

## Letter to the Editor

# METALLOGRAPHIC RESEARCH OF THE LATE BRONZE AGE SICKLES FROM SLOVENIA

MIRKO DOBERŠEK, NEVA TRAMPUŽ OREL

Sickles dated to the Period HaA (12-11 cent. B.C.) discovered in three hoards of Slovenia, in Črmošnjice, Debeli Vrh, and Jurka Vas were investigated by optical microscopy, electron probe microanalysis (EPMA), and inductive coupled plasma-atomic emission spectrometry (ICP-AES). The sickles were made of tin bronze and „pure“ copper. All the sickles were cast in sandstone moulds. The presence of  $\text{Cu}_2\text{S}$  and  $\text{FeS}$  in all the investigated sickles indicates that the artefacts were prepared directly from primary raw materials. The concentration of four impurities (As, Sb, Ni, Co) suggests that the same raw material was used in manufacturing sickles. Some of them were cold smithed, while the others were not. The distribution of ( $\alpha$ + $\text{Cu}_2\text{S}$ ) eutectics permits a conclusion that the sickles were cast in the upright position, with the blades downwards.

## 1. Introduction

As in other parts of Europe in the period between 1700–800 BC, the Bronze Age appeared also in the south-eastern Alpine region (present territory of Slovenia). The extraction and working of metals (copper, tin, and gold) made significant advances, and the organization of supplying these metals had reached a high level. With the discovery of bronze, the production of various objects increased significantly, and the market of bronze products started flourishing.

The relative closeness of Slovenia to large metallurgical centres and cultures, which were the leading ones in development of bronze technology, i.e. Aegeia, the Carpathian Basin, and later the Italic peninsula, had an effect on developments only in the Late Bronze Age (1300–800 BC). The majority of hoard-finds in Slovenia from LBA is from the period between 13<sup>th</sup> and 11<sup>th</sup> century BC. There are almost twenty of them, as Cerovec, Pušenci, Črmošnjice, Debeli Vrh upon Predgrad, Jurka Vas, and others. There are many archaeological indications that

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Prof. Dr. M. Doberšek, Institute of Metals and Technologies, Lepi pot 11, 1000 Ljubljana, Slovenia.

N. Trampuž Orel, Senior Curator, National Museum, Archaeological Department, Prešernova 20, 1000 Ljubljana, Slovenia.

these hoard-finds belong to the large group of Urnfield hoards of Central Danubian area, Northern Croatia, Austrian Styria, and Carinthia. They have been classified chronologically into the BrD to HaA period, according to the chronology of Muller-Karpe [1], i.e. 13<sup>th</sup> to 11<sup>th</sup> century BC, and they show close connections between the south-eastern Alpine region and Pannonian regions. The extensive chemical analyses of Late Bronze Age (LBA) metal artefacts from Slovenia were carried out by the National Museum and the Institute of Chemistry in Ljubljana. With metallographic analyses, microanalyses of the distribution of elements in single phases, and chemical compositions of the studied sickles, we detailed the technology of their production and evaluated their functions.

## 2. The interpretation of results

The sickles presented in this paper were selected from three HaA hoard-finds: Črmošnjice, Debeli Vrh upon Predgrad, and Jurka Vas. They belong to the HaA period (12<sup>th</sup>–11<sup>th</sup> century BC) after Muller Karpe, and to the Uiora type of sickles with regional variants after Primas [2]. Their basic characteristics are: an elongated, plastically shaped blade, a handle with decorated ribs, a pronounced handle spur, and no rivets. Sickles of this type were wide spread in the Carpathian basin, in northern Balkan peninsula, Lower Austria, and south-western Slovakia in the period from BrC2 to HaA1, i.e. 14<sup>th</sup> to 11<sup>th</sup> century BC. The selected sickles from Slovenia can be incorporated into the above mentioned distribution.

The chemical analyses of metal artefacts from some of the LBA hoards in Slovenia showed that tin content changed in the BrD – HaA period according to the type of the artefact [3]. Sickles contained 3–7% tin, axes 7–8%, and swords and spearheads 9%. The above differentiation of alloys is very interesting, as modern knowledge of copper alloys with tin ascertains that strength and hardness of the alloys increases with tin content. Maximum ductility is obtained with alloys containing 4% Sn [4]. The possibility of thinning, which is a characteristic of alloys with a high elongation and a greater hardness, are properties which are necessary for a successful preparation of sickle blades (frequent forging and whetting).

