

Mail links from the de Soto *entrada* of 1540

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Abstract

Among 22 links recovered in Florida from Hernando de Soto's expedition, four were of tin bronze and the remainder of iron. Most of the iron links were completely mineralized but sectioning revealed remnant areas of sound metal in eight links. The microstructures indicated that the links were fabricated from drawn wire by rivetting. There was no conclusive evidence that high-phosphorus iron had been selected for the wire.

Introduction

Wire has traditionally been a high-tech product, thus it can be a sensitive indicator of technological development. Since iron wire is especially difficult to produce, ethnographic accounts have given close attention to traditional processes of making iron wire wherever it occurs¹. In Europe wire mills represented one of the earliest industrial applications of water power for other than flour milling². In mail armour the transition to the use of drawn iron wire, a mill product, from the hand forging of links is such an indicator. Earlier iron links were either punched or cut whole from sheet, or riveted or welded from strands slit from sheet; later they were welded or riveted from wire.

Mail ('chain mail' is considered incorrect terminology) comes from the latin word *macula*, meaning the mesh of a net. Several classical authors quoted by Piggott³ refer to mail as originating with the Gauls and pre-Roman finds in Britain and on the continent support this origin.

Medieval mail and its construction have been studied by E Martin Burgess, Cyril Stanley Smith, Alan R Williams, and Colin Brewer⁴, but, with the exception of Burgess' examination of some Roman period links from a Scottish hoard, all these examples were well preserved. Until the recent study by Lang, Craddock and Hook of two well-preserved links from the 8th-century Coppergate helmet⁵, there were no published metallographic studies or chemical analyses of mail from archaeological contexts.

The de Soto *entrada* of 1539-1543 heralded the end of mail as effective armour. According to Garcilaso de la Vega⁶ a test was performed during the winter encampment of 1540 in which it was shown that Apalachee Indian arrows easily pierced mail armour. The site of this encampment was discovered in Tallahassee,

the capital of Florida, in 1987⁷, and a number of mail links were recovered from it. The excavator, Charles R Ewen, generously made 22 links or fragments of them available for study⁸.

A number of questions were asked about these links. One was whether the excavated links were lost or, in light of the 1540 test results, intentionally discarded. The excavators concluded from the scattered nature of the finds that the links had been lost accidentally. Another question was whether the links were fabricated from wire or cut from sheet. A further question was the quality of the iron itself and its selection and refining. These questions could be addressed in the laboratory.

Date of Westphalian fining

Earlier research in the Conservation Analytical Laboratory of the Smithsonian Institution showed that iron used for wire in the 17th and 18th centuries was selected for high phosphorus content and refined by techniques unique to the making of iron for wire. These techniques, which we called Westphalian fining, had the effect of removing both carbon and slag from the iron before it was submitted to drawing⁹.

We know from documentary sources that the special 'fining' (*ie*, refining) of wire-iron began about 1390, and that the selection for high-phosphorus pig iron to be fined to wire-iron was standard processing practice by 1677¹⁰. The date this selection was introduced was conjectured, on the basis of a change in the proportions of wire-strung musical instruments, to be about 1600. The links from the de Soto encampment of 1540 were examined for direct evidence that this procedure was followed earlier, *ie* no later than 1539.

In general wire cannot be dated intrinsically, neither can dated examples of mail be sampled for original links because of repairs; broken or missing links in this kind of armour must be replaced in order to maintain it. So it seemed that securely dated links were best obtained from closely-dated, sealed archaeological contexts. This in turn replaced the problem of dating wire with the difficulties of examining very small pieces of rusty iron.

The de Soto links

The iron links were heavily corroded, with accretions of soil and particles of quartz (sand) embedded in the corrosion product. Including these accretions, they

weighed from 0.15-0.3g, measured 5-10mm across, and were roughly 1.5-2mm thick except at the rivets where they were nearer to 3-4mm thick. All the links were radiographed, but this failed to indicate whether or not sound metal remained.

An initial examination of link 87.1 after surface cleaning was made in a scanning electron microscope. The X-ray emission spectrum from its fracture surface (Fig 1) indicated the presence of iron, silicon, aluminum, and phosphorus, all elements that to a certain extent may be in accretions from the soil. Another fragment (87.2) gave a similar result. Both were attracted by a magnet, but their fracture sections had a black, mineralized appearance, not a metallic one.

