IRON-BASED BAR - FE ALLOY - MODERN TIMES - FRANCE

Artefact name	Iron-based bar
Authors	Christian. Degrigny (HE-Arc CR, Neuchâtel, Neuchâtel, Switzerland) & Mathea. Hovind (University of Oslo, Department of archaeology, conservation and history (IAKH-UiO), Oslo, Oslo, Norway)
Url	/artefacts/404/

The object

Credit UiO-IAKH, M.Hovind.

Fig. 1: Wrought iron bar. Surface “a” and “b”, to the left and right, respectively,

Description and visual observation

Description of the artefact	Wrought iron bar with a circular pierced tip (Fig. 1). Its surface is covered by brown and orange-red corrosion products in addition to localized deposits of soil and charcoal. Dimensions: L = 374mm; W = 21mm; T = 6mm; WT = 293g.
Type of artefact	None
Origin	Château de Germolles, Mellecey, Bourgogne, France
Recovering date	Unknown
Chronology category	Modern Times
chronology tpq	1801 A.D.
chronology taq	2000 A.D.
Chronology comment	19th - 20th century
Burial conditions / environment	Outdoor atmosphere
Artefact location	Haute Ecole Arc Conservation-Restauration
Owner	Château de Germolles, Mellecey, Bourgogne

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Inv. number	No inventory number.
Recorded conservation data	Not conserved

Complementary information

Nothing to report.

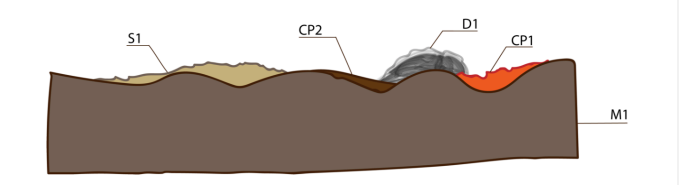
Study area(s)

Credit UiO-IAKH, M.Hovind.

Fig. 2: Zones of the artefact submitted to visual observation and location of sampling area (a cross-section of the metal, marked by a stippled line),

Binocular observation and representation of the corrosion structure

The schematic representation below (Fig. 3) gives an overview of the corrosion layers encountered on the object from a first visual macroscopic observation.



Captions	Description
S1	Light brown soil with a porous and friable consistency.
D1	Black, powdery deposit. Very loosely bound to the surface. Probably charcoal.
CP2	Dark brown and strongly adherent corrosion crust. Almost uniform in appearance - except in areas where it is disturbed by corrosion blisters.
CP1	Bright to dark orange corrosion products.
M1	Remaining metal. Dark grey to brown and matte in appearance. Highly irregular surface with dark pits ranging from approximately 1 – 5 mm in diameter.

Credit UiO-IAKH, M.Hovind.

Fig. 3: Preliminary stratigraphy corresponding to the overall surface of the artefact. CP = Corrosion Product, D = Deposit, S = Soil, M = Metal,

MiCorr stratigraphy(ies) – Bi

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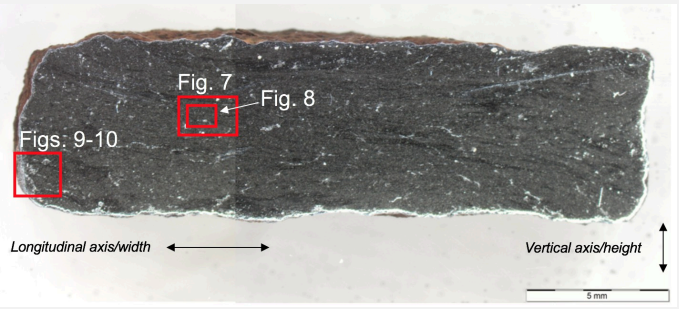
Sample(s)



Credit UiO-IAKH, M.Hovind.

