A Metallographic Study on Iron and Steel Arrowheads from Kaman-Kalehöyük Stratum II

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ABSTRACT

This paper demonstrates preliminary results of a new technical study of iron and steel artefacts selected from established, dated contexts from Kaman-Kalehöyük Stratum II. Seven arrowheads were analysed by metallographic observation and Vickers micro-hardness testing. Through a deliberate planning of sample selection and sample preparation, this study revealed features and changes in production techniques of iron and steel arrowheads. Even with only a small number of samples, a technological change in between IIc and the later periods was confirmed. On checking the recent updates of archaeological investigation, consequently, this study suggested new viewpoints on technological aspects and cultural contexts of Kaman-Kalehöyük Stratum II.

1. INTRODUCTION

The perception of early iron or steel use in Anatolia which was mainly established by philological studies seems to have made little real progress for several decades. Anatolian ironworking is, indeed, conspicuous several early textual evidence well before the beginning of the Iron Age. For example, Assyrian merchants at Kanesh, dated to the Middle Bronze Age, included several descriptions about iron (=‘*amūtu*’) as a fascinating merchandise to them (Maxwell-Hyslop 1972: 159; Siegelová 2008: 53). Another document recording the KI.LAM festival in the Old Hittite period attests the existence of ironsmiths as LÚ^{MEŠ} AN.BAR (.DÍM.DÍM) (Kammenhuber 1996: p.215; Košak 1983/6: 126; Siegelová 2008: 53). Some other Hittites’ written records dated to the sixteenth to thirteenth century BC also seem to have partially indicated the existence of iron objects as well as changes in use and its social meaning. Despite such tangible evidence, there seems to have been little investigation about the smelting and production technologies, namely how a blacksmith would acquire iron and steel to make such objects. Transition of the term for iron in the texts does not address such questions related to the beginning of the iron and steel. This is simply because, as Siegelová states, ‘Regrettably, the technology is not elucidated in texts’ (Siegelová 2008: 54).

Scientific investigation of excavated archaeological iron and steel objects from securely dated contexts is expected to be an alternative approach towards the technological aspect of the ancient iron and steel production. Especially metallographic observation is often used for distinguishing between iron and steel and examining further material characteristics. Maddin (1982: 304) concisely explains that ‘Even with corroded objects information is still obtainable through careful examination by both optical and electron microscopic methods.’ In fact, published scientific examinations of a limited number of archaeological iron and steel objects seem to have already paved the way toward illuminating the technological nature of the ancient Near Eastern iron production in Mesopotamia, Levant, Cyprus, Western Iran and Egypt[1]. Nevertheless, it should also be emphasized that such informative approaches are not conclusive without accurate dating and sufficient archaeological contextualization of the analysed samples. Even if the blacksmiths' activities are fully reconstructed for a single object, its significance must be subjected to the basic archaeological information such as dating, cultural background and artefact types. Under what socio-cultural influence was the object produced? For what kind of artefacts was steel used and why?

From Stratum II, dated to the Iron Age, Kaman-Kalehöyük is endowed with a relatively large quantity of iron and steel finds. In regard to scientific research, Akanuma has reported hundreds of metallographic and chemical analyses of the iron and steel objects. Especially he has proposed interesting views on the development of ancient Central Anatolian steel production[2]. Steel, usually referred to an alloy of iron with up to 2% carbon, is often considered as an important material whose regular use and production could indicate utilisation and manipulation of the new material replacing bronze in the ancient times. However, steels have not necessarily superior mechanical properties to bronze. Looking at Vickers hardness, for instance, remarkable domination of steel over bronze is restricted only for the quenched steels (see Fig. 1). Physical properties of steel are dependent on complex factors such as carbon concentration, atomic structure, grain size, *etc*. which are responsive to production circumstances in technological terms. Consequently, such complexity makes it difficult to determine how the steel was achieved: was it produced by accident or design with carburization and case hardening? Detailed material characterization, therefore, necessary to discuss steels in its technological context.

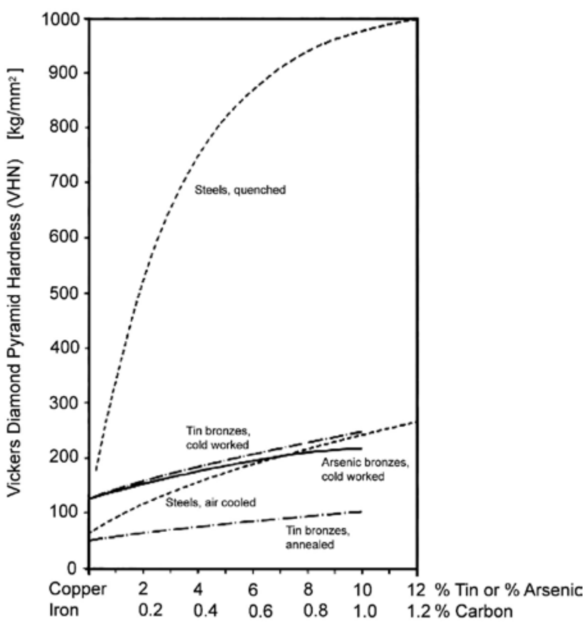

Fig. 1 Hardness-composition curves of different alloys (tin bronze, arsenic bronze, and air-cooled and quenched steels), showing effects of alloying elements' concentration and heat treatment upon Vickers micro-hardness. There seems to be no remarkable difference between air cooled steels and bronzes in their hardness readings. However, only quenched steel demonstrates extremely high hardness even if the carbon concentration is relatively low (after Williams 2002: 6).

