Metallurgical analysis of iron artefacts from Thailand

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Summary

Four iron artifacts excavated in Northeast Thailand have been subjected to metallographic examination. Three of the artifacts, dated to the Late Iron Age (eg 300-400 AD) exhibit competent forging and welding of wrought iron, with cutting edges hardened by cementation. The fourth artifact was undated but exhibits a difficult blacksmithing technique, involving welding an ultra-high carbon content steel onto a wrought iron core to form a high quality axe blade. Details of heat treatments are reconstructed and invoke the suggestion that the undated axe-head may be contemporary with the other artifacts.

Introduction

This paper describes four iron artifacts discovered in Northeast Thailand and shown in Fig 1, numbered 1 to 4. Artifact 1, a crescent-bladed axe, was found on the surface of the ground a short distance from the Late Iron Age mound site of Non Phrik. The other three artifacts, a chisel (2), a spearhead (3) and an adze (4), were excavated from the mound and are dated to the first and second millennia AD.

Fig 1: Appearance of the four artifacts after removal of the metallographic samples. Arrows show sample sites.

The Non Phrik mound, which represents a prehistoric village site, is located near Ban Hua Na village, Phu Luong sub-district, Loei Province (latitude 17° 08′ 13″ N; longitude 101° 43′ 28″ E) (Fig 2). The mound rises a maximum of 3.6m above surrounding paddy fields. Non Phrik means "chili mound" as it has been under cultivation with this crop for at least two decades. The surface area of the site is about $257m^2$.

The site was originally located and excavated by the Thai Fine Arts Department in 1975 during the Pa Mong Archaeological Programme, a salvage project that was to precede the construction of a dam on the Mekong River (1). Further archaeological investigations which led to the discovery of the iron artifacts were undertaken by Rutnin in 1985 (2). The Fine Arts Department reported that the mound's stratigraphy was disturbed and this conclusion was partly confirmed by subsequent excavation. In particular, the uppermost levels were disturbed by ploughing and it is not known how much of the deposit has been lost because of the activities associated with cultivation.

The area excavated by Rutnin comprised $8m^2$ in the northwest sector of the mound. The details of the stratigraphy of the site, methods of excavation and the associated finds are reported elsewhere (Rutnin 1988) and only a brief description of the context of the excavated iron artifacts will be given here.

Two shattered pots laid on their sides with the mouth of one inserted over the other were encountered at a depth of 40-60cm in the main cultural layer. The sediment in this layer consisted of sandy clay loam with a pH of 5.5. A small socketed axe or chisel (artifact 2) was recovered directly above one of these vessels, which was decorated with "cord-marking", and could have come from inside it. A larger socketed axe or adze (4) and a socketed spearhead (3) were found inside the second vessel which was distinctively decorated with four appliqué spotted lizards (probably geckos). This form of appliqué decoration is also found at other Iron Age sites in the region. Local manufacture of the pots is also indicated by the nature of the sand temper.

Three 14c dates were obtained from charcoal samples taken from inside both vessels. These dates are as follows: (BP date is uncorrected 5568 year half-life; 2-sigma BC/AD ranges use Klein corrections (3):

(ANU-5028) 1690+/-120 BP (AD 60-575). Sample taken from immediately above the broken vessels.

(ANU-5032) 930+/-520 BP (AD 605-1420). Sample taken from the level of the sherds.

(ANU-5033) 1630+/-160 BP (AD 60-575). Sample taken from inside the broken appliqué-decorated pot.

Stratigraphically ANU-5033 is the most reliable date, and there is no significant statistical difference between this determination and ANU-5028. It is most probable then that the second millennium AD determination, which is for the sample that is stratigraphically least secure, is affected by contamination, or is not directly associated with the pots and iron artifacts. It is concluded that these artifacts date to the Late Iron Age. This conclusion is supported by the character of the associated finds (Rutnin 1988). As there is no evidence for iron working activities at Non Phrik it is likely that these artifacts were obtained through trade. Iron was produced in Thailand from 500 BC or earlier (7). A millennium later small scale production of bloomery iron was widespread but steel was more likely to be imported from either India or China (8).

Metallographic Examination

The iron artifacts were submitted to one of the authors (LMH) for metallographic examination.

One section was taken from each sample at points evident as triangular notches in Fig 1. Examination was by optical and scanning electron microscopy. Grain size was estimated by comparator. Representative hardness readings were taken by Knoop Microhardness apparatus and converted to Vickers scale (HV). Carbon content was estimated in representative areas by point counting of pearlite and carbide areas under the microscope. These quantitative observations are listed in Table 1. All specimens were ferromagnetic. In each specimen the bulk of the original metal was preserved under layers of surface corrosion of thickness 1mm or more.

Table 1

Artifact	Area	ASTM Grain Size	% Carbon	Average Hardness
1. Axe	Fig 4	8	1.8 ± 0.3	HV231
	Fig 5	3	0.01*	HV180
2. Chisel	Fig 9	6-7	0.03 ± 0.01	HV151
3. Sp'head	Fig 11 (centre)	7	0.13 ± 0.02	HV190
	Fig 11 (outer layers)	2-3	0.01*	HV163
4. Adze	Fig 16 (Lower edge)	8	0.2 ± 0.02	HV327
	Fig 15	6	0.05 ± 0.01	HV207

* Visual estimate

Notes re Table 1:

1. % carbon was estimated from the volume of carbide or pearlite determined by point counting after ASTM standard E562-76. The error range was obtained by statistical analysis of the counts made but the small samples, and the necessity to select different representative areas within any one sample, would be expected to increase the range of error.

