# THE MIDDLE BRONZE AGE IRON PUNCH OF SOUTHEAST DRENTHE

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ABSTRACT: The iron object of awl or punch form recovered from the MBA southern plank footpath XVII(Bou) (dendro-age around 1350 B.C.) in the raised bog near Emmen, Drenthe, Netherlands, has been examined metallurgically. It was produced from an iron source by a reduction process and was then forged to the required dimensions. There is thus clear evidence of iron production and usage some 500 years before the Iron Age officially begins in this area.

KEYWORDS: The Netherlands, Southeast Drenthe, Middle Bronze Age, iron smelting, iron punch, metallurgical research.

## 1. INTRODUCTION

The 'punch', shown in figure 1, is 38 mm long and of roughly square cross-section (2-3 mm). One end is pointed with a pyramidal tip (fig. 2), apparently produced by ground or hammered flats. At the other end the square form has been reduced to a chisel or cutting edge (fig. 3). It has suffered some corrosion but is of generally sound internal structure. Presumably it was originally straight but has been bent at some stage, perhaps in use.

The investigation to establish the type of material and the method of manufacture had to be carried out in a way consistent with the object being preserved as a museum display specimen. A wedge-shaped section was cut from a point near the centre using a jewellers saw (fig. 4). In this way material was obtained for metallographic examination and hardness testing, which could be subsequently glued back into place with suitable filling without affecting the original overall dimensions or shape of the object. Compositional analysis was also obtained from this small removed section by means of electron probe microanalysis. Although chemical or spectrographic methods might have been preferable for the analysis, the amount of material destroyed for any significant improvement in accuracy would have been prohibitive.

A procedure which allows the replacement of sections taken for investigation is considered by the author to be an important requirement for all investigations of this type. Not only does it restore the object as far as is finally possible but, more importantly, it ensures the availability of a specimen in its original context for subsequent re-examination at some future date if developments in theory or available analytical and metallographic techniques suggest that more useful information might be obtained. In an examination of South East European copper axes from the Ashmolean Museum undertaken by the author (Charles, 1969) it was discovered by X-rays that a specimen had been removed previously during the examination by Voce reported by Coghlan (1961) and the gaps filled with a suitable camouflaging putty. It was therefore necessary to take a further specimen for the examination, a process which clearly cannot be repeated very often. In the present work it was possible to carry out a second examination to check if there was any copper dissolved in the iron object a considerable time after the first examination, by dissolving out the glued-in specimen, and without any further damage.

## 2. COMPOSITION

The specimen cut from the object was mounted in plastic and polished to a metallographic finish. Analysis was effected by use of the 'Microscan 5' (Cambridge Inst. Co. Ltd.) electron probe micro-analyser and the only element detected in the iron matrix was manganese at a level of 0.52%. The few very small non-metallic inclusions in the matrix contained manganese and usually some sulphur and appeared to be manganese oxide/manganese sulphide. There was very little variation in the manganese content from point to point in the matrix ($\pm$0.01%). Analysis for carbon content and the ferritic microstructure clearly indicated a value below 0.1%, probably in the range 0.05-0.07%. Phosphorus was also absent in significant amount. On the

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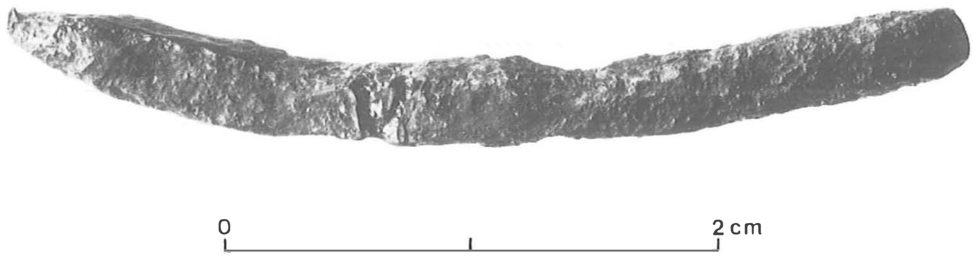


Fig. 1. The iron punch.

second occasion when the section was submitted to electron probe microanalysis, a specific purpose was to determine any copper content. No significant content existed (0.003-0.022%).

