Monitoring of archaeological and experimental iron at Fiskerton, England

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Abstract

The Iron Age site at Fiskerton is under threat of desiccation. In order to understand the present condition of the wooden structure and the metal and other artefacts, a project has been set up to monitor the ground water levels and quality, and the changes in condition of experimental modern materials placed in the vicinity of the causeway. This paper describes those aspects of the project which relate to the iron samples, including characterization of the corrosion products of three archaeological artefacts from recent excavations using XRD and SEM-EDS.

Abstract (Spanish)

Fiskerton, un yacimiento de la edad del hierro, está en peligro de deshidratación. Para comprender mejor el estado actual de la estructura de madera, de los objetos metálicos y otros hallazgos, se ha iniciado un proyecto para controlar los niveles freáticos, la calidad del agua y unas estructuras realizadas con materiales modernos situadas cerca del yacimiento. Este trabajo se centra en los aspectos relacionados con las muestras de hierro e incluye la caracterización, por medio de XRD y SEM-EDS, de los productos de corrosión de tres objetos arqueológicos recuperados recientemente.

Keywords: iron, corrosion, monitoring, XRD, SEM

1. Introduction

The pre-Roman Iron Age wooden causeway at Fiskerton was excavated in part in 1981 (Field and Parker Pearson 2003). Associated with the causeway are ferrous weapons, tools and other artefacts, and finds of bronze, pottery, stone, bone, jet and amber. The causeway appears to run from the site of the modern village of Fiskerton (five miles east of the city of Lincoln), down to the now canalised River Witham. Dendrochronology has dated the causeway to have been in use from at least 457 to 321 BC, although its precise function has not yet been established

The causeway has been under a fairly intensive arable farming and drainage regime for a number of years, and concern has been raised that this may have led to the desiccation of this internationally important site. Recent changes to land management with reversion to grassland will reduce plough impact, and a further planned land-use change will reduce the drainage of the site, allowing water-tables to rise. Whilst this recent initiative (which will see the creation of a natural wildlife habitat under a local Countryside Stewardship Scheme) would at first appear beneficial for the site, the effects of raising the water levels on the already desiccated soil and archaeological structures and artefacts are not known. It is possible that this may be detrimental due to the introduction of different water chemistry and oxygen regimes on site.

In 2001, a research committee was established to assess the survival and threats to the archaeology in the immediate vicinity of the causeway and in the wider area of the River Witham Valley (Catney and Start 2003).

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Through its direction, and under the initiative and project management of English Heritage (the government's advisor on the historic environment in England), a number of surveys of the site at Fiskerton have now been completed including geophysical, auger and field walking surveys. The purpose of these surveys was to provide as much information about the site at the point of this significant land-use change. In addition to those projects aimed at characterising the archaeological and palaeoenvironmental aspects of the site, a conservation management project was also set up with the following aims:

- To assess the current state of preservation at the site
- To determine the impact of re-watering on the preservation of archaeological materials at Fiskerton
- To test methodologies for assessing conditions of a variety of archaeological materials so that these methodologies could be used in the future at other sites in the Witham Valley and elsewhere.
- To provide guidance on future management of archaeological material at Fiskerton.

The project involves the assessment of the condition of previously excavated archaeological materials and also the deterioration of a range of modern experimental samples buried in the vicinity of the causeway. Additionally, the groundwater levels and quality are being monitored through sampling at a number of points at the causeway site. Research and monitoring will be on-going for a period of at least three years. The results of this work will help inform decisions about the precise nature of future land-use change, as well as provide a proxy measure for the preservation of the site.

This paper describes the project and presents the initial results to date which relate to the archaeological and the experimental iron. The first samples of the buried experimental materials were removed from the ground in June 2004 and the preliminary results are included and briefly commented on. The three main components of the project are the analysis of previously excavated (archaeological) iron artefacts, analysis of experimental iron samples, and the monitoring of the groundwater.