2. ASTM grain size number = N where n = 2N-1 and n is the number of grains per square inch as seen under the microscope at a magnificationof 100x. Thus a higher number represents a finer grain size and n increases logarithmically with N as in the table below.

ASTM Grain Size

	ASTM No.	Mean Number of Grains per in.^{2} at 100X	Grains per mm^2
	-3	0.06	1
	-2	0.12	2
	-1	0.25	4
	0	0.5	8
Usual Range	1	1	16
Usual Range	2	2	32
Usual Range	3	4	64
Usual Range	4	8	128
Usual Range	5	16	256
Usual Range	6	32	512
Usual Range	7	64	1024
Usual Range	8	128	2048
Usual Range	9	256	4096
	10	512	8200
	11	1024	16400
	12	2048	32800

Artifact 1: Iron Socketed Crescent-bladed Axe

The sample was taken from the rear shoulder of the blade (arrowed in Fig 1) after an attempt to cut into the cutting edge appeared likely to cause excessive damage. Fig 3 shows the polished section through the thickness of the blade, encased in oxide on three sides. The straight side free of oxide was the final break at the base of the cut.

Microstructure

Fig 4 shows a representative microstructure of the darker areas in Fig 3, adjacent to the surface oxide. Fig 5 represents the whiter core region. In Fig 4 fine grains of ferrite (pale grey) are encased in heavy grain boundary deposits of carbide (dark). A black stringer of slag is prominent in the centre. Fig 6 is a scanning electron micrograph at high magnification of an area in Fig 4. The contrast is reversed, so that ferrite appears black and carbide (Fe_3C) is white. It is evident that the grain boundary deposits are almost continuous carbide, with patches of very fine lamellar pearlite. The carbon content was estimated as 1.8% carbon (Table 1) on the assumption that the grain boundary material was all massive carbide. The estimate is therefore too high, but the high carbon area (Figs 3 and 4) is certainly hyper-eutectoid, perhaps averaging 1.5% carbon.

Fig 5 at the same magnification as Fig 4, shows coarse

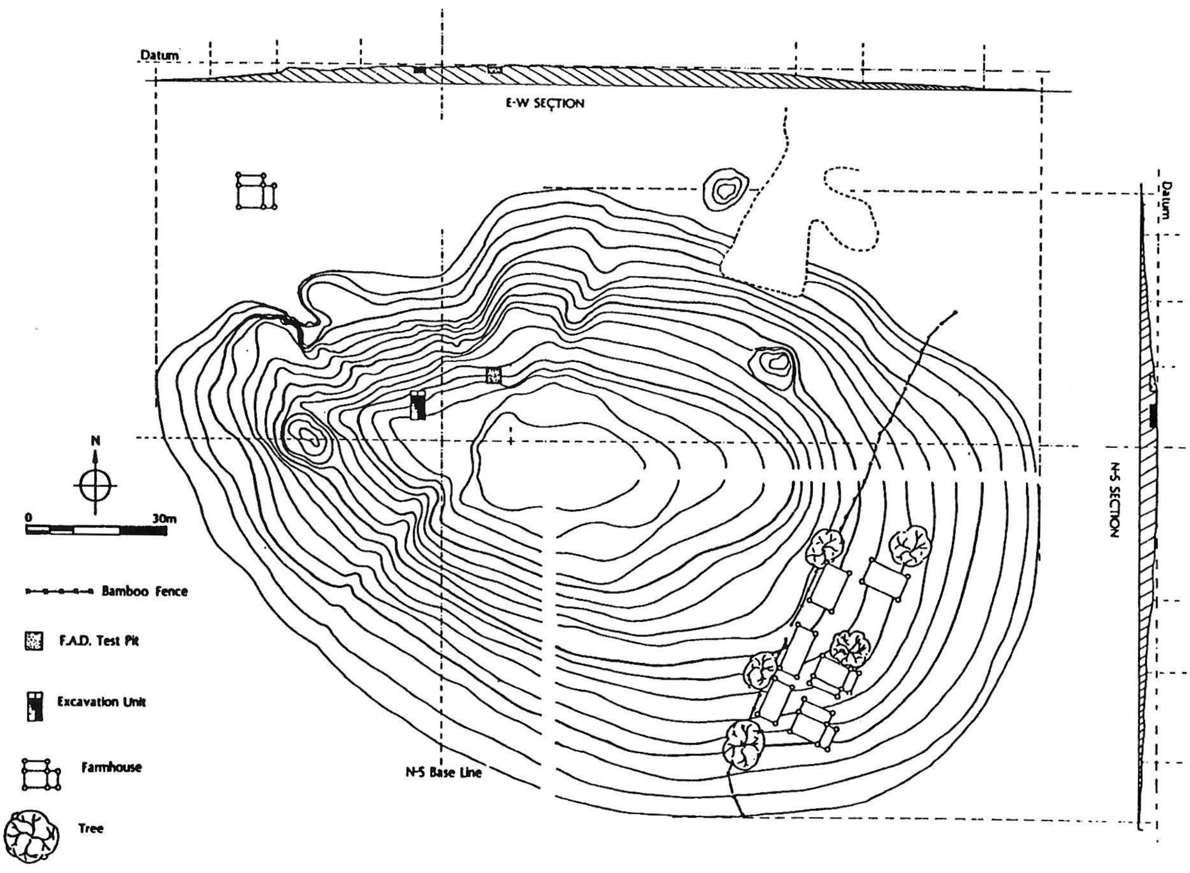

Fig 2 Plan of the Non Phrik site.

ferrite grains with thin films of carbide at the boundaries and a fine precipitate within the grains. Hence the carbon content is very low, perhaps 0.01%. The straight lines crossing the grains are twins, probably resulting from deformation during extraction of the sample. Fig 7 shows a sharp boundary between the high and low carbon regions, indicating a welded junction. A string of oxide particles was observed along the weld interface.