These analysis results are interesting. Firstly, the absence of nickel rules out any meteoritic source for the raw material, since all meteorites contain significant proportions of nickel. Secondly, it is unusual to find in primitive iron such a manganese content with the virtual absence of carbon. As Tylecote (1962: pp. 182, 191) has pointed out, in the primitive hearth processes manganese present in an ore hardly ever appears in the iron, but rather in the slag, and the strongly reducing conditions required for manganese to appear in the iron would also give take-up of carbon from the charcoal fuel. Certainly, however, manganese is frequently present in iron ores which would give 0.5% Mn in the iron with a substantial level of recovery. There are, of course, also many ores high in manganese ($\sim$10%), particularly bog iron ores which are amorphous mixtures of hydrated iron and manganese oxides, where in some the manganese may even be present in greater proportions than iron. In any event, whilst it is accepted that whilst the occurrence of such a manganese content in early iron is unusual, modern experience in the production of sponge iron clearly indicates that such compositions can be readily obtained by a very simple system.

The oldest, and still the largest, plant for the production of sponge iron powders is at Höganäs in Sweden where a high grade magnetite ore is mixed with anthracite or coke breeze and put into ceramic containers ('saggers') which are subsequently heated in a furnace. After lengthy heating at $\sim$1200 °C and cooling, the products are emptied out and the sponge iron pieces separated from the residue and then pulverised to powder. The pieces of sponge could equally well, of course, be consolidated and perhaps welded together in the presence of a suitable slag into small pieces of high density iron. Leadbeater *et al.* (1947) give the composition of eleven 'oxide-reduced' iron powders and for five of these the carbon contents lie in the range 0.06-0.01% with manganese from 0.33-0.75%. Goetzel (1949: p. 179) similarly gives the specification of some sponge iron powders as being C 0.1% max, Mn 0.5%.

Fig. 2. The pyramidal tip of the punch.

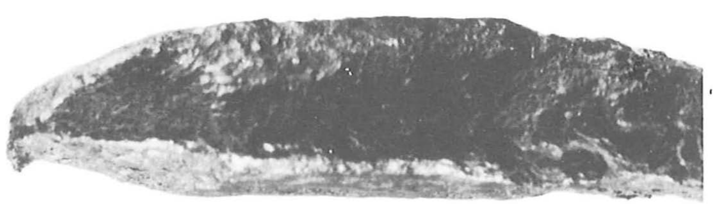

Fig. 3. The chisel or cutting edge of the punch.

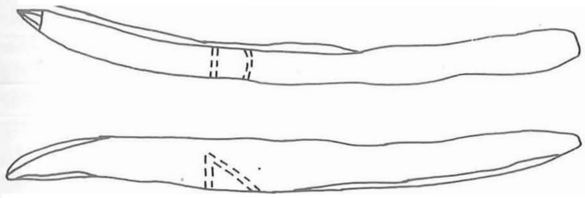

Fig. 4. Sketch showing the position of the wedge-shaped section.

It is our present view that the significance of this information rests in the small, dispersed form of the product. With a high manganese content in a specific ore (*i.e.* a high thermodynamic activity of MnO, possibly unity) the manganese contents in the iron (reflecting a relatively low activity of Mn) can be achieved with the very strongly reducing conditions (high $CO/CO_2$ ratios) obtained in the presence of charcoal at temperatures in excess of 1200 °C. It is also well known that although the substantial blooms recovered from primitive furnaces are carburised to varying extents, they usually exhibit surface decarburisation. As the bloom cools to lower temperatures before removal from the furnace the $CO/CO_2$ ratio in equilibrium with remaining charcoal falls and below ~700 °C the conditions become positively oxidising. Since carbon is present as an interstitial solute it can still diffuse relatively quickly to the surface at such a temperature, a process which continues as carbon is removed from the surface through oxidation by $CO_2$. The manganese in the iron, on the other hand, although not now stable in relation to the oxidising character of the furnace environment, which becomes more so with decreasing temperature, being in substitutional solution will not diffuse at a significant rate through the iron sponge. In a small object, therefore, one could finally expect to find a carbon-free iron but with the manganese content largely unaffected and with, perhaps, small internal particles of largely manganese oxide remaining from incomplete initial reduction if the initial MnO content of the ore were high. This explanation fits both the powder and the examined artifact.

We now have to consider under what conditions such small pieces of iron would be produced. Clearly, if a furnace were being worked purposefully for the reduction of iron - using iron ore exclusively, certainly with any degree of development, it would be likely that the resultant bloom would be considerably larger than represented by the mass of the 'punch', and as a result would be more likely to be extracted earlier from the furnace, since its bulk would be easily found in the debris. Such bulk, early extraction and chilling by moving into cold air would prevent further decarburisation. Small pieces, as represented by the punch, could only be recovered readily once the furnace was cold, with maximum decarburisation.

