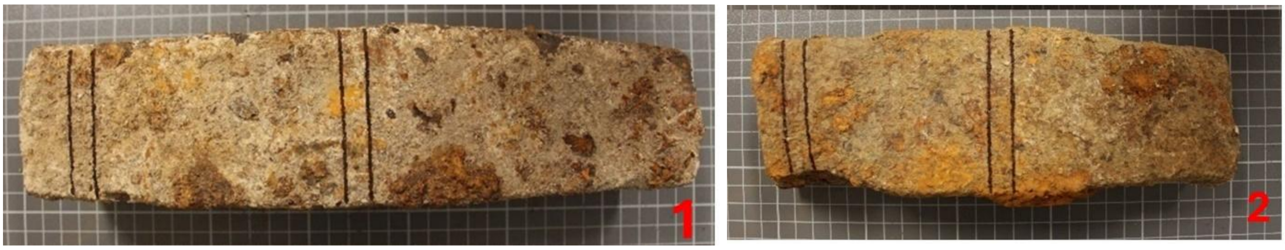

Fig. 1: Marking of sampling locations for iron bars 1 and 2. Two slices were made from each sample for a complete examination of the objects.

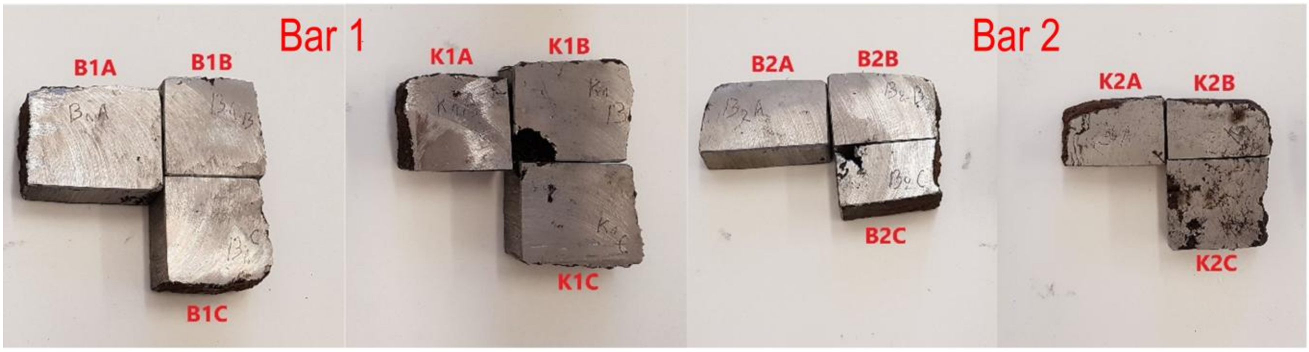

Fig. 2: Pieces that have been cut and prepared from the slices. The slices in Fig. 1 were large in one for metallographic preparation. B denotes the section at the edge of the bars and K is the section taken from the centre of the bars. The number (1, 2) denotes the bar tested. The letter designation is the identifier of the pieces as shown.

RESULTS

Figure 3 shows a mosaic image (reconstructed tiled images taken by the optical microscope) of sample K1B as a characteristic microstructure of block 1. The sample is very inhomogeneous. A perlite - secondary carbide area (1) and a completely ferritic area (4) can be found next to each other. The consequence of this is that it is not possible to determine the average carbon content only by estimating the volume fractions of the microstructural elements, but as simple data, it is not informative. An area with a typical microstructure is outlined in the mosaic image, which is marked with numbers. The microstructures associated with the numbers are shown in larger magnification in Fig. 4. The carbon content of area 1 is very high, 1–1.1 wt%. Perlite grains are bounded by a secondary cementite net. Area 1 itself is very small compared to the others. Area 3 is completely pearlitic with a carbon content of 0.8 wt%. The area of perlite occupies nearly a quarter of the sample. It is not uniform because the areas 1 and 2 are wedged into the pearlitic structure. Area 4 is a fully ferritic area with a medium grain size. The carbon content of the ferritic area is extremely low. The ferritic area occupies less than a quarter of the section. The largest area is occupied by Type 2, where ferrite and perlite are found together in varying volume fractions.

