

# Early Iron from Kinet Höyük, Turkey: Analysis of Objects and Evidence for Smithing

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## Keywords

Kinet Höyük, Iron Smithing, Slag, Basalt, Iron Age

## Abstract

Kinet Höyük (ancient Issos), located in Cilicia on a narrow corridor between the seacoast of the East Mediterranean and the Amanus mountains (Nur Dağları), is an archaeological site with 29 excavated occupational levels, starting from the Early Bronze Age. The successive settlements at Kinet Höyük faced many military campaigns and changed hands frequently because of its strategic position with access to shipping routes, and the availability of rich mining and forestry resources. However, iron smithing was one of the activities that changed least during the transformations occurring in the region, according to iron-related finds from different occupational levels at the site. In this paper, iron objects and smithing slags from the Iron Age period at Kinet are introduced from their metallurgical perspective. The results of metallography and SEM-EDX analysis of Iron Age steel objects are discussed in light of previous studies on iron metallurgy of the neighboring regions. Additionally, slags which are dated to the site's Neo-Assyrian phase are evaluated by petrography, ICP-MS and XRD analysis. Although all of the iron objects are fully corroded, remnant metal observations point to a variety of microstructural phases. The majority belong to medium- and high-carbon steel structures that were affected by heat treatments, i.e. normalizing, and annealing. Plentiful hammerscale were detected in thin sections of smithing slags. Basalt attachments to slags are considered to reflect the presence of basalt in the smithing hearths and other pyrotechnical settings.

## Introduction Historical and Technological Background

Cilicia has been an important location for iron metallurgy throughout its history. As one recent indication of this fact, modern mining reports still note its exception-

al richness. Together with Adana and Kayseri, the city Hatay is located in a region with Turkey's greatest concentration of iron ore beds: 24.98 % of all beds in Turkey (Cihnioğlu, et al., 1994). Historical records emphasize the importance of the region also. The iron mentioned in the famous Hittite Kizzuwatna letter from the second half of the 2<sup>nd</sup> millennium BC, must have been smelted somewhere in Cilicia (Pleiner and Bjorkman, 1974, p.283; Yalçın, 2005, p.499). Many centuries later, as can be seen from the texts concerning taxes and tributes to the Assyrian kings Tiglath-pileser III (744- 722 BC) and Sargon II (722 – 705 BC), Eastern Cilicia was not only an important source of iron-bearing deposits to smelt quality iron, but also a passageway to reach other iron sources in Anatolia (Maxwell-Hyslop, 1974, pp.151-152). Moreover, Cilician steel was one of the most popular types for trade (Pleiner, 2000, p.22).

Although archaeological evidence shows that the number of iron finds increased significantly during the latter half of the 2<sup>nd</sup> millennium, this trend is thought to have occurred because workshops at sites in the East Mediterranean and Near East increasingly dealt with bronze and iron together (Harrison, 2013; Pigott, 1989, p.67). During the 9<sup>th</sup> - 7<sup>th</sup> centuries BC, iron became the preferred material in Cilicia as can be observed from its change in lifestyle to a stage of fully-fledged iron production and use (Pleiner, 2006, p.12). The reasons for this transformation are still open to question, with possible answers being easier access to the mining sources of iron in place of copper and tin, the main components of bronze; and/or superior properties of iron/steel compared to bronze. These properties were either provided by the mastery of technological steps; which involved alloying carbon with iron (transforming it into steel) and applying thermal treatment, two techniques that make iron a harder material than bronze;<sup>1</sup> or were the natural outcome of uncontrolled smelting operations creating blooms, a mixture of iron/steel. The technological sig-

nificance of these possibilities explains why it is important to understand the material properties of early iron objects and how they were produced. Researchers have therefore focused their attention on determining the amount of carbon, the homogeneity, and the hardness of early iron objects from Anatolia, East Mediterranean and Near East.

In ancient cultures, iron had decorative, ceremonial and prestigious functions until the end of the 2<sup>nd</sup> millennium BC (McConchie, 2004, p.18). The earliest known Anatolian iron objects are a bracelet from Tilmenhöyük (Gaziantep), and especially a group from Alacahöyük in Central Anatolia. The Alacahöyük objects, which include a gold-handled dagger and two ornamental pins with golden heads were found in a royal tomb dated to the Early Bronze 3<sup>rd</sup> millennium BC. Their analysis shows that the origin of some of the Alaca iron is meteoric (Yalçın, 2008, p.177). Texts from early 2<sup>nd</sup> millennium Kültepe suggest that, for its Assyrian traders, iron was seven times more valuable than gold (Maxwell-Hyslop, 1972, p.159). Hittite texts from later in the same millennium support the high value of iron for cultic and religious settings, but numerous references also indicate that iron began to serve in manufacturing utilitarian items such as tools (Muhly, et al., 1985, p.73). Although some texts distinguish between good quality iron (steel) and bad iron, analytical studies have yet to prove which smelting and smithing factors were responsible for creating an iron type that qualified as “good” during the Late Bronze Age. Deliberate production of steel did develop during the following centuries and became common by the Middle Iron Age. For instance, metallographical studies on 9<sup>th</sup> - 7<sup>th</sup> century arrowheads from the Urartian castle at Anzaf, in eastern Anatolia, noted their wrought iron and medium carbon steel micro-structures. Slag inclusions separating different layers suggest that their heterogeneous structure was formed by forge welding (Yalçın, Belli and Maddin, 1994, pp.44-45). The analysis of contemporary arrowheads from Kaman-Kalehöyük, in Central Anatolia, noted controlled thermal processes and deliberate carburization already in the early examples, and a change to a softer forging material by the mid-8<sup>th</sup> century BC (Masubuchi, 2008, p.291). From a contemporary context, Urartian iron objects from Toprakkale were classified, on the basis of their manufacture, into three groups by Piaskowski and Wartke (1989). In the first group, represented by different types of objects including an iron ingot, the microstructure consisted of irregular areas of high carbon (0.8 %) steel mixed with pure iron. The second group is the soft one, containing only pure iron structures (ferrites). The last group was characterized by

phosphorous iron and irregularly carburized areas. According to this classification Kaman-Kalehöyük arrowheads from the mid-8<sup>th</sup> century BC fit in Piaskowski and Wartke's 2<sup>nd</sup> group.