This leads to the suggestion that such small pieces of iron were produced adventitiously in the smelting of copper. Whether an oxidised copper ore, such as malachite, was employed with an iron oxide flux (- perhaps the gossan (limonite) from the copper deposit itself), or a roasted sulphide such as chalcopyrite ($Cu_2SFe_2S_3$), the iron activity in the system would be high and the conditions at high temperature would be sufficiently strongly reducing to produce small amounts of iron locally within the charge. It may well be, therefore, that small amounts of iron were produced in association with copper smelting, to be recovered from the spent charge when breaking it down for recovery of the copper prills which would be subsequently agglomerated by melting in a crucible.

Small pieces of iron produced in this way might not necessarily contain appreciable copper, particularly if resulting from the reduction of pieces of separated iron oxide flux which had not come into contact with the copper ore. An alternative theory, however, resting on comment originally by Tylecote (1974), is that small amounts of iron could have been made in association with copper smelting, through simultaneous reduction to give a copper/iron liquid melt, from which iron would be rejected during cooling. With slow solidification in a crucible, such iron would form as an iron 'rim' to the crucible. In this case the iron would retain a copper content equivalent to the maximum solid solubility, which is quite substantial (1.8% in ferrite at 835 °C with little change at lower temperatures). The significance of the very low copper contents, for which a specific second examination was made, is clearly to exclude this possible mechanism of iron formation.

In view of the provenance of the object it is not unlikely that the source of iron oxide flux in copper smelting was hand-picked lumps of high grade bog iron ore (limonite) of moderate manganese content. The use of this material, which is porous and readily reduced to iron, is already established for many early iron-making activities. It is, of course, still possible that even this small 'punch' was the product of a purposeful smelt for iron, using exclusively such iron ore as the sole charge, and certainly with the recognition of the source of the metallic material so different from copper, but sometimes produced with it, there would have been the development in this direction. Once iron was produced in large enough pieces to retain substantial carburisation and where practice had been modified to assist in maintaining higher carbon levels, the product would be superior to bronze for many of the functional tools, and the Iron Age begins. It is not generally recognized that only when carbon contents in the range 0.4-0.5% are achieved do the properties become superior to a cold worked bronze in terms of hardness (*i.e.* yield stress). With an equal or superior product the easy accessibility to rich iron ores, as compared to the less concentrated occurrence of copper minerals, would clearly be attractive to a rapidly increasing population.

Although not readily explicable it is also interesting that at a later date the ancients distinguished between ores which produced a soft iron when reduced and those which produced a carburised iron, steel, as discussed by Sisco & Smith (1951) in relation to a treatise by Ercker (1580). Ercker's text indicates that siderite ($FeCO_3$) which he calls 'steel stone' tended to give steel on reduction by solid state processes as compared with magnetic ore which he distinguishes as an 'iron ore'. As Sisco & Smith comment, the physical or chemical basis for this would make an interesting study, for it is clear that the possible variations in composition which could arise from primitive processes with differing ores are not yet fully understood.

## 3. METALLURGICAL MICROSTRUCTURE

The section cut from the object was mounted in perspex successively so that two planes of the section could be examined in turn after polishing. In the unetched condition some small non-metallic inclusions could be seen, but at a relatively low volume fraction. One plane examined was that of the section cut at approximately 45° to the vertical and in this the inclusions were somewhat deformed from the original round to ellipsoid (fig. 5), consistent with a section at this angle through a more elongated form. A second plane was prepared and examined from the tip of the section, roughly coinciding with the centre of the object, in a direction as near parallel to the length as was possible in handling and mounting such a small fragment of metal. In this the inclusions were mainly markedly elongated in the lengthwise direction, with a few showing voids associated with harder inclusions where iron flow had occurred around them during working, without they themselves deforming to the same extent (fig. 6). In the etched condition the sections showed ferrite grains elongated in the direction of the length, clearly indicating fabrication into the present shape by mechanical working below the recrystallisation temperature, *i.e.* cold-work (fig. 7).

Fig. 6. Markedly elongated inclusions, in the lengthwise direction.

