HISTORICAL METALLURGY

Investigation of a broken pile-shoe from a Roman bridge

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ABSTRACT: A Roman pile-shoe made from four iron bars had breaks in three bars. One break was a recent impact fracture. A sample containing one of the fracture surfaces was broken into large fragments with a hammer. These were investigated fractographically, metallographically, and by surface and bulk chemical analyses. The fractures were brittle and primarily intergranular. The metal was a coarse-grained phosphoric wrought iron (0.52wt% P) with very low silicon, manganese and sulphur contents, and extremely low carbon content (0.0033wt% C). This extremely low carbon content and coarse grain size indicate decarburisation during smithing. Furthermore, the combination of extremely low carbon and high phosphorus contents is concluded to be the most probable reason for the impact brittleness. This could have been facilitated by a notch effect due to surface corrosion. The significance of the embrittlement and surface corrosion is considered with respect to conservation of archaeological iron objects, including similar pile-shoes.

Introduction

Obstacles in the Maas riverbed, near the town of Cuijk in the Netherlands, were recognised in the early 1990s to be the remains of a Roman bridge. An archaeological recovery programme was set up (Goudswaard 1996; Goudswaard *et al* 2000), with the primary assistance of divers from the amateur archaeological organisation Mergor in Mosam (MiM). The recovery programme resulted in finding many stone blocks and more than 100 oak piles. The remains of the piles were 2–3m long, 0.3–0.4m square above the pointed lower ends, and some were still covered at these ends by iron pile-shoes. The piles (and hence the pile-shoes) have been dated to the 4th century AD. Dendrochronological investigations of the wooden piles showed that the bridge was built in three stages between 340AD and 400AD (Haalebos *et al* 2002).

Each pile-shoe was made from four iron bars, joined by heating and hammer-welding to form a point. The bars had approximate dimensions of 13×40×500mm. One pile-shoe was observed to have three broken bars, and at least one break was recent. This was an impact fracture owing to the pile-shoe falling onto the floor of a storage area. MiM sent the pile-shoe to the museum, Het Valkhof, for initial examination. Figure 1 shows the breaks and a sawn-off slice containing the upper fracture surface of the recent break; Figure 2 shows the lower fracture surface. Over the centuries the bar had corroded to varying depths, up to about 0.5mm. The largely internal fracture had shiny facets, some of which were up to 3mm in size. This unusual and obviously brittle fracture prompted a detailed investigation.

Experimental scope of the investigation

The sawn-off slice (Fig 1) was struck on a side surface by a hammer, resulting in brittle fracture into large fragments. These fragments were then used for the investigations summarised in Table 1. This 'sampling', though highly unorthodox, was effective and confirmed the ambient temperature impact brittleness of the bar.

As the results accumulated, it became clear that *fresh* fracture surfaces should be examined, if possible, for evidence of elemental (phosphorus) segregation to grain boundaries. Unfortunately, specimens could not be made for *in vacuo* fracture and examination by Auger electron spectroscopy (AES). The alternative was to break a
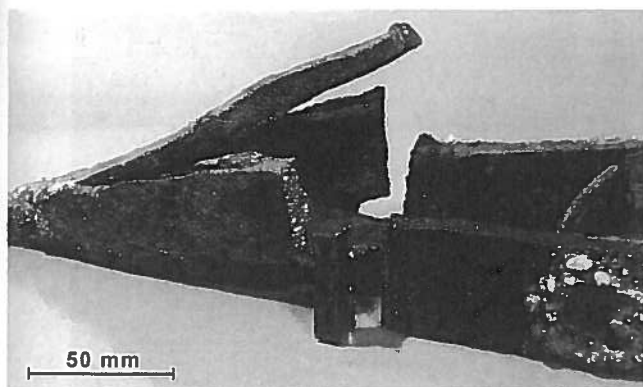
Figure 1: The broken pile-shoe with a sawn-off slice containing the upper fracture surface of the recent break.
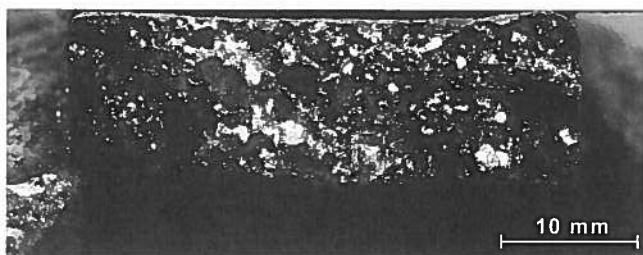

Figure 2: The lower fracture surface of the recent break.