Fig. 4: Micrograph of the cross-section with description of its orientation and location of Figs. 7-10,

Description of sample	A rectangular section (Fig. 4) was cut out from the tip of the iron bar. Its longitudinal axis corresponds to the width of the iron bar while the height/thickness of the bar is represented by the vertical axis.
Alloy	Fe Alloy
Technology	None
Lab number of sample	WIB2018 (Wrought Iron Bar sampled in 2018)
Sample location	Haute Ecole Arc Conservation-Restauration
Responsible institution	Haute Ecole Arc Conservation-Restauration
Date and aim of sampling	March 2018, study of corrosion stratigraphy and chemical analyses

Complementary information

The fact that the artefact was considered as test material enabled extensive sampling that would not otherwise be possible.

Analyses and results

Metallography

Microscope: Leica DMi8 (a metallographic, inverted, reflected light microscope) with magnification up to 500X.
Camera: Olympus SC50 connected to the software “Olympus Stream”, version 1.9.4. Illumination modes: bright field and cross-polarized light.

SEM-EDS

Instrument: Jeol 6400; voltage: 20 kV; working distance: 18 and 24mm; sample preparation: palladium depot.

Non invasive analysis

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Metal

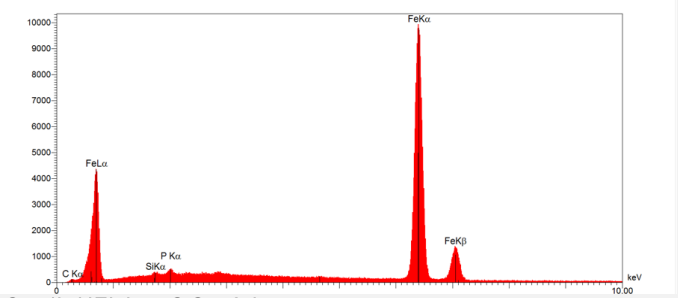
The metal is a wrought iron consisting of Fe, with some P and C (Fig. 6). The presence of Si is due to slag inclusions. The inclusions appear elongated[1] (Fig. 7) and filled with phases appearing light grey, medium grey and dark grey both in bright field and in SEM in BSE-mode (Figs. 7 and 8). Punctual analysis by SEM-EDS (Table 1) revealed that the light grey phase consists mainly of Fe and O with some C and has a composition similar to Wüstite (FeO). The medium grey phase has a similar composition but contains more P and C in addition to Si (Table 1). This phase is probably Wüstite in a Fe-P matrix. The dark grey phase corresponds to the glassy matrix and contains significally higher concentrations of Si and P, in addition to the usual Fe and O (Table 1). The relatively high Si-content indicates that this phase might be Fayalite (FeSiO4) in a Fe-P matrix.

Smaller inclusions/nodules are evenly distributed throughout the metal (Fig. 7 and 8). They appear dark grey and have a composition similar to the dark grey phase in the elongated inclusions (Table 1).

Elements mass%	Fe	O	P	Si	C	V	S	Mn	Al	Cr	Mg	Ca
Light grey phase	83	12	0.1	0.2	2	2	0.1	0.4	0.2	0.4	-	-
Medium grey phase	74	15	4	3	3	0.8	0.7	0.6	0.3	0.1	-	-
Dark grey phase	52	22	13	8	3	-	0.2	-	0.1	-	0.2	0.1
Nodules	49	27	11	5	6	-	1	0.9	0.1	-	-	-

Table 1: Chemical composition of the different phases in the slag inclusions and the nodules in the metal matrix. Method of analysis: SEM-EDS. Lab. of Electronic Microscopy and Microanalysis, Néode, HEI Arc, credit MiCorr_HEI Arc, C.Csefalvay.

[1] As the section was cut across the iron bar – it is the cross section of the inclusions that are visible. Thus, their length and direction cannot be deduced directly from the sample.



Credit HEI Arc, C.Csefalvay.