For the same period, numerous Neo-Assyrian tablets mention iron, although none of them describes technical information about smelting or smithing iron. Nevertheless, they make frequent references to the types of iron objects and their usage. Assyrian accounts of the early 9<sup>th</sup> century suggest that iron daggers had replaced the bronze ones which were standard as earlier military equipment. Similarly, reports written by Assyrian officers of the 8<sup>th</sup> century BC from Guzana-Tell Halaf refer to 500 iron arrowheads and 5 daggers (Pleiner and Bjorkman, 1974, pp.285-287). It is likely that the thousands of iron objects discovered by excavations in western Iran at Hasanlu, including 700 arrow-points, 500 spear-points and 70 swords, were produced by blacksmiths travelling with the troops of Assurnasirpal II (883-859 BC) during the Assyrians' first military campaign into that region. Analysis of one of the Hasanlu swords concluded it was entirely made of “mild steel” so as to enhance its strength as a weapon. However, most of the other iron objects showed a mixed structure of wrought iron and mild steel (Pigott, 1989, p.76). Assyrian ironwork is also well attested from its major cities. The collection of analyzed iron objects from Room 84 of Sargon II's Palace at Khorsabad, dated to the late 8<sup>th</sup> BC, includes a hoe, an adze and two iron bars. Metallographic examination reveals that the iron bars (ingots) consisted of heterogeneously, incidentally carburized steel of good quality. The carbon content of the carburized zones surpasses eutectoid composition (0.8 %). Also present are decarburized or non-carburized areas whose main metallographic structures are equiaxed ferrites. In contrast and despite its skillful shape, the adze was made from wrought iron with poor carbon and numerous slag inclusions. The hoe contains some steel strips but is nonetheless of poor quality. No heat treatments like quenching or tempering were applied to any of these objects (Curtis, et al., 1979; Pleiner, 1979). The iron objects from Nimrud are dated to the late 7<sup>th</sup> century, from the sack of the palace. Iron bars from there, similar in shape to the ones from Sargon's palace, also share the same structure with heterogeneous heavily carburized zones. Finally, the analysis of eight Neo-Assyrian finished objects from Nimrud of various types (e.g. sickle, arrowhead, blade, armor scales etc.) noted some carburization zones observed as relics of cementite from the pearlite phases.

With the exception of commentary on the arrowheads from Anzaf castle and from Kaman-Kalehöyük, the research results on early iron objects have been

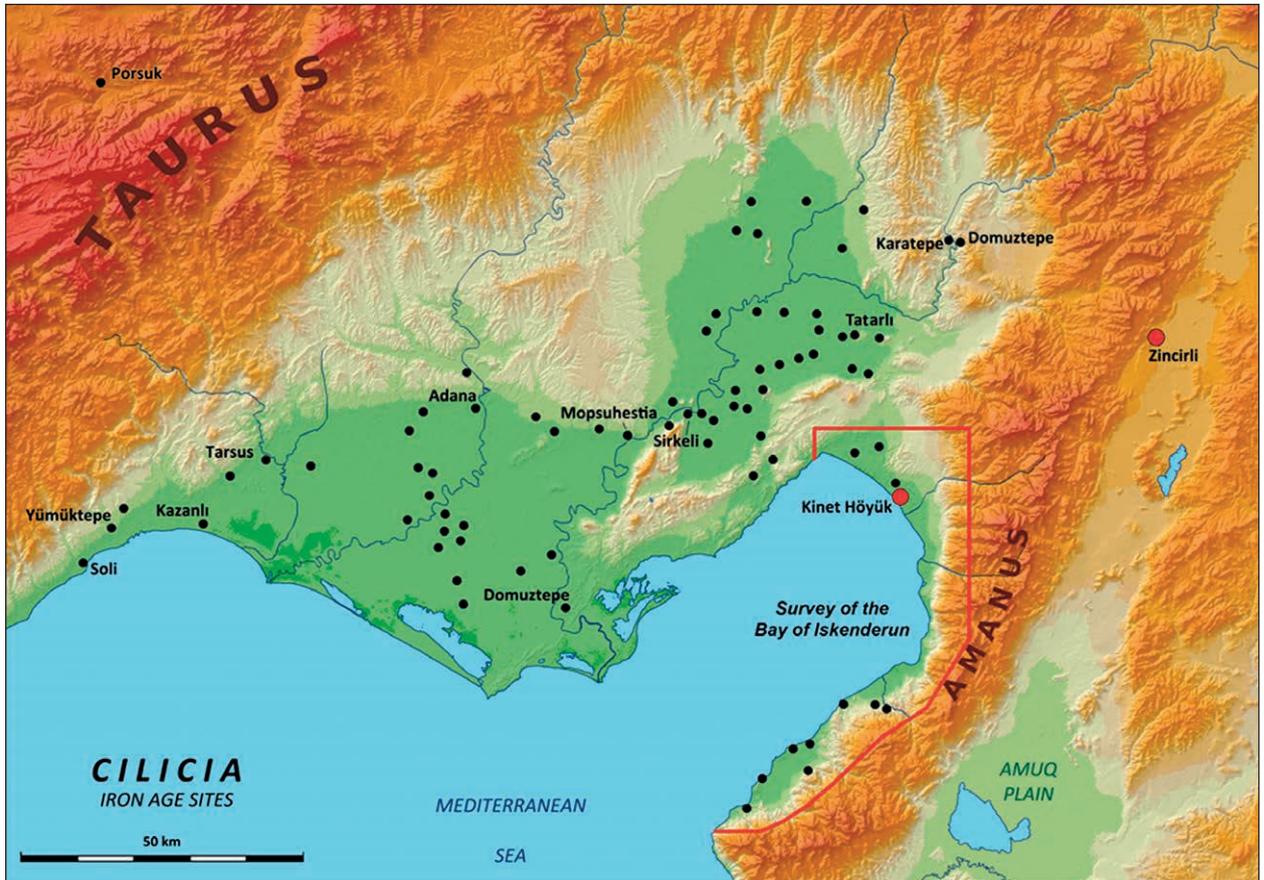


Figure 1. The location of Kinet Höyük, in the northeastern corner of the Mediterranean (courtesy G. Lehmann).

thought to show that Iron Age blacksmiths did not have good control on the quality of their steel, based on the heterogeneous structures of incidentally carburized zones. Moreover, it is stated that they did not have enough knowledge to apply thermal treatment processes like quenching, and tempering, or hot welding to make layered steel. The examination of iron objects from the Cilician port of Kinet Höyük gives an opportunity to compare previous information about Iron Age blacksmiths with manufacturing practices at this particular site. Although the Kinet sample presented here can admit to partial or inconclusive evidence, it nonetheless presents features that may be recognized as belonging to a broader metallurgical tradition.

### Archaeological Contexts and Early Iron Materials at Kinet Höyük

The ancient seaport of Kinet Höyük is situated in the northeastern most corner of the Mediterranean at the back of İskenderun Bay (Hatay, Turkey), where it provided boats with shelter from winds, and a protected access to overland routes and resources (Figure 1). Its long set-

tlement history, beginning in the Late Neolithic 6<sup>th</sup> millennium BC, can be attributed to this advantageous location, which facilitated long-distance connections along maritime circuits. Thus, despite Kinet's small size and apparent remoteness, its material culture reflected the larger economic trends and technological developments of the times. The site likewise attracted major regional powers, such as the Hittites and Assyrians, to occupy and exploit its harbor facilities.

Excavations by a Bilkent University (Ankara) project from 1992 to 2012 were carried out mainly on the 26 m-high and 3.3 ha mound, with additional soundings in the surrounding fields at its foot. They determined that by at least Early Bronze II (ca. 2900 BC), the site covered ca. 5 ha, encompassing port installations at sea level, and a citadel on the mound behind it, in antiquity as today ca. 450 m distant from the sea. This configuration and scale were maintained, with occasional interruptions, into the first centuries of the Iron Age (7<sup>th</sup> /6<sup>th</sup> c BC), after which the lower town was abandoned and occupation retreated to the mound proper for another 400 years of the Persian and Hellenistic periods. Occupation ceased ca. 75 BC, and was revived for a short time only during the Middle Ages (12<sup>th</sup> - 14<sup>th</sup> c AD).

