RAIN GUTTER FRAGMENT – GREY CAST IRON – MODERN TIMES – FRANCE

Artefact name	Rain gutter fragment
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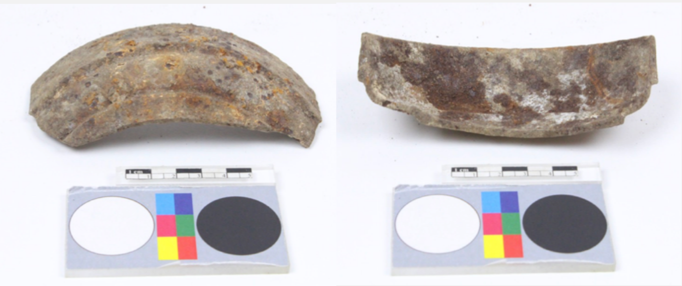
Url	/artefacts/358/

The object



Credit UiO-IAKH, M.Hovind.

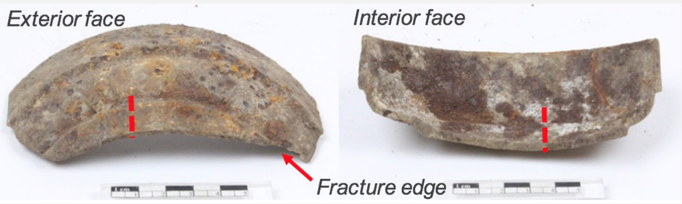
Fig. 1. Cast iron gutter fragment, exterior and interior faces (to the left and right, respectively),

Description and visual observation

Description of the artefact	Rain gutter fragment (Fig. 1), possibly part of the extension of a roof drain pipe. Both the exterior and interior surfaces are covered by heterogeneous corrosion crusts. The underlying metal seems however to be well preserved, including features such as a difference in thickness etc. L = 55mm; W (interior) = 120mm; Tmax. = 6mm; WT = 176g
Type of artefact	Architectural element
Origin	Château de Germolles, Mellecey, Bourgogne, France
Recovering date	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
Inv. number	No inventory number.
Recorded conservation data	No recorded conservation data.

Study area(s)

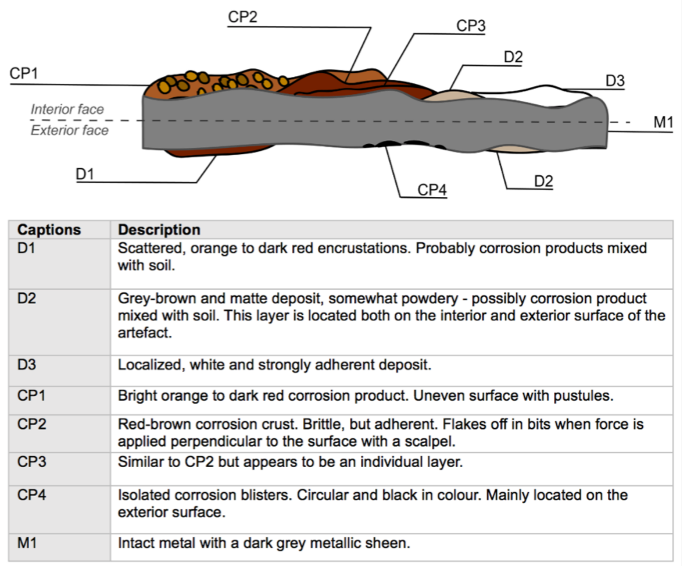


Credit UiO-IAKH, M.Hovind.

Fig. 2. The exterior and interior faces before sampling. The dotted line represents the sample location (a cross-section of the metal). The fracture edge corresponds to the edge that was formerly attached to the remaining artefact,

Binocular observation and representation of the corrosion structure

The schematic representation below (Fig. 3) gives an overview of the strata encountered by visual macroscopic observation.