Fig. 6: EDS-spectrum showing the chemical composition of the metal (M1). The surface area analyzed was approx. 2*2 mm. Method of analysis: SEM-EDS. Lab. of Electronic Microscopy and Microanalysis, Néode, HEI Arc,

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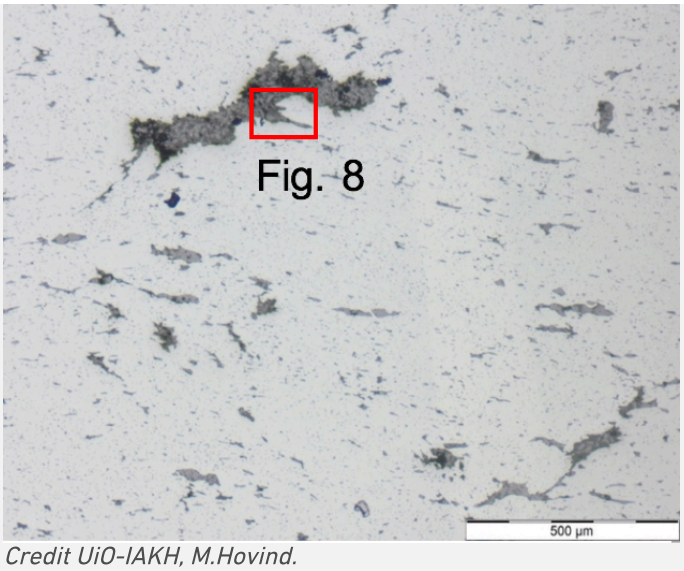
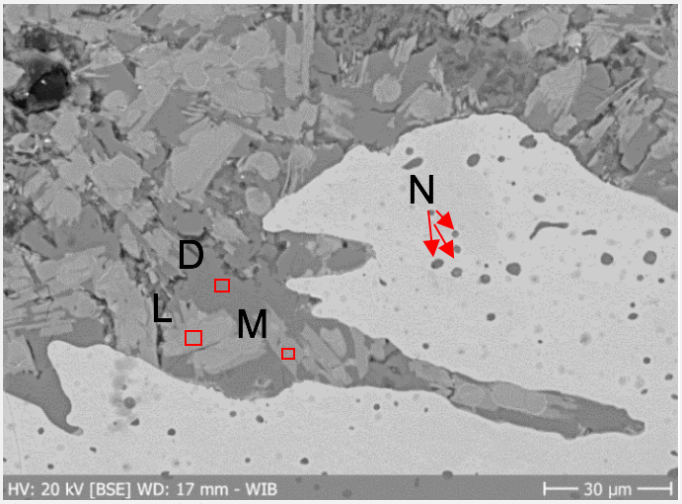


Credit UiO-IAKH, M.Hovind.

Fig. 7: Micrograph of the metal sample from Fig. 4 (detail), unetched, bright field. The microstructure of the metal with slag inclusions and the location of Fig. 8,



Credit HEI Arc, C.Csefalvay.

Fig. 8: SEM-image (BSE-mode) of the selected area from Fig. 7 (detail). The different phases in the slag inclusions are clearly visible: L = light grey phase, M = medium grey phase, D = dark grey phase/matrix, N = nodules,

Microstructure	None
First metal element	Fe
Other metal elements	C, P

Complementary information

Nothing to report.

Corrosion layers

The corrosion crust is relatively thick and consists of two layers: CP1 and CP2. The latter is a dense product layer appearing dark grey under both bright field and polarized light (Figs. 9 and 10). The outermost layer (CP1) is a porous crust, appearing dark grey under bright field and bright orange under polarized light (Figs. 9 and 10). The corroded metal (CM1) appears as isolated areas of corrosion within the sound metal.

The composition of the corrosion products shows a varying content of Fe and O throughout the crust with an increasing O-content towards the surface of the corrosion layer CP1 (Table 2). Elemental mapping by SEM-EDS (Fig. 11) shows that Ca and Mg are present in cracks which penetrates the outer corrosion crust (CP1). These elements are probably exogenous in origin and could originate from the layer of soil (S1) that is covering the metal surface.