Manufacturing Process

The axe has a relatively massive cutting head formed by welding a layer of high carbon steel onto a wrought iron core. It is likely that the core and the socket were made in one piece by beating out on the anvil a fan-shaped piece of soft iron, at a working temperature of the order 1200°C. At the narrow part of the fan the iron would be upset to thicken it and form the shape of the core. The wider part could be wrapped round a mandrel and the overlap welded to form the socket.

To provide the cutting edge a piece of high carbon steel, perhaps 2-3mm thick, was wrapped round the previously shaped core and welded on. This hyper-eutectoid steel would have to be the product of specialist steelmakers. The Indian Wootz steel may have been traded in this period (4), but a source in China is also feasible. In either case its shaping and welding required the blacksmith to have special knowledge. Welding would require a temperature of about 1200°C (light yellow heat) but between this temperature and about 850°C the high carbon steel passes through a brittle range. The favourable temperatures for final shaping would lie between 850-650°C (cherry red to blood red, when hypereutectoid steel is superplastic (5). It is therefore suggested that, after welding, the smith allowed the temperature to fall to a cherry red heat, then forged continuously for final shaping as temperature fell to, say, 750°C. This would break up the plates of pro-eutectoid carbide as they formed and also refine the austenite grain size. The presence of the fine pearlite (Fig 6) suggests that the tool was finally quenched into water from about 750°C, though the quenching rate was not sufficient to form martensite in the sample examined. The product is a very tough and shock-resistant tool which would retain a sharp cutting edge.

Artifact 2: Iron Socketed Chisel

A triangular section was cut from the side edge of the blade, as marked in Fig 1, and mounted for polishing

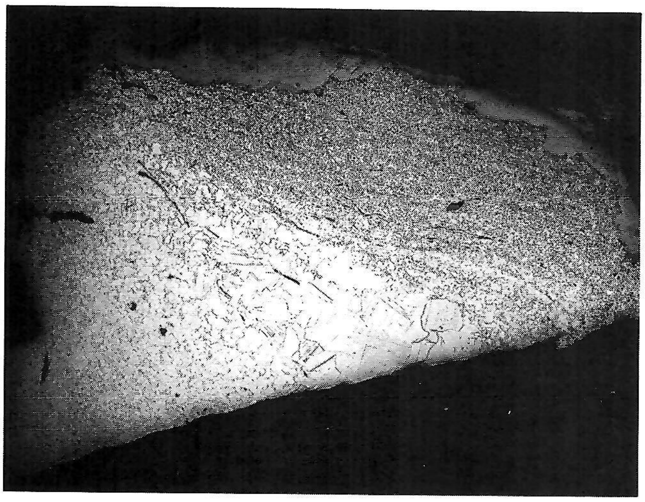

Fig 3: Full area of polished and etched sample from the axe, item 1, Mag. X17. Grey oxide visible on three sides. The straight lower side was the final break at the base of the cut. The direction left to right is through the thickness of the blade. Dark areas are rich in carbon, light area almost carbon-free.

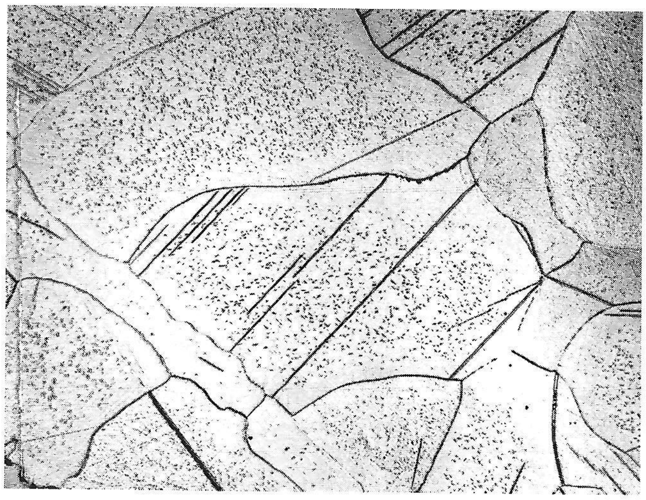

Fig 5: Coarse-grained ferrite in centre of Fig 3. Mag. X160. Straight lines crossing grains are Neumann bands, or twins, caused by cold deformation. Fine dots within the grains are precipitated particles, probably carbo-nitrides. The grain boundaries are emphasised by thin films and small particles of carbide (Fe_3C).

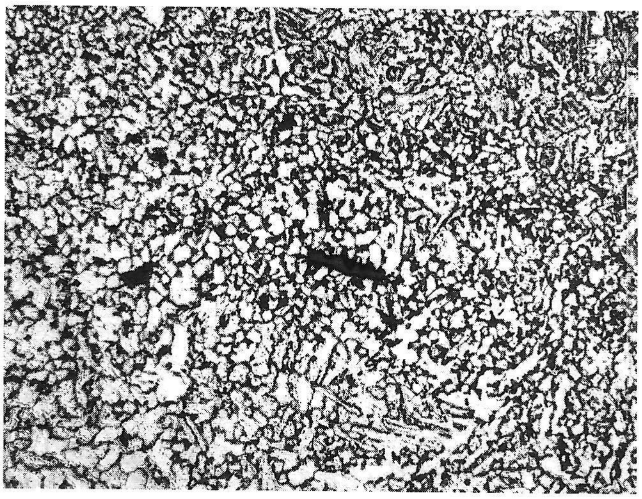

Fig 4: Fine-grained material at top right of Fig 3 Mag. X160. Light ferrite grains with dark carbide at grain boundaries. Black-outlined particle at centre is slag.

