METALLOGRAPHIC INVESTIGATIONS OF A HISTORICAL BLOOM FOUND IN STYRIA – AUSTRIA

S. Strobl¹, R. Haubner¹, S. Klemm²
¹ University of Technology Vienna, Institute of Chemical Technologies and Analytics, Getreidemarkt 9/164-CT, 1060 Vienna, Austria
² Prehistoric Commission, Austrian Academy of Sciences, Fleischmarkt 22, 1010 Vienna, Austria

Received 10.02.2010
Accepted 26.03.2010

Corresponding author: Dr. Susanne Strobl Technische Universität Wien Institut für Chemische Technologien und Analytik 164-CT Getreidemarkt 9 A-1060 Wien tel.: 0043-1-58801-16167 fax: 0043-1-58801-16199 email: sstrobl@mail.zserv.tuwien.ac.at

Abstract
In Palfau (Styria/Austria) a bloom was found during archaeological investigations of a historical trade route, the so-called ‘Dreimärktestraße’. As iron smelting sites have not been recorded in the vicinity, the original production site remains unknown; it is assumed that the bloom was lost during transport.

The bloom has a diameter of about 18 to 19 cm and shows corrosion products and stones on its surface. A fragment from one side of it was used for metallographic investigations.

The bloom contains many pores and inclusions like oxides and also charcoal used in the metallurgical process. From the metallurgical point of view microstructures from cast iron over steel to pure iron were observed. Several profiles through the sample showed that there are strong carbon gradients in the metallic iron responsible for the various microstructures.

The results are in comparison with the technological aspects of a historical bloomery.

Keywords: bloom, Fe-C microstructures, archaeometallurgy

Introduction
The so-called ‘Dreimärktestraße’, today called B 25 ‘Erlauftal Straße’, is an old historical trade route for the transport of raw iron, iron products and other goods from Eisenerz in Styria to Lower Austria since the Mediaeval Period. During archaeological investigations of this route near Palfau (Styria) in the years 1999 and 2000 [1] also a bloom was found (Fig.1). As iron smelting sites have not been recorded in the close vicinity, the original production site remains unknown; it is assumed that the bloom was lost during transport.

In Austria the first aggregates for iron production were bloom furnaces too, but less is known about the era between 1000 and 500 B.C. [2]. From the Roman period already iron blooms were found and investigated [3]. From later time periods up to the 14th century several bloomeries were documented [4-6] in Carinthia and Styria. South-east of Palfau, at a location called Dürrnshöberl near Admont in Styria, the remains of a mediaeval iron shaft furnace was excavated [7]; archaeological experiments [8], copying this type of shaft furnace, produced a typical bloom, similar to the one found at Palfau. Following the description of Otto Johannsen (1925) [9] bloom furnaces were still used in Styria in the 18th century mainly for small productions called ‘Waldeisen’ [2].
The investigated bloom has an irregular oval form and a diameter of 18 to 19 cm (Fig.1). Its bottom side has a smoother surface than the rough and irregular upper side. Some spots of rust indicate that corrosion occurred. A small fragment of the bloom was used for the examinations (Fig.1d).

**Experimental**

The sample was cleaned in an ultrasonic bath and cut. Metallographic preparation started with grinding, using SiC paper (P 120, 320, 600 and 1200), followed by polishing with 6 μm diamond and 1 μm Al₂O₃ suspension. For etching, Nital (2 % HNO₃ in methanol) was used. The microstructures were examined by light optical microscopy (LOM).

**Results and Discussion**

**Cross sections of the investigated sample**

The investigated part of the bloom was very porous, contained oxides and charcoal and showed a broad variety of Fe-C microstructures [10]. To illustrate this diversity four profiles are shown in Fig.2.

In Fig.2a oxide inclusions, mainly surrounded by ferrite can be seen (left side) and the carbon content increases from the left to the right side. Similar microstructures are observed in Fig.2b but there are more pores and corrosion products. Fig.2c represents a profile with higher carbon content consisting of pearlite and acicular cementite. Massive cementite (highest amount of carbide) was observed in Fig. 2d (see on the left side of the picture). Details of these microstructures are shown below.

**Fe-C microstructures with a content < 0.8 % C**

In Fig.3 Fe-C microstructures with < 0.8 % C are shown. In Fig.3a oxides, beside large ferrite grains, can be seen and only small amounts of pearlite are observed. During the reduction of the iron oxide in this region the carbon concentration decreases because it gets consumed. Some of the smaller inclusions seem to be slag impurities. Nearby the oxides large ferrite grains become visible and the microstructure changes from ferrite to pearlite due to a carbon gradient (Fig.3b). In the ferrite-pearlite region small cementite needles are precipitated within the large ferrite grains (Fig.3c). These needles have a length of about 1 μm and can be explained by the solubility of carbon in ferrite at the eutectoid temperature. The cementite precipitation takes place at ambient temperature during long time storage [11]. Fig.3d and e show regions with higher amounts of pearlite and less ferrite. In Fig.3d the ferrite grains are long and organized
needle clusters with three main orientations tilted for 120°. This microstructure can be described as “Widmannstätten” ferrite formation, which normally occurs when very large austenite grains are cooled rapidly [10]. Fig.3e represents a surface region with ferrite at the outside and inside pearlite, Fig.3f shows an area with nearly pure pearlite.