In archaeological terms, the previous scientific studies of Kaman-Kalehöyük iron and steel objects seem to still remain several specific crucial aspects to be carefully considered. First of all, vast majority of the samples previously investigated were undefinable and fragmental objects, so it is almost impossible to follow diachronic technological changes even in a single artefact type. Inevitably, technological differences among various artefacts types has not yet been addressed. Also, it seems that little attention has been paid to the cultural contexts of chronological subdivisions in the Iron Age Stratum II. Iron Age levels at Kaman-Kalehöyük span quite a long period, about 1100 years, and divided into four subdivisions: IId, IIc, IIb, and IIa (Omura 2004: 114-134). Needless to say, this stratum contains diversity of cultural phases from the Early Iron Age even into the Hellenistic period. Furthermore, the stratigraphic and artefact studies on the Iron Age levels at Kaman-Kalehöyük has been recently updated with much more detailed stratigraphic information of cultural sequence (see Matsumura 2005). Therefore, it must be obvious that we need to investigate our understanding of Iron Age iron technology better into such comprehensive chronological and cultural contexts.

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[1] see the following publication for Mesopotamia: Curtis *et al*. 1979; Pleiner 1979, Levant: Muhly *et al*. 1977; Pigott *et al*. 1982, Cyprus: Maddin 1982, Western Iran: Maxwell-Hyslop and Hodges 1966; Pigott 1980, and Egypt: Williams and Maxwell-Hyslop 1976.

[2] see papers in previous AAS contributed by Akanuma in 1993, 1995, 1997-2004, 2006, and by Akanuma and Sasaki in 1996.

Table 1 Locus information for examined seven iron and steel arrowheads from Kaman-Kalehöyük Stratum II.

YNo.	Artefact type	Building level	Sector	Grid	Provisional layer	Date of excavation
87001168	Arrowhead	IIa3-b	N-III	XLI-54	㉔	870727
87001166	Arrowhead	IIa5-1	N-V	XXXVI-54	㉒	870624
88001437	Arrowhead	IIa7(1)	N-VI	XXXIV-54	㉛	880630
87001163	Arrowhead	IIb2(1)	N-V	XXXVII-54	㊲ - ⓒ	870820
89002178	Arrowhead	IIc2or3	N-VII	XXXIII-55	⑦ - ⓐ	890831
89002179	Arrowhead	IIc2or3	N-VII	XXXIII-54	⑧ - ⓐ	890908
89002187	Arrowhead	IIc3-3	N-VI	XXXIX-54	㊻	890829

This paper demonstrates preliminary results of a new metallographic study on iron and steel artefacts selected from established, dated contexts from Kaman-Kalehöyük Stratum II. This study aims at illuminating diachronic technological changes of ironworking in Stratum II, especially focussing on the following research questions:

1) Is it possible to find any evidence indicating steel production with carburization, case hardening, quenching and any other heat treatment techniques employed in the Iron Age?
2) How do we characterise the technological development of iron and steel production in light of cultural sequences at Kaman-Kalehöyük?

In order to investigate production techniques on the basis of the recent, more detailed archaeological interpretation of Stratum II, samples in a single artefact type 'arrowhead' with different chronological contexts have been selected for the technical examination.

Table 2 Approximate dating, building levels at Kaman-Kalehöyük Stratum II attested by pottery and stratigraphic analyses (referred to Matsumura 2005: 561)

Date	Building level	Sample
ca. 100		
	IIa1- IIa2	
ca. 300 ca. 550	IIa3 - IIa5	YNo.87001168 YNo.87001166
ca. 700	IIa6 - IIa7	YNo.88001437
	IIb	YNo.87001163
	IIc1	
ca. 800	IIa2 - IIa3	YNo.89002178 YNo.89002179 YNo.89002187
ca. 900	IId1 - IId3	
ca. 1200	IId4 - IId6 (III12 - III13)	

2. SAMPLE AND PREPARATORY PROCESS

In this study, a group of arrowheads have been selected for the investigation. Of thousands of iron and steel artefacts excavated from Kaman-Kalehöyük, the majority are, in reality, fragments. In addition, even if a group of objects can be identified as a single artefact type, they may not necessarily be distributed over a long period of time (*e.g.* from IId to the latest building level of IIa). It is also important to suppose that the majority of such a group of prospective samples do not retain metallic structures any longer. A preliminary survey, therefore, was undertaken to select appropriate artefacts for the later investigations. Through a deliberate statistical survey and preparatory assessments including x-radiography, seven arrowheads listed in Table 1 were selected as initial samples for this project. Chronologically they are able to be contextualized from around 900 to 300 BCE as shown in Table 2.

Preparatory Observation

Documentation through careful visual observation is an essential step for metallographic observation. Excavated iron and steel artefacts are often morphologically elusive because of massive corrosion products and soils over the surface. Close visual observation and investigative conservation cleaning sometimes help to find morphological features and important materials such as organic remains (wood, textile, *etc*.), and other metals (copper, silver, gold,

Table 3 X-ray cabinet working condition (for radiography)

Instrument	Todd Research Ltd. X-ray Inspection Cabinet
Accelerating voltage	80 kV
Filament current	3 mA
Exposure time	90 sec.
Filter	0.2 mm Aluminum plate

etc.) which provide important aspects to consider the usage or, in some cases, the social value of the artefact. Documentation is also important to deal with contradiction between analytical requirement and conservation of the object. In order to obtain good photomicrography in high resolutions, preparatory sample processing is generally thought to be inevitable even though it usually requires sectioning of archaeological finds. In addition, large sample sections across a complete object possibly provide useful evidence for technological interpretation (*e.g*. a gradient pattern in steel sometimes indicates evidence of carburization or case hardening). There is no perfect resolution, but documentation is useful to leave records of material condition.