Figure 3: FEG-SEM fractograph at, and near, the external surface of a sample from the sawn-off slice. The fracture is almost entirely intergranular.

sample in a nominally inert atmosphere and investigate the fracture surface by XPS.

Fractography

Figure 2 shows the macrofractographic appearance of the recent break, discussed above. Figures 3 and 4 are FEG-SEM fractographs of a sample obtained from the sawn-off slice. These show brittle intergranular and cleavage fracture. Intergranular fracture predominated at, and near, the outside surfaces. The grain size varied from about 0.25mm to more than 2mm, which is very coarse.

Metallography

Two samples were examined. The first was used to examine the microstructure, iron and inclusion compositions and hardness. The microstructure after polishing and etching with nital (Fig 5) showed large undeformed ferrite grains, with an unusual etching effect in Zones

Table 1: Techniques used to investigate the pile-shoe

| Technique	used for investigation of ...	Institution
Fractography		
Macrofractography	brittle fracture, corrosion	Het Valkhof
Scanning electon microscope (SEM)/Field emission gun SEM (FEG-SEM)	intergranular + cleavage fracture	PR-MA / NLR
Optical metallography	microstructure, hardness	CORUS, NLR
Chemical analysis		
Bulk		
X-ray diffraction (XRD)	iron and corrosion products	PR-MA
X-ray fluorescence spectroscopy (XRF)	iron composition	CORUS
Combustion + infra-red (IR) detection	carbon and sulphur content of iron	CORUS
Metallographic surfaces		
SEM+Energy-dispersive X-ray analysis (EDX), Electron probe micro-analysis (EPMA)+wavelength-dispersive X-ray analysis (WDX)	iron and inclusion compositions, phosphorus and oxygen line scans	CORUS / PR-MA
Fracture surfaces		
X-ray photoelectron spectroscopy (XPS)	grain boundary segregation	PR-MA
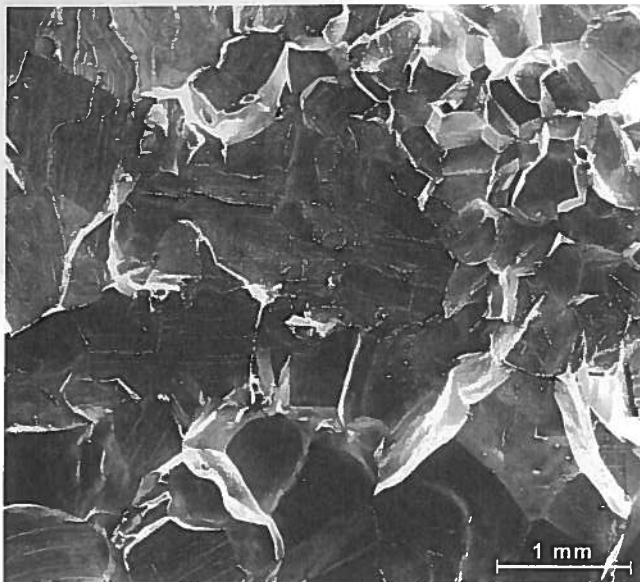
Figure 4: FEG-SEM fractograph nearer the centre of a sample from the sawn-off slice. The fracture is mixed intergranular and cleavage.

2 and 3 which was due to phosphorus segregation, and typically occurs in ancient phosphoric iron (Stewart *et al* 2000b; Godfrey *et al* 2003; Godfrey pers comm). Other images consistently showed the same three zones, which are undoubtedly the result of hot-welding three strips of iron together to form the bar.

Table 2 shows that the metal was a phosphoric iron with very low manganese and sulphur contents. These results are consistent with an ancient origin, *ie* they are what would be expected from smelting a phosphorus-containing iron ore in a furnace fuelled by charcoal rather than coke. The hardness values are typical for annealed phosphoric iron (Tylecote 1986, 145); note that the fracture location was about 100mm from the weld area (Fig 1), and was therefore not included in the final making of the pile shoe.