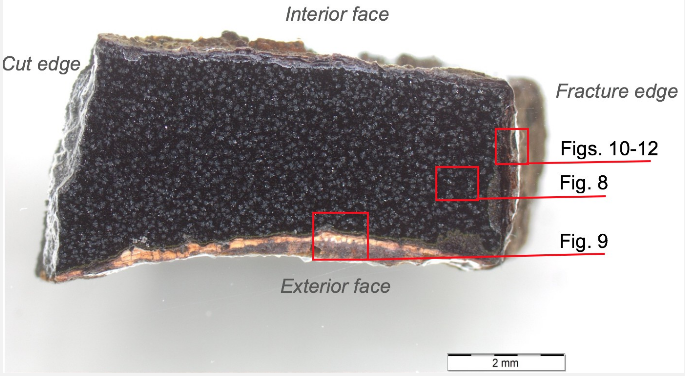
Captions	Description
D1	Scattered, orange to dark red encrustations. Probably corrosion products mixed with soil.
D2	Grey-brown and matte deposit, somewhat powdery - possibly corrosion product mixed with soil. This layer is located both on the interior and exterior surface of the artefact.
D3	Localized, white and strongly adherent deposit.
CP1	Bright orange to dark red corrosion product. Uneven surface with pustules.
CP2	Red-brown corrosion crust. Brittle, but adherent. Flakes off in bits when force is applied perpendicular to the surface with a scalpel.
CP3	Similar to CP2 but appears to be an individual layer.
CP4	Isolated corrosion blisters. Circular and black in colour. Mainly located on the exterior surface.
M1	Intact metal with a dark grey metallic sheen.

Credit UiO-IAKH, M.Hovind.

Fig. 3. Preliminary stratigraphy corresponding to the overall surface of the artefact. The interior surface is shown above the stippled line and the exterior surface below. D = Deposit, CP = Corrosion products, M = Metal,

MiCorr stratigraphy(ies) – Bi

Sample(s)



Credit UiO-IAKH, M.Hovind

Fig. 4. Micrograph of the sample (a cross-section of the metal) showing its orientation relative to the artefact and the locations of Figs. 8-12,

Description of sample	The sample consists of a rectangular section (Fig. 4) which was cut out from the fractured edge of the gutter fragment. It is representative of three surfaces: the exterior and interior surface, as well as the fracture edge. L = 7mm; Wmax. = 5mm; Tmax. = 4mm (approx.).
Alloy	Grey cast iron
Technology	Cast
Lab number of sample	CIG2018 (Cast Iron Gutter, 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 a 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

Metal

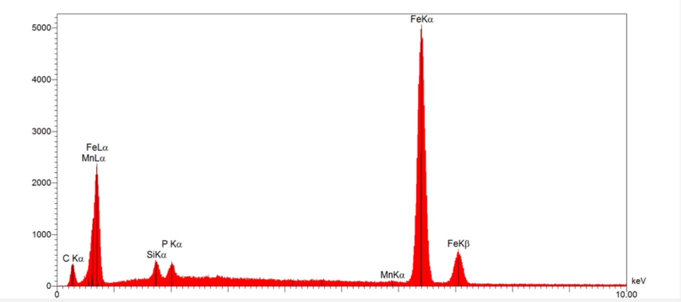
The metal is a P-rich grey cast iron with Si and Mn (Fig. 7). Its microstructure is characterized by evenly distributed graphite flakes in a matrix composed of dendrites and an interdendritic eutectic phase (Fig. 8). What appears to be irregular graphite flakes/porosity is in fact deformed graphite flakes; graphite is soft and prone to destruction by smearing and/or preferential removal during polishing (Scott 1991:38-39).

The dendrites appear light grey under polarized light, while the eutectic phase appear light brown (Fig. 8). The eutectic phase is rich in Fe and P and contains small amounts of C and Si, whereas the dendritic phase contains much less P (Table 1).

Elements **Mass%***	**Fe**	**C**	**P**	**Si**
Eutectic phase	78	7	14	0.5
Dendritic phase	91	5	0.5	3

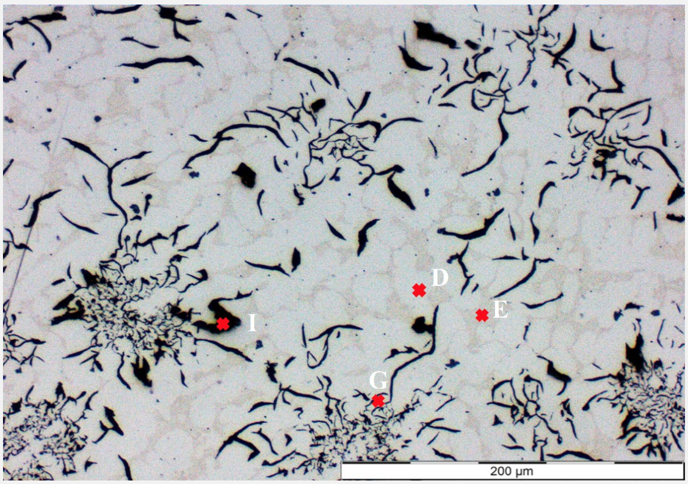
Table 1: Chemical composition of the matrix (eutectic and the dentritic phase). Method of analysis: SEM-EDS. Lab. of Electronic Microscopy and Microanalysis, Néode, HEI Arc, credit MiCorr_HEI Arc, C.Csefalvay.