The frequency of sickles as a function of tin content in sickles of Slovenian hoards has a maximum in the range 2.5–4.5% Sn. It was found that 80% of all the investigated sickles has concentration of tin in bronze below 5%. The above research led us to the conclusion that the manufacturing of bronze alloys for sickles from the south-eastern Alpine region in the HaA period was intentional and controlled which manifests a high technological level of the smelters. The concentrations of impurities as As, Sb, Ni, Co were found to be in the order As < Sb < Ni < Co in 62% of the investigated sickles confirming that the same raw material has been used for their making.

The morphology and the distribution of the phases in the alloys was determined by the optical microscopy. The samples for metallographic analyses and

microanalyses (EPMA) were taken transversally across the blade of the sickles (Fig. 1a,b,c). Metallographic samples were prepared according to standard metallographic techniques. Alkaline etching reagents were used to reveal the microstructure: 5% solution of  $\text{FeCl}_3$  in alcohol and 10% solution  $(\text{NH}_4)_2\text{S}_2\text{O}_8$  in  $\text{H}_2\text{O}$ .

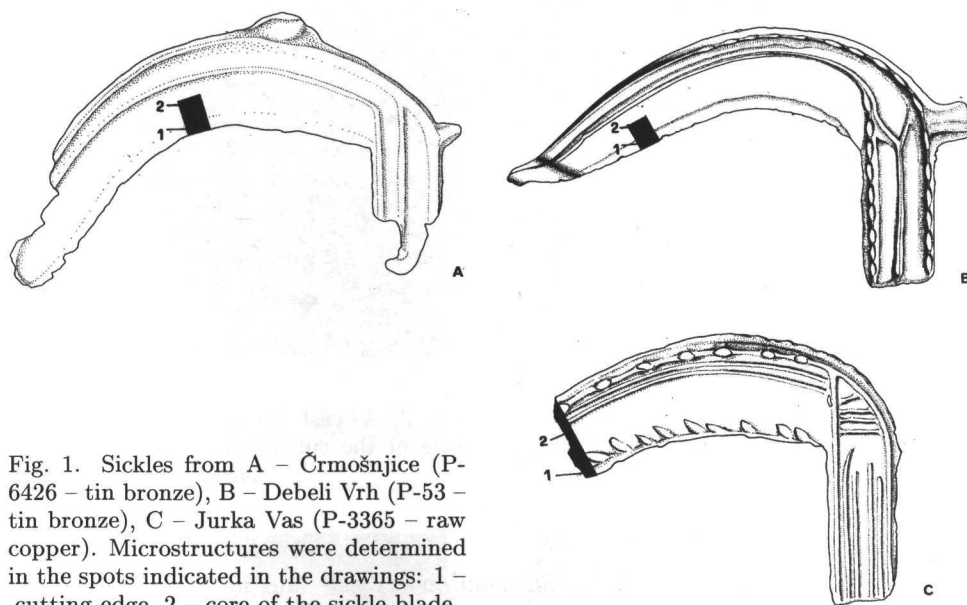


Fig. 1. Sickles from A – Črmošnjice (P-6426 – tin bronze), B – Debeli Vrh (P-53 – tin bronze), C – Jurka Vas (P-3365 – raw copper). Microstructures were determined in the spots indicated in the drawings: 1 – cutting edge, 2 – core of the sickle blade.

The microstructure of the sickle from Črmošnjice was composed of primary crystallized solid-solution copper dendrites and a second phase of  $\text{Cu}_2\text{S}$  in dendrite pockets (Fig. 2). The quantity of the  $\text{Cu}_2\text{S}$  phase was essentially lower than that in the sample from Jurka Vas. The gas porosity was a result of the gas trapping during the casting process. The undeformed dendritic grains on the edge of the sickle blade indicate that these sickles were cold worked by smithing yet (Fig. 3). The surface roughness of the sample indicates that the sickle was cast in a sandstone mould. The microdistribution of the elements in individual phases confirms the presence of the  $\text{Cu}_2\text{S}$  phase with the greatest concentration of sulphur in the globules of this phase. In certain places, there is an increased concentration of sulphur and iron which indicates the presence of an  $\text{FeS}$  phase. It can be concluded that the Črmošnjice sickle was made by alloying „raw“ copper with tin and not by remelting scrap. The very weak coring of tin around the edges of the casted structures and the even distribution of antimony, arsenic, and nickel in the microstructures indicate slow cooling after the casting.