To understand the history of iron usage, smithing and the development of iron metallurgy in Kinet Höyük during the Iron Age, iron objects and slags spanning a wide time range were selected for sampling: 4 blades (KT17086, KT16003, KT17715, KT7955), 3 iron rods (KT8548, KT13325-L, KT13325-S), and a slag assemblage consisting of 23 pieces. Initial examination of the slag assemblage determined that it fell into two groups: the first, material of basaltic origin that was subjected to heating; and the second, a group of smithing slags. The present study will discuss the 7 objects, 5 smithing slags (KT15662, KT15985, KT16287, KT16622, KT17007) and one basaltic block (KT20471), which is thought to be the wall fragment of a pyrotechnological setting i.e. a furnace.

The first evidence at Kinet for iron metallurgy occurs with the onset of the Iron Age (later 12<sup>th</sup> c BC), and continues throughout the following occupational periods. Iron was introduced during the site's only non-architectural phase, Early Iron Age Period 12 (ca. 1150/1130-1000 BC). It is represented on the mound's west side by a 1 m thick accumulation of ash and refuse, eventually cut into by storage pits and open hearths. These deposits may have resulted from seasonal use of the site, rather than year-round settlement; but they reflected much industrial activity for processing livestock and other materials. The finds include a fragment of the site's earliest iron blade (KT7955) (Figure 2). After a gap of uncertain length, the next two levels (Middle Iron Age Periods 11-10, ca. 1000-800 BC) saw a return to a built environment. Although preservation is poor, their architectural remains were clearly suited to permanent structures. The exposed sector of Period 11, again on the west side, included the sunken chambers of several furnaces: oval pits lined with mud plaster and bricks, and heavily burnt (Lehmann, 2017, p.237-238; Gates, 2013, p.104-107). KT20471, which resembles a brick with one slightly curved side and a glossy dark brown surface, was found in one of the sunken chambers (Figure 3).

The two later Middle Iron Age levels, Periods 9 and 8, were in contrast well characterized with coherent architectural plans. They were recovered in exposures on the west (Area E/H) and east (Area A/D) sides of the mound, and both produced traces of metallurgical activities. Period 9 (ca. 800-725 BC) was represented on the west side by a monumental building, and on the east by kilns and the two pieces of iron rod discussed below (KT13325-L, KT13325-S). The settlement ended in a severe fire, after which the buildings' remaining contents were dumped outside walls or into pits (Gates 2000, pp.196-198; 2004, pp.407-408). The two blade samples recovered from this post-destruction episode mark the transition into



Figure 2. The tang section of blade KT7955.



Figure 3. Cross-section of slaggy block KT20471.

the next period and are labelled here “9/8 transition” (KT17086, KT17715). However, they can be considered to belong with the samples from Period 9 proper. The destruction's late-8<sup>th</sup> century date, documented by associated ceramic finds, corresponds to the period when Cilicia came under the control of the Neo-Assyrian Empire (ca. 730 - 640 BC; Lehmann, 2016, p.323). It can be assumed that the Period 9 settlement was destroyed during military campaigns leading to the region's conquest, since the succeeding Period 8 occupation displayed an intrusive material culture of Assyrian origin.

A Neo-Assyrian affiliation for the Period 8 level at Kinet (ca. 730 - 650 BC) is recognized by a change in building orientation and masonry; an Assyrian ceramic repertoire dominated by thick-walled, undecorated vessels of utilitarian purpose; cylinder seals with Assyrian motifs and iconography; and unusual numbers of equids (Lehmann, 2016, pp.324-327; Gates, 2004, pp.406-407). On the west side, a large and irregular structure included small rooms around a cobbled court, many of them

furnished with an oven. Associated finds produced evidence for the first industrial activity which was evidently related to iron smithing. The largest selection of slags analyzed in the present study was found abandoned in the debris of these rooms and its courtyard when the building was emptied and burnt (KT15662, KT16622, KT17007). Two further pieces of slag reasonably belong to the foundation phase of this structure, rather than the preceding level (KT16287, KT15985); and the slags are supplemented by the analytical results of one finished object, an iron blade (KT16003). Together, they provide an instructive record for provincial workshop production, during a historical period when iron metallurgy became a prominent industry.

After the Assyrian episode ended in destruction, iron production continued at Kinet during its Late Iron Age Periods 7 and 6 (ca. 650 - 575 BC), from which were recovered a number of iron tools, especially blades. This century was marked by the emergence of Aegean commercial interests, manifested in the eastern Mediterranean and at Kinet by a ceramic repertoire dominated by Aegean/East Greek-style tableware. Architectural remains for the two periods combined industrial and residential functions on both sides of the mound. Area A/D again included a group of kilns and/or lime furnaces, this time dated to Period 6 (Gates, 1999, pp.261-264; 2003, pp.283-285). The sample selected to represent this production stage is an iron rod from a gravel-paved street on the mound's east side (KT8548, Period 7).

## Analytical Procedures and Results

### Metallographic and SEM Observations

The seven metal objects which were examined by metallography are fully corroded. None of these iron objects could survive in their original shapes. It is therefore not possible to determine the type and size of the blades, when tips and handles have lost their original definition. However, after sectioning and mounting, the back and cutting edges of blades can be clearly distinguished in cross-section. The iron rods differ in length from 3 to 10 centimeters and seem to be broken from longer pieces. After photographs of the objects were taken, the cross-sections were prepared by cutting with air-cooled diamond discs. To prevent overheating the samples, the cutting procedure was carried out at slow speeds with regular pauses. After mounting, grinding was done using wet silicon carbide papers with grit sizes starting from 60 up to 1200. Diamond pastes with 6, 3 and 1  $\mu\text{m}$  particle sizes were used for polishing. Since no uncorroded metal

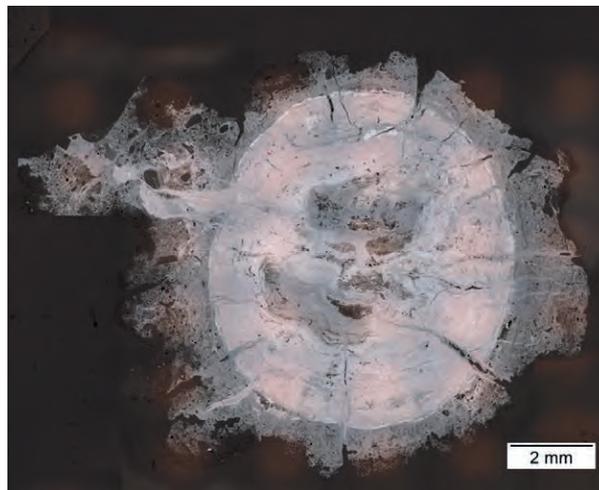


Figure 4. Cross-section of iron rod KT13325-S.

body was found during first observations with a metallography microscope, no etchant was used.

