

WEST STOW, SUFFOLK
ANALYSIS OF METALWORK FROM
A BURNT RECONSTRUCTED
GRUBENHÄUS (WSW 060)

ARCHAEOLOGICAL CONSERVATION REPORT

Vanessa Fell



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Reconstructed Grubenhäus (WSW 060)**

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Summary

A fire during February 2005 destroyed a modern reconstructed Grubenhäus at West Stow Anglo-Saxon Village. The remains were subsequently excavated by Suffolk County Council archaeological Service and analysed with the aim of enhancing our understanding of a range of site formation processes. This report presents the results for selected metal artefacts that were investigated to determine the nature of their surface oxidation and corrosion layers, using x-ray diffraction analysis. The ferrous items oxidised mainly to haematite, wüstite and magnetite whereas the brass item produced zinc oxide. Interpretations are limited owing to the presence of modern protective coatings on some of the items.

Keywords

Burial Environments
Conservation
Modern
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Copper Alloy

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Introduction

The Anglo-Saxon Village at West Stow is an educational and research resource devised on modern reconstructions of an early Anglo-Saxon settlement. It is sited at the original settlement (NGR: TM 7970/7132) excavated by Stanley West between 1965 and 1972 (West 1985). The site incorporates a number of reconstructed buildings based on the archaeological evidence, including several *Grubenhäuser* or 'Sunken-Featured Buildings' (SFB). The site is owned and managed by Suffolk County Council.

During the early hours of 19 February 2005, one of the *Grubenhäuser* constructed between 1992 and 1999 was destroyed by fire (Fig 1). The original building (SFB 12) was interpreted by West as a six-post structure with a suspended floor and a lined pit or cellar – the only SFB discovered at West Stow that had a lined cellar (West 1985, 20–22, 117, fig 59). Within the original building were found 76 artefacts, the largest number of any of the buildings, and this was a distinctly 'female' assemblage including metalwork, bone combs, glass beads and weaving equipment.



Figure 1. The Grubenhäuser on 20 February 2005 looking east (Photo: Jess Tipper).

The reconstructed building measured 5.5m x 4.3m, height 4m, with a plank-lined pit 1.6m depth accessed by a trap door and ladder from the planked floor above (Tipper 2005). It was furnished with a variety of artefacts although it is worth noting that the items recovered after the fire do not bear any resemblance to the original Saxon metals assemblage recovered by West (1985, 20–21). After the fire in February 2005, the building collapsed and the remains were fenced off and left undisturbed (Fig 2) until excavation a few months later. Although monitored by the fire services, no dousing with water occurred.

The building was meticulously excavated as soon as possible after the firing, during the summer of 2005 (see Appendices 1 and 2). Archaeologists and archaeological scientists were invited to submit project outlines to investigate any aspects that might facilitate understanding of the construction of SFBs, the nature of destruction of these by fire, and site formation processes. The current project was initiated towards a better understanding of high temperature oxidation of metalwork within an archaeological environment.

Aims of the metalwork study

A project was devised to investigate the effects of burning on a selection of the metal objects such as nails and other fittings. These include both ferrous and non-ferrous artefacts, on their own and in conjunction with organic materials such as leather and wood.



Figure 2. The Grubenhäus on 30 March 2005 looking east (Photo: Brian Kerr). Note the chain lying over the cill beam (arrowed) and the fire extinguisher (centre right).

Previous studies of burnt archaeological metalwork

One of the first people to comment on burnt metalwork was Leo Biek who noted in particular the presence of bright red deposits on archaeological ironwork (Biek 1963, 133-4). The oxide, haematite, was noted to be a component of fire-scale on freshly forged iron, and could also be formed as a consequence of conflagrations of buildings and other structures (eg Blackwell and Biek 1985). Since Biek's observations, the generally good condition of burnt ironwork has been noted from cremations in particular. For example, at King Harry Lane cemetery, Hertfordshire, iron nails were found to be in 'pristine condition having been burnt with the 'calcined bones' (Stead and Rigby 1989, 111). Only rarely though have artefacts been scientifically analysed, for example those from a cremation at White Horse Stone, Kent (Fell 2004)

Description of the finds analysed

The site was visited by the author on 20 July 2005 towards the end of the excavation through the invitation of Dr Jess Tipper. At this stage in the excavation there were still numerous artefacts in the ground that had not yet been recorded and lifted.

Ten of the artefacts that had already been recorded and recovered were selected at random for examination and analysis (Table 1), to include:

- Ferrous items with surface layers in a variety of colours (yellow, red, through to dark greys), in particular a chain (Table 2, Fig 3).
- Ferrous items that had had been attached to wood or other organic material – a shield boss (Fig 4), a hinge (Fig 5), and five nails and a screw (Fig 6).
- The single non-ferrous metal object (Fig 7).