1.1 Analysis of archaeological iron artefacts

The metal artefacts from the 1981 excavation were examined and recorded during the programme of investigative conservation in 1982 (White 2003). In subsequent years, selected ferrous artefacts were analysed by metallography to investigate their metal structure (Fell 2003), and corrosion layers were analysed by x-ray diffraction (XRD) together with comparative samples from other waterlogged sites (Fell & Ward 1998). The presence of iron sulphides was concluded to be one factor in the unusual preservation of some of the ferrous artefacts at Fiskerton, although clearly the corrosion processes which cause the different corrosion types in the ironwork assemblage are complex. A separate study on a single artefact concluded that the presence of ferrous carbonate and complex calcium iron silicates was significant in the preservation of the artefact (Biek et al. 2003).

Subsequently, there have been a few small-scale excavations and casual finds of metalwork close to the causeway in response to dredging work in the dykes. In particular, an excavation in 2001 yielded ten iron artefacts of which three were selected for study of corrosion products and for metallographic examination (Figure 1). Selection was based on their robust condition and because they seemed to have been waterlogged probably continuously since burial. The three artefacts analysed were a currency bar (10), a shafthole axehead (11) and a socketed axehead (131), each of which provided:

- Five samples of surface corrosion products for XRD analysis
- One metallographic sample comprising a continuous corrosion layer from metal outwards, for the technological study of the metal (to be reported elsewhere) and for the study of corrosion products by scanning electron microscopy with energy-dispersive x-ray analysis (SEM-EDS).

1.2 Burial of experimental iron samples

To assist our understanding of the corrosion processes, modern test pieces of iron, serving as analogues for the archaeological artefacts, were inserted in the ground at two locations adjacent to the causeway. Samples will be extracted from both locations at intervals over three years. The installations also included materials being studied by other researchers (copper, bone, antler and horn), mounted separately and according to schemes devised for previous burial experiments elsewhere (e.g. Simpson et al. 2004). Analyses of the iron will include:

- Corrosion rate, as a measure of the weight of corrosion products generated per sample over time (cf. Matthiesen et al. 2004).
- Corrosion products to be analysed by XRD with any heterogeneous regions in the metal samples examined by SEM.
- Characterization of corrosion type and comparison with archaeological samples from Fiskerton analysed previously (cf. Fell and Ward 1998).

Figure 1. Axeheads 11 (upper) and 131 from Fiskerton excavations 2001. The lower axehead (131) is socketed and lugged. The 'V' notches from the cutting edges (left) are where the metallographic samples have been removed.

1.3 **Monitoring of groundwater**

The groundwater was monitored via peizometers (dipwells), which are tubes with finely perforated tips to permit the ingress of water. Clusters of peizometers were placed at five locations in the vicinity of the wooden causeway, with the following aims:

- To establish base-line data for groundwater levels at monthly intervals
- To measure on-site pH, redox potential, temperature and conductivity at monthly intervals
- To provide samples for detailed chemical composition of the groundwater at one monitoring point at monthly intervals
- To measure changes in these data over time.

In each of the five peizometer clusters, water levels have been recorded monthly since August 2003, whilst pH, redox potential, temperature and conductivity have been measured since October 2003. The chemical composition of the water is measured in the laboratory by the Environment Agency, the government body responsible for water use in England, with results available from February 2004.

The relationship between the water levels in the peizometers and the archaeology on site is less than clear. Crude estimates suggest that most of the archaeological material recovered from excavations came from the level or above the level of the highest peizometers. However, the base of any vertical timbers of the causeway would be likely to be much lower than the lowest peizometer. During the 1981 excavations only one vertical timber was excavated (due to the difficulty of such an operation), which was five metres long (Field and Parker Pearson 2003, 41). Given that the depth of "soft" sediment in some areas of the causeway is as deep as six metres, the potential for the survival of at least some of these vertical wooden structures is highly likely. This is important as it is often the working of the points of such timbers that is of most interest to wood and tool specialists (*ibid*). In the course of the next year, it is likely that small excavations will take place on site to investigate the condition of the preservation of unexcavated sections of the causeway.

Aside from the information that the monitoring programme will provide for understanding the conservation of the site, it also provides essential data for the experimental iron samples described previously.