During light microscope examination of objects, micrographs were prepared of each cross-section by “stitching” them digitally. The areas evaluated as interesting were then photographed at greater magnitude.

Stitched micrographs of the rods show that these objects have elliptic or circular cross-sections with a radius measuring 10-13 millimeters including the corrosion crust. Their surfaces are coated with a corrosion crust nearly 2 mm thick, consisting of soil and corroded matter (Figure 4).

Because of the objects' heavily altered microstructure, observation mostly showed destructive corrosion and its by-products. A few areas with a lighter color were noted as candidates for the benign corrosion stage, however. These areas were mostly available dispersed at the inner parts of the objects. Observation on these less corroded areas was done by Scanning Electron Microscope (SEM) to search for partial survival of the original metallographic structure, such as carbide particles in a corroded ferrite matrix, or as fossil or ghost structures assumed by the slightly differential appearance of corroded ferrite and cementite respectively (Rehren, Asderaki and Lemos, 2007). High-contrast images created by the Back Scatter Detector (BSD) of the SEM enabled detection of remnant or ghost structures (Notis, 2002, p.261). Interpretation of ghost structures could then be achieved by comparing the BSD images with uncorroded micrographs (Figure 5). According to the grain sizes, lamellar spacings in pearlite (fine or coarse), shape and distribution of ferrite (equiaxed or acicular) and proeutectoid cementite (linear or globular), cooling rates of the objects and possible heat treatments were interpreted.

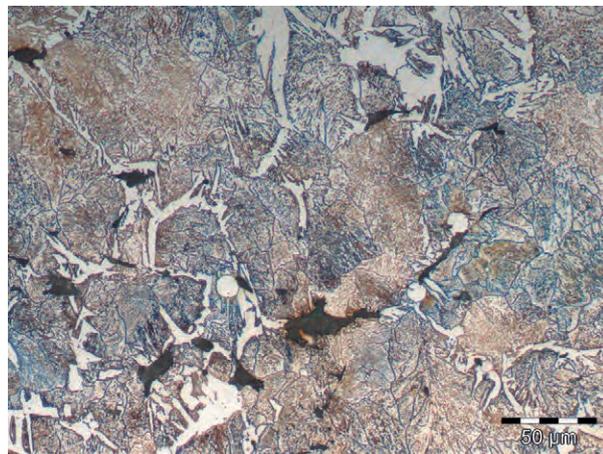
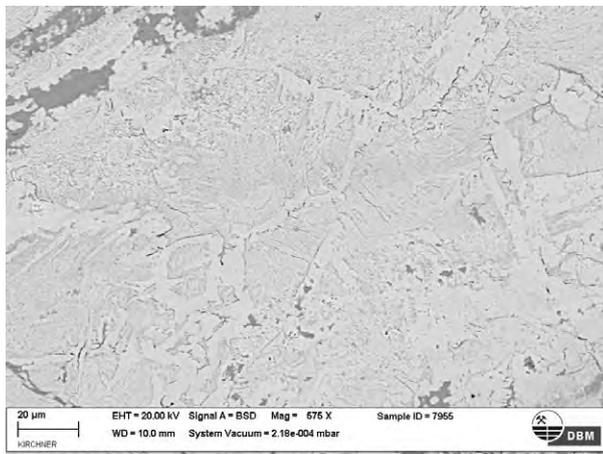


Figure 5. Ghost structures from BSD image of KT7955 (left), and metallography photo of an uncorroded medieval object (right) showing that the ghost microstructure (pearlite and proeutectoid grain boundary ferrite) would look similar if not corroded.

ed as well. The microstructure observations are summarized in Table 1.

According to metallography and SEM analysis, the microstructure observed in iron rod KT8548 consisted mainly of normalized<sup>2</sup> pearlite and ferrite grains (Figure 6). Grain sizes are very fine. The composition can be estimated as medium-carbon steel (reaching 0.4% C). The dispersed areas detected as bearing ghost structures contain a similar amount of carbon, suggesting a homogeneous composition. The microstructure of iron rod KT13325-S, from the same context, is more heterogeneous. Two different microstructures were observed: one with a similar character to KT8548; and the other, a

hypereutectoid steel with a cementite network. In high carbon areas, the carbide particles tend to spheroidize. This could be the effect of normalizing.

The carbon amount of iron rod KT13325-L approximates eutectoid composition. The proeutectoid phase is observed at the grain boundaries, which are ferrite in some parts and cementite in others. In both cases, the lamellar spacing in pearlites is large and their grain size is coarse (Figure 7). This circumstance indicates another heat treatment, which is called annealing.<sup>3</sup>

Similar to the iron rods, the sampled blades did not preserve their original surfaces and metal bodies. From their cross-sections, however, some anatomical prop-

Table 1: Analyzed objects and microstructure observations (\*EIA: Early Iron Age, MIA: Middle Iron Age, LIA: Late Iron Age).

No.	Sample Code	Level*	Phase	Type of Object	Type of Ferrous Structure	Microstructure	Heat Treatment
1	KT7955	EIA	12	Handle of Blade	Medium Carbon Steel	Ferrite + Pearlite	Annealing
					High Carbon Steel	Cementite Network	
2	KT13325-S	MIA	9 or 8	Iron Rod	Medium Carbon Steel	Ferrite + Pearlite	Normalizing
3	KT13325-L	MIA	9 or 8	Iron Rod	Medium Carbon Steel	Ferrite + Pearlite	Annealing
					High Carbon Steel	Globulized Cementite Network	
4	KT17715	MIA	9/8 Transition	Blade	Low Carbon Steel	Ferrite + Pearlite	Normalizing
5	KT17086	MIA	9/8 Transition	Blade	N/A	N/A	Normalizing
6	KT16003	MIA	8	Blade	High Carbon Steel	Globulized Cementite Network	Normalizing
7	KT8548	LIA	7	Iron Rod	Medium Carbon Steel	Pearlite + Ferrite	Normalizing

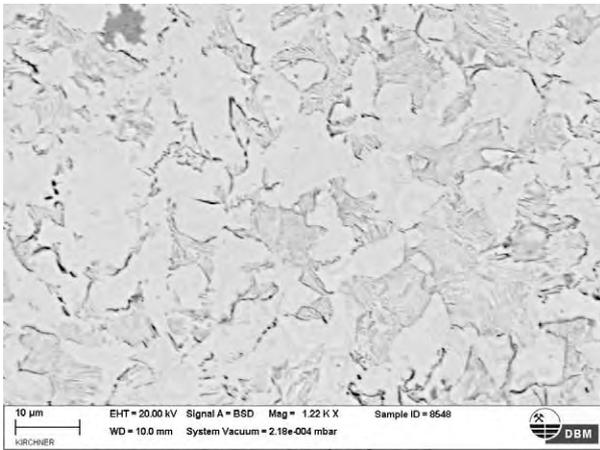


Figure 6. Normalized ferrite and pearlite grains in KT8548.