Table 1. Metal items selected for analysis

No	Identity	Material	Description	Context
177	Chain	Iron	Length 2.27m made of 66 elongated links, each 44 x 20mm, metal diameter 5mm. Fig 3. Table 2	Found partly draped over a cill beam (see Fig 2), but in use it was well raised above the floor.
344	Shield boss	Iron	OD c.155mm. Two of three studs survive <i>in situ</i> . Much flaking of grey surface, with uneven brown corrosion beneath. Fig 4	Near to and above the nails described below.
455	Hinge	Iron	Length 210mm (with hinge bent). Width max at hinge, 105mm. Smooth grey surface with some red deposits. Fig 5	From a wooden chest. From collapsed material recovered from the base of the pit, adjacent to shield boss.
462	Nail	Iron	Length 42mm (with slightly bent stem). Fracturing longitudinally. Brown products on grey stem. Fig 6	From collapsed material recovered from the base of the pit, adjacent to shield boss. Possibly from the chest although some of the 20 or so nails found there will be from the shield and other objects.
463	Nail	Iron	Length 54mm (bent at 90°). Brown products on grey stem, plus sand. Fig 6	As nail 462 above
464	Nail	Iron	Length 61mm (bent). Brown products on grey stem. Fig 6	As nail 462 above
465	Nail	Iron	Length 55mm (bent). Brown products on grey stem. Fig 6	As nail 462 above
468	Nail	Iron	Length 49mm (straight). Fracturing at the top (where there are brown products). Lower part of stem is grey and appears stable. Fig 6	As nail 462 above
469	Screw	Iron	Length 65mm (bent). Brown products on grey stem. Fig 6	As nail 462 above
639	Brooch	Copper alloy	OD 45mm. Survives in part only. One rivet is in place. Pin seems to be of the same metal. Grey surface over pale yellow metal. Fig 7	Found at interface between collapsed material and <i>in situ</i> deposits on the cellar base. Probably had been on the cellar floor.

Table 2. Description of chain 177 links according to colours

Links * In zone	No of links	Length (mm)	Distance from link 0 (mm)	Description
1–4	4	140	0–140	Bright red
5–8	4	140	140–280	Red + brown
9–12	4	140	280–410	Mainly red
13–17	5	180	410–570	Grey + red,
18–19	2	70	570–650	Grey + red, plus with brown
20–27	8	28	650–920	Grey + red
28–36	9	31	920–1230	Yellow
37–38	2	8	1230–1310	Yellow with brown
39–43	5	17	1310–1480	Grey + red + brown
44–66	23	80	1480–2270	Brown over compact grey, with occasional red deposits (eg Link 59)

* Numbered from Link 1 as in Fig 3, top left



Figure 3. Chain 177, numbered at the links where samples of surface layers were removed for XRD analysis.



Figure 4. Shield boss 344. Left, inside the boss; right, outer side.



Figure 5. Hinge 0455.



Figure 6. Nails 0462 (lower right), 0463 (lower left), 0464 (centre right), 0465 (centre left), 0468 (top, second row); screw 0469 (top).



Figure 7. Brooch 639 (diameter 45mm).

Methods of analysis

Corrosion layers of different colours or texture were targeted for analysis (Appendix 3). Samples were removed from the artefacts by scalpel and with the aid of a low-power binocular microscope. Samples in the order of 1mg were ground in an agate mortar and mounted on a glass slide.

X-ray diffraction data were collected on a Philips PW 1840 powder diffractometer using cobalt K α radiation (wavelength K α 1 = 0.178896nm, K α 2 = 0.179285nm) incorporating a solid-state silicon detector. The running parameters were 40kV 40mA for X-ray generation. Data was collected between the angles 7 and 100 °2 θ , at step size 0.05 °2 θ per step, time per step 1 second, with a receiving slit width of 0.3mm. A search-match computer programme was used to identify unknown components in the diffraction patterns by comparison with standards in the powder diffraction file (International Centre for Diffraction Data, ICDD). Initially powder diffraction files database version PDF-1 was employed; later, in 2006, version PDF-2 was used. Minerals named in this report, their formulae and PDFs are shown in Table 3.