* The value is the calculated average of three analyses of the same feature, but in different areas.



Credit HEI Arc, C.Csefalvay.

Fig. 7: EDS-spectrum showing the chemical composition of the metal. Method of analysis: SEM-EDS. Lab. of Electronic Microscopy and Microanalysis, Néode, HEI Arc,



Credit UiO-IAKH, M.Hovind.

Fig. 8. Micrograph of the metal sample from Fig. 4 (detail). Unetched, bright field. Graphite flakes (G) in black, the dendrites (D) in light grey and the eutectoid phase (E) in light brown. Deformed graphite flakes are visible as irregular black inclusions (I),

Microstructure	Dendritic structure with graphite flakes and a P-rich eutectic phase
First metal element	Fe
Other metal elements	C, Si, P, Mn

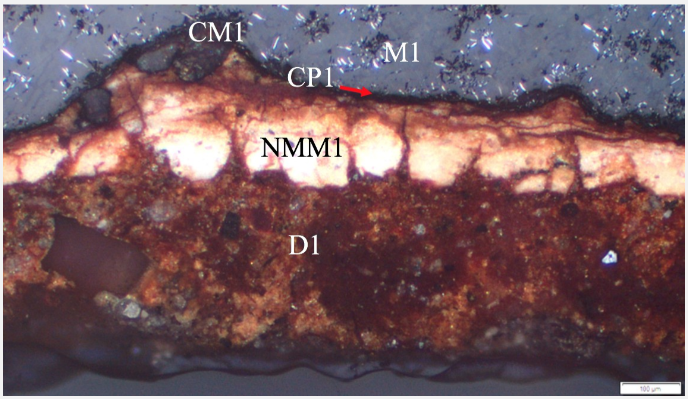
Corrosion layers

The exterior face (Figs. 2, 4 and 9) consists of a white cracked layer (NMM1 in Fig. 5) superimposed by a layer of deposit mixed with orange-red corrosion products (D1 in Fig. 5). Punctual analysis by SEM-EDS (Table 2) revealed that the white layer is rich in Ba, O, S, Fe, C and Zn. It has a chemical composition similar to Lithopone (BaSO4 ZnS), a preparatory paint layer. Fe is probably a contamination from the superimposed deposit/corrosion product D1 (Table 2). The latter (D1) is Fe- and O-rich and contains Si, Al and some Ca, in addition to a range of elements (K, P, Na, Cl, S and Ti) present in minor amounts (Table 2). A thin layer of corrosion products (CP1) is located just beneath the paint layer, appearing dark grey under polarized light (Fig. 9). This layer is followed by a corroded metal phase (CM1). Their exact composition was not analyzed but is likely to correspond to the corroded metal phase of the fracture edge, described below.

The interior face (Figs. 2 and 4) shows similar characteristics but does not include a white layer similar to NMM1. The fracture edge however (Figs. 2, 4 and 10), shows a more complex stratigraphy consisting of a dense product layer which can be further divided into two individual strata (CP3 and CP2), superimposed by a porous corrosion crust appearing orange under polarized light (CP1) (Fig. 11). The corroded metal (CM1) is located just beneath CP3 and contains remnants of the P-rich eutectic phase (Figs. 10-12). The M/CP ratio (metal to corrosion products) is rather high, implying extensive internal corrosion. Furthermore, a simple crack is traversing the corrosion layers – indicating a fragilization of the structure. As for the composition of the corrosion products CP1, CP2 and CP3, they all contain Fe and O (Fig. 12). A marbling effect within CP2 and CP3 indicates a variation in the concentration of Fe and O (particularly visible in bright field - Fig. 10). The white deposit (D1 in Fig. 6) might originate from exposure to calcareous water (Table 2). The presence of exogenous elements such as Si, Ca, Al, Na and O (Table 2) was confirmed by elemental mapping (Fig. 12).