In this study, the samples were photographed with appropriate scales and then illustrated through careful visual observation. Then, they were examined with x-ray radiography. The operating conditions of the x-ray cabinet were listed in Table 3. Figure 2 shows the photographs of seven arrowheads arranged in chronological order from the later object to the earlier object (A to G). Just through the visual observation, the majorities of the arrowheads seemed to be made up of simple forms with roughly pointed heads and slender shafts, except for an arrowhead YNo.87001163 which seems to be in double-blade style. On the other hand, the radiographic images (Fig. 3) provided much clearer profile of the other six arrowheads, YNo.87001168, YNo.87001166, YNo.88001437, YNo.89002178, YNo.89002179 and YNo.89002187.

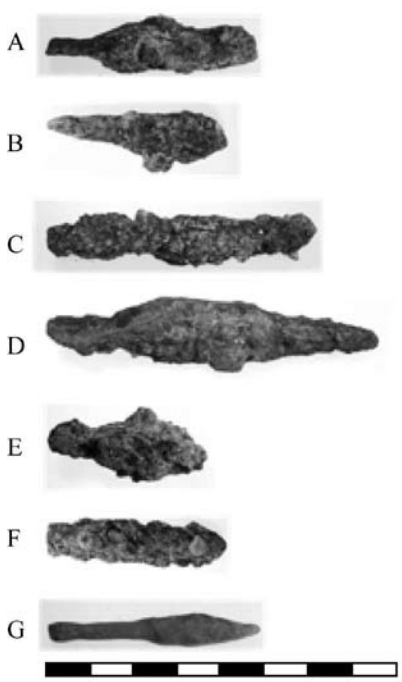


Fig. 2 Photographs of examined iron and steel arrowheads from Kaman-Kalehöyük Stratum II. A: YNo.87001168 from the IIa third building level, B: YNo.87001166 from the IIa fifth building level, C: YNo.88001437 from the IIa seventh building level, D: YNo.87001163 form the IIb second building level, E: YNo. 89002178 and F: YNo. 89002179: from the IIc second to third building level, G: YNo.89002187 from the IIc third building level.

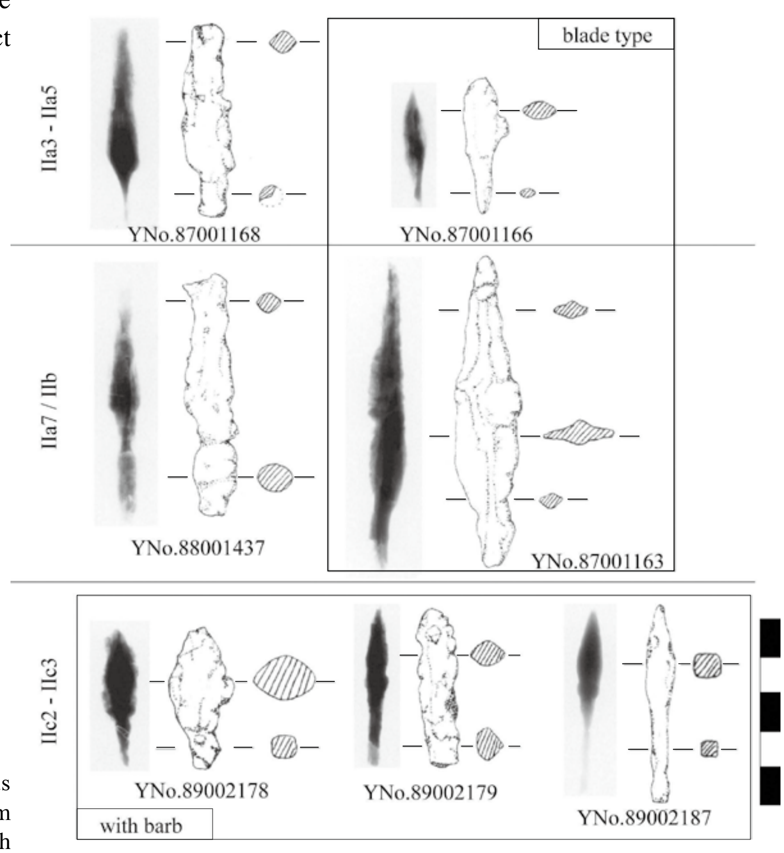


Fig. 3 Drawings (left) and x-ray images (right) of seven arrowheads. Darker shadow on the x-ray images represents dense structure or thicker parts of each sample. It sometimes provides useful information to find areas where metallic structure possibly remains.

Table 4 Summary of metallographic examination: ferrous structure and results of Vickers micro-hardness testing.

YNo.	Building level	Type of ferrous material	Area near the core		Area near the surface	
			Structure	Hardness/Hv	Structure	Hardness/Hv
87001166	IIa5-1	Low carbon steel	F (+ P)	110	-	-
87001163	IIb2(1)	Low carbon steel	F (+ P)	105	-	-
87001168	IIa3-b	Medium carbon steel	P	264	P	308
88001437	IIa7(1)	Very low carbon steel (Iron?)	F (+ P)	152	F (+ P)	163
89002178	IIc2or3	Low carbon steel (carburized)	F (+ P)	98.7	P + Fang	171
89002179	IIc2or3	Low carbon steel (carburized)	F (+ P)	98.4	P + Fang	171
89002187	IIc3-3	High Carbon Steel	P + C	189	P + C	256

F: Ferrite, Fang: Ferrite in angular shape, P: Pearlite, C: Cementite

3. METHODOLOGY

Sample Processing

Through careful observations of the x-ray images (Fig. 3), an entire or a half cross section of each sample was taken with a diamond impregnated cut-off wheel from the area in which dense structure was observed. Consequently, all sediments were taken from around the middle of the head not from the thiner shaft of each arrowhead (Fig. 4). Then each section was embedded in acryl resin and polished with wet emery papers (#120/ #320/ #600/ #1200), and finished with diamond powders (9 μm/ 3 μm/ 1 μm/ 0.25 μm) on a rotating polisher. Finally, the metallic surfaces were etched with 2% nital: a solution of nitric acid concentrate (conc. HNO_3) in ethanol (C_2H_5OH).