Table 3. Principal oxidation products and other minerals detected by XRD

Mineral name	Formula	PDF (Ver-1)
Goethite	α -FeOOH	29-0713
Haematite	Fe ₂ O ₃	33-0664
Maghemite	γ -Fe ₂ O ₃	39-1346
Magnetite	Fe ₃ O ₄	19-0629
Wüstite	FeO	06-0615
Zinc oxide (Zincite)	ZnO	36-1451
Calcite	CaCO ₃	05-0586
Quartz	SiO ₂	31-1233

X-ray diffraction analysis detects only crystalline phases and therefore amorphous components will not be determined. Nevertheless, it is a standard analytical method for determining minerals and corrosion products on archaeological artefacts, as well as numerous other applications

Three items were checked to determine the elements present using X-ray fluorescence (XRF). In two instances (S4 and S10), the powder that had been used for XRD analysis was placed in the specimen chamber; the third item, the brooch, was placed whole in the chamber. These were analysed under vacuum at 40kV, 220 μ A in an Eagle II X-ray fluorescent spectrometer with a lithium-drifted silicon detector. The results are qualitative and not standardised.

Results

The results of the XRD analyses are summarised in Table 4. On the ferrous items, the iron oxides haematite, wüstite and magnetite were the commonest oxidation

products determined. The iron oxyhydroxide, goethite, was also found on several items.

Zinc oxide was detected two iron artefacts, chain 177 and nail 0463. In the case of chain 177, the zinc oxide comes from the yellow coloured links 33 (S4) and link 34 (S21). The spectra for 177 also produced minor peaks equivalent to aluminium iron vanadium (AlFe_2V), aluminium nickel (AlNi) and other compounds. Other unusual peaks occurred on hinge 0455, nails 056, 0465 and 0468, and boss 344. Selected results for 177 are shown in Figure 8. The single copper alloy item, brooch 639, yielded zinc oxide in the sample investigated, shown in Figure 9.

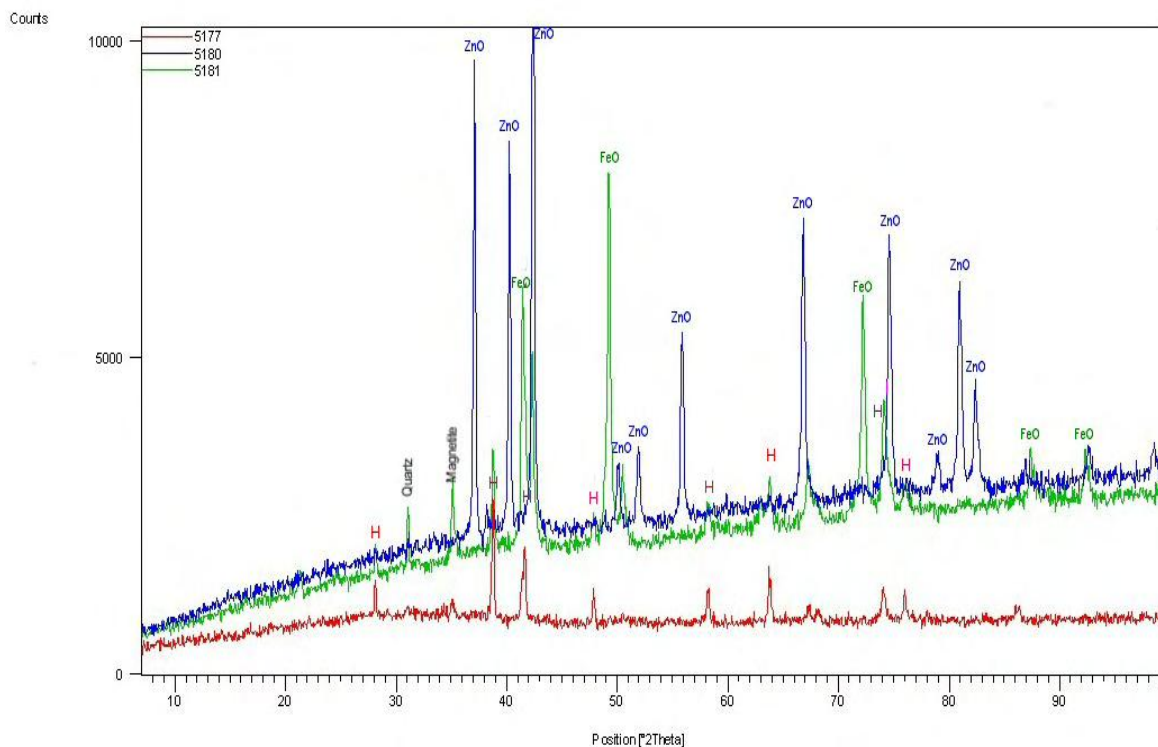
The three items in which zinc oxide was detected (chain 177, nail 0463 and brooch 639) were checked by X-ray fluorescence, and were all shown to contain zinc (eg Figure 8, lower, and Figure 9, lower). Brooch 639 was shown to contain primarily copper and zinc, with traces of lead, suggesting that it was a brass.