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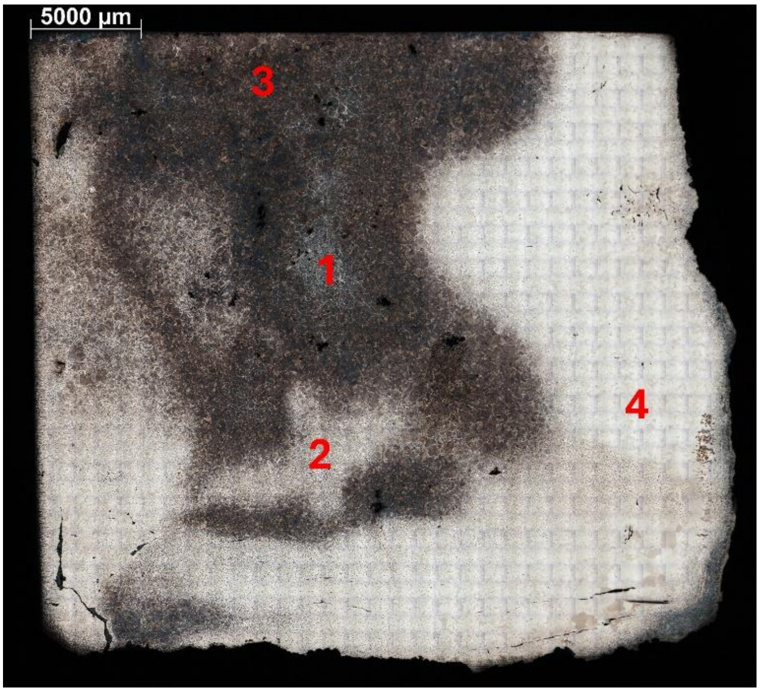

Fig. 3: Mosaic image of K1B sample at 100x magnification. The mosaic image shows an optical microscopic image of the entire section at low magnification. There are 4 denoted areas where significant inhomogeneity of the carbon content formed different microstructures.

The largest amount of ferrite is shown in Fig. 4, at point 4. At area 2 the ferrite forms a net structure, where the perlite grains are surrounded by a ferrite shell from which a needle ferrite has grown toward the interior of the perlite grains. This formation is called Widmanstätten ferrite. The transition between the smaller and higher carbon content areas is diffuse and continuous, but also can be found as wide transition and narrow transition. The transition is due to diffusion initiated as a consequence of the homogenization of the carbon content. The width of the transition zone indicates how much time was available for the diffusion of carbon.

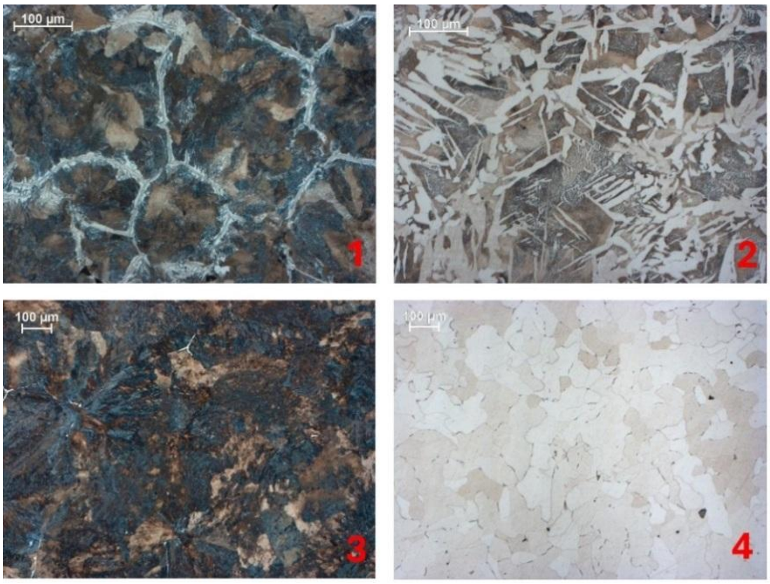

Fig. 4: Characteristic microstructures of K1B piece. Point 1 shows perlite colonies surrounded by cementite (iron carbide) net. In addition, at point 2 perlite colonies surrounded by ferrite net can be seen. The needle-like growth of ferrite refers to Widmanstätten ferrite, indicating an intense cooling but not quenching of the object. At point 3 pure perlitic structure can be observed, at point 4 denotes almost completely ferritic structure.

At room temperature, the rate of carbon diffusion is extremely low; however, at high temperatures, the movement of carbon atoms is significantly accelerated. The mentioned wide transitions between different areas suggest that at high temperatures, the two different

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volumes with different carbon content spent more time next to each other, while the narrow transition indicates a short tempering.