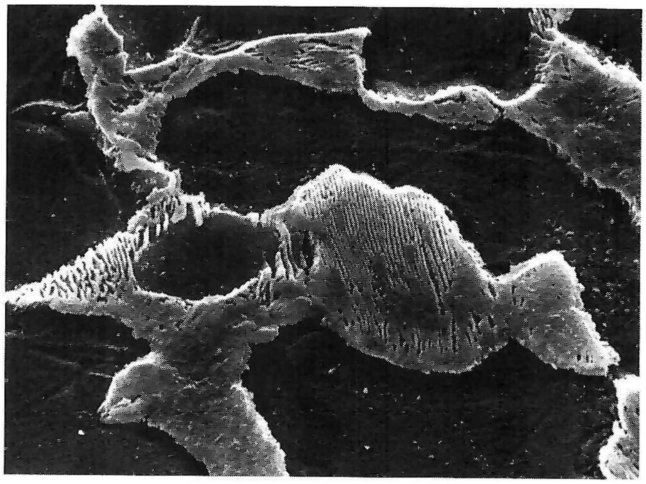

Fig 6: Details of grain boundary carbides which etch dark in Fig 4. Scanning electron micrograph. Mag. X4020. The ferrite appears dark and contains small spheroids of precipitated carbide. The light carbide (Fe_3C) areas are predominantly continuous, but contain patches of two-phase fine pearlite (Ferrite + carbide).

so that the section shown in Fig 8 is through the thickness of the blade. The irregular shape is due to loss of thickness by corrosion.

Microstructure

A laminated semi-circular pattern is readily visible in Fig 8 and seen in more detail in Fig 9, where ferrite grain boundaries are visible, with dark carbide-rich particles and black, mostly elongated, stringers of slag. Fig 10 shows that most of the ferrite grain boundaries are emphasised by films of carbide (Fe_3C), and pearlite appears as dark grey angular areas. The black rounded areas are slag. The features described are typical of wrought iron with a very low slag content, but a less usual feature is a precipitate of very fine particles through the ferrite grains, seen as light dots in Fig 10. The low hardness, HV151, and carbon content 0.03% (Table 1) are consistent with the microstructure.

Manufacturing Process

The chisel is a simple shape made by a simple procedure. The starting material was piled wrought iron, made by hammering sponge from the smelting furnace into thin sheets, then folding and re-folding while hot and hammering them together to form a

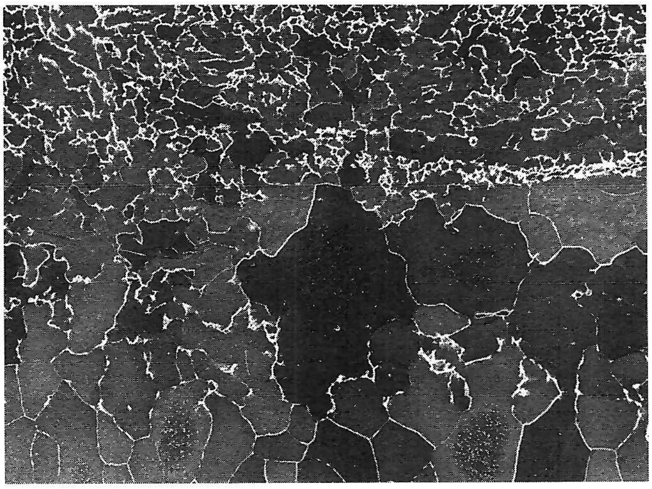

Fig 7: Showing sharp boundary between high carbon, fine-grained area and low carbon coarse-grained area (ref. Fig 3). Scanning electron micrograph. Mag. X110. The ferrite grains show much higher colour contrast than in optical micrographs.

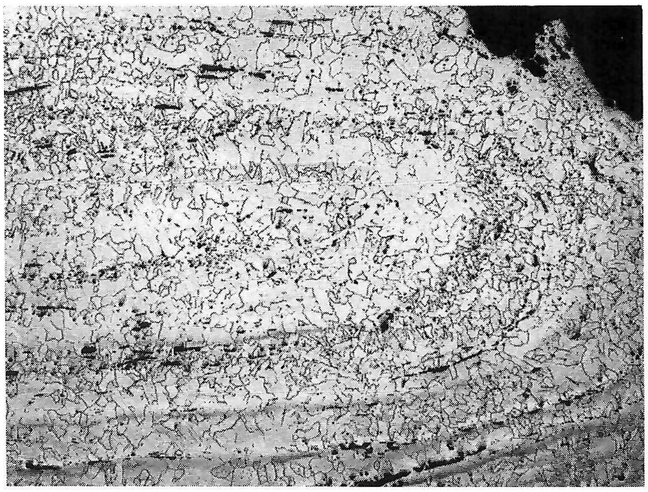

Fig 9: Enlarged area from Fig 8. Mag. X40. Shows uniform ferrite grain size. Elongated black particles are slag. Semi-circular pattern of laminations indicates folding to form the blade and the line of small dark inclusions centre left represents oxide inclusions along the weld line.