2. Methods

2.1 Archaeological iron

Since excavation in 2001, the artefacts have been stored under desiccated conditions at the conservation laboratories of Lincolnshire County Council Heritage Service. The three artefacts selected for analysis were each sampled in 2004 to provide surface corrosion products for XRD analysis and a metallographic sample for the study of the corrosion layers by SEM-EDS.

The corrosion products for XRD analysis, chosen at random but biased towards black or darker coloured products, were selected as those that could be readily detached from the surface of the artefacts. Samples in the order of 1mg were ground in an agate mortar and mounted on a flat single-crystal silicon sample holder, designed to reduce background scatter.   X-ray diffraction data were collected on a Philips PW 1840 diffractometer using cobalt K_α radiation (wavelength 0.179026 nm) incorporating a solid-state silicon detector. A search-match computer programme (Philips, based on JCPDS files) was used to identify unknown components in the diffraction patterns by comparison with standards in the powder diffraction file.

The samples for metallography and investigation of the corrosion layers were removed from the cutting edges of the two axeheads and through the end of the currency bar, using a jeweller's piercing saw. The samples were mounted in Buehler epoxy resin and ground and polished to 0.25 μm fineness in diamond slurry according to standard metallographic techniques.   The mounted samples were examined firstly under a metallurgical microscope at magnifications up to x400, and then by SEM-EDS in a Leo 4401 Stereoscan electron microscope with Isis EDS software at 25 kV accelerating voltage and probe current of 2.0 nA with a germanium detector. Back-scattered electron images were obtained from unetched carbon-coated surfaces.

2.2 Experimental iron samples

The iron test pieces for burial at Fiskerton were mounted on rods of inert material and installed as two groups of six rods each and placed adjacent to the two clusters of groundwater monitoring peizometers nearest to the River Witham (clusters 1 and 2). One sample rod will be extracted from both locations at intervals of six months, 12 months and then probably yearly thereafter depending on initial results and changes to water levels and chemistry.

The installation system was similar to that devised for the experimental work at Nydam Mose, Denmark, for which we are very grateful to David Gregory for advice. Our system is rather simpler however, comprising core rods of glass fibre reinforced polyester, spacers of polypropylene, and the samples attached with nylon cable ties in recesses along the rods. Each two metre rod was fitted with eight samples of iron spaced over a length of 1.2 m. Different soil conditions and depths were factors in the positioning of the experimental samples on the rods. In total, 96 test pieces were required and these were made as strips of annealed ferritic wrought iron each measuring approximately 50 mm long, 10 mm wide and 4 mm thick. Each strip of iron was marked with a punch before accurately recording the weight and dimensions.

The rods were installed on 22 December 2003 and the first (6 month) extraction was made on 22 June 2004. Samples were initially stored wet at 4°C but were later transferred to desiccated storage at ambient temperature. Because the site is 400 km away from our laboratories, it is not possible to analyse samples immediately and these difficulties must of course be allowed for in our conclusions.

2.3 Groundwater monitoring

Clusters of either three or four Casagrande porous plastic peizometers, with a modified tip length of 130mm were installed at each of the five selected locations. Using results from an auger transect adjacent to the line of the causeway, the peizometers were installed at various depths, ranging from 0.6 m below the surface, to a maximum of 1.9 m below the surface depending on the depth and complexity of the deposits.

In almost all of these clusters the deepest sample was deliberately placed much lower than the known archaeological horizons to ensure that water would be available even in the driest months. The uppermost

peizometers were installed just below the bottom of the plough soil and most degraded peat horizons, but still within deposits that were for the most part in a less than optimal state of preservation. The general state of preservation of organic material within the deposits was found to improve with depth, with well preserved peat occurring somewhere between 1m and 1.3m below the ground surface. Three additional peizometers measure the water levels at positions east of the causeway, to provide a non-linear picture of groundwater levels.

Water levels are measured using an electronic dip-meter with audible depth indication and the other data recorded from water samples recovered in the field using samples drawn from the peizometers using a bailer. Water chemistry data were monitored using a Pro-Sys portable multi meter for pH/mV, TDS, conductivity and temperature, with individual probes for each test.