There are deformation twins and a few elongated or ovoid inclusions in Zones 1 and 3 (Fig 5); Figure 6 shows deformation twins and inclusions in Zone 3, and the unusual etching effect in more detail. The twins, which are the sharp lines confined to the near-surface regions, are the result of the hammer blow to obtain the
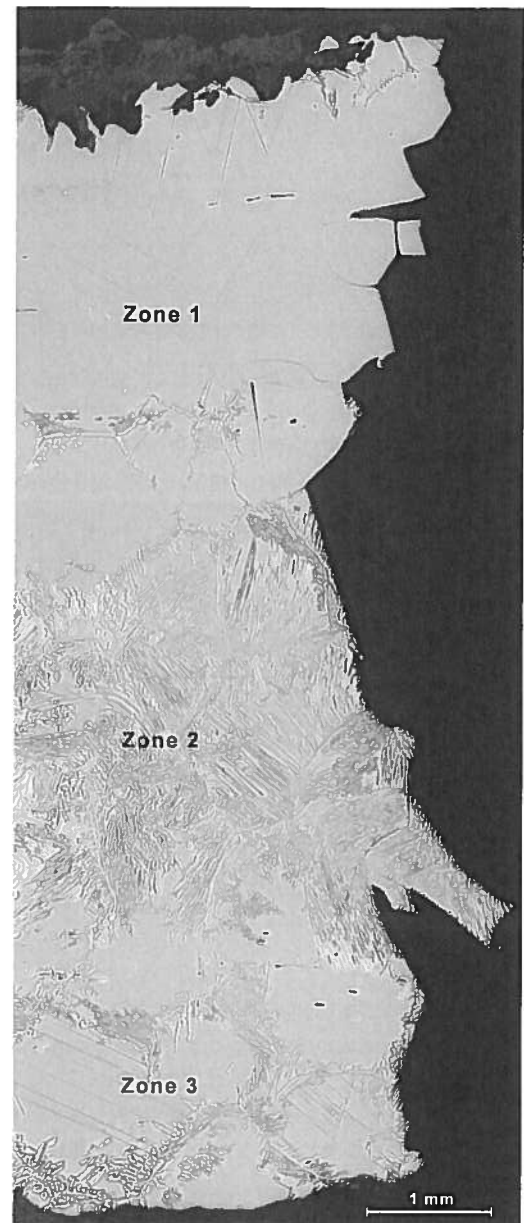

Figure 5: Through-thickness microstructure of a broken sample: the top and bottom edges are the original surfaces.

sample. Some of the inclusions were analysed and found to contain iron, phosphorus and oxygen.

Another area of the first sample had much larger elongated inclusions in Zone 1. These contained predominantly iron, oxygen and silicon, and also aluminium, calcium, magnesium, phosphorus, potassium and sodium. They

Table 2: Iron composition (wt%) and Vickers microhardness for the zones in Figure 5

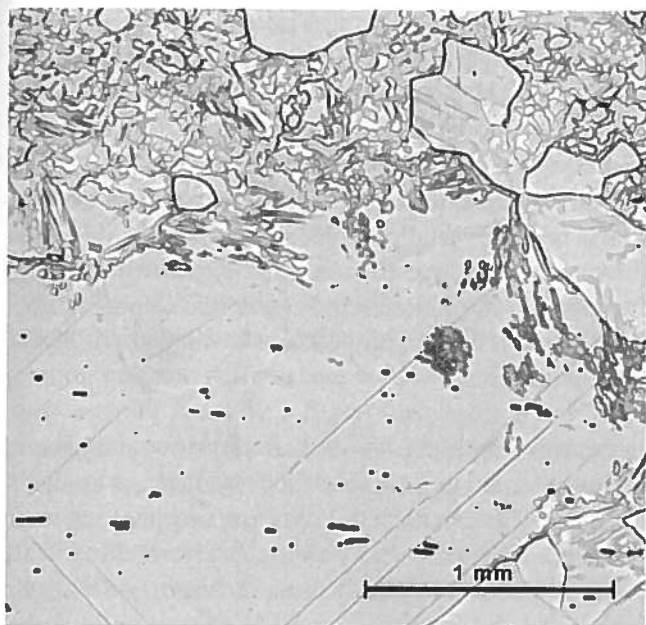| Zone | Composition (wt% determined by EPMA+WDX; 5 measurements per zone) | | | | | H_v (1kgf load) |
	Fe	P	Si	Mn	S	
1	98.75–99.68	0.45–0.52	0.06–0.07	0.002 max	0.010 max	169, 171, 194
2	98.92–99.44	0.25–0.40	0.05–0.07	0.013 max	0.012 max	141, 143, 150
3	98.55–99.07	0.28–0.40	0.06–0.07	0.014 max	0.009 max	144, 155, 159