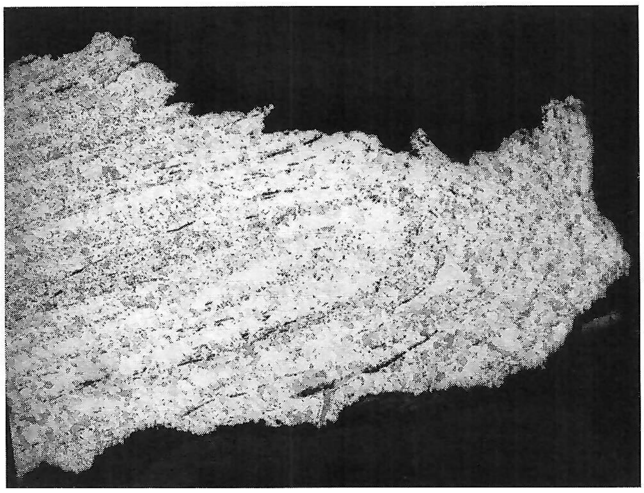

Fig 8: Full area of polished and etched sample from item 2, the chisel. Mag. X20. Lamellar appearance due to alternating lower and higher carbon layers and to elongated slag inclusions. Width (top to bottom) shows thickness of remaining metal after substantial surface corrosion.

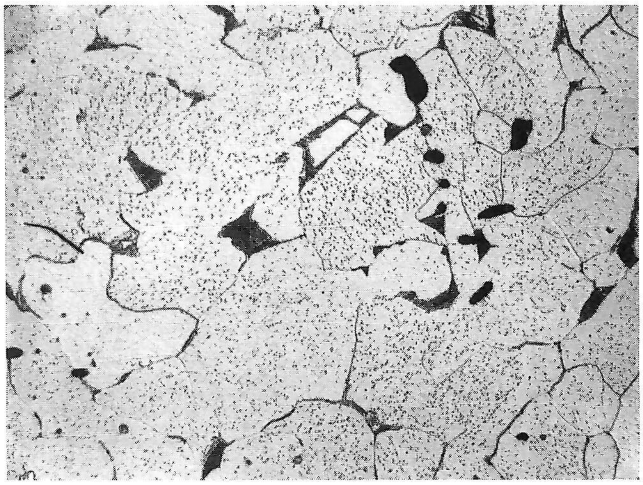

Fig 10: Representative microstructure in higher carbon laminations of Fig 9. Mag. X400. Most ferrite grain boundaries contain carbide films. Angular areas of intermediate grey at grain boundaries are pearlite colonies. Rounded darker inclusions are slag particles. Pale dots thoughout ferrite grains are precipitated particles of carbo-nitride.

shape required for sale to blacksmiths. When re-heated in the forge the surface of each sheet may be either oxidised or reduced, so that the carbon content is different in surface and centre of the sheets. When these sheets are welded together the laminated appearance results.

The swirling laminations suggest that the chisel was very simply made by folding a strip of piled wrought iron along its longitudinal axis and hot-forging to weld the overlap. The socket would be formed round a mandrel in the same operation, as described for the axe.

In the sample taken there is no evidence of any attempt to harden this soft metal. However, the sample was some distance from the cutting tip (Fig 1) and it is likely that the cutting tip was treated by cementation to increase its carbon content. Both the spearhead and the adze provide evidence that cementation was known and practised.

Artifact 3: Iron Socketed Spearhead

This artifact was in two pieces, with a corroded fracture well behind the point (Fig 1). The sample was taken by cutting back from the fracture surface and thus

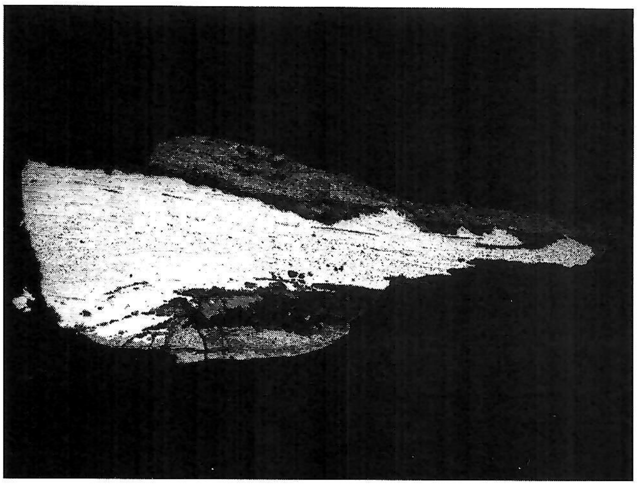

Fig 11: Full area of polished and etched sample from the spearhead, item 3. Mag. X10. Dull grey marginal areas are corrosion product. The section is through the thickness of the blade. The right-hand end, thinned by corrosion, was adjacent to the fracture surface in Fig. 1.

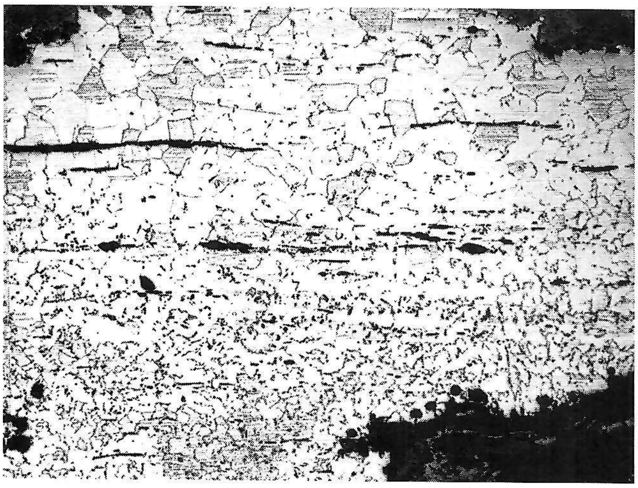

Fig 12: Section near thinner end of Fig 11. Mag. X80. The upper part shows coarse ferrite grains (ASTM 2-3), low in carbon, which were near the outer surface visible in Fig 1. The lower part shows fine ferrite grains (ASTM 7) and higher carbon content. These formed a central layer through the thickness of the spearhead. Black, elongated particles are slag.

represents the structure in the centre of the blade and through its thickness. In Fig 11 the thin end is adjacent to the fracture and the tapered appearance is due to the heavier corrosion at the fracture surface. Dull grey corrosion products are visible in the photograph.