Table 4. Summary results of XRD analysis

S	Object	XRD no	Results - Major minor (trace) ■
1	Chain 177	5177	Haematite (Magnetite)
2		5178	Quartz Magnetite Wüstite Zincite Goethite
3		5179	Haematite Wüstite (Magnetite, Quartz)
4		5180	Zincite (unknown*)
5		5181	Wüstite Haematite Magnetite (Quartz)
6		5183	Magnetite, Goethite Wüstite (Quartz)
21		5198	Zincite (Quartz, unknown*)
7	Hinge 0455	5184	Haematite, Magnetite Wüstite (unknown*)
8		5185	Magnetite Wüstite (Haematite, unknown*)
9	Nail 0462	5186	Quartz Haematite, Goethite Magnetite
10	Nail 0463	5187	Zincite Quartz
11	Nail 0464	5188	Quartz Maghemite (Goethite, Hematite?, unknown*)
12	Nail 0465	5189	Quartz Magnetite (Goethite, unknown*)
13		5190	Magnetite Zincite, Wüstite (Calcite, Quartz)
14	Nail 0468	5191	Quartz, Magnetite Goethite, Haematite (unknown*)
15	Screw 0469	5192	Magnetite (Haematite, Wüstite, Quartz)
16	Brooch 639	5193	Zincite (Quartz)
22		5199	Zincite (Quartz, unknown*)
17	Boss 344	5194	Haematite, Magnetite (Wüstite)
18		5195	Wüstite, Goethite Lepidocrocite (Magnetite, unknown*)
19		5196	Haematite, Quartz (Magnetite, unknown*)
20		5197	Haematite, Wüstite Magnetite
23		5200	Haematite
24		5201	Magnetite, Wüstite (unknown*)
25		5203	Magnetite, Goethite, Wüstite Lepidocrocite
26		5204	Haematite

■ Results shown bold for **Major** components, normal text for minor, bracketed for (trace)

* Traces of uncertain compounds such as aluminium iron vanadium (AlFe_2V)



H = haematite peaks; **FeO** = wüstite peaks; **ZnO** = zinc oxide peaks

XRD no	Link no	Sample	Result
5180	33	S4	Mainly zinc oxide
5181	40	S5	Mainly wüstite, with some haematite and magnetite
5177	4	S1	Mainly haematite

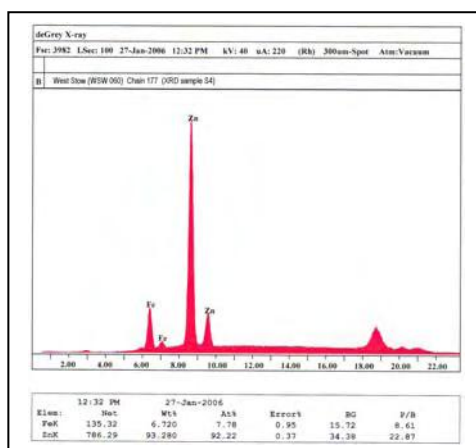


Figure 8. Chain 177. Upper: XRD spectra of three samples, shown from top to bottom; S4 (5180) shown blue, S5 (5181) shown green, and S1 (5177) shown red. Lower: XRF spectrum produced from S4, which confirms the presence of zinc in the yellow links