Elements	Fe	O	C	Ca	Si	P	S	Mg	Al	V	Cr	Mn

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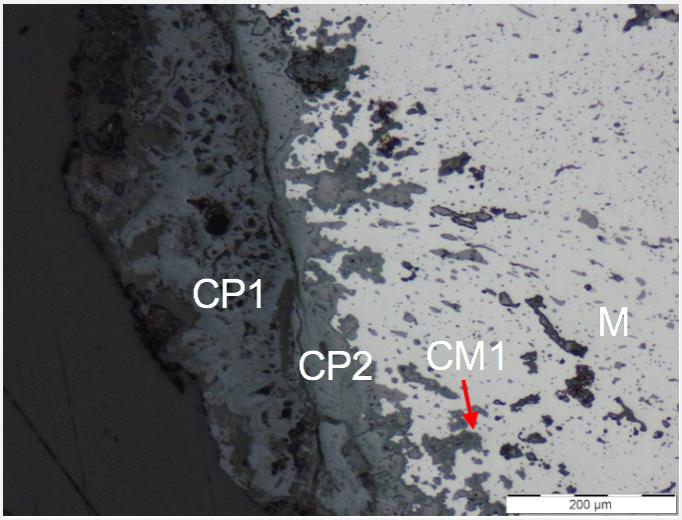
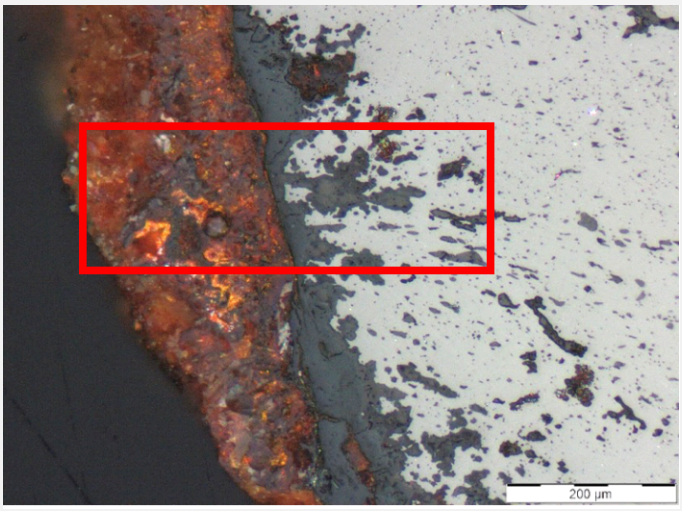
mass%												
CP1	57	36	5	1.0	0.3	0.3	0.3	0.3	0.1	-	-	-
CP2	68	27	3	0.5	0.4	0.4	0.1	0.1	-	-	-	-
CM1	70	25	3	0.1	0.5	0.5	0.3	0.1	0.1	0.3	0.1	0.1

Table 2: Chemical composition of the corrosion layers from Figs. 9 and 10. Method of analysis: SEM-EDS. Lab. of Electronic Microscopy and Microanalysis, Néode, HEI Arc, credit MiCorr_HEI Arc, C.Csefalvay.



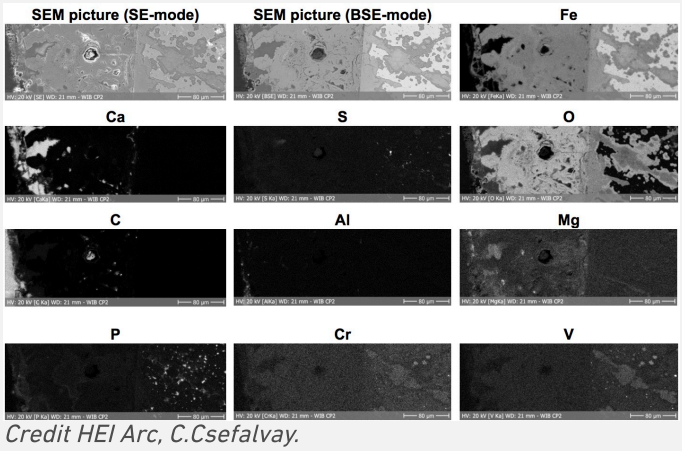
Credit UiO-IAKH, M.Hovind.