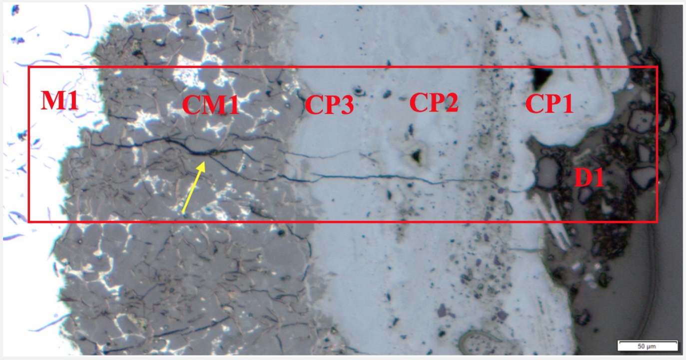
Elements **mass%***	**Fe**	**O**	**Ba**	**C**	**Si**	**S**	**Zn**	**Al**	**Ca**	**P**	**K**	**Na**	**Cl**	**Ti**
Deposit (D1)	22	35	-	26	10	0.3	-	3	1	0.8	0.8	0.5	0.4	0.1
Paint layer (NMM1)	10	23	37	9	0.5	12	7	-	1	-	-	-	-	-

Table 2: Chemical composition of the strata from Fig. 9. Method of analysis: SEM-EDS. Lab. of Electronic Microscopy and Microanalysis, Néode, HEI Arc, credit MiCorr_HEI Arc, C.Csefalvay. *The value is the calculated average of three analyses of the same feature, but in different areas.



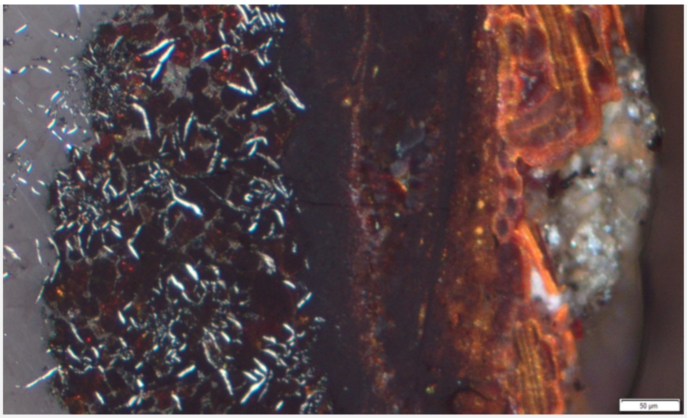
Credit UiO-IAKH, M.Hovind.

Fig. 9. Micrograph of the metal sample from Fig. 4 (detail), unetched, polarized light, 5x. The stratigraphy of the exterior surface, from top to bottom: the metal (M1) in grey, a thin layer of corroded metal (CM1) and a white cracked layer (NMM1) superimposed by a porous dark-red deposit (D1),



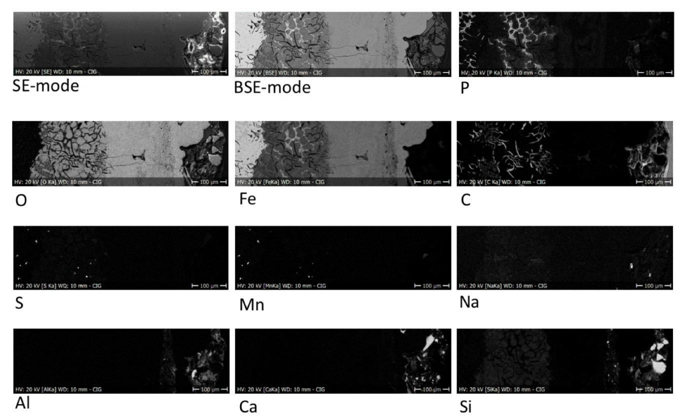
Credit UiO-IAKH,M.Hovind

Fig. 10. Micrograph of the metal sample from Fig. 4 (detail), unetched, bright field. From left to right: intact metal (M1) in white, followed by preferentially corroded metal (CM1) and dense product layers (CP3 and CP2) in various shades of grey. CP1 is slightly porous and covered by grain-like particles (D1). The red square corresponds to the area selected for SEM-EDS elementary mapping (Fig. 12). A simple crack (yellow arrow) is traversing the corrosion layers,



Credit UiO-IAKH, M.Hovind.