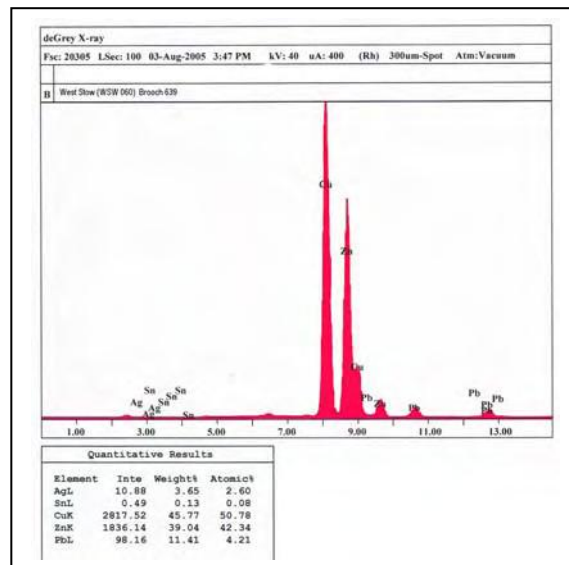
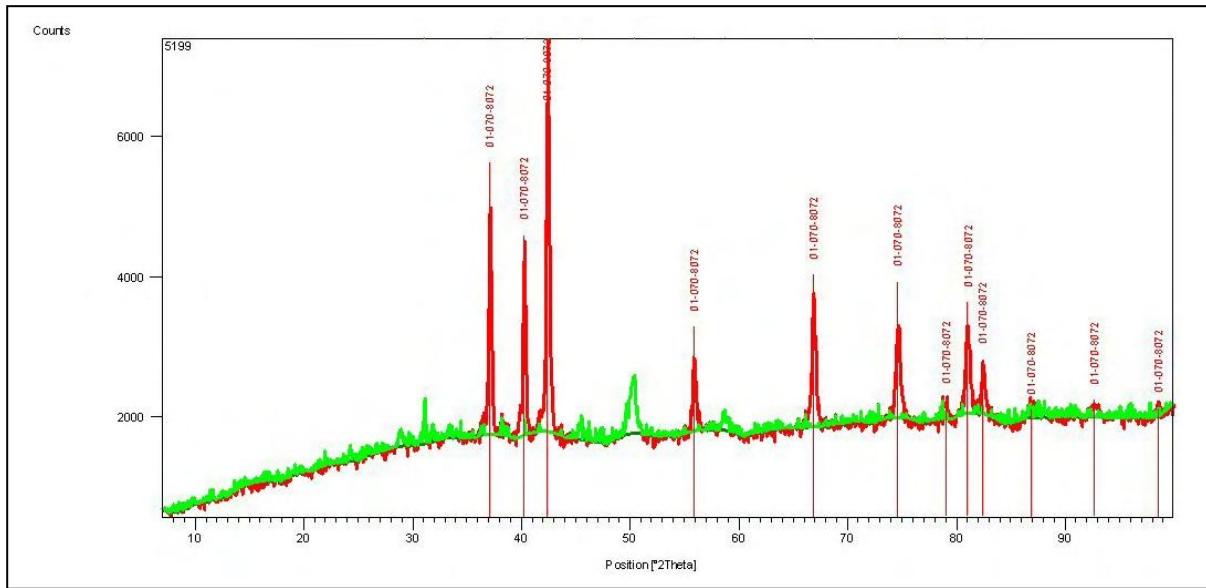


Figure 9. Brooch 639. Upper: XRD spectrum in which the identified peaks (zinc oxide) are highlighted in red, and the baseline and unidentified residue peaks are green (S22). Lower: XRF spectrum of the brooch surface showing mainly copper and zinc with a trace of lead, indicative of a brass.

Discussion

Ironwork

When iron is heated in an oxidising atmosphere such as air it usually forms multi-layered oxide scales comprising wüstite (FeO), magnetite (Fe₃O₄) and haematite (Fe₂O₃), the proportions of which are temperature dependent (Birks and Meier 1983, 75). Below 570°C, a two-layered scale develops consisting of magnetite (the least oxidised phase) next to the metal, and haematite (the most oxidised phase) on the surface. Above 570°C, wüstite forms below the other two iron oxides, next to the metal, giving the sequence: metal, wüstite, magnetite, haematite. These scales increase in thickness particularly at elevated temperatures when the wüstite will form the thickest layer. However, the latter is unstable and tends to break down to iron and magnetite below 570°C under certain conditions, although if wüstite is quenched or rapidly cooled, it can be retained without transformation (Kofstad 1988, 9). It is commonly found in ironworking slags (Wingrove 1970) including archaeological examples where it can be recognised through metallography and by scanning electron microscopy (eg Starley 2003, fig 102).

In the West Stow finds, these three oxides – wüstite, magnetite and haematite – were identified on chain 177, hinge 0455, screw 0469, and boss 344. One or more of these oxides were identified on the remaining iron artefacts except nail 0463 (see below). To some extent, however, the incidence and proportions of these oxides reflect the selective sampling procedure that targeted unusual coloured or textured deposits. Goethite was found on most of the ferrous items and this is a common oxide found on corroding iron (Cornell and Schwertmann 2003). Its presence here therefore is not surprising and is very probably due to corrosion in the ground between the time of the fire and the excavation.

On two iron artefacts, chain 177 and nail 0463, zinc oxide was identified. On the chain, the zinc oxide comes from the yellow coloured links 33 and 34. The most plausible explanation for the zinc on both items is that these were galvanised or otherwise coated to protect the iron from corrosion. In such processes, the zinc serves as an anodic sacrificial metallic coating, and other metals are often added to improve properties, commonly aluminium, tin, nickel and copper-based alloys (Higgins 1973, 427; Shrier et al 1994, 12:55). Very probably, therefore, the unusual compounds detected on several of the iron finds (eg aluminium iron vanadium) can be accounted for by these coatings, which on heating, would probably form a range of oxides.

In the three items with zinc oxide, no other oxidation products were detected and the reason may be that this oxide has strong peaks, like quartz from soil, which can tend to over-ride other minor components (such that their peaks do not show). The yellow colour of the samples removed from 177, for example, may well be due to the presence of trace amounts of iron oxides.