Further determination of the chemical composition of the groundwater is provided from a 1.5 litre water sample drawn from one peizometer at cluster 2, the only sample point which has shown slightly faster rates of recharge, with water availability throughout most of the year. The analysis of these samples is funded and measured off-site by the Environment Agency. Measurements include lead, ammonia, nitrate, sulphide, chloride ion, orthophosphate, silicate, sulphate, calcium, tin, manganese, iron and copper.

3. Results

The XRD results from the samples of corrosion products from the three archaeological artefacts are summarised in Table 1. The dominant crystalline phases determined from each artefact were greigite (Fe_3S_4) and siderite ($FeCO_3$). Greigite formed a major component of the crystalline phases in nine of the 15 samples, often in conjunction with siderite, although the latter was only a major component in six samples. Other components detected were pyrite (FeS_2), mackinawite ($Fe_{1+x}S$), goethite (α-FeOOH), magnetite (Fe_3O_4), vivianite ($Fe_3(PO_4)_2 \cdot 8H_2O$) as well as small amounts of calcium carbonate and other soil constituents such as quartz.

Table 1. Summary of XRD results of archaeological samples

Sample	Object	Crystalline components
1	Bar 10	**greigite** (siderite calcite)
2	Bar 10	**greigite** **calcite**
3	Bar 10	**vivianite** **calcite** siderite (?goethite ?magnetite)
4	Bar 10	**calcite** **siderite** (greigite pyrite ?magnetite)
5	Bar 10	(greigite goethite siderite + other)
6	Axe 11	**greigite** (siderite) (calcite)
7	Axe 11	**greigite** magnetite
8	Axe 11	**greigite** (calcite)
9	Axe 11	**greigite** **siderite**
10	Axe 11	**siderite** greigite magnetite goethite
11	Axe 131	**greigite** **siderite** **pyrite**
12	Axe 131	**greigite** **siderite** **pyrite** (?mackinawite)
13	Axe 131	goethite magnetite calcite quartz
14	Axe 131	**greigite** **siderite** pyrite
15	Axe 131	**pyrite** vivianite calcite (goethite)

Major constituents shown bold; minor shown normal and trace levels are bracketed

The corrosion layers on the metallographic samples were very thin, a consequence presumably of little active corrosion of the artefacts in the waterlogged burial environment. Adjacent to the surviving metal (which in all three artefacts comprised ferrite with small amounts of pearlite or grain boundary cementite) there were occasional traces of remanent cementite. Remanent structures are not uncommonly preserved in less corroded iron artefacts such as those from anoxic environments at near neutral pH. Elemental mapping of part of the corrosion layer from axehead 11 distinguished in particular, concentrations of S in the outer corrosion layer including occasional circular forms, presumably framboidal sulphides (Figure 2).
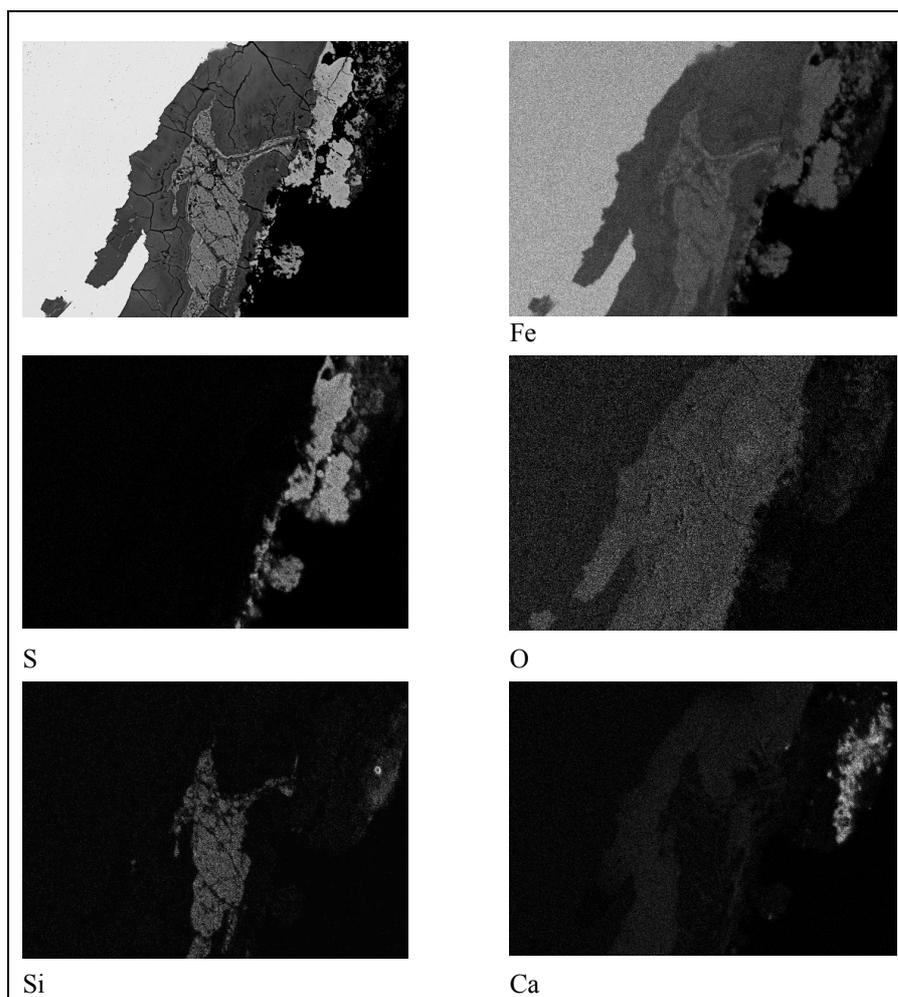