An example of cold working after casting is the sickle from the hoard of Debeli Vrh. The microstructure of the sample is composed of very strongly microseg-

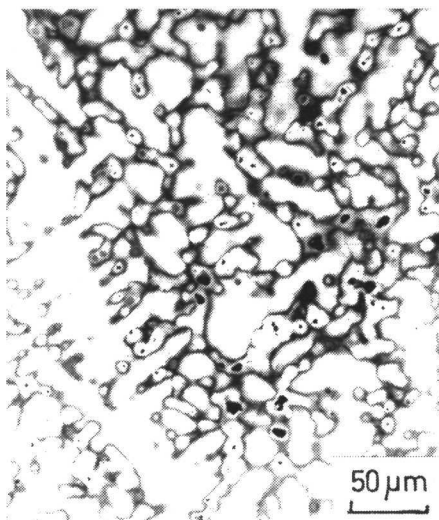


Fig. 2. Microstructure of the cutting edge of the sickle from Črmošnjice (P-6426).



Fig. 3. As cast, not deformed microstructure of the cutting edge of the sickle P-6426.

regated layers of tin in copper solid solution crystals and an interdendritically dispersed fine grey  $\text{Cu}_2\text{S}$  phase (Fig. 4). The very strong microsegregation of tin indicates a rapid dissipation of heat during cooling. The form of the  $\text{Cu}_2\text{S}$  phase in the lower part of the sample is elongated, i.e. deformed, whilst in the upper part it is globular, i.e. undeformed. The blade of this sickle, etched with 10%  $(\text{NH}_4)_2\text{S}_2\text{O}_8$ , exhibits deformed dendritic grains (Fig. 5) which shows the effect of cold working of the artefact. The microanalysis of this sample confirmed the presence of copper solid-solution crystals with very significant microsegregation of tin and the phases of  $\text{Cu}_2\text{S}$  and  $\text{FeS}$ .

The sickle from Jurka Vas was interesting because we have found that it was made by casting „pure“ copper. The surface of the sample and the coarse quasipolygonal grains with very weak microsegregation indicates the casting in the sandstone mould (Fig. 6a). The microstructure is composed of large primarily crystallized grains of copper solid solution and of the precipitated globular  $\text{Cu}_2\text{S}$  phase which was found both inside the grains and on the grain boundaries (Fig. 6b).

In the lower part of the sickle, the  $(\alpha+\text{Cu}_2\text{S})$  eutectics was not observed though it was present in the middle part. The portion of the eutectics increases towards the upper part of the sample (Fig. 7), so it may be concluded that the artefact was cast in an upright position (with the blade downward) which positively effected the

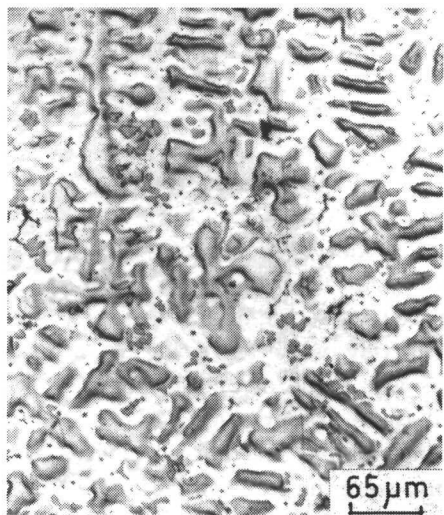


Fig. 4. Microstructure of the etched core of the sickle from Debeli Vrh (P-53).

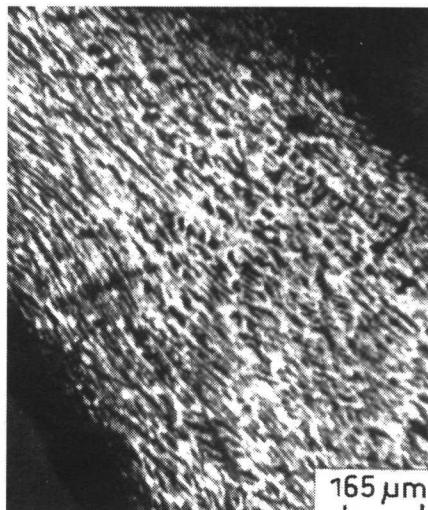


Fig. 5. Microstructure of the cold hammered cutting edge of the sickle P-53.