Microstructure

In Fig 11 laminations are evident parallel to the length of the spearhead and emphasised by dark-etching stringers of slag, which show the direction of working. A central layer which is slightly darker, representing higher carbon content, is sandwiched between lighter-

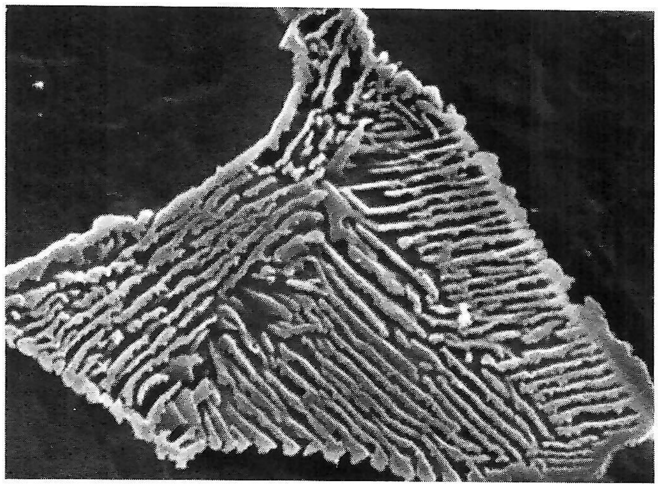

Fig 13: Scanning electron micrograph. Mag. X12,400. Enlargement of one of the small dark carbide-rich particles in lower part of Fig 11. The alternate lamellae of white carbide and dark ferrite form the constituent called pearlite.

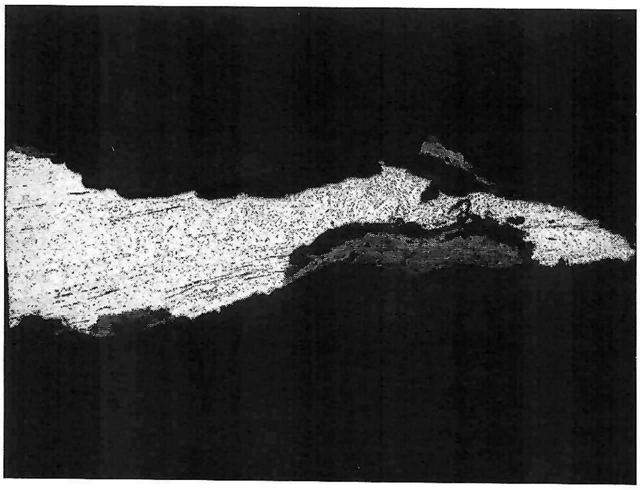

Fig 14: Full area of polished and etched sample from the adze, item 4. The sample shows a cross-section through the thickness of the cutting edge of the adze, where corrosion was severe. Dull grey areas are corrosion product. Right hand end is nearest to blade edge. Mag. X10.

etching low carbon layers which are adjacent to the outer surfaces of the spearhead. This can be seen more clearly in Fig 12, which excludes the lower part of Fig 11. The outer, light-etching, layer comprises coarse-grained ferrite (ASTM 2-3) with negligible carbon content. The central layer is fine-grained (ASTM 7) and contains a substantially higher carbon content of 0.13% (Table 1). Fig 13 shows one of the dark carbide-rich spots in Fig 11 at high magnification.

Manufacturing Method

The spearhead appears to have been made in a sandwich construction, commencing with a relatively high carbon strip of the iron, lying parallel to the top surface seen in Fig 1. This was sandwiched between strips of soft, low carbon iron and the sandwich forge welded and shaped. The socket was probably formed by making one of the soft iron strips twice as long as the other, wrapping the extended portion around a mandrel and welding the overlap.

The use of a higher carbon core ensures that the point of the spear can always be resharpened to give a relatively hard, sharp point, while the soft outer layers are more easily ground to remove the bulk of the metal required for sharpening. The higher carbon central strip would have been prepared by the smith by cementation, ie by burying a thin strip of low carbon iron about 1mm thick in a charcoal fire with limited air access to give a reducing atmosphere, rich in carbon monoxide.

Artifact 4: Iron Socketed Adze

The metallographic sample (Fig 14) was cut by hacksaw from the cutting edge of the tool (Fig 1) and mounted to show the structure through its thickness, ie a section normal to the plane of Fig 1. There was severe thinning by corrosion at this point, and some dull grey corrosion product is visible in the photograph.

Microstructure

Fig 15 shows an enlarged area from the centre of Fig 14 and Fig 16 shows the right hand end, which is all that remains of the cutting tip. Fig 15 shows a typical wrought iron microstructure with fairly fine ferrite grains and elongated stringers of slag. There is a progressive increase in carbon content from 0.05% average in Fig 15 to 0.2% at the lower edge of Fig 16 where a relatively high proportion of the dark, carbide-rich particles can be seen. This progressive carbon gradient is typical of diffusion of carbon from the surface and it can be assumed that the original cutting edge, mostly removed by corrosion, contained a carbon content considerably higher than 0.2%.