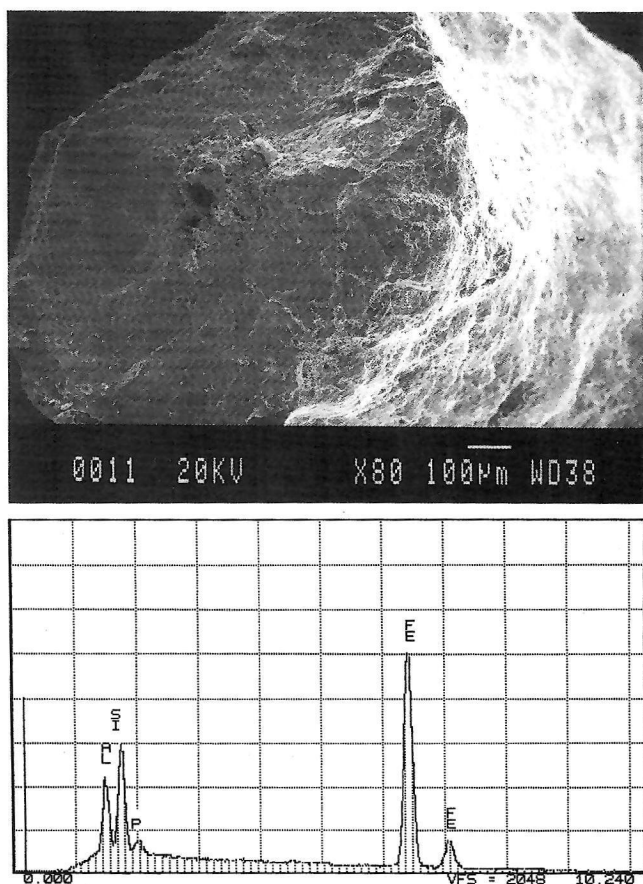


Figure 1: Scanning electron micrograph of the fractured end of a completely mineralized link (a), and x-ray emission spectrum from this surface (b).

The specific gravity of link 87.1, which weighed less than 0.2g after cleaning, was measured using perfluoro-1-methyl decalin as the immersion fluid¹¹. The result was 4.1 Mg/m³ at 25°C, somewhat less than that of magnetite (5.2 Mg/m³). This was taken as evidence that the link, and most probably the fragment as well, was totally mineralized. To locate sound metal in such small and badly corroded objects it seemed necessary to section them.

Sectioning rusty iron

In practice good sectioning and polishing required mechanical cleaning of the specimens before mounting metallographically in plastic. Cleaning was done with a fine dissecting needle while the link was submerged in a drop of distilled water. The mineralised areas were brittle, and most difficult to remove were the grains of quartz (sand, SiO₂) that had become heavily concreted with corrosion products. The greater hardness of these grains caused the most difficulty in obtaining a flat section and a controllable abrasion rate during polishing.

The small size and fragility of the cleaned links called for mounting in a plastic hard enough to control the abrasion rate, yet one fluid enough to fill cracks in mineralised areas; Buehler's Epo-Thin proved satisfactory. Using a small automatic polishing machine (Buehler's Minimet) with 15µm diamond paste followed by 3µm and 0.1µm diamond suspensions, each link was sectioned progressively at a slow and controlled abrasion rate, inspecting the surface being prepared at frequent intervals so as to avoid polishing away any metal that may have remained in the matrix of corrosion products.

Sections were made of nine links whose strength during mechanical cleaning suggested metal was still present¹². According to Burgess, round wire, 'a sign of degeneracy in mail, was not generally used until very late – after 1500'¹³, so the first step in examining the original microstructure of the links was to establish that they were made from drawn wire. This was done from several lines of observation.

The microstructure of the remnant metal had the texture characteristic of drawing and showed no sign of annealing or having been brought to welding heat. Morphologically the nonmetallic inclusions – which often were also observable within completely mineralised samples – were lengthened as in wire drawing, but not broadened as well, as in hammered sheet. Further evidence of drawn wire was joining, not uniformly required where links were punched whole from sheet. One link (EEa) was sectioned parallel to the plane of the link, through the joint (Fig 2), revealing a rivet and remnant metal in both link and rivet.