Fig.2 Cross sections through the investigated sample of the bloom (Nital etching)

**Fe-C microstructures with a content > 0.8 % C**

Fe-C microstructures with > 0.8 % C observed in the cross sections of the bloom are shown in Fig.4. A nearly pure microstructure of pearlite with only one cementite needle can be seen in Fig.4a. The cementite is surrounded by a shell of ferrite formed during cooling at the eutectoid temperature. In Fig.4b the amount of cementite with surrounding ferrite is higher and also some pores and inclusions (oxides) can be seen. With increasing carbon content the cementite regions become thicker but pearlite remains the dominant microstructure (Fig.4c). Further increase of carbon results in a cementite network and the pearlite is located in-between (Fig.4d-f). Fig.4d shows a region with a distinct carbide gradient from pearlite to a cementite network. The
network of massive cementite and the amount of pearlite in-between can vary but for these regions carbon contents of about 2.5-3.0 wt.% are assumed (Fig. 4c-f). In the common nomenclature of iron alloys such a microstructure would represent a white cast iron.

Fig. 3 Fe-C microstructures with < 0.8 % C observed in the cross sections of the bloom. (a) ferrite and oxides, (b, c) ferrite and pearlite, (d, e) pearlite with elongated ferrite inclusions, (f) pearlite

Fig. 4 Fe-C microstructures with > 0.8 % C observed in the cross sections of the bloom. (a) pearlite with a large cementite needle, (b, c) pearlite with cementite, (d) region between pearlite and a carbide network, (e, f) massive cementite network and pearlite.

Microstructures of charcoal and corrosion products
In some surface regions of the bloom charcoal from the bloomery process and also regions with corrosion products are observed. Very different microstructures of charcoal are shown in Fig. 5a, b and c. From these observations we can conclude that the charcoal used was produced of different kinds of wood, as was typical for late mediaeval charcoal production in the area [12]. It is also obvious that there was an excess of charcoal in the bloomery process responsible for the carbon gradients in the bloom.

The corrosion products are the result of storage within the soil for several hundred years. In principal, the results correspond with the corrosion phenomena of modern steels and cast irons.
At first the ferrite corrodes and the cementite remains. **Fig.5d** shows that the large ferrite grains corrode faster and the pearlite, because of the cementite lamella, corrodes slower. In **Fig.5e** a region can be seen where the network of ferrite is strongly attacked and pearlite remains in between. In case of higher carbon content and formation of a massive cementite the ferrite within the pearlite is corroded (**Fig.5f**). The cementite lamella can be seen clearly between the corrosion products of the ferrite.

![Charcoal and corrosion](image)

**Fig.5** Charcoal and corrosion; (a-c) different cross sections of charcoal, (d-f) corroded regions of the bloom

*Considerations about the bloom production and the bloom microstructures*

Following the descriptions by Gabriell Jars [13] and Harald Straube [14] for the production process in a bloomery the different microstructures in a bloom can be explained as follows:

The bloomery is heated by burning fresh wood or charcoal and after a certain temperature is reached the iron ore and additional charcoal are added. At lower temperatures the ore is reduced by the CO, which is formed during firing. At higher temperatures the reduction takes place by the direct contact of ore with charcoal. Reduced metallic iron, remaining ore and charcoal form the bloom; the bloom is placed at the bottom of the bloomery.

In Europe until the late Mediaeval Period the temperature achieved for the smelting process was not high enough to produce cast iron, therefore a homogeneous alloy could not be formed. All processes within the bloom are diffusion controlled and so very inhomogeneous microstructures can be observed. The carbon content in the metallic part of the bloom is dictated by the overall carbon balance. In case of smaller bloomeries due to the more inhomogeneous temperature distribution and shorter process time the local variations in a small bloom can be larger compared to a large bloom.

Normally, the bloom gets forged after production; this is not true for the bloom presented here. As described by Johannsen [9] blooms with diameters up to 55 cm were produced in Styria up to the 18th century. From this point of view the bloom investigated in our study was either produced in a bloomery, which was not state-of-the-art in the 18th century, or the bloom found is much older, possibly dating to the Mediaeval Period.
Conclusions
The investigated bloom shows the typical size for small bloomeries used until the Mediaeval
Period. There are also no hints that the bloom was forged after having taken out of the furnace. It
seems that this bloom represents the status directly after reduction. The bloom is very
inhomogeneous, containing cavities, oxides and charcoal beside of metallic iron. Also the
metallic parts are very inhomogeneous caused by different carbon concentrations. Regions
containing nearly carbon free ferrite change to regions containing up to 3 wt.% C. Different
microstructures of hypo-eutectoid and hyper-eutectoid steel were observed and described.

Acknowledgements
We like to thank Doz. Dr. Bernhard Hebert, Bundesdenkmalamt/Landeskonservatorat Graz,
Austria, for the financial support.

References
128, 1983, 163-168
183-204
Aquileia, Italy, paper 76
43–63.
M.B.H.
mbH, Düsseldorf
Eisen- Stahl- Blech- und Steinkohlenwerke in Deutschland, Schweden, Norwegen, England,