The project aimed to investigate also if the presence of organic materials, such as wood and leather, may have had any effects on the nature of the oxidation products that formed on the metalwork. Evidence for this was not found and would in any case not be easily detectable on the items selected for examination because these

did not retain any charred organic materials apart from small pieces of charcoal collected within the boss (see Fig 4, left). Oddly, though, insect cuticles did survive on the hinge. The fire seems to have swept through the centre of the building including the pit, removing wood from all or most composite finds (eg Fig 10), leaving some unburnt material only where concealed underneath beams, for example unburnt wood (Fig 11) and soil (Fig 12).

Copper alloy

The brooch 639 was shown to be made of brass. When heated, a brass will melt in the range from 900°C for a 40% brass (wt % zinc) to 1040°C for a 10% brass (Brandes 1983, table 14.4). In brasses of low zinc content (below 10%), oxidation usually produces scales that contain mainly copper oxides, whereas at higher zinc proportions, the scales usually comprise zinc oxide (Kofstad 1988, 357–9).

In the West Stow brass brooch, the presence of only zinc oxide in the grey surface deposits suggests that this was a moderate or high zinc brass that was heated in oxidising conditions to temperatures less than the melting point.

Corrosion after excavation

The ferrous items that were visible in the ground were distinctly red in colour (eg chain 177 in Fig 2, and bed frame in Fig 10) and this colour persisted during exposure to the weather through the excavation, and later until at least November 2006 when the items were returned to SCCAS. During this 21 month period, some deterioration of the iron was visible – flaking continued on certain items such as the hinge and boss, beneath which there was active corrosion. The flaking may be due to the combined effects of active corrosion producing voluminous corrosion products, exacerbated by contraction of surface layers of scale. A sample of the grey scale layer from the boss was tested for likelihood of corrosion by placing in a container of tap water in the laboratory and after 12 months this appears visually not to have corroded.



Figure 10. Part of bed frame before excavation.



Figure 11. Charred and unburnt wood from the underside of a beam.



Figure 12 Unburnt soil from beneath a beam (where iron in the soil has not been oxidised).

Conclusions and summary

This project aimed to characterise the metal oxidation products resulting from the fire. As expected, analysis showed that haematite was common, as previously noted by Biek (1963) and other authors. One oxide not normally recorded on archaeological artefacts was wüstite, although this is commonly found in ironworking slags. Also found on two ferrous items was the oxide of zinc, due presumably to the burning of protective coatings that had been applied to the modern iron. Specific oxidation effects were not noted due to the proximity of organic matter in contact with the metalwork. The brass brooch had burnt to produce zinc oxide, which suggests that it had a high zinc content.