Figure 2. Scanning electron micrograph of metallographic sample from axehead 11, showing top left, that there are two corrosion layers, an outer layer (centre) in contact with the metallic iron (left), and an outer layer (right). The back-scattered electron images show element distributions for Fe, S, O, Si, P. Width of each image, 0.4 mm

In terms of the analysis of the modern experimental samples of iron and the ground water monitoring, only preliminary results can be presented at this stage.

Visual appearance of the iron samples extracted at six months suggests that there has been considerable oxidation of the upper samples from both groups and probably in all the samples from cluster 1. The latter are nearest the delph of the river, within clay deposits which seem to suffer from frequent changes in water levels. The cluster 2 samples, however, are buried in peaty layers and water levels are more consistent. The smell of hydrogen sulphide was very noticeable when these samples were removed. Pronounced orange staining was visible on the plastic installation rod at a position equal to 0.8 m below ground level. Because of the extensive oxidation, only the lower six of the eight samples on the rods removed from each cluster were analysed by XRD (Table 2). These were determined a few weeks after extraction from the ground and may therefore reflect some recent corrosion. There are two principal findings. Firstly, iron sulphides are present in samples from cluster 2 but not in the samples from cluster 1. Secondly, iron carbonate was not detected in any samples.

Table 2. Preliminary XRD results for 6 month experimental samples

Depth* (m)	Crystalline components #	
	Cluster 1	Cluster 2
0.98	**quartz calcite** goethite	**goethite** quartz
1.19	**goethite** (magnetite)	**goethite calcite** (?akaganeite)
1.32	**goethite calcite**	**mackinawite calcite** goethite (magnetite)
1.45	**quartz calcite** goethite (magnetite)	**mackinawite** calcite
1.58	**quartz** calcite	**goethite mackinawite calcite** greigite
1.61	**quartz calcite** goethite (magnetite)	—

 # Major components shown bold, minor shown normal, trace levels are bracketed;
*Approximate depth below ground to mid sample; — Not analysed because of insufficient sample