Figure 6: Detail of the microstructure in Zones 2 (above) and 3 (below), showing deformation twins and elongated inclusions (black) in Zone 3, and the unusual etching effect.
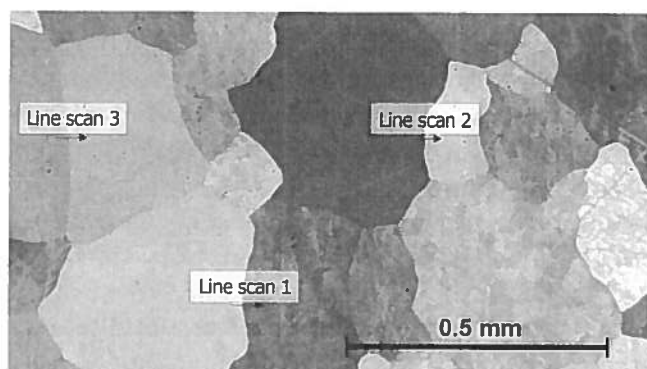

Figure 7: Positions of three EPMA + WDX line scans for phosphorus and oxygen contents.
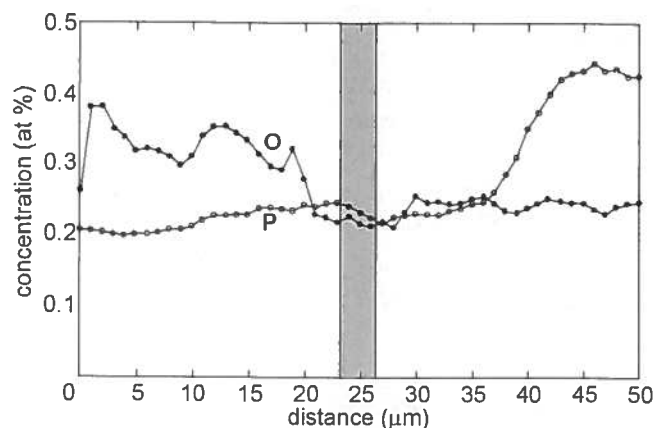

Figure 8: EPMA + WDX line scan (No 2 in Fig 7) showing variations in oxygen and phosphorus concentrations across the grain boundary (represented by the grey band).

were probably the remains of smelting slag that had not been fully removed during smithing the iron bloom and subsequent smithing of the wrought iron stock. Similar slag inclusions were reported for a pile-shoe from the Roman bridge at Minturnae (Campbell and Fahy 1984).

The second sample was used for three EPMA line scans of phosphorus and oxygen contents across grain boundaries (Figs 7 and 8). There were no indications of pronounced segregation of either element at the grain boundaries. In total, the line scans showed phosphorus contents varying between 0.2wt% and 0.44wt%, which agrees well with the data in Table 2. The oxygen contents varied between 0.17wt% and 0.44wt%.

Bulk chemical analysis

The XRF and combustion + IR bulk analyses on

another sample gave the following results: 0.06wt% Si; 0.52wt% P; 0.01wt% Mn; 0.004wt% S; 0.0033wt% C; with the balance nominally being 99.4wt% Fe (oxygen content was not determined). The analyses are consistent with the EPMA+WDX results in Table 2. The extremely low carbon content is important (see below).

An XRD bulk analysis was also done for the corroded surface of a sample and a clean fracture surface. The corrosion layer closest to the metal surface was identified as akaganeite, while the fracture surface was virtually pure ferritic iron. The identification of akaganeite is important. It forms after excavation, when oxygen gets unhindered access to the wet corrosion layer (Selwyn 2004), which must contain chloride ions (Balasubramaniam et al 2003; Neff et al 2005). Since iron chloride is hygroscopic, the corrosion on the pile shoe will probably continue unless preventative measures are taken.

Fracture surface analysis

XPS was used to look for evidence of elemental (phosphorus or oxygen) segregation on grain boundary fracture facets. A saw-notched new sample was inserted into a glove box in an atmosphere of nitrogen containing 0.1ppm water and 0.3ppm oxygen. The sample was then broken and one fracture surface transferred directly to the vacuum chamber of the XPS equipment.