Fig. 9: Micrograph of the metal sample from Fig. 4 (detail), unetched, bright field. Corrosion layers CP1-CP2 and the corroded metal (CM1) in grey,



Credit UiO-IAKH, M.Hovind.

Fig. 10: Micrograph similar to Fig. 9, but under polarized light. CP1 in orange, CP2 and the corroded metal phase (CM) appear dark grey. The red square corresponds to the area selected for elementary mapping by SEM-EDS (Fig. 11),



Credit HEI Arc, C.Csefalvay.

Fig. 11: SEM image and elemental chemical distribution of the selected area from Fig. 10. Method of analysis: SEM-EDS. Lab. of Electronic Microscopy and Microanalysis, Néode, HEI Arc,

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Corrosion form	Pitting
Corrosion type	Type II (Robbiola)

Complementary information

Nothing to report.

MiCorr stratigraphy(ies) – CS

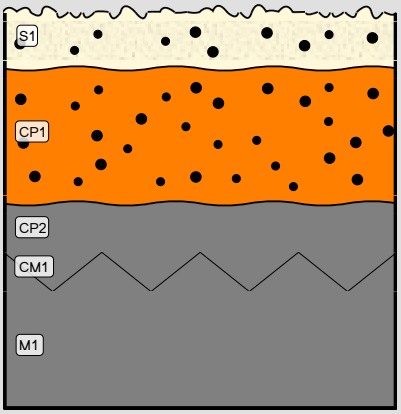
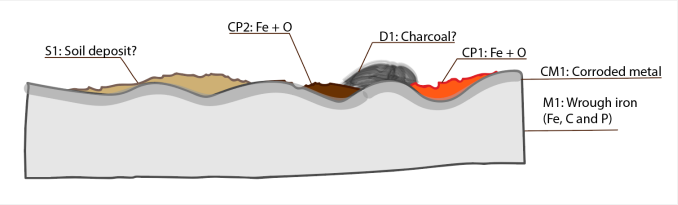


Fig. 5: MiCorr stratigraphy of the corrosion crust, to be related to Figs. 9 and 10, Credit UiO-IAKH, M.Hovind.

Synthesis of the binocular / cross-section examination of the corrosion structure

Nothing to report.



Credit UiO-IAKH, M.Hovind.

Fig. 12: Corrected stratigraphic representation with results from analyses by SEM-EDS and visual microscopic observation. The colour of the metal was changed to grey as this was the colour of the cross-section of the metal, CP = corrosion product, D = deposit, M = metal S = soil, CM = corroded metal,

Conclusion

The artefact is a wrought iron with evenly distributed inclusions of what appears to be wüstite in a fayalite matrix. Wrought iron containing slags was readily available until World War II, after which it was substituted by low-carbon steels (Selwyn 2004:96). This indicates that the artefact can be dated no later than the first half of the 20th century. As regards the production method, it has been suggested (Dr. Phil. M. Senn, 2018, personal communication the 26th of April) that the artefact was produced by puddling, an indirect process for the conversion of pig iron to wrought iron, while decreasing the level of impurities (Selwyn 2004:112-113).

The corrosion products on the surface of the iron bar are typical for iron exposed outdoors with varying contents of Fe an O in addition to a layer of Ca-containing soil.

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References

References sample:

1. Selwyn, L. (2004). Metals and corrosion: A handbook for the conservation professional. Ottawa: Canadian Conservation Institute.

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