The mosaic image of sample B2A (Fig. 5) shows a similar character. The mostly pearlitic area is denoted by 2. No pure perlite is found in this sample. A small ferrite net covered the perlite grains (Fig. 6), so the estimated carbon content of this area is approximately 0.7 wt%. Also, no pure ferritic area is found in area 4. Small perlite islands are embedded in the ferrite grains. The estimated carbon content of this area is 0.15 wt%. In terms of their proportion, the mostly pearlitic area is slightly smaller than seen in the previous sample. The estimated carbon content of this bar is slightly higher than that of the previous sample. However, it should still be borne in mind that the structure is so inhomogeneous that it is difficult to determine the average carbon content for the entire sample. In addition, this data is also irrelevant in this form.

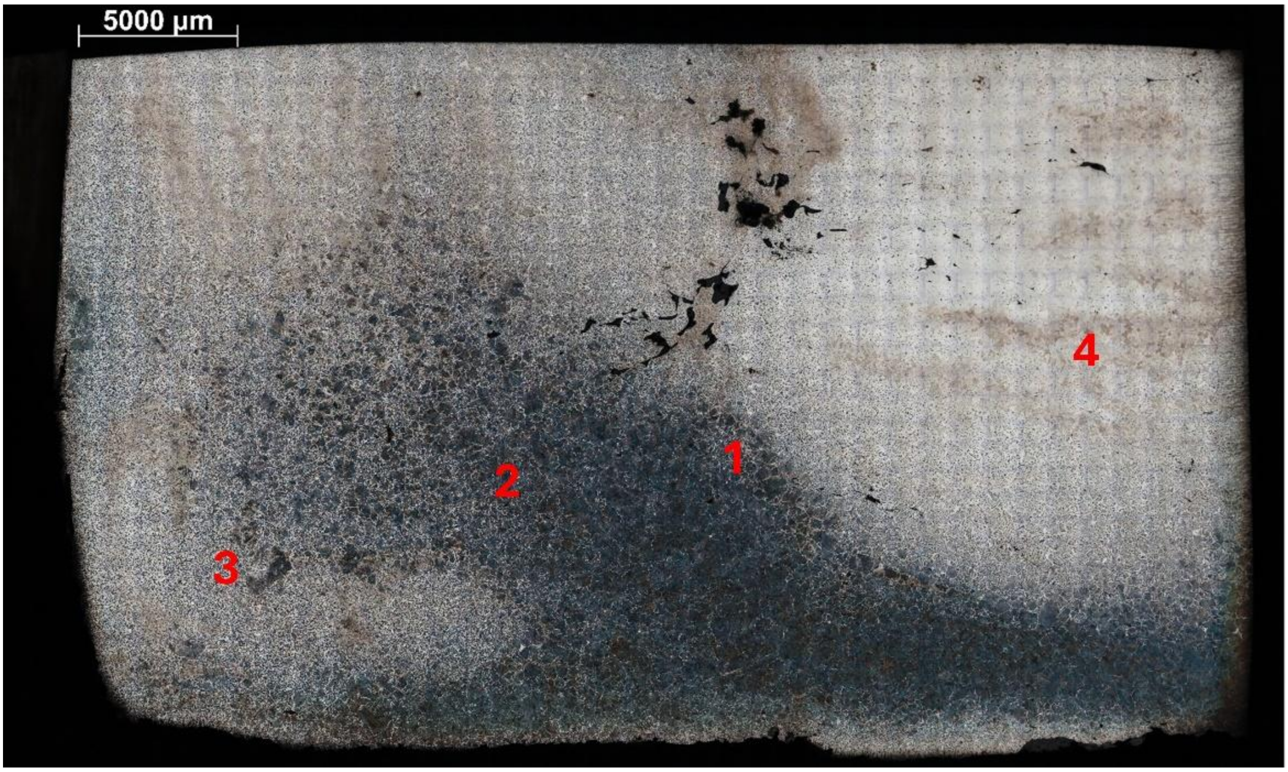

Fig. 5: Mosaic image of B2A sample at 100x magnification. In the mosaic image, 4 areas can also be identified, where different microstructures can be seen due to the inhomogeneity of the carbon content.

In this sample, a transition between low and high-carbon areas also can be seen. The wide transition indicated at 3 in this sample is extremely extensive, and the decrease in perlite content in the mosaic image with the decrease in carbon content can be seen. A narrow transition also can be found (1). Both transitions are also characterized by the ferrite net surrounding the perlite grains and the Widmanstätten ferrite (Fig. 6), as in the first sample.