Figure 9 shows a Secondary X-ray Image (SXI) of the measurement positions on the fracture surface, whereby it was intended to analyse grain boundary facets only (however, post-analysis SEM fractography showed that the fracture surface consisted mainly of cleavage facets). Depth profiles of elemental concentrations were obtained from measurements using monochromatic Al Kα radiation, spot size 100μm, alternating with argon
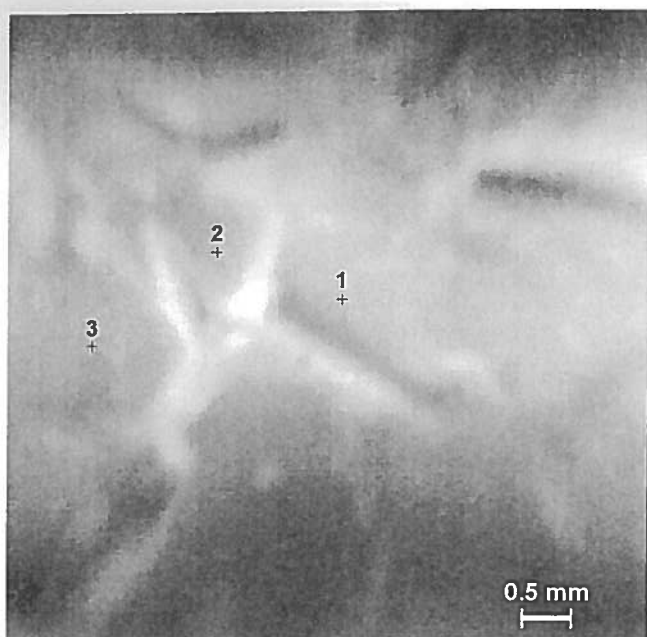
Figure 9: Secondary X-ray image image of the XPS measurement positions on a fracture surface.
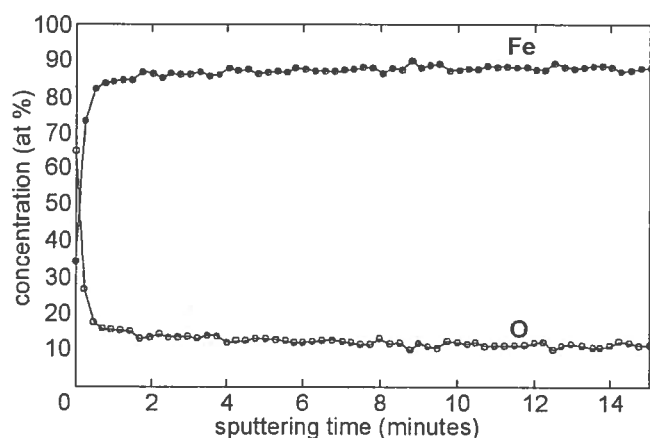

Figure 10: Representative example of the XPS measurements (No 3 in Fig 9). Sputtering depth about 10^{-4}mm after 15 minutes.

sputtering (Fig 10). Only iron and oxygen were detected, with a very high oxygen concentration on the initial fracture surface and persistent oxygen concentrations ~12at% (3.5wt%) after sputtering times of 2–15 minutes. This result must have been due to contamination of the fracture surface by adsorbed oxygen, since otherwise it would signify large amounts of iron oxides in the bulk metal, and this is inconsistent with the metallography and EPMA+WDX line scan analyses.

Discussion

The metallography and chemical analyses show that the recently broken bar is a phosphoric iron with very low silicon, manganese and sulphur contents; and extremely low carbon content *at the fracture location*. For comparison, analysis of the weld area of a pile-shoe

from the Roman bridge at Trier gave the following results: 0.03–0.06wt% Si, 0.21–0.22wt% P, 0.01wt% Mn, 0.008–0.009wt% S, 0.07–0.11wt% C (Cüppers 1969, 211).

The caveat about the carbon content at the fracture location is because metallographically examined pile-shoes have shown a range of iron microstructures, ranging from ferrite to predominantly pearlite (Cüppers 1969, 210; Campbell and Fahy 1984), *ie* varying widely in carbon content.