Water levels on site during the 10 months for which data are available are highly variable (Figure 3). For peizometer A in cluster 2, water levels were recorded between only 0.69 m below ground in January 2004, to as low as 1.62 m below ground two months earlier, a difference of nearly one metre. Similar results were recovered from the other monitoring positions, with the highest level recorded only 0.09 m higher, from peizometer C, cluster 3. Some of the shorter peizometers, and those towards the north end of the site have only recorded water during part of the year, even though every attempt was made to ensure that at least one peizometer would be long enough to sample water even in the driest conditions. For the months of October and November, water level data (and therefore samples for other analyses) was only recovered from 10 out a possible 34 peizometers, less than 30% of the number installed. Water levels in cluster 1 are in general slightly lower than those for cluster 2 reflecting the dewatering effect of the adjacent delph (drainage ditch).
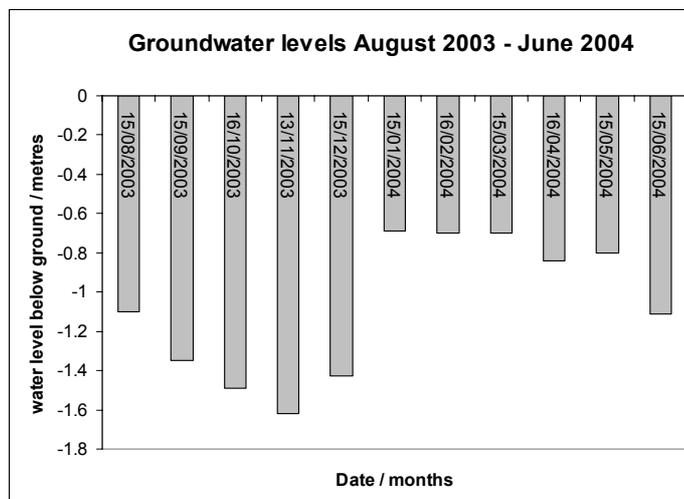
Groundwater levels August 2003 - June 2004

Figure 3. Water levels from peizometer A, cluster 2, from August 2003 to June 2004. The bars on the graph are a visual representation of the empty peizometers. The bottom of the bars mark the water level below ground level.

There is only a limited amount of data so far collected for pH, redox potential, conductivity, temperature and chemistry and these results require careful interpretation and may in time need to be calibrated. However, it is worth noting that, as far as can be established, the tests undertaken are providing a proxy measure of the state of preservation of the deposits. Where deposits are below the level of the groundwater year round, pH values vary between about 6 and 7. Redox values for these samples usual fall between +100 and -200 mV. In contrast to this, in deposits that have only recently been re-wetted, following a period of time where the groundwater level was below the depth of the peizometer, a different set of results are seen. In the first month that a sample is re-wetted, pH readings are usually as low as 3.5 – 4.5, and become less acidic with the increasing time that the deposits are below the level of the groundwater. In these samples, redox values are typically between +330 and +125 mV.

4. Discussion

The range of crystalline corrosion products detected on the archaeological artefacts, and in particular the preponderance of greigite and siderite, is very similar to the previous results for the analysis of artefacts from the 1981 excavation at Fiskerton (Fell and Ward 1998). In those analyses, made many years after excavation of the artefacts, pyrite and greigite were recorded as the major sulphide phases in equal numbers of artefacts, whereas in the present study, greigite dominated. Pyrite appeared on one artefact in particular (axehead 131), and this was the artefact which appeared to be most corroded of the three. This is probably in agreement with the generally accepted pathway for the conversion of the monosulphide, mackinawite, to other metastable phases including greigite, and finally to the disulphide phase, commonly pyrite (Morse et al. 1987; Vaughan and Lennie 1991; Walker 2001).

The second major constituent of the corrosion layers, siderite, was found in 10 samples. This is a similar ratio to the previous analyses where six of nine sampled artefacts from Fiskerton were found to have siderite in their corrosion layers. The formation process for siderite at Fiskerton may be related to sulphide production through the reduction of organic matter (Fell and Ward 1998, 114). Siderite is a major corrosion product on iron artefacts from Nydam Mose, Denmark, whereas sulphides are absent (Matthiesen et al. 2003). The authors suggest three possible mechanisms for the formation of siderite and that the layered appearance in the corrosion layers may indicate that it is not a simple formation process (Matthiesen et al. 2003, 190). They further discuss the possible protective nature of siderite on ironwork at Nydam. At Fiskerton, the presence and possible

significance of siderite was noted by Biek et al. (2003). Clearly, however, the corrosion processes and siderite formation at these two sites are not the same and further research and monitoring of the site conditions at Fiskerton is necessary to better understand the corrosion mechanisms.