Analysis

Once it could be seen that the links were fabricated by rivetting wire stock, the next question was the identity of the iron alloy: wrought iron, high-phosphorus low-carbon iron, or steel. Certainly these links were not steel; cementite (iron carbide, Fe₃C) was observed in neither the metal nor the corrosion product¹⁴.

The levels of phosphorus sought (*c* 0.15-0.22%) were not

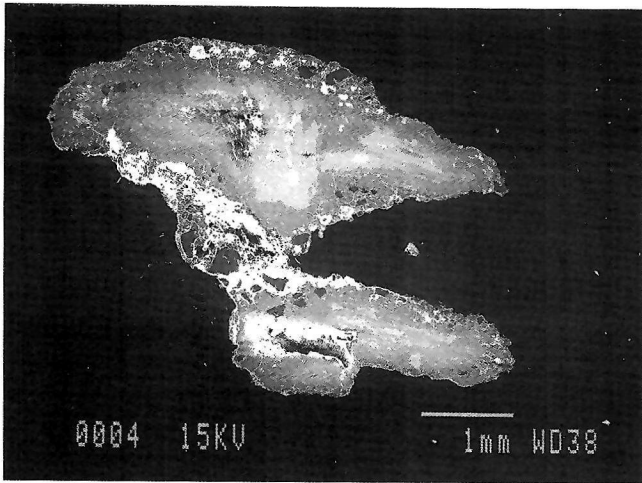


Figure 2: Section through link EEa, showing remnant sound metal in both link and rivet.

detectable in the metal by the analytical method used (X-ray emission). It was, however, already known that the selection and fining process that produced a high-phosphorus low-carbon alloy resulted in wire-iron having a diagnostic distribution of nonmetallic inclusions,

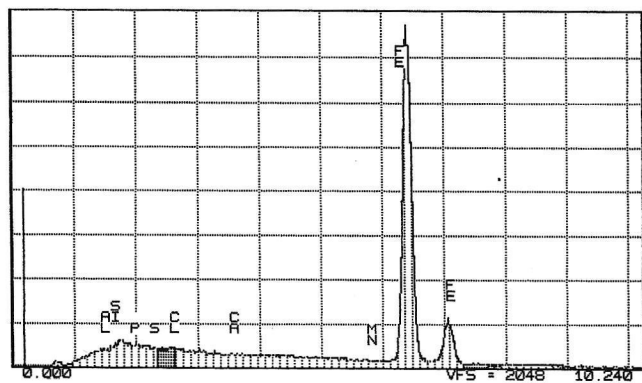
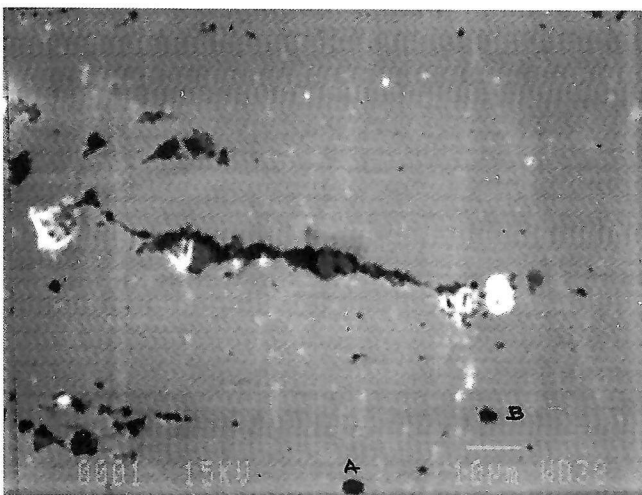


Figure 3: Area of sound metal in the rivet of link EEa (a) that produced the x-ray emission spectrum in (b). Spectra of inclusions indicated by A and B are shown in Figure 4.

specifically an abundance of iron phosphide inclusions (Fe_3P) but a virtual absence of the slag (SiO_2 , Al_2O_3) found in wrought iron¹⁵. Thus the two can be distinguished from each other solely on the analyses of their inclusions¹⁶.