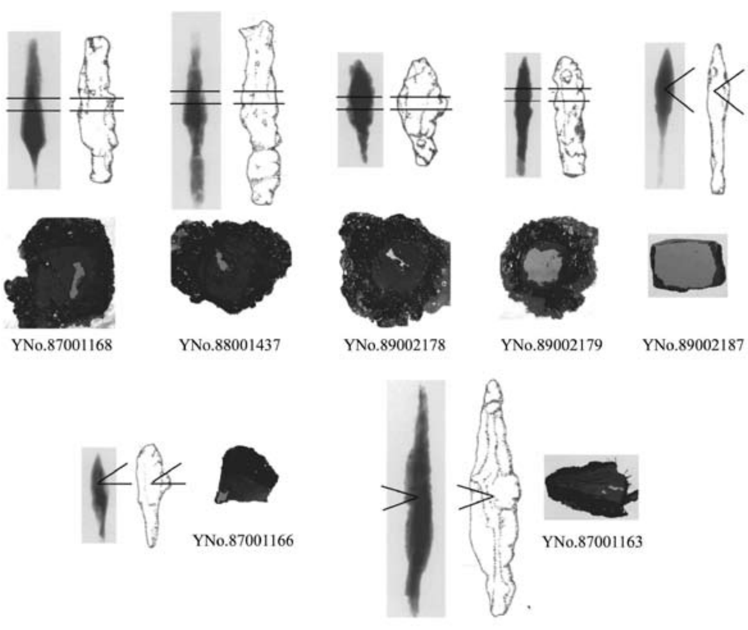


Fig. 4 Sampling points and photographs of cut-off sections (a whole section: YNo.87001168, YNo.88001437, YNo.89002178, and YNo.89002179; a half section: YNo.89002187 and YNo.87001163; a quarter section: YNo.87001166).

Metallography

The analytical method employed in this study was a standard process for metallography with microscopic observation and micro-hardness testing[3]. Samples were firstly examined under optical microscope and distributions of ferric structures were recorded carefully with magnification of x 50, x 100, x 200, and x 500. Then micro-hardness was measured by Vickers micro-hardness tester with 100 gramme load. Micro-hardness testing is a effective physical examination which often used to reinforce, revise, or interpret the information obtained by metallography (Gilmour 2000: p.476). In this study, it played an important role for the interpretation of ferrous structures and comparisons to micro-hardness data for copper alloy.

4. STYLISTIC OBSERVATION

While typological studies on metal arrowheads from Kaman-Kalehöyük have already been published (see Yukishima 1998), conclusive archaeological explanation has not been given for the typology of the iron and steel

[3] in point of fact, some other analytical methods such as XRF and SEM-EDS were also used but they were not to be dealt with in this preliminary report.

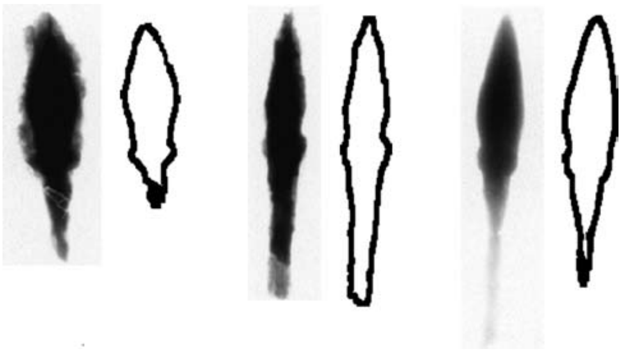


Fig. 5 Illustration of x-ray profiles of three arrowheads from IIc period. The small arrows point out the barb-like structure of each object.

arrowheads. This study, also, can make only preliminary remarks on the stylistic characteristics of a restricted number of samples. The preparatory observation illuminated that the arrowheads could be divided into two groups: two arrowheads were made of single or double blade but the others were not. Of the latter three arrowheads seemed to have protuberance on their shaft which might be barbs of the arrowheads (Fig. 5). A portrait of each sample is described as follows.

Arrowheads with blade edges

Single-blade

YNo.87001166: A relatively small arrowhead with 4.3 cm in length unearthed from the fifth building level of IIa period which is the early phase of Achaemenid and Lydian cultural influence at Kaman-Kalehöyük. A flat and roundish form seemed to be a unique feature of this sample. The x-ray revealed that the profile of the leaf-shaped head and a small part of the shaft (Fig. 3). The head was well-proportioned but seemed to be very small; the length of the head can be estimated about 2 cm.

Double-blade

YNo.87001163: An arrowhead with 7.8 cm in length unearthed from the second building level of IIb period which belongs to the Phrygian cultural phase. As mentioned above, this arrowhead kept morphological characteristics of double-blade style. There have been some typological studies on bronze and iron arrowheads from Kaman-Kalehöyük, though all the double-blade arrowheads already investigated were made of bronze (see Yukishima 1998). Further typological investigation must be necessary to mention about chronological and cultural background of YNo.87001163.

Arrowheads without blade edges

YNo.87001168: An arrowhead with 4.8 cm in length unearthed from the third building level of IIa period. According to the recent chronological studies, this building level is thought to belong to the later phase of Achaemenid and Lydian cultural influence at Kaman-Kalehöyük. The x-ray reflected a simple and linear outline of the artefact that were totally covered up by thick corrosion products (Fig. 3). As far as the radiograph shows, this arrowhead retains the whole part of the head and a part of the shaft though mostly corroded. Fig. 4 shows a cutaway section of the each arrowhead. The distribution of a dense corrosion layer (a medium dark area around the white metallic remains on Fig. 4 YNo.87001168) signifies a squarish shape of the original cross section.