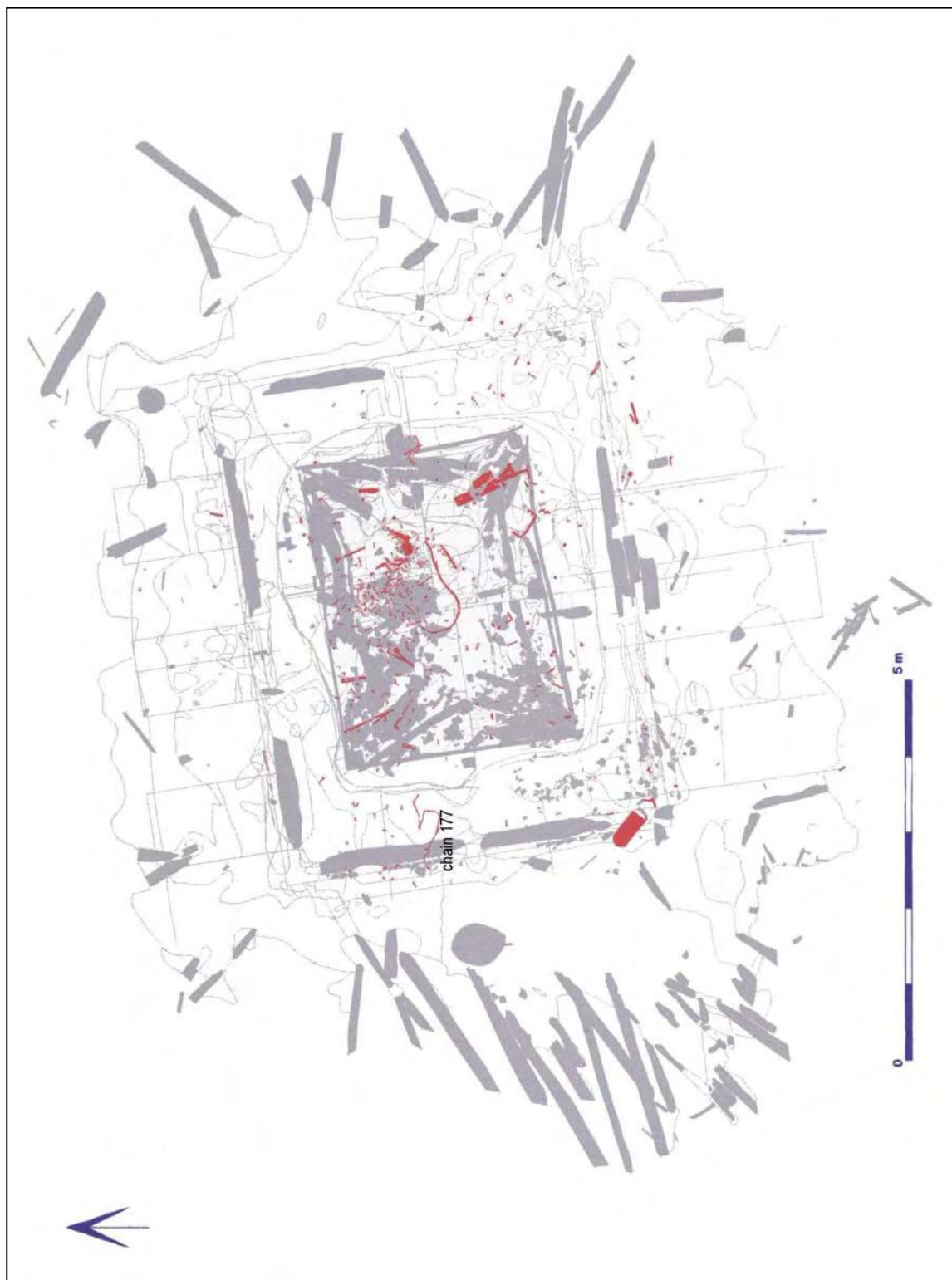
Acknowledgements

I thank Dr Jess Tipper for assistance and interest throughout the project, for welcoming me on site, and for supplying illustrations.

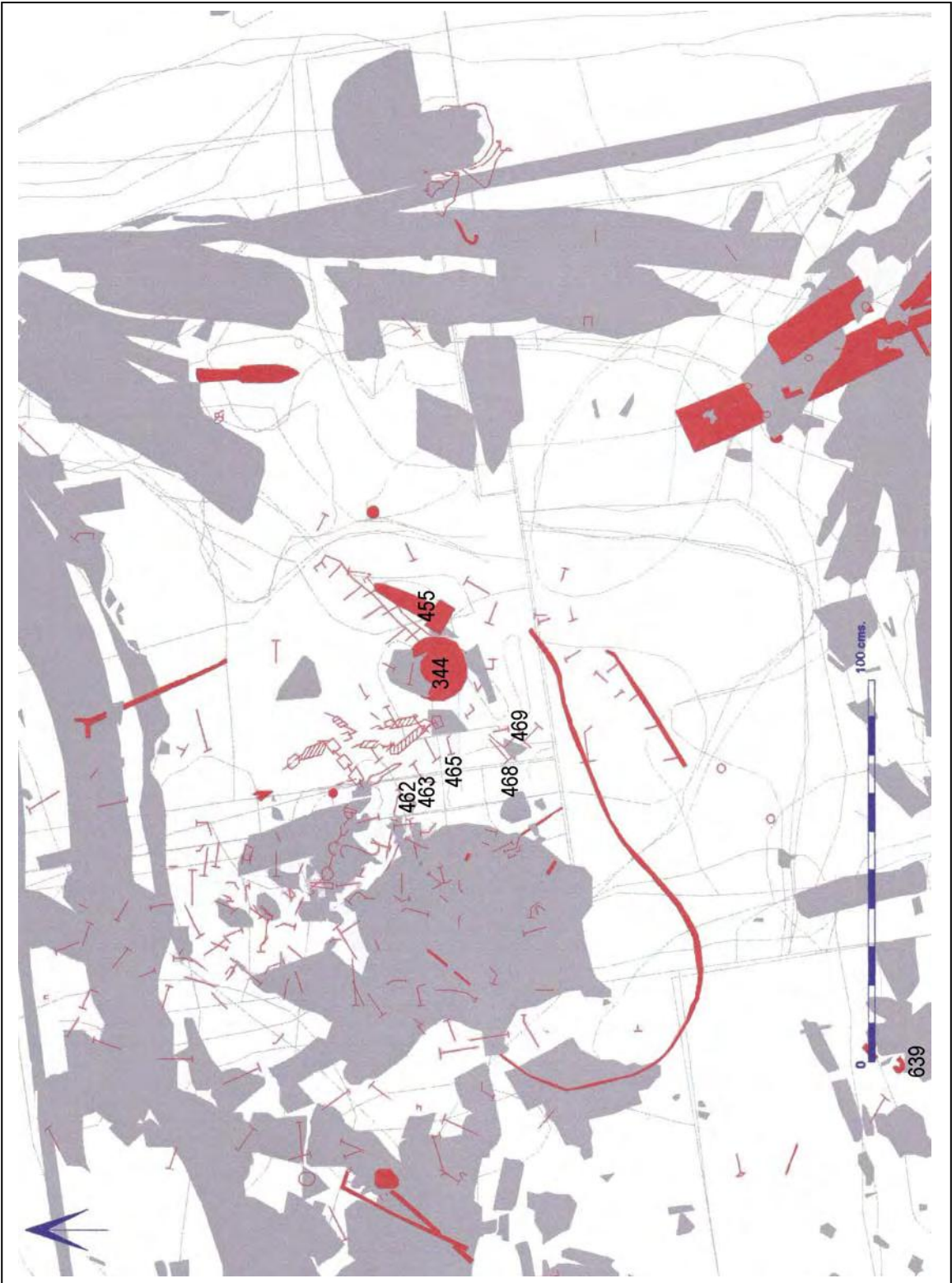
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Appendix 1. Plan of the excavated Grubenhäus showing metal finds, coloured red (adjusted from plan supplied by Dr Jess Tipper). Note chain 177 across cill beam on west side.