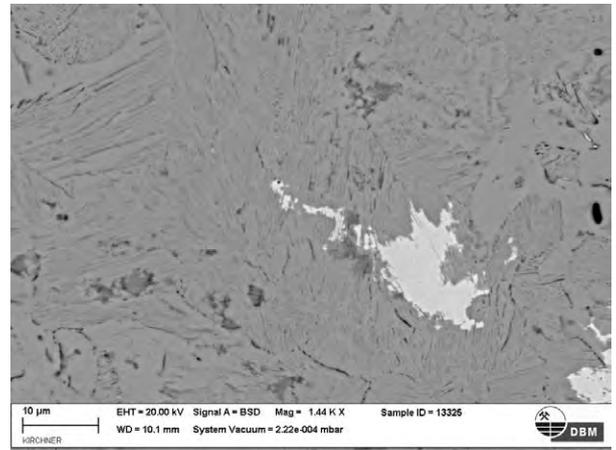


Figure 7. Coarse pearlite grains in KT13325-L.

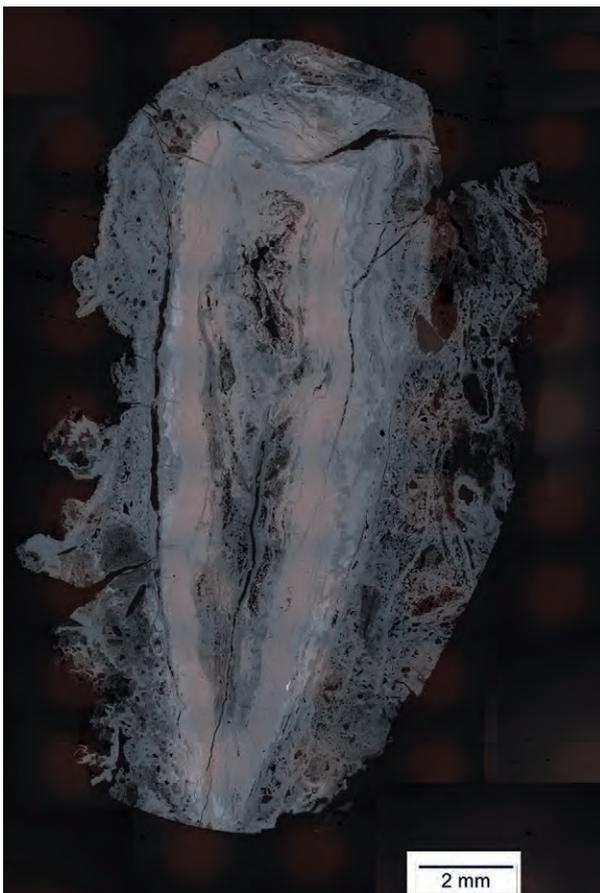


Figure 8. Cross-section of KT17715.

erties of the tools can be detected. In the case of blade KT17715, its width from cutting edge to back can be estimated at ca. 2 centimeters. Some organic residues were visible in the corrosion crust, besides the soil particles and by-products of corrosion (Figure 8). At SEM analysis in between the corroded matrix, pearlite areas were observed as thin borders around ferrite grains (Figure 9). The amount of pearlite indicates that this blade contains

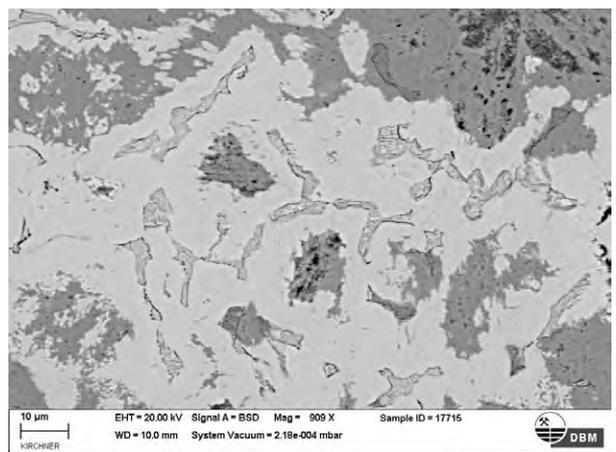


Figure 9. In KT17715, the dominant microstructure is ferrite.

the lowest amount of carbon (around 0.2 % C), in contrast to the other sampled objects

Metal remnants of proeutectoid cementite were useful to evaluate the metallographical micrographs of the Kinet blades. In the microstructure of KT16003, the remnants and ghost structures of cementite surround the original pearlite boundaries. Although from this image the size and shape of grains can be recognized, the lines are broken and tend to globulize. This image can be compared to a medieval blade from Kinet, which shows a border between a similar type of corrosion effect and the surviving original structure (Figure 10 and 11). This comparison shows the tendency to spheroidization of the cementite network and pearlite layers in the grains.

In the microstructure of blade handle KT7955, seen in Figure 5 to consist of coarse pearlite with widmanstatten ferrite, hypereutectoid compositions are observed in other sectors as a dark ghost network of proeutectoid cementite (Figure 12).

Neither metal remnants nor ghost structures are sufficient to determine the original structure of blade

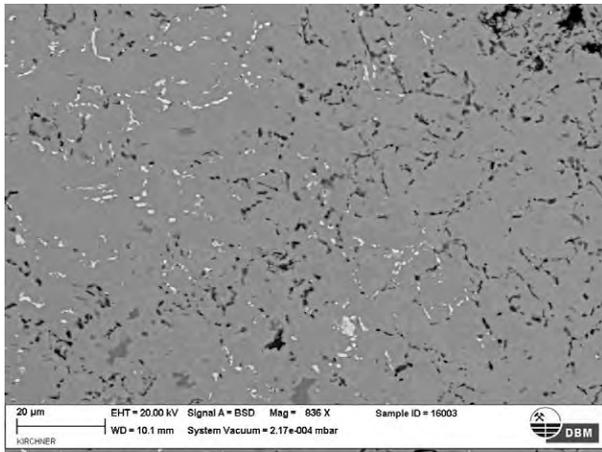


Figure 10. Cementite network seen as remnant (white) and ghost (black) structures in KT16003.

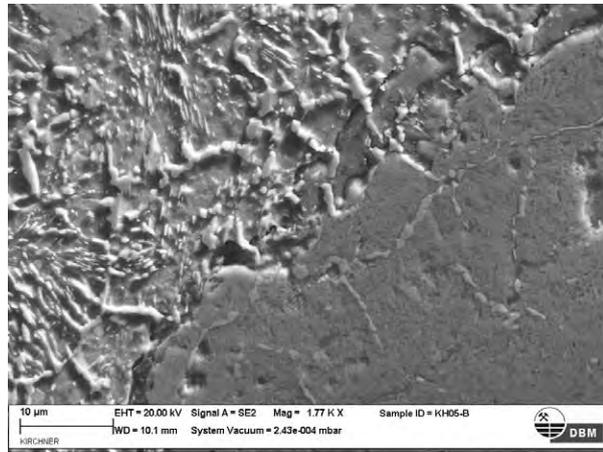


Figure 11. A secondary electron image of a medieval blade from Kinet shows how corrosion changes semi-globulized hypereutectoid steel.