In the case of both samples it can be stated that the characteristic microstructure of the blooms and the objects were formed during hot forging. The difference compared to the bloom is that the perlite, sometimes secondary cementite net with perlite microstructure is

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found in the cemented surface region of the bloom. It is characteristic of objects forged from bloom that areas with high carbon content can be found within the object. The bars were made of blooms, by hot forging. Inhomogeneity was found originally in the bloom. To see the wide transition zone, the bloom itself spent a long time during the manufacturing at high temperatures. In contrast, narrow transitions were formed during the forging of blooms into a thicker one, when the parts with different carbon content were forged at high temperatures. Both the microstructure and the significant number of narrow transitions suggest that several blooms were used to form the bar.

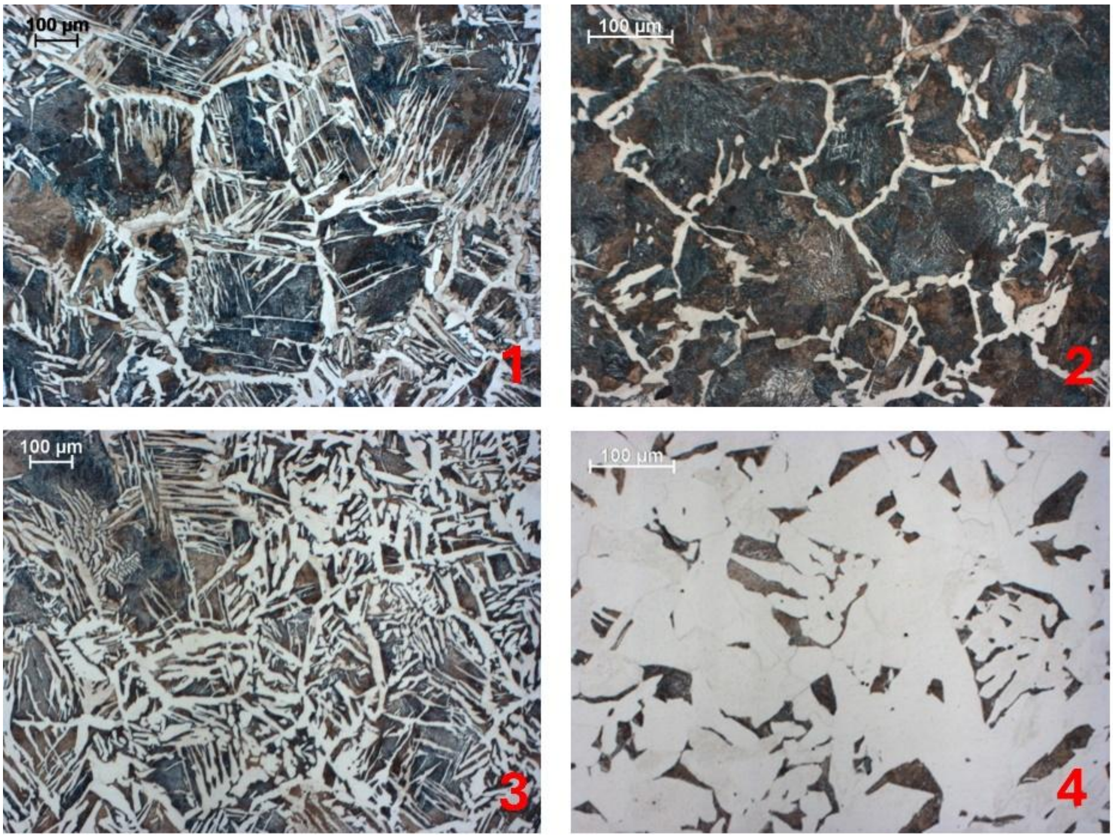

Fig. 6: Characteristic microstructures of the B2A sample. In the sample, a mainly ferritic-perlite structure can be seen. The amount of ferrite depends on the carbon content. In areas with high carbon content, few ferrites form a net (point 2), in areas with low carbon content perlite between the ferrite grains can be found (point 4). Medium carbon areas are characterized by Widmanstätten ferrite (points 1 and 3).

CONCLUSIONS

From the test results, the assumed method of production is the production and compaction of blooms, and then their hot forging into blocks. Whether the blooms were used at the same time or cut in parts cannot be answered from the series of tests. Forging did not mean a high degree of deformation in terms of mosaic images. After hot forging, the blocks cooled freely in the air from the forging temperature, which is inferred from the formation of Widmanstätten ferrite.

In terms of their microstructure, one of the goals of forging the bars could be homogenization, where the aim could be to reduce the inhomogeneous nature of the

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blooms. Another side of the deformation of the irregularly shaped blooms is that they could be brought into a more regular, more manageable, unified shape and size. The bars can in any case be considered as forging pre-products and in this form were suitable both for processing by full forging and for processing via the hot forging process by folding. In terms of supplying blacksmiths, it can be considered a higher-value pre-product than a bloom itself. This made it possible to separate the metallurgical and the processing process, as well as its expertise, which still characterizes metal processing today.