YNo.88001437: An arrowhead with 6.2 cm in length unearthed from the seventh building level of IIa period in which Phrygian cultural influence has been prominently observed. The x-ray illuminated the outlines of the head and a part of the shaft (Fig. 3). While the head had a linear forms similar to YNo.87001168, YNo.88001437 showed a longer shaft with something swelling at the middle. Inferred from the formation of a dark dense corrosion on the cutaway section (Fig. 4 YNo.88001437), the original cross section of YNo.88001437 was also thought to be square in shape .

Three arrowheads with barb-like decoration

YNo.89002178: An arrowhead with a round cross section (Fig. 4 YNo.89002178): about 3.6 cm in length, unearthed from a locus which belongs to the second or third building level of IIc period. This period is thought to be under the influence of south-eastern cultural tradition. Relatively thick body had seemed to be a remarkable characteristics at first sight but the x-ray image (Fig. 3) revealed that it had a slender profile under massive corrosion products. Moreover, it became clear that YNo.89002178 has a small decoration which seems to be a barb of the arrowhead. The similar feature was also observable on the x-ray images of other two arrowheads from IIc period (Fig. 5).

YNo.89002179: An arrowhead about 4.0 cm in length with a rounded rectangular cross section unearthed from a locus belonging to the second or third building level of IIc period. As is seen in YNo.89002178, the x-ray image of YNo.89002179 also disclosed the decorative part around the area between shaft and head (Fig. 5). Original cross section of YNo.89002179 seemed to be angular rather than round in shape (a medium dark area on Fig. 4 YN0.89002179).

YNo.89002187: An arrowhead about 4.9 cm in length unearthed from the floor of an architecture remain named Room 38 which belongs to the third building level of IIc period. A very thin and dense corrosion product covered over the surface. Only through the visual observation, this arrowhead seemed to be made up of simple ridges and smooth surface and, indeed, a cutaway section of the head seemed rectangular (Fig. 4 YNo.89002187). However, the x-ray revealed an elaborate form under the smooth corrosion. This arrowhead also had a barb-like protuberance between the head and shaft, just same as the former two IIc arrowheads had (Fig. 5).

5. RESULTS AND DISCUSSIONS

A variety of metallographic structure was observed on the sampled sections. In accordance with the carbon concentrations estimated by the metallographic structure, most of the samples were categorised as hypo-eutectoid steel, yet a hyper-eutectoid steel and an eutectoid steel were also identified. While such basic characterisation differentiated each arrowhead to some extent, it should be also important to hypothesise possible technological background, namely forging and hardening techniques adopted to the artefacts. This section discusses such technological aspects correlating to the results of stylistic characterisation and chronological contexts. First of all, results of the technical examination are described in the same order as the stylistic description already presented.

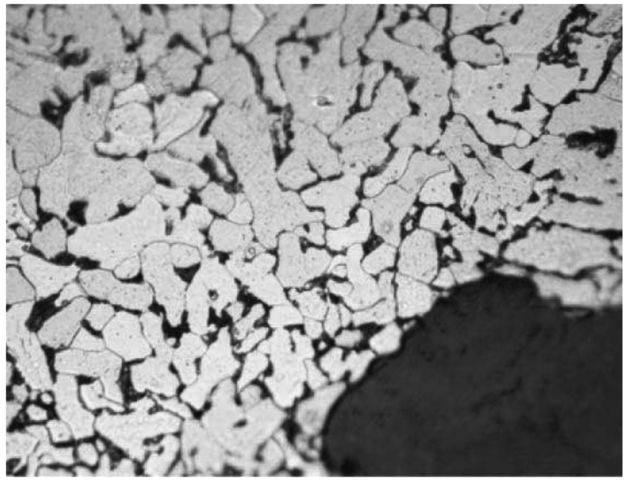


Fig. 6 YNo.87001166: a photomicrograph showing a typical structure of relatively low-carbon hypo-eutectoid steel: ferrite grains (pale) and pearlite grain boundaries (dark). Magnification x 500; etched 2% nital.

Arrowheads with blade edges

Single-blade

YNo.87001166: Metal was retained only in a limited area which is equivalent to the core of the artefact (the pale gray area on Fig. 4 YNo.87001166). The metallographic structure was basically composed of ferrite grains (white grains on Fig. 6) and grain boundaries were filled with pearlite (black particles on Fig. 6). This is a typical metallographic structure for hypo-eutectoid steel with relatively low carbon concentration. Table 4 summarised the results of metallographic observation and micro-hardness testing. According to the results of Vickers micro-hardness testing and the composition-hardness curves shown in Fig. 1, the micro-hardness reading 110 Hv for YNo.87001166 seems to be relevant to the hardness of air cooled steel with about 0.15 %C.

Double-blade

YNo.87001163: Metal was only preserved in the inner area of the artefact (the light gray strip and dot on Fig. 4 YNo.87001163). A typical structure for hypo-eutectoid steel with relatively low carbon concentration was observed. Pearlite was spread over the ferrite matrix as dark grain boundaries. Compared to YNo.87001166, relatively large amount of slag inclusions were observed. Angular glassy slag inclusions (black strips on the top

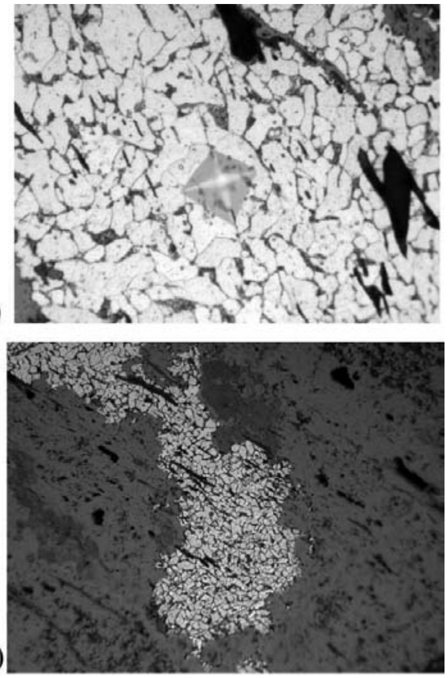
(a)
(b)


Fig 7 YNo.87001163: photomicrographs showing hypo-eutectoid structure with pale ferrite grains and dark pearlite boundaries. A gray square at the center of picture (a) is a trace of an indenter for Vickers micro-hardness test. Black angular substances on the right side of picture (a) are glassy slag inclusions. Magnification x 200 (a), x 500 (b); etched 2% nital.

and right edges of Fig. 7 (a)) were ranged along the long side of the cross section (Fig. 7 (b)). Vickers micro-hardness marked 105 Hv, which corresponds to the hardness of air cooled hypo-eutectoid steel about 0.1 %C as the composition-hardness curves shown in Fig. 1.