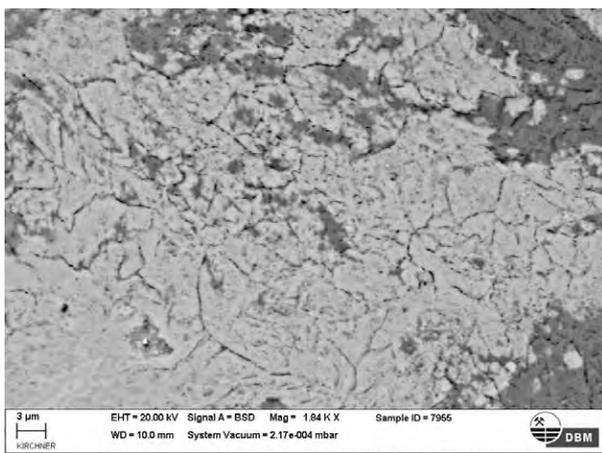


Figure 12. Cementite network of KT7955.

KT17086. A few metal remnants were observed appropriate to the size and shape of spheroidized cementite. Towards the tip of the blade were noticed elongated glassy slag inclusions inside the fully corroded matrix (Figure 13). The slag inclusions, which were broken and deformed during the forging stage, could either have originated from the smelting operation, or were introduced into the body during folding or welding operations. Due to the corrosion it was not possible to follow the distribution of slag inclusions across the whole cross-section.

### Chemical and X-ray Diffraction Analysis of Slags

The sampled slags discussed in this paper were recovered in area E/H on the west side of the mound, in a Middle Iron Age level dated from the mid-8<sup>th</sup> to mid-7<sup>th</sup> century BC (Kinet Period 8). The fragment of a furnace wall,

KT20471, was found in the same area but belonged to an earlier Middle Iron industrial context (10<sup>th</sup> century BC, Period 11). When sectioned, it was found to contain a light-colored sandy material (Figure 3). All of the slags have different morphologies: some with irregular lumps, others with sharp corners and plane surfaces looking like fragments from larger pieces. Only KT16622 has a noticeable plano-convex shape with a radius of ca. 8 centimeters. Dominant colors are dark brown and grayish, and most are randomly colored rusty red.

To sample the slags, water-cooled diamond discs were used to prepare two segments of similar nature and appearance. One was reserved for the preparation of thin sections to use in petrographic analysis, and the second was ground up in a cylindrical mill for powder samples. Chemical bulk analysis was applied on powdered samples by inductive coupled plasma-mass spec-

Figure 13. Elongated slag inclusions towards the tip of blade KT17086.

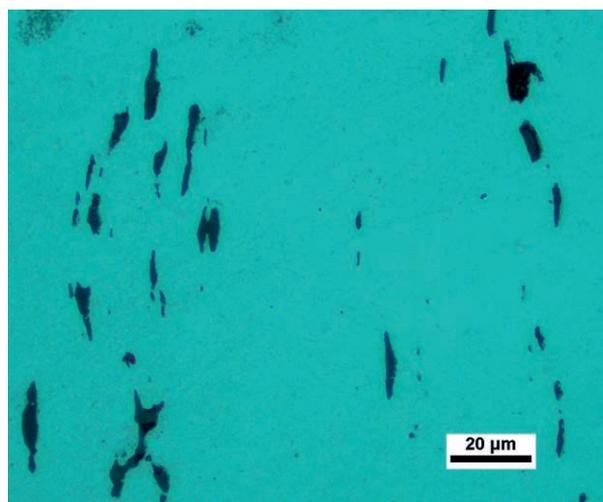


Table 2. Chemical compositions of slag assemblage (normalized to 100% according to Fe<sub>2</sub>O<sub>3</sub> value).

Sample Code	Mass %											ppm					
	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	CaO	TiO <sub>2</sub>	MnO	FeO	Fe <sub>2</sub> O <sub>3</sub>	K <sub>2</sub> O	Ba	V	Cr	Co	Ni	Sr
KT15662	0.82	5.00	2.33	13.83	1.15	5.70	0.36	0.06	62.51	69.47	1.27	239	80	410	20	230	390
KT15985	0.16	5.15	0.80	12.14	0.51	2.92	0.01	0.06	69.47	77.21	1.04	68	20	320	30	420	100
KT16287	0.22	4.93	0.86	19.92	2.14	4.34	0.00	0.07	59.23	65.83	1.69	370	40	670	35	390	260
KT16622	1.32	6.83	5.53	24.65	0.35	10.49	0.78	0.14	42.69	47.45	2.47	125	110	890	100	430	310
KT17007	0.10	4.75	0.84	12.73	0.20	2.86	0.01	0.05	69.97	77.76	0.70	97	30	420	40	250	85
KT20471	3.02	6.94	17.67	45.20	0.53	9.38	2.61	0.20	11.66	12.96	1.50	263	250	230	50	280	480

Table 3. List of minerals and oxides identified by XRD (X-ray diffraction) and petrographic examination.

Sample IDs: KT15662 . KT15985 . KT16287 . KT16622 . KT17007	
Wüstite	FeO
Magnetite	Fe <sub>3</sub> O <sub>4</sub>
Fayalite	Fe <sub>2</sub> SiO <sub>4</sub>
Quartz	SiO <sub>2</sub>
Diopside	CaMgSi <sub>2</sub> O <sub>6</sub>
Sample ID: KT20471	
Anorthite	CaAl <sub>2</sub> Si <sub>2</sub> O <sub>8</sub>
Albite	NaAlSi <sub>3</sub> O <sub>8</sub>
Forsterite	Mg <sub>2</sub> SiO <sub>4</sub>
Magnetite	Fe <sub>3</sub> O <sub>4</sub>
Diopside	CaMgSi <sub>2</sub> O <sub>6</sub>

trometry (ICP-MS), and the X-ray diffraction method (XRD) gave support to mineralogical definition for petrography.

As seen from Table 2, four slags are very rich in iron oxides (59.23 % - 69.97 %). In a fifth sample, KT16622, the amount of FeO in is present in lower levels (42.69 %), while its CaO, MgO, Na<sub>2</sub>O and TiO<sub>2</sub> quantities are higher and show a parallel tendency with KT20471.

XRD examination of the samples (Table 3) reveals that the main mineral constituents of the slags are iron oxides (wüstite, magnetite) and olivine group minerals (fayalite and forsterite). On the other hand, in KT20471 were observed feldspar group minerals (plagioclase), pyroxene (diopside), and olivine (forsterite).

A polarizing microscope was used to observe the thin sections. Selected thin sections were then viewed and described under the scanning electron microscope (SEM), and chemical analysis of selected regions from these sections was carried out by SEM using energy dispersive X-ray fluorescence spectroscopy (EDX).

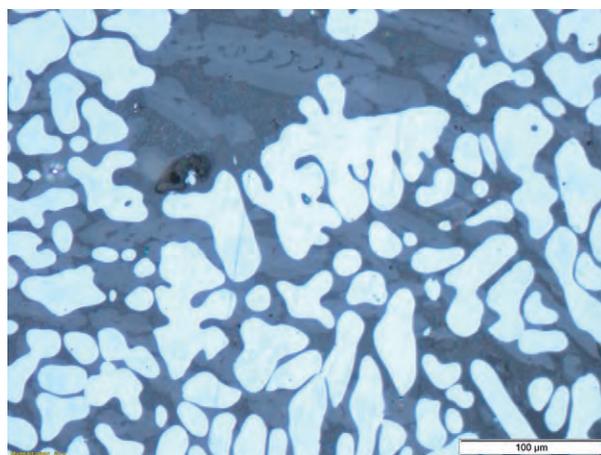


Figure 14. Semi-globular wüstite, fayalitic skeleton and glass phase of KT15985.