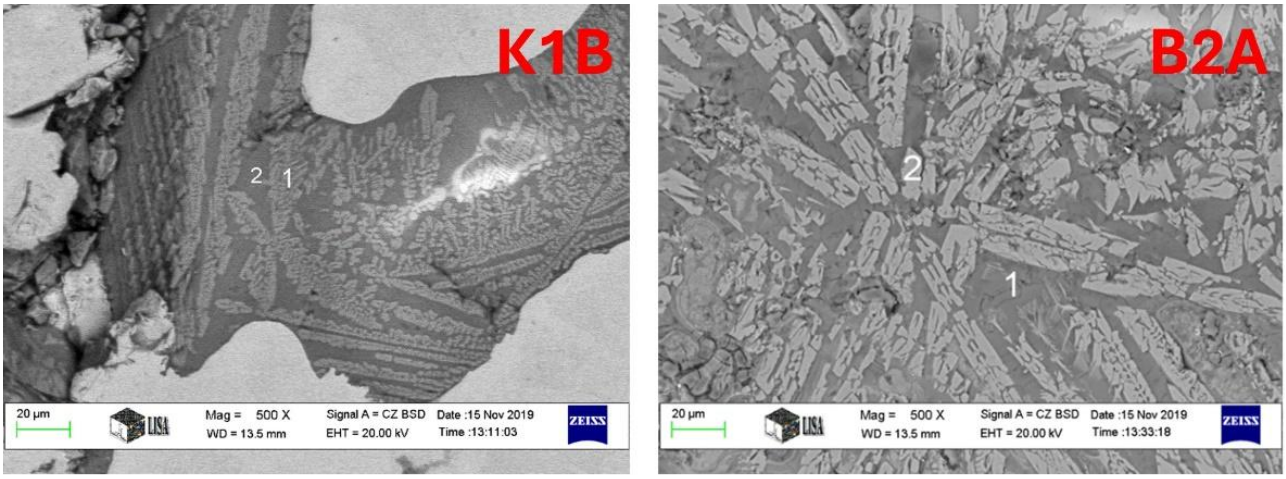

Fig. 7: SEM images of inclusions in the samples. Chemical composition analysis was performed at the marked points. The results are summarized in Table 1.

Location	O	Na	Mg	Al	Si	P	S	K	Ca	Ti	Mn	Fe
Fig. 7 K1B/1	25,08	0,22	2,47	3,12	21,05	0,20		1,45	4,68	0,42	11,50	29,81
Fig. 7 K1B/2	25,33	0,88	0,38	11,39	26,43	0,65	0,97	3,91	11,87	1,00	4,13	13,05
Fig. 7 B2A/1	38,83	0,14	0,30	11,29	11,16	0,55	0,14	0,03	0,33	0,86	0,76	35,60
Fig. 7 B2A/2	28,35		2,04	1,61	17,84	0,24	0,06	0,68	2,35	0,22	8,44	38,16

Tab. 1: Results of EDS chemical composition analysis by w/w%. The analysed points are denoted in Figure 7.

As indicated in the introduction, relatively few findings exist, and an extremely small number of reported test results could be found. Consequently, any new data sets like the present one are extremely important. Even comparing the results of the analysis of the two iron bars, interesting questions arise. Each sample contained several large inclusions (Fig. 7). The chemical composition of these was determined by EDS analysis (Tab. 1). The inclusions are multiphase, so both the glassy phase and the crystalline phase were analysed. Both inclusions can be said to be of metallurgical origin. However, the inclusion of sample K1B has a much higher calcium content and manganese content than the inclusion of sample B2A. In addition, the Si/Ca ratio is different, and lower in the K1B inclusion. This indicates that the two inclusions were not formed under the same smelting parameters, i.e., it can be assumed that the raw material is possibly not from the same origin. This also supports the hypothesis that bars from different smelters served to supply local blacksmiths. This also

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seems to be supported by the significant weight differences observed between the Croatian and Kapos Valley bars. However, based on recent test results for the two iron bars, which differ slightly in shape and proportions, it is still possible that one of the smaller smelters may have tried to produce a product like the better-known shape and quality, probably at a lower price. At the same time, these further nuances in the picture emerged during the research, which we will be able to outline even more accurately once we are in the possession of a larger data set that is suitable for statistical analysis.

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