Fig 17 is a scanning electron micrograph showing detail in an area from Fig 16 identifiable by the slag stringers. In the the usual reversal of contrast the ferrite grains appear black to grey and the carbide-rich particles along the grain boundaries are white. In Fig 18 a few of these carbide-rich particles are enlarged to reveal a plate-like mixture of carbide and ferrite identified as bainite. Another area in Fig 19 shows a slag particle exhibiting two phases, carbide films at the grain boundaries, some patches of very fine pearlite and a uniform precipitate through the grains.

Method of Manufacture

The adze is a relatively massive object which would be subject to shock loading in service, so that the tough wrought iron is a very suitable material for the body of the tool, though too soft to retain a good cutting edge. Hence special treatment has been applied to provide a good cutting edge.

Wrought iron would have been available on the market, possibly in small bars which could be readily welded together and shaped on the forge and anvil. The bars would contain parallel slag stringers along their length and the straight, parallel stringers in Fig 14 and 17 suggest that the solid body of the adze was formed by simply hammering a hot bar or bars into the desired shape. The socket would be formed integrally with the body as described for the other artifacts. Fig 15 is probably representative of the microstructure of body and socket.

It seems clear that the cutting edge was then hardened by carburising (cementation). For this purpose the body of the tool would be masked by coating it with clay. The exposed edge would then be immersed in a charcoal fire which is maintained in a reducing condition so that carbon from the combustion gases can diffuse into the metal.

There is an apparent anomaly in the microstructure in that the ferrite grains are equi-axed, which is indicative of a slow cooling rate, whereas the colonies of bainite and pearlite in the grain boundaries could only form by very fast cooling, eg quenching in water. This combination could result if, after carburizing at perhaps 1200°C, the tool was cooled slowly to, say, 750°C, when most of the austenite would be transformed to ferrite. Fast quenching into water from this temperature could then cause the small amount of remaining austenite to transform to a mixture of bainite and fine pearlite, and perhaps martensite in the thin edge which has been lost by corrosion.

During this final quench the solubility of carbon (and nitrogen) in the ferrite would fall, but both elements could be expected to remain in supersaturated solid solution. The presence of a fine precipitate (Fig 19) suggests that quenching was followed by a brief low temperature tempering treatment, eg at 200-300°C, which would permit the excess carbon and nitrogen to diffuse out of solution and form carbonitrides. Although this treatment would scarcely change the properties of the microstructures actually observed, it would greatly improve the toughness of any martensite that may have been formed in the original sharp edge.

Discussion

With the exception of the axe, the tools were formed by conventional blacksmithing methods that were common to Europe, the Middle East and the Indian sub-continent since the early Iron Ages (6), but the blacksmithing was of high quality and the microstructure of each tool is adapted to its application. Interest attaches to the method used to provide a durable cutting edge in each case. Hardening the edge demands, firstly, that the carbon content of the metal be increased and this must be followed by a suitable heat treatment.

It is evident that the smith was familiar with the concept of cementation to increase carbon content, and practised it in two different ways in making the spear and the adze and probably the chisel. However, the maximum carbon level actually observed in these three

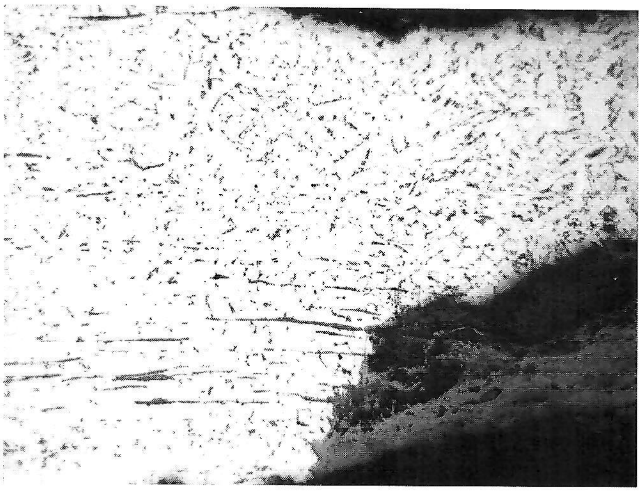

Fig 15: Section left centre of Fig 14. Mag. X45. Ferrite grains with dark bainite-pearlite colonies at grain boundaries. Elongated black stringers are slag.

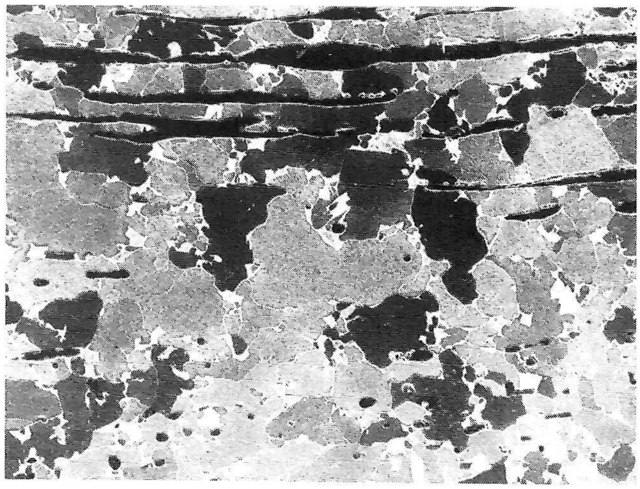

Fig 17: Scanning electron micrograph of area in Fig 16 recognisable by slag distribution. Mag. X212. Contrast is reversed relative to optical micrograph so that ferrite grains are dark, carbides white. Carbon content increasing towards foot of photograph.