In contrast, both pile-shoes had very low manganese and sulphur contents despite the very different analysis locations. These results indicate iron ore smelting with charcoal, rather than post-18th century smelting with coke, *ie* the iron is not of modern origin. As already mentioned, the Cuijk piles and pile-shoes date from the 4th century AD while the Trier piles and pile-shoes date from the 2nd century AD.

The iron ore would most probably have been local, and locally smelted. Phosphorus-containing bog iron ores were widely available and used for making bloomery iron in the region now known as the Netherlands (Joosten 2004; Godfrey and van Nie 2004). The likelihood of local iron production would not have depended on the different smelting techniques employed within the Roman Empire and by contemporary Germanic people, since the main differences were in the method of slag separation from the iron bloom—as discussed by Godfrey and van Nie (2004).

The carbon content (0.0033wt%) of the recently broken bar is important with respect to the smithing of the iron and its present impact brittleness, and is discussed below.

Carbon content and smithing

Together with the large ferrite grain sizes, up to 2–3mm, the extremely low carbon content of the recently broken bar suggests that decarburising conditions pertained during smithing (Dinnetz 2003), specifically at the fracture location and during heating in a forge in order to hammer-weld the four bars to form the pile-shoe point. Actually, there would have been several smithing steps, starting with the cut-up iron bloom and continuing with forging the iron into bars and strips, welding strips into bars, and welding four bars together to make the pile-shoe. Any or all of these steps could have contributed to reducing the carbon content.

The alternative is that the original iron bloom was extremely low in carbon. There is a strong tendency for

high-phosphorus ancient iron to be low in carbon, with carbon contents in the range 0.01–0.1wt% (Tylecote 1986, 145, 149, 151 and 169). This range in carbon content is significantly above the 0.0033wt% of the recently broken bar, suggesting that it is more likely that the extremely low carbon content resulted from smithing.

Be that as it may, the fact that the bar, and presumably others like it, was used for making a pile-shoe shows that the strength was adequate despite the extremely low carbon content. As Tylecote states, there is no reason why a smith working a high-phosphorus iron should be dissatisfied with the strength of the product, since phosphorus will harden iron almost as much as carbon. Smiths would have known that this kind of iron was readily hot-worked and did not require special forging skills. They would also know that it was difficult to cold-work, which is why, in general, there seems to have been a move away from high-phosphorus iron ores during the Roman period (Tylecote 1986, 169). However, they might not have recognised any tendency for the cold iron outside the weld area to be very brittle under impact loading. This is further discussed below.

Carbon content and segregation-induced brittleness

Modern metallurgy has shown that the effects of carbon and phosphorus on the mechanical properties of iron alloys are linked (Hopkins and Tipler 1958; Erhardt and Grabke 1981). If phosphorus is present in unalloyed iron it will tend to segregate to the ferrite grain boundaries, resulting in brittle fracture, particularly under impact loading (Inman and Tipler 1958; Hopkins and Tipler 1958; Ramasubramanian and Stein 1973; Erhardt and Grabke 1981). However, carbon can displace phosphorus from the grain boundaries, even if the bulk concentration of phosphorus is relatively high (Hopkins and Tipler 1958; Erhardt and Grabke 1981). This is called the *site-competition effect*, and explains why modern unalloyed carbon steels, which generally contain no more than 0.15wt% phosphorus, are not embrittled. Only if the carbon content is unusually low can phosphorus embrittle the grain boundaries (Erhardt and Grabke 1981).

Besides phosphorus, oxygen can cause intergranular brittle fracture in irons having carbon contents less than 0.005wt% (Rees and Hopkins 1952; Kumar and Raman 1981), and has also been shown to segregate to grain boundaries (Kumar and Raman 1981). As in the case of phosphorus, the site-competition effect between carbon and oxygen should play an important role (Krasko 1997). Very low carbon contents are thus required for segregation-induced embrittlement by phosphorus or oxygen. Since

fracture surface analysis by AES was not possible, and the XPS results are inconclusive, the question remains whether to attribute brittle intergranular fracture of the pile-shoe bar to grain boundary segregation of phosphorus or oxygen.

The many tensile and impact test results in the literature are complicated by different heat treatments, testing temperatures, notch effects and qualitative assessments of the fracture characteristics. On the whole, it appears that phosphorus is a more potent embrittler of iron than oxygen, though this does not necessarily mean that fracture is intergranular, since cleavage and partial ductile fracture can also occur.