Fig. 11. Micrograph similar to Fig. 10 but under polarized light. The external deposit (D1) appears grey-white, CP1 in bright orange, while CP2 and CP3 appear dark red to brown. The corroded metal (CM1) contains remnants of graphite flakes (in white) and the P-rich eutectic (in grey),



Credit HEI Arc, C.Csefalvay.

Fig. 12: 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,

Corrosion form	Multiform
Corrosion type	Mostly type II with locally type I (Robbiola)

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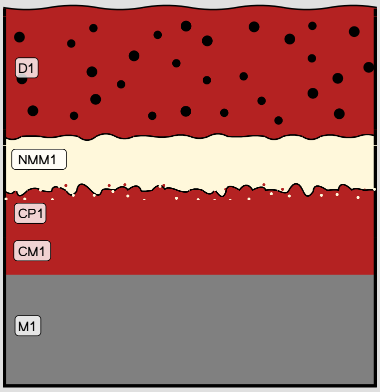


Fig. 5. MiCorr stratigraphy of the exterior face, to be related to the micrograph of Fig. 9. CP1 corresponds to CP2 in Fig. 3, Credit UiO-IAKH, M.Hovind.

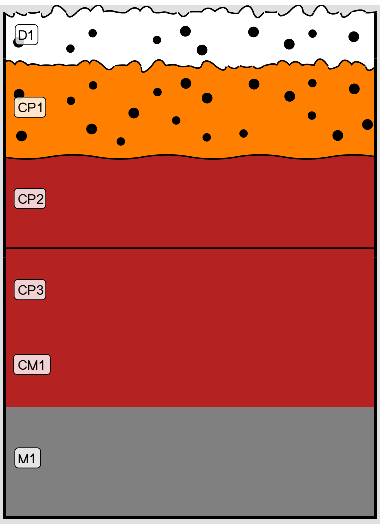
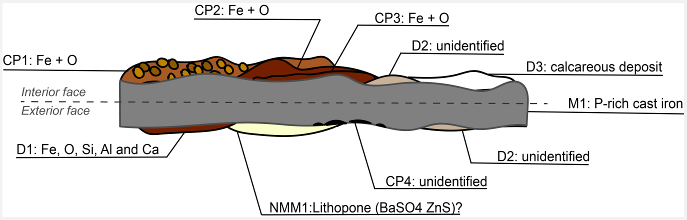


Fig. 6. MiCorr stratigraphy of the fractured edge, to be related to the micrograph of Fig. 11. D1 corresponds to D3 in Fig. 3, Credit UiO-IAKH, M.Hovind.

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

Based on the analyses carried out, the stratigraphy of Fig. 3 has been corrected.



Credit UiO-IAKH, M.Hovind.

Fig. 13. Corrected stratigraphic representation with results of SEM-EDS analysis and visual microscopic observation,

Conclusion

The rain gutter fragment is a grey cast iron with a microstructure consisting of graphite flakes and a P-rich eutectic phase. The exterior surface contains a preparatory paint layer, located beneath a layer of corrosion products mixed with soil. It seems likely that the cast iron gutter has been covered with a protective layer of paint (e.g. Lithopone) to slow down the rate of atmospheric corrosion. The presence of Lithopone reinforces the suggested dating of the artefact (20th century) as this pigment was developed and used from the 1880s and onwards (Lithopone 2007). It seems however that the protective layer has none or only very limited effect as (pitting) corrosion is present also in these areas.

The corrosion layers consist mainly of Fe and O, indicating atmospheric corrosion with presence of none or only a very small amount of contaminants/pollution. The scattered white deposit is possibly originating from exposure to calcareous water as it is localized mainly on the interior face of the fragment, where rain water would hit the metal before entering the ground.

References

References sample:

1. Scott, D. A. (1991). Metallography and microstructure of ancient and historic metals. Marina del Rey, Calif.: Getty Conservation Institute in association with Archetype Books.

2. Lithopone (2007). In: Gooch J.W. (eds) Encyclopedic Dictionary of Polymers. Springer, New York, NY