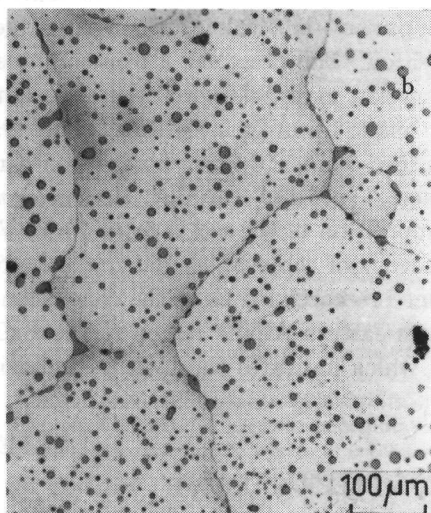
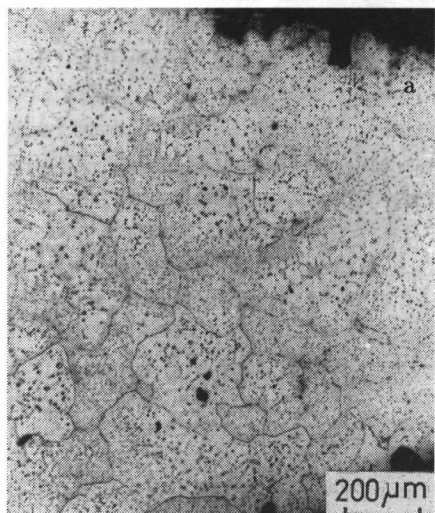


Fig. 6a,b. Microstructure of the cutting edge of the sickle from Jurka Vas, a – the surface of the sickle, b – the quasipolygonal grains without significant segregations.

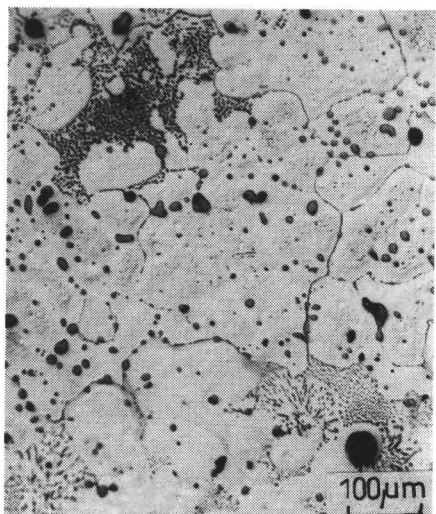


Fig. 7. Microstructure of the core of the sickle from Jurka Vas showing the existence of  $(\alpha + \text{Cu}_2\text{S})$  eutectics.

increase of  $(\alpha + \text{Cu}_2\text{S})$  eutectics content from the tip of the blade towards the upper part of the sickle (Jurka Vas) suggests that this object was cast in an upright position. The presence of iron sulphide, FeS, in all three samples shows that primary raw materials were used in making these sickles, and that the artefacts were not made from remelted scrap. The blade of the sickle from Debeli Vrh was cold smithed while the sickle blades from Črmošnjice and Jurka Vas were not. The absence of traces of smithing, the decoration on the edge of the blade, and chemical composition of the sickle from Jurka Vas (Sn 0.02%) places this artefact into the category of tools which did not have function of a usable tool. The above mentioned research led us to the conclusion that the making of bronze sickles from the south-eastern Alpine region in the BrD-HaA period was controlled and purposeful which indicates the high technological level of the smelters who supplied this region.

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quality of the casting. There are no traces of shrinkage porosity in the sample, and also gas porosity is relatively low.

### 3. Conclusion

The sickles from Črmošnjice and Debeli Vrh were made by casting tin bronze with 2.85, and 3.30 m.% Sn, respectively, while the sickle from Jurka Vas was made of „pure“ copper cast. All three artefacts were cast in sandstone moulds. The different microstructures were the result of different chemical compositions and different cooling rates. The presence of very strongly microsegregated cored crystals in the sickle from Debeli Vrh indicates rapid cooling rates of the casting. The in-