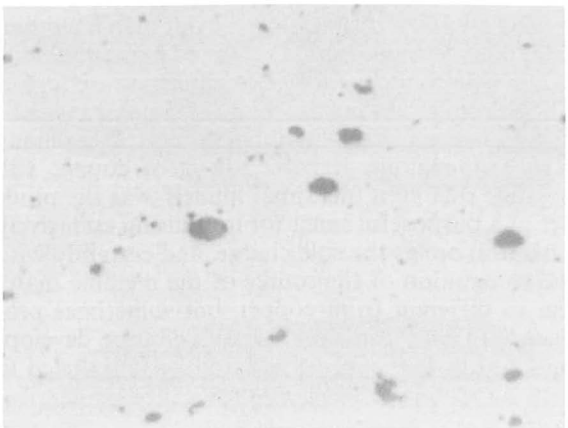

Fig. 5. Inclusions deformed from the original round to ellipsoid, in the 45° section.

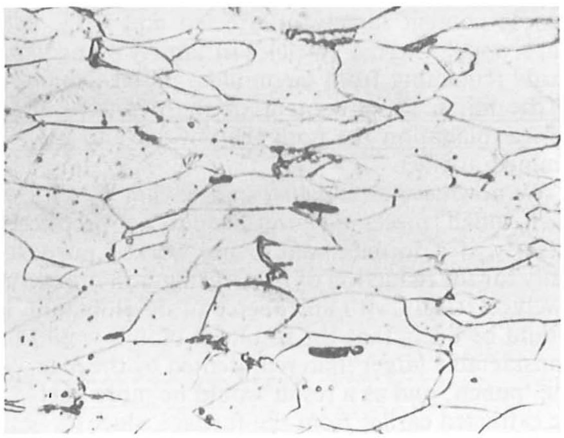

Fig. 7. Ferrite grains, elongated in the direction of the length.

That extensive work at low temperatures has taken place is also suggested by the elongated manganese sulphide inclusions which generally have a maximum relative plasticity, *i.e.* their hardness most nearly approaches that of the iron, with reduced temperature. Measurements of axial ratio for both the elongated grains and the non-metallic inclusions indicate similar degrees of deformation, indicating that cold work alone has been effected. Any work above the recrystallisation temperature will give an equiaxial structure in the room temperature ferrite but would still effect some continued deformation of the inclusions. Whilst measurements in both ferrite grains and inclusions would suggest at least 100% extension of the iron in length (50% reduction in area) during working, such an observation is greatly affected by the accuracy of the alignment of the examined plane to the working direction. With the very small amount of material taken for examination this was very difficult to do. However, hardness measurements indicated a very high level of work hardening and substantiated the metallographic observations.

## 4. HARDNESS MEASUREMENTS

The second metallographic plane was hardness tested over its whole area using the Leitz Miniload microhardness tester. The values for Vickers Hardness ($H_V$) obtained ranged from 254-272 with an average value at 262. The pyramidal point tip of the punch was cleaned of corrosion product by placing against a polishing wheel carrying $45\mu$ diamond paste and then also tested for hardness. Values varying between 290-317 were obtained at this point.

The hardness of annealed ferrite is ~100 and the value of 262 for the cold-worked main body of the piece corresponds to 50-60% reduction in area for such material, in agreement with the metallographic observation. Clearly, also, the working point has been further hardened in its formation by hammering or abrasion. It could also, of course, have become somewhat harder as a result of use, although its form is still fairly intact and does not indicate much subsequent blunting.

It is interesting to note that the Vickers Hardness for annealed copper is ~50, increased to 100-120 by cold work (30-70% reduction in area). Bronze will be harder, depending on composition and the amount of cold work, but values of 60-80 in the annealed or as-cast condition rising to 250 for cold-worked forms would be typical. It is clear, therefore, that this piece of pointed cold worked iron could have been used as a punch for decorating the softer copper or bronzes, but there are many other possible uses for an artifact of this form.

## 5. CONCLUSIONS

This iron object was produced by the solid-state reduction of a rich iron ore to produce a piece of iron sponge which was subsequently consolidated by hammering and then worked out to its final length. This cold working to final form involved of the order of 100% extension in length (50% reduction in area). Presumably the initial form of the sponge was such that consolidation yielded a piece of iron approximately 14×4×4 mm which was then lengthened. The similar extension observed for sulphide inclusions would indicate that hot working above the recrystallisation temperature was not involved at any stage in this lengthening process. The hardness developed, and the form, suggest that a possible use was as an engraving tool, awl or punch.

## 6. REFERENCES

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