The petrography of these slags presents three constituent features: mainly semi-globular wüstite dendrites, fayalite skeletons, and glass phase (Figure 14).

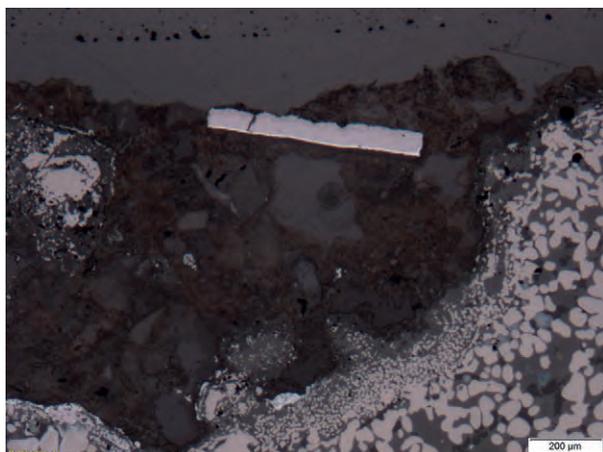


Figure 15. Hammerscale (white flake) surrounded by sediments adhering to the wüstite-dominated structure of KT16287.



Figure 16. Dominant structure is prismatic laths of anorthite in KT20471.

This observation suits well with the XRD examination. In most of the thin sections, hammerscale were visible (Figure 15).

The main constituents of the mineralogical structures observed in thin section of furnace wall KT20471 are plagioclase minerals (Figure 16). The plagioclase minerals detected as anorthite and albite are mostly prismatic laths. Numerous vesicles were manifested as dark in crossed polarized light. According to XRD, the main olivine mineral is forsterite which occurs as phenocrystals. Chemical and mineralogical analysis confirms that this block is made out of basalt.

One significant image of this study shows the occurrence of minerals belonging to both groups in the thin section of KT16622. This sample documents the mixture of microstructures like forsterite minerals, wüstite and hammerscale in a single slag (Figure 17). The combination of minerals also explains the derivation of the slag's chemical composition as distinct from the others.

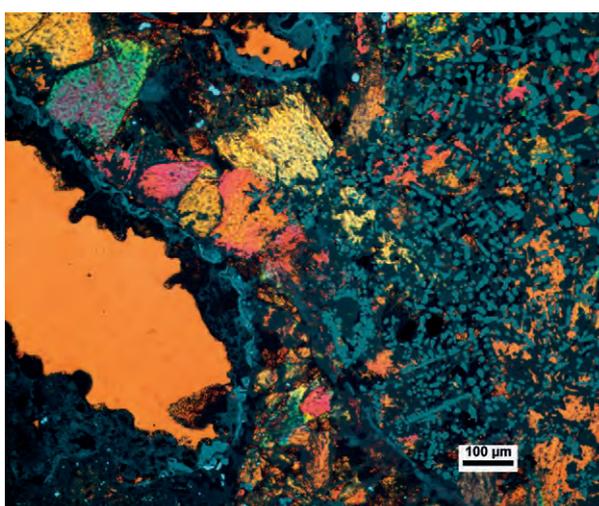


Figure 17. Wüstite formations (on the right) and basalt area (forsterite phenocrystals) seen together in KT16622.

## Discussion of Results

Opinions diverge about how innovations in iron metallurgy at the beginning of the first millennium BC determined the preference for iron to produce utilitarian tools and military equipment in the Eastern Mediterranean and the Near East. Specialists have focused on demonstrating, for early iron objects, the application of thermal processes that produced homogeneous steel structures, providing iron with qualities superior to bronze. Although most of the archaeological iron finds are fully corroded, microstructure analysis to reveal their original material has succeeded in supply-

ing valuable information on this issue. Assyrian and Urartian iron ingots from Nimrud, Khorsabad and Toprakkale consisted of pure iron within dispersed high-carbon spaces. The ingots were produced either by the primary smithing of heterogeneous blooms or by joining blooms coming from different smelting operations. This situation shows that carburization was realized during the smelting operation without deliberate control. Production of high carbon steel is mostly attributed to a developed metallurgical technology. On the contrary, experimental smelting experiments have shown that advanced control over the ore, fuel and air supply is necessary only to produce homogeneous pure iron blooms (Sauder and Williams, 2002, p.130). Most probably it was acceptable for ancient smelters to have carbon in some parts of the bloom, but only up to a

certain level, since excessive carbon will result in cast iron, a non-refinable product of smelting which has to be discarded.<sup>4</sup>

From this perspective, the microstructures of ancient iron objects produced from heterogeneous iron ingots can be expected to show parts with different concentrations of carbon. After the refining stage, such a material should have an overall hardness suitable to use in the production of specific tools. The difficulty arises when the differences in the smithing starting materials are high, since the carbon amount determines optimum smithing temperature. Pure iron becomes soft enough to forge when the temperature exceeds the ideal smithing temperature of high-carbon steel by ca. 150 - 200 degrees (Sherby and Wadsworth, 2001, p.351). Smithing a mixed iron-steel material creates the risk of burning the steel, which brings a dramatic end to the material's workability (Pleiner, 2006, p.53). Moreover, keeping high-carbon steel too long in the oxidation atmosphere of a hot smithing hearth brings about decarburization, which results in softening at the outer edges of the object. Decarburization also causes heterogeneous structure inside the object. This factor's relevance to the skills of ancient black smiths is another aspect to consider while discussing the material properties of iron objects from Kinet Höyük.

Although the iron objects from Kinet are heavily corroded and retain no solid metal parts, careful sample preparations made it possible to obtain images of remnant and ghost structures of their original microstructures using metallography and SEM techniques. Relic structures belonging to pearlite and cementite structures show that all of the sampled objects are steel, with a carbon amount of 0.2 - 0.8%. In the majority of the objects, the material composition is hypereutectoid steel. Their carbon amounts differ slightly. Since the relic objects were noticed randomly throughout the structures and the corrosion crust has destroyed all surfaces, it is difficult to say whether there is an increasing or decreasing pattern showing diffusion or decarburization of carbon from the surface. A specially-designed furnace to carburize iron materials from the Assyrian citadel at Tel esh-Shari (7<sup>th</sup> - 6<sup>th</sup> century BC), in the north-western Negev, could reach temperatures of 1150 °C and carburize surfaces as high as 1 % carbon (Rothenberg and Tylecote, 1991, p.14). However, the Kinet samples do not give enough evidence for the usage of such a furnace providing controlled carburization.

Other than carburizing, heat treatments identified as normalizing and annealing are attested in the microstructures of the Kinet iron objects. All of the analyzed objects' microstructures are belonging to softer versions

of medium- and high-carbon steel. Normalized pearlite and globulized cementite were created after exposing the object to heat for a specific length of time. The normalized structures of blade KT17715 show that it was removed from the smithing hearth after being heated beyond the critical upper temperature, while knife KT7955 experienced a similar operation but was left in the smithing hearth for cooling.