Appendix 2. Plan of the excavated Grubenhäus showing metal finds, coloured red, and the locations of those analysed (adjusted from plan supplied by Dr Jess Tipper).



Appendix 3. Description of samples for XRD analysis

S	Location	Colour	Description of sample
1	Chain 177 (top row in Fig 3, link 4)	Red	Outer red layer (only), which sits above dark grey, well-formed layer
2	Chain 177 (top row in Fig 3, link 7)	Dark brown	Scraped down to shiny metal. Could include metal
3	Chain 177 (2nd row in Fig 3, link 14)	Grey + red	As S2, plus some white deposit (more of which exists on Link 15)
4	Chain 177 (4th row in Fig 3, link 33)	Yellow	Powdery (scraped down to metal)
5	Chain 177 (5th row in Fig 3, link 40)	Very grey and shiny	Mainly the grey and shiny layers but could include some red or brown deposits
6	Chain 177 (6th row in Fig 3, link 55)	Brown	Mainly brown powder although adjacent links do include grey and red under
7	Hinge 0455 (grey scale flake near middle hole)	Grey with some red	A flake was prised from the surface
8	Hinge 0455 (grey flake from edge)	Grey with some brown and red	Flake of scale from the edge of the hinge, between centre hole and hole at the end (away from hinge itself)
9	Nail 0462	Brown	Outer fracturing layer near head, plus soil and quartz grains
10	Nail 0463	Grey + orange	Poor sample: much quartz and very little corrosion products.
11	Nail 0464	Brown + white	Brown iron corrosion products plus whitish deposits on the bend of the stem near the tip
12	Nail 0465 (a)	Orange – brown	Mainly the outer layer but some of the inner layer too
13	Nail 0465 (b)	Dark grey + other	Inner layer, adjacent to thin brown-black which is next to metal
14	Nail 0468	Brown	Already fractured strips from stem centre and tip
15	Screw 0469	Dark grey	Surface layer; well-formed, near head. Has thin orange corrosion active below
16	Brooch [639]	Yellow + grey	The yellow might not be metallic. The grey is probably an oxidation product
17	Shield boss [344]	Grey + red + brown	A flake of scale that has fallen off the curved edge from the convex (outside)
18	Shield boss [344]	Brown	From the convex (outer) side, near the long rivet. Powdery deposit under the scale flakes
19	Shield boss [344]	Black	Bubbly black scale layer plus soil – which overlies a very smooth black layer on the inside of boss near the top
20	Shield boss [344]	Black + some brown	Inside the boss towards the top: bubbles of corrosion with soil over, which are lifting away with brown corrosion products visible at the bubble edges (and beneath)
21	Chain 177 (4th row in Fig 3, link 34)	Yellow + some red	Link 34 scrapings. (Sample analysed in order to check results of S4)

22	Brooch [639]	Pale grey – black	First sample was whole thickness of deposit from the back of the pin (down to bare metal). Second sample was scrapings from the ring.
23	Boss [344]	Grey	Top layer of scale, convex (outer) side of boss
24	Boss [344]	Grey	Under layer of scale, convex (outer) side of boss
25	Boss [344]	Orange	Orange loose particles between scale layers and metal, convex (outer) side of boss
26	Boss [344]	Grey on both sides	A blister, on outside of convex (outer) side of boss.



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