In metallurgical terms, metallographic structure of the blade edge is often to provide important information of heat treatment techniques such as case hardening, carburization, quenching *etc*. but, unfortunately, such observable evidence might have been lost into the outer corrosion areas of these two arrowheads.

Arrowheads without blade edges

YNo.87001168: Metallographic structure was mainly comprised of pearlite units in several different forms. Fine pearlite particles were structured into radial patterns (Fig. 8 (a)) while small lamellar pearlite was also identified partly (Fig. 8 (b)). No gradient in carbon concentration was identified. The radial pattern of fine

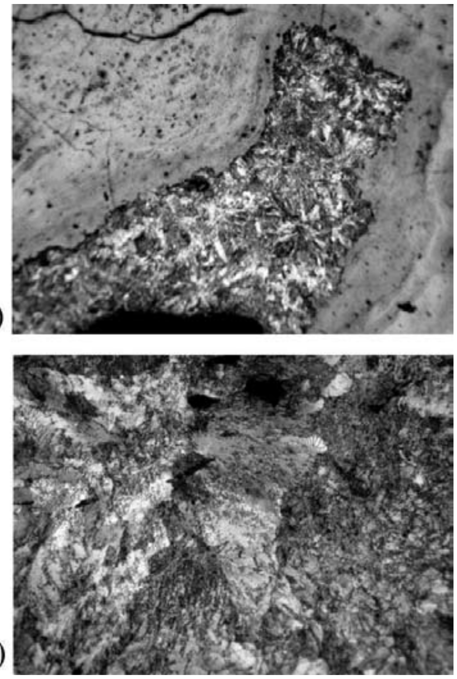
(a)
(b)


Fig 8 YNo.87001168: microstructure showing radial patterns of very fine pearlite (a). Small lamellar pearlite is also identified (b). Magnification x 50 (a), x 500 (b); etched 2% nital.

pearlite was reminiscent of the structure composed of martensite. This feature indicates that several heat treatment processes, probably including quenching, have been employed during the forging. Indeed, some standard metallographic structures acquired through isothermal transformation at around 600 ºC show good resemblance to this pearlite structure. However, exact techniques must be anticipated in connection with the observation of samples produced by experimental smithing. Focusing on physical properties, YNo.87001168 possessed relatively high hardness compered to other arrowheads. The micro-hardness of this arrowhead was 246 Hv for the core area and 308 Hv for the area near the original outline. Although the pearlite-rich structure indicated that this sample could be categorized as eutectoid steel, the micro-hardness readings are higher than those of air cooled steel but lower than the water quenched steel of 0.8 %C in Fig. 1. This should be another evidence indicating complex heat treatments that YNo.87001168 might have experienced.

YNo.88001437: Preserved metal was stretched from the core to around the original outline (the pale gray area on Fig. 4 YNo.88001437). Slag inclusions, black dots and spots seeming like spilt ink on Fig. 9 were scattered on the metal. Metallographic structure was basically formed by ferrite. Nevertheless, fine grain boundaries observed under high magnification were pearlite or possibly cementite. This is usually considered to be a typical structure for very low carbon steel. Despite such a metallographic structure, this sample had relatively high hardness. As listed in Table 4, the Vickers micro-hardness was 152 Hv for the core area and 163 Hv for the area near the original outline. They are more than fifty points higher than the hardness of air cooled carbon steels around 0.05 %C, which can be estimated on the basis of composition-hardness curves shown in Fig. 1.

Three arrowheads with barb-like decoration

YNo.89002178: Preserved metal structure was observed from the core to the area near the original outline (the pale gray area on Fig. 4 YNo.89002178). Blank spots on the sampled section (black shadows on Fig. 10 (a)) could have been caused by the fall of slag inclusions or corrosion, otherwise original defect of the metal. The metallographic structure illustrated that it is a typical hypo-eutectoid steel with relatively high carbon concentration. A fine ferrite network (white lines on Fig. 10 (b)) was surrounding pearlite colonies (dark areas on Fig. 10 (b)). A gratitude in micro-hardness readings which possibly signifies carburization was detected. The micro-hardness increased from the inner area to the outer area: 98.7 Hv for the point around the core and 171 Hv for the point close to the original outline (Fig. 10 (a)). These hardness readings are fully applicable to the hardness-composition curve of the air cooled steel with 0.3 to 0.5 %C (Fig. 1).

YNo.89002179: Metallic structure was comparatively well preserved but there were many black spots of slag inclusions spread over the cross section (Fig. 4 YNo.89002179). Metallographic structure was classified into hypo-eutectoid steel composed of ferrite and pearlite. However, the pearlite components seemed not to be distributed homogeneously. Especially in the inner area, ferrite structure dominated over pearlite (Fig. 11 (a)). This differentiates YNo.89002179 from the former arrowhead YNo.89002178. By contrast, in the outer area, colonial pearlite surrounded by angular ferrite was partly observed (Fig. 11 (b)). Micro-hardness ranged from 98.4 Hv at the center point to 171 Hv at the point near the original outline. Given this fact, YNo.89002179 is also thought to have been slightly carburized or case hardened.