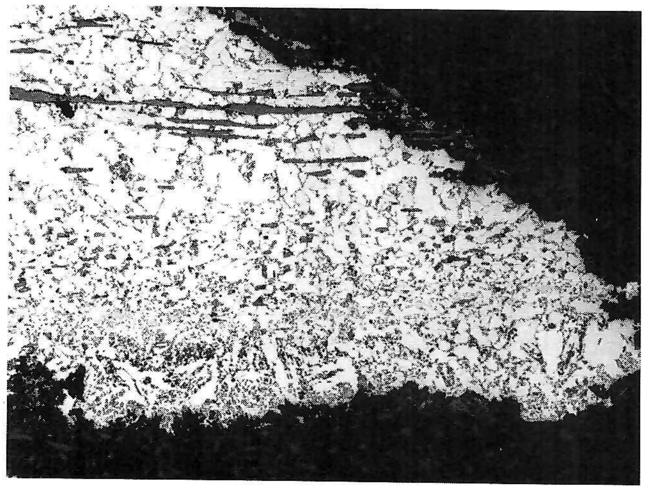

Fig 16: Section at right hand end of Fig 14. Mag. X80. Lower margin contains remnant of high carbon content (0.2%) iron which represents the original hardened cutting edge. Upper section is low carbon iron (0.05%) similar to Fig 15. Note that slag stringers extend into remnants of corrosion product top right.

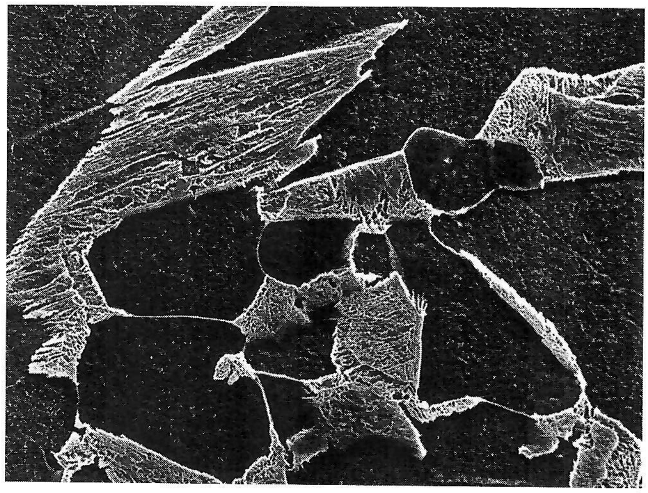

Fig 18: Grain boundary carbides in high carbon tip region of Fig 16. Mag. X2720. Ferrite dark. White carbide rich areas are mainly bainite. Sharp white lines are grain boundary carbide films.

tools was about 0.2% and, although higher levels would have been achieved in the original edges, they would still be low compared with that in a modern cutting tool, which might range above 0.8% carbon. Hence edge retention would not be very good. The smith may have been aware that longer cementation times would give him better properties, but control of constant conditions over long periods was probably not feasible. It would certainly not be feasible for the smith to produce the high carbon content of about 1.5% observed in the axehead (Artifact 1). This high carbon layer must have been produced separately in a crucible process, which may have been the Wootz steel produced in southern India and used in later centuries to produce the renowned Damascus swords. If the axe is contemporary with the other three tools its production at that period was a remarkable achievement. Although a much later date seems likely, a comparison of heat treatments applied suggests that it may have been contemporary.

The heat treatment deduced for edge hardening of the adze involved welding and shaping at about 1200°C, slow cooling to 750°C (during which shaping may have been completed), a water quench to room temperature and a final low temperature tempering treatment. The spearhead has a similar microstructure and was therefore treated similarly. The optimum treatment for the low carbon content involved would be to complete the working and quench from above about 900°C, while the steel was still fully austenitic. The edge could then transform to hard martensite and the final tempering treatment would have value to toughen the martensite. It might be expected that this sequence would be known from long experience and tradition, even though the structural changes involved were unknown. However, the treatment applied was essentially identical with that used for the very high carbon steel in the axehead. For the latter material the low temperature working, below 850°C, and final quench from about 750°C were essential for the production of a strong and tough product. If quenching to martensite were successful the final tempering would also be necessary.

It is therefore tempting to suggest that the technique for successful treatment of very high carbon (hypereutectoid) steel was known at the time that the other three artifacts were produced (300-400 AD) and was automatically applied to all steels. Although not the optimum procedure for low carbon steels it still

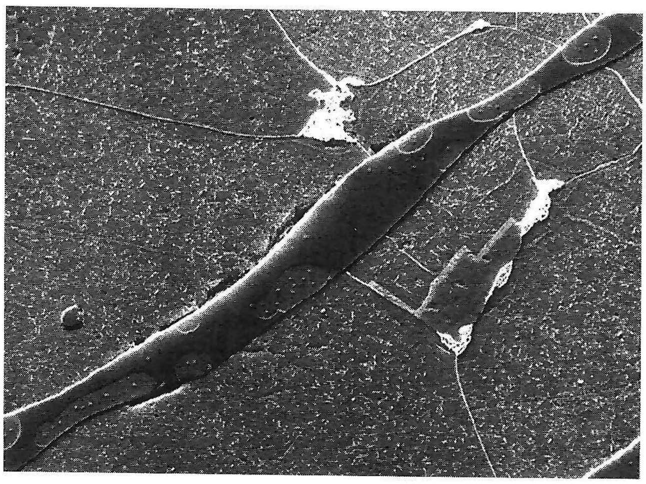

Fig 19: A small slag stringer in area of Fig 15. Mag. X1690. Shows typical two-phase structure.

produced useful results. It needs to be remembered that, although the smith could have quite accurate control of temperature through observation of temperature colours, his understanding and control of what we now know to be the effects of carbon content was necessarily very limited. Only with the development of phase diagrams at the end of the nineteenth century was this understanding developed.

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