However, there is one observation that favours phosphorus-induced embrittlement of the pile-shoe bar, namely that oxygen-induced intergranular fracture requires the iron to have a high yield strength (Kumar and Raman 1981). As stated earlier, the hardness values in Table 2 are typical for annealed phosphoric irons. These hardnesses suggest low yield strengths in the range 250–350 MPa (Gale and Totemeier 2004; Stewart *et al* 2000a).

Phosphorus and manganese contents and smithing

As mentioned earlier, Figure 5 shows that the unusual etching effect was confined to Zones 2 and 3. Table 2 shows that these zones had lower phosphorus and higher manganese contents than Zone 1. Since phosphorus stabilizes the α-iron phase and manganese stabilizes the γ-iron phase, these metallographic and compositional correlations might reflect the extent to which the three zones were heated into the $(\alpha + \gamma)$ or γ phase fields during the final smithing process.

As a first approximation, the bar material illustrated in Figure 5 can be considered as a binary alloy of iron and phosphorus. From the Fe-P phase diagram (Baker and Okamoto 1992) we may infer that in order to transgress the $(\alpha + \gamma)$ and γ phase boundaries this bar material would have had to reach temperatures above 912°C and probably above 1000°C. The higher the temperature, the more likely that the material would have a large final (ferrite) grain size, as was observed.

Cause of embrittlement

The results of modern metallurgical research and the present investigation lead us to conclude that the most probable reason for brittleness of the recently-broken bar of the pile-shoe is the local combination of extremely low carbon and high phosphorus contents. Obviously, the brittleness did not manifest itself when the pile-shoe was made and subsequently attached to an oak pile. Two

possible reasons can be given. Firstly, there would have been no reason to apply impact forces directly to the bar during its attachment, which was done using a nail driven through a hole in the bar (Figure 1, bottom right). Secondly, the surface of the bar would have been smooth, which is no longer the case owing to surface corrosion. Over the intervening centuries the corrosion penetrated up to 0.5mm into the bar, and the depth of corrosion was not uniform (Fig 2). There could thus have been a notch effect added to the impact force caused by the pile-shoe falling to the floor during storage, and this combination could have facilitated the brittle fracture.

Once intergranular cracking began, the cracks would be subject to dynamic effects, *ie* the crack velocity would contribute to the brittleness. This is possibly why there was a transition to a mixture of intergranular and cleavage fracture in the interior of the bar.

Conservation of other pile-shoes

There are two aspects to conserving other pile-shoes. Firstly, the presence of akaganeite closest to the metal surface suggests that corrosion will continue unless prevented by drying out the corrosion layer and storing the pile-shoes in a low-humidity environment (Selwyn 2004) or applying a protective (organic) coating. Secondly, the pile-shoe's brittleness needs to be considered in the context of conservation of archaeological iron objects. As mentioned above, ancient phosphoric irons have carbon contents typically in the range 0.01–0.1wt% (Tylecote 1986). These amounts of carbon should prevent the type of impact embrittlement shown by the pile-shoe, as may be concluded from impact tests on phosphoric iron and steel containing 0.01–0.015wt% carbon (Josefsson 1954; Suzuki *et al* 1985). Carbon-containing phosphoric irons can be embrittled (Rellick and McMahon 1974; Stewart *et al* 2000a), but this requires specific heat-treatments which are unlikely to have been employed in antiquity for 'everyday' iron objects (see also Gouthama and Balasubramaniam 2003). Even so, some or many of the other pile-shoes stored in Cuijk could have extremely low carbon contents at similar locations outside the weld area. If any are to be removed from storage, they should be handled and transported with care to avoid breakage, despite their fairly robust appearance.

Conclusions

Impact brittleness of the pile-shoe bar was most probably caused by the combination of extremely low carbon and high phosphorus contents. Surface corrosion could have facilitated the brittle fracture by providing a notch effect during impact. The extremely low carbon content and the coarse grain size are most probably due to

decarburisation during smithing, though a high heating temperature could also favour coarse grains. Similar pile-shoes should be handled and transported with care, and also protected against further corrosion.

It would be most interesting to take further samples from some of the pile shoes, and from different locations, for chemical analysis, controlled impact testing, fractography and metallography as they would most probably provide further insights into the techniques of manufacturing what are essentially low-quality, mass-produced artefacts.

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