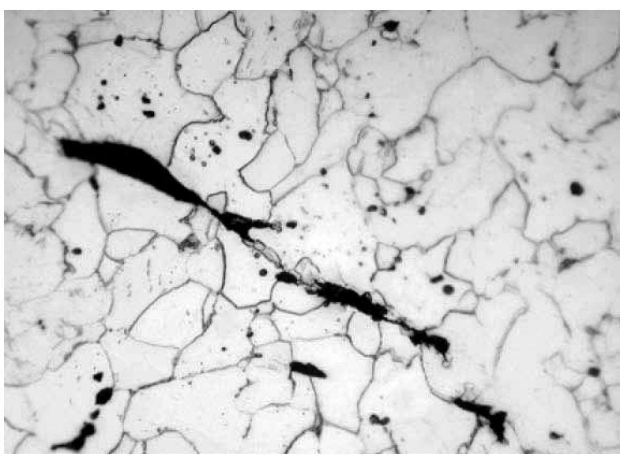


Fig. 9 YNo.88001437: microstructure showing pale ferrite grains and a needle-like boundary network of pearlite or cementite. Entrapped slags are scattered unevenly. Magnification x 500; etched 2% nital.

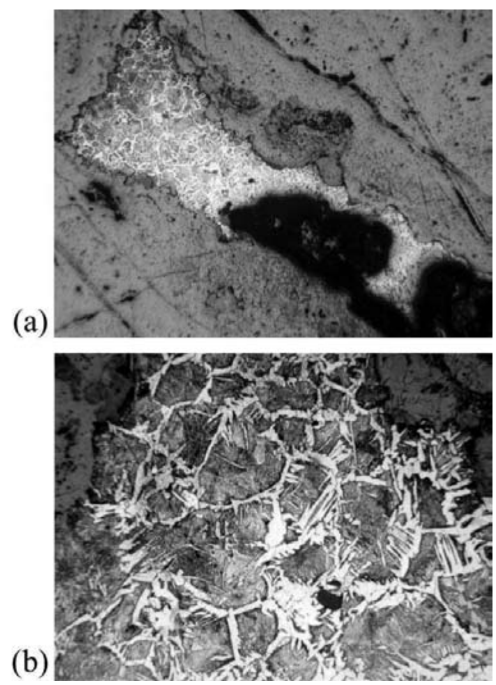


Fig. 10 YNo.89002178: photomicrographs showing a typical hypo-eutectoid structure of relatively high carbon content: a network of ferrite and dark lamellar pearlite. Magnification x 50 (a), x 200 (b); etched 2% nital.

YNo. 89002187: Metal was well preserved under the thin and stable corrosion layer (Fig. 4 YNo.89002187). Metallographic structure showed a different feature from the other arrowheads. Colonial pearlite (dark areas on Fig. 12 (a)) was surrounded by a network of needle-like cementite (white lines on Fig. 12 (a)). This is a typical structure for hyper-eutectoid steels. Although the pearlite-cementite units seemed to be homogeneously spread over the section (Fig. 12 (b)), hardness testing revealed that there was a gradual increase in micro-hardness from the core to the area near the original outline: from 189 Hv to 256 Hv. Compared to the other arrowheads, the micro-hardness readings were high, yet they were still applicable to the hardness curve of air cooled steel on Fig. 1.

Microscopic observation, thus, distinguished a variety of metallographic structure enclosed in the arrowheads. Even among the samples belonging to the same cultural period and have similar morphological features, clear sign of highly standardized production was not confirmed as similarities of metallographic structure. At the moment, it must be impossible to give further concrete interpretation about the technological varieties of early iron and steel arrowhead making, which might have derived from the differences of individual craftsmen or workshops in a community, alternatively, different geographical location of the production place. Nevertheless, technological discussion is still possible if giving attention to the other aspects.

Firstly, focusing on the micro-hardness of the non-bladed arrowheads, YNo.87001168, YNo.88001437, YNo.89002178, YNo.89002179 and YNo.89002187, the outer hardness was ranging from 163 Hv to 308 Hv. They all achieved the hardness equivalent to or higher than that of bronze. However, different hardening techniques could have been adopted. In particular, a technological change can be observed between the arrowheads from IIc period and the later arrowheads. As mentioned above, three arrowheads from IIc period, YNo.89002178, YNo.89002179 and YNo.89002187, were air cooled steel but possibly experienced some sort of carburization. Although it is still difficult to ascertain whether they were intentionally hardened or consequently hardened

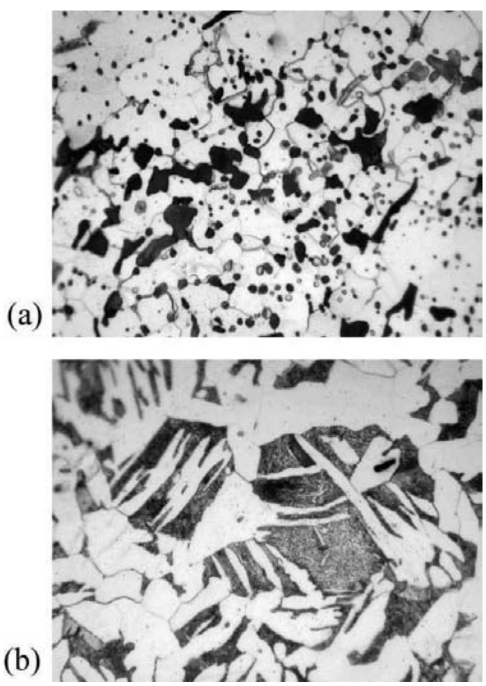


Fig. 11 YNo.89002179: photomicrographs showing heterogeneous hypo-eutectoid structure: ferrite grains with dark pearlite boundaries and black glassy slag inclusions (a), and pearlite colonies with angular ferrite (b). Magnification x 500 (a) (b); etched 2% nital.