The rivet metal of link EEa when analyzed by X-ray emission had only iron peaks in the spectrum (Fig 3). Inclusions, separately analyzed (Fig 4), contained significant amounts of phosphorus. The analyses were confirmed by microprobe. The same procedure was followed with the metal of the link itself (Fig 5) with the same results: iron metal containing phosphide inclusions but no slag. Similar results, however, were obtained from only two other links (CCb, DDa). Other sections were not characterized by phosphide inclusions.

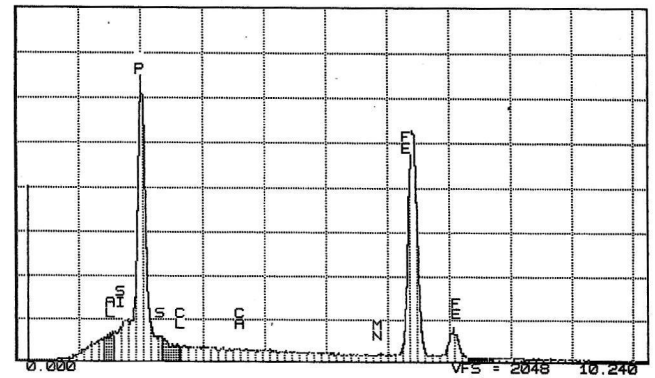
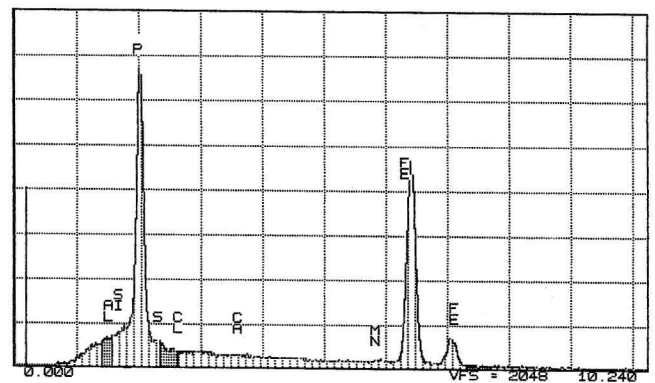


Figure 4: Spectra of the inclusions A (above) and B (below) indicated in Figure 3.

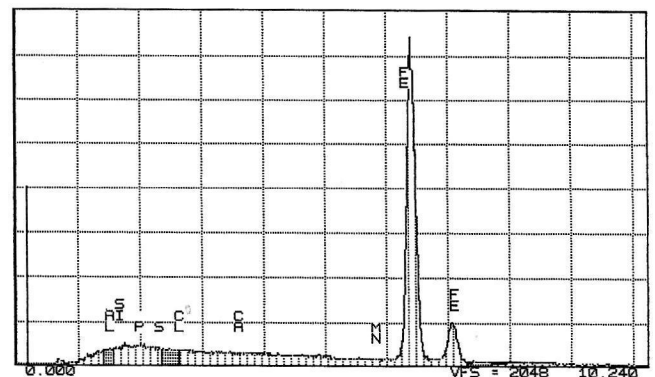


Figure 5: Spectrum from metal in the link of EEa (compare with Figure 1).

Conclusion

The metallographic examination and analysis of these links determined that three iron links were fabricated from a high-phosphorus, low carbon alloy that had been drawn into wire and a rivet that was wire of the same alloy. Had these been the only links available for sectioning and analysis, selection for phosphorus in wire-iron as early as 1539 would have seemed substantiated. However, phosphorus often appears in old iron as an impurity, depending on the ore, or the fuel. To establish that it is present intentionally – *ie*, that the special selection of iron for wire making documented in 1677 was being practised – phosphorus must be present in every link. Instead it appeared inconsistently in the samples examined, so a date for the selection process earlier than 1677 was not supported by the de Soto links. Nevertheless they did demonstrate a consistent use of wire for mail by 1539¹⁷.

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- 16 Before analysis by microprobe became generally available, inclusions in mail were identified simply as slag; for earlier mail this may be a fair assumption.
- 17 I am grateful to Dr Ewan for providing the samples. I am also grateful to Alan R Williams for the

generous loan of his metallographic sections of mail links, and to Janet Lang for copies of her micrographs of Sutton Hoo mail.

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