Elemental mapping for axehead 11 showed sulphur concentration in the outer corrosion layer and this result is similar that for axehead 131 from the 1981 excavations (Fell and Ward 1998, fig 3). This may explain why many of the corrosion products analysed by XRD showed high sulphide levels and it is worth reiterating that the sampling procedure for corrosion products was biased towards the darker deposits. Of the few artefacts examined by metallography, the layering in the corrosion mantle seems less complex than at Nydam. In our artefacts, the outer layer containing sulphides could result from changes in burial conditions, in particular pH, at some time in their history.

The preliminary results for the experimental iron samples show the sulphides mackinawite and greigite forming at cluster 2, but no siderite. Groundwater measurements at cluster 2 have indicated readings of pH 6.2 and of calcium carbonate 125mg/l – conditions which are not too favourable for the formation of siderite. However, it is clearly premature to attempt elaborate interpretation of the data at this early stage. The presence of goethite and other iron oxides in most of the samples is probably due principally to post-excavation corrosion .

Of the site monitoring data available from Fiskerton, the current indication from the water level results suggest that much of the archaeological site is likely to be above the level of the groundwater. This is very much seasonally affected, with the driest months in October and November and the wettest (or months where the water levels were highest) occurring during January, February and March. This is not likely to provide a particularly beneficial environment for the preservation of organic deposits, nor inhibit the corrosion of any surviving archaeological metals, or the recently introduced experimental samples. When water levels are low, the potential for oxygen diffusion to previously anoxic deposits increases, which would in turn encourage activity of aerobic bacteria, and also further advance the corrosion of metals. At Fiskerton, this potential was certainly exacerbated during the driest months, when much of the upper 0.5 m of soil on site was completely dried out, and in many places deep cracks to a similar depth were visible. Given that the recent land-use change is from arable to grassland, there should be fewer periods when the field is fallow with no crop cover. Whilst this should reduce the amount of direct moisture loss through evaporation from the bare soil, the results from June 2004 would suggest that other mechanisms, such as evapo-transpiration are likely to be contributing to the reduction in water levels.

Redox data are the hardest to measure in any monitoring project, and the type of data collection method that we have employed on site has been suggested to be less reliable than other, non-water sample based methods of redox measurement (Smit 2002, 96). However, the same author also recognises that redox data from dipwells do bear some similarity to the results from other, 'more accurate' methods. The principal concern with redox results from groundwater is that oxidation/reduction reactions occur during the time that the water is in the borehole. This is similar to our experience, and early results indicate lower redox potential readings in peizometers before purging, and similar to Smit, particularly where water samples had collected in the dead-end of the peizometer and had not been effectively emptied. By purging the water the day before, changes in redox within the borehole will only relate to reactions occurring over a 24 hour period. In the near future we hope to also further calibrate our results, through comparison with results from *in situ* redox electrodes, or modelling the potential for oxygen (and other electron-acceptor species) transfer within the closed dipwells. The pattern of low pH and high redox potential in deposits where waterlogged conditions have recently been re-established, is similar to that recorded by Caple and Dungworth (1998) in laboratory experiments on the re-hydration on organic deposits. If nothing else, this would suggest that at least the basic trends identified in the redox data do replicate those from earlier work.

Since the burial of the experimental materials, water levels on site have been rising. Potentially this suggests that to begin with, samples would become exposed to water with high acidity and, as indicated by the high redox values, the presence of significant numbers of electron acceptors. This may also have been the scenario in antiquity – variations in water levels and chemistry, resulting in complex corrosion processes on the ironwork. Over the next three years, we will be monitoring site conditions and the deterioration effects on the experimental materials, as well as possibly recovering additional archaeological artefacts. This will provide data towards better understanding of the corrosion sequence as well as informing on the best system to preserve the site and the artefacts for the future.

Acknowledgements

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