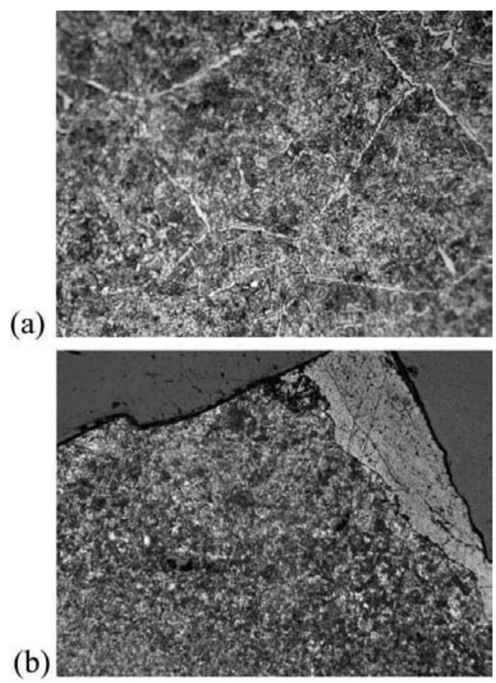


Fig 12 YNo.89002187: photomicrographs showing a typical hyper-eutectoid structure: a network of cementite and a dark pearlite background matrix. Magnification x 500 (a), x 50 (b); etched 2% nital.

after elaborate forging, at least metallography did not evince complex heat treatment process for the arrowhead production in this period. However, hardening technique seems to have transferred from such a simple process to rather complex material control in the later phases of IIa period. The metallography of the later two arrowheads, YNo.89001168 and YNo.88001437, suggested that quenching process could have been operated. Accepting the possibility of isothermal transformation for YNo.87001168 from the third building level of IIa period of under Achaemenian and Lydian cultural influence the production steps can be supposed that, firstly, the eutectoid steel was heated over 800 ºC in a furnace where austenite can exist in stable condition. Then it moved to the place around 600 ºC (*e.g.* corner of the furnace, another furnace, *etc*.) kept there for a moment and finally water quenched. Quenching technique itself might have begun in earlier times, the seventh building level of IIa where YNo.88001437 was found. Nevertheless, it should also be supposed that another hardening effect was used for achieving relatively high hardness of this object. Hardening effect of minor elements such as P or Ni must be examined by compositional analysis such as EPMA in the near future[4].

Comparison between stylistic and technological characteristics profferes another important view point. While all the arrowheads from IIc period had a similar stylistic feature with the barb-like decoration, their metallographic structure did not show close affinity. This must reflect the nature of the socio-cultural entity at that time. Again, there seemed to be no strong standardization in the technological context. On the contrary, it can be said that the barb-like decoration might not represent strong organisational control because variation in detailed forms: round, roundish and angular cross section, still exists. The stylistic feature, therefore, should be considered as not a standardised form but a trend of the cultural tradition in IIc period, just same as the painting tradition of the pottery sherds. Further investigation of iron and steel artefacts in light of stylistic and technological aspects could help characterization of the socio-cultural entity in IIc period.

Finally, it is difficult to give remarks on the structure and physical property of the bladed arrowheads, YNo.87001166 and YNo.87001163. Yet, as far as the available structure showed, there is no evidence indicating intensive carburization or case hardening (no gradients in the formation pattern of pearlite and ferrite) around the core area. Even if carburization or edge hardening was undertaken, it must have affected only the surface area which were lost into corrosion. Careful observation of ghost structure in the corrosion product must be inevitable for further investigation.

6. CONCLUSION

This paper explained technical examinations of seven iron and steel arrowheads from Stratum II at Kaman-Kalehöyük. Through a deliberate planning of sample selection as well as sample preparation, this study seems to have achieved some important information about the features and changes in production techniques of iron and steel arrowheads as follows:

1) In the earlier times of Stratum II (IIc2 and IIc3), arrowheads were produced through carburization of steel or relatively careful processing in a pyro-environment.
2) In sometime between IIc and IIb periods (around the mid of eighth century BCE), the former production style was changed. The mainstream of new arrowhead production was probably undertaken without carburization or long-time forging.
3) Quenching techniques were adopted to the arrowhead making at latest in IIa period, probably in order to obtain adequately hard material.

Thus, technological nature reflected in the arrowhead production could be seemingly divided into at least two streams in the Iron Age Stratum II. The period when earlier iron and steel making tradition was adopted can constitute the second and third building levels of IIc period at Kaman-Kalehöyük. Then, the later tradition seems to have started in IIb or early IIa period. From technological point of view, it might be still

[4] A preliminary compositional analysis has already been operated with SEM-EDS. A very low concentration of phosphorous was detected. However, even if considering the hardening effect of this phosphorous content, it is still probable that this sample has experienced rapid cooling on the basis of hardness-phosphorous content curves in Buchwald 2005: p.144.

incomprehensible whether these two different natures can be situated on a single cradle of technological development. However, it could be worth noticing that we can find similar changes around the end of IIc period in the results of other artefact studies at Kaman-Kalehöyük. For example, in light of stylistic studies of Iron Age painted potteries, painting styles as well as technological tradition changed between IIc and IIa periods. While the mainstream of painted ware in IIc period at Kaman-Kalehöyük can be categorized as Alişar IV type, it changed in IIa period to the Phrygian polychrome ware (Matsumura 2000: 126). Through technological studies of Iron Age ware, Matsumura (2000: 128) also suggested that cultural influence from southern region was thought to be observed in the pottery making tradition of IIc period, and also Phrygian cultural influence was conspicuous in the technology of IIa period. Such recent information will provide helpful suggestion for the further interpretation of the technological traditions of iron and steel production in the Iron Age.

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