Similar structures were observed in iron rod KT13325-L. Annealing, a technique whose origin can be traced back to working native copper, was then applied to bronze by ancient metallurgists as well (Kienlin, Bischoff and Opielka, 2006, p.456). The practice of annealing aimed to decrease the hardness incurred during the cold-working of bronze. Considering that Iron Age metal workshops were processing iron and bronze together as seen at Tayinat and from the metallurgical wastes found in a large 7<sup>th</sup> century BC Assyrian pit at Tel Dor (Roames, 2011, p.154; Eliyahu-Behar et al., 2008, p.2907), it can be suggested that the techniques of annealing and/or normalizing were learned and developed from an old metal and transferred to a new one to resolve similar problems, i.e. to soften high-carbon steel for further manipulation. On the other hand, the corroded sections of the Kinet blades provide only a few images resembling the needle martensite structures that are signs of quenching. Since most parts of the tools were not affected by quenching, it is difficult to propose that a technique such as partial quenching was used to shield other parts of the tool from heat. Secure evidence for quenching is therefore unavailable for the Kinet samples, even if quenching has been attributed to Assyrian iron-smiths (Lang, 1975, p.200). Since quenching was not involved in bronze working, it represented a major technological step that ancient metalworkers must have achieved by trial and error.

Slag inclusions are observed frequently in the microstructures of ancient iron objects, although the blooms were hammered to expel as much of the slag as possible by ancient blacksmiths. The concentration of observed slag inclusions shows the efficiency of the refining stage. Kinet's fully corroded samples make it difficult to determine the presence or absence of slag inclusions, none were observed near the ghost structures. The only instance was the elongated slag chains around the tip of blade KT17086.

The sampled iron objects from Kinet all date to the Iron Age, but span a 500-year range from the earliest example, KT7955 (Early Iron Age Period 12, 12<sup>th</sup> - 11<sup>th</sup> century BC) to the latest one, KT8548 (Late Iron Age, Period 7, 7<sup>th</sup> century BC). Although the number of analyzed objects is limited, the changes detected among them were minor from a metallurgical point of view. In

all periods, the presence of medium- and high-carbon steel structures in most of these items shows that these Iron Age blacksmiths were skilled in choosing the right materials for producing tools. Moreover, the consistency in procedures for applying heat during the manufacturing process indicates the deliberate transfer of metallurgical knowledge over many generations.

A significant percentage of the slag assemblage collected from all Iron Age levels in Area E/H were found on analysis to be heat-affected fragments of basalt. Kinet produced much evidence for cold-processed basalt, i.e. stone-carving to make stone bowls, braziers and grinding stones in all periods, as well as blocks in Late Bronze and Iron Age masonry (Gates, 2006, pp.295-298). The one slag-like example discussed in this study, KT20471, was chosen because of its unusual form and appearance. It was cut into the shape of a rectangular plano-convex brick, and its dark-brown surface has a glossy, vitreous texture, as if from intense exposure to fire. Since it was recovered on the sunken floor of a furnace, basalt may have served in the construction of this and other pyrotechnic features at Kinet. The petrographic and chemical analysis of basalt shows close affinities to the basaltic formations of the Delihalil volcano at Turunçlu, 15 km northwest of Kinet as the crow flies, and conveniently accessible by boat (Yalın, 2009, pp.74-85).

Analysis of the remaining slags (KT15662, KT15985, KT16287, KT16622, KT17007) points to high amounts of iron oxide (42 – 70 %) and a dominance of iron oxide minerals in their structure. Thus, they represent wastes of an iron-related process. Hammerscale visible in the petrographic images are proof of an iron smithing process as the source of these wastes. Additionally, the plano-convex form of KT16622 is a characteristic shape which results from the accumulation of slag at bottom of the smithing hearth (McDonnell, 1984, p.48).

The smithing slags discussed here were recovered in Area E/H, from different sectors of a single large building occupied over three separate phases, all dated to Kinet's Neo-Assyrian Period 8. This building was well equipped with ovens, and with evidence for industrial activities in small rooms around a paved courtyard. The slags were found scattered in waste deposits rather than in the fills and context of the ovens; however, the building had been cleaned out with each successive phase, so that little remained in situ.<sup>5</sup> At minimum, these findings show that iron smithing activities were taking place at Kinet in this architectural compound during the later 8<sup>th</sup> to mid-7<sup>th</sup> centuries BC, when Assyrians were controlling the site.

This association between the slag-like basalt finds and iron smithing wastes is not limited to their Period 8 occupational context. Similar evidence can be cited

from the petrographic image of KT16622 (see above, and Figure 17), where wüstite-dominated parts are interlaced with basaltic parts. Another significant finding is the excessive amount of magnesium oxide (MgO) in the smithing slags. The magnesium oxide concentration is between 4.93 and 6.83 % in the sampled smithing slags. This range is considered atypical for smithing slag composition.

The elevated value of MgO is specific not only to Kinet's Neo-Assyrian smithing slags, but also to those from its Crusader-period occupational levels, and to slags from the neighboring "Tüpraş Field Site," early Islamic Hisn al-Tinat (Güder, et al., 2015).

The main component of smithing slags derives from the oxidation of the forged iron object. Iron particles that did not consolidate well with the object may also contribute to the slaggy matter. In addition, its silica, alumina and other oxides are absorbed from the smithing hearth lining, its interior, the hearth's surroundings, and the fluxes which are intended to protect the object from further oxidation (Serneels and Sebastien, 2003, pp.471-472). The use of solid basalt as construction materials for furnaces and smithing hearths at Kinet would contribute only a limited concentration of oxides. A second possible source is the local, magnesium-rich clay, which served to make tuyères, to plaster ovens and smithing hearths, and was occasionally tempered with crushed basalt as seen in some Kinet pottery.<sup>6</sup>

A third possible source that should be considered here for oxides in smithing slags are the fluxes added during smithing. The analysis of smithing slags from medieval Hisn al-Tinat noted white powders occurring in clusters within the slag matrix. P-XRF analysis of different clusters gave them MgO values as high as 35 %, and a CaO content of ca. 6 % (Güder, 2015, pp.230). Other relevant comparisons can be cited from the Iron Age settlements at Rehov, Beer-Sheba and Hazor in the southern Levant, where the cross-sections of slags revealed similar clusters of white inclusions. They were found to be composed almost entirely of Ca and O, or concentrations of Ca, O and Si (Eliyahu-Behar, et al., 2013, p.4325). These examples show that fluxes rich in calcium and magnesium (i.e. dolomite) contributed mineral components to the by-products of operations, and can be discerned from elements contained in their slag.

## Conclusions

The current study supports the consensus on Iron Age metallurgy in the eastern Mediterranean and Near East that carburizing was realized during the smelting pro-

cess, without any evidence for quenching. It especially provides new data and insights on local workshop practice during this period of developing iron technology. The research conducted here demonstrates that Kinet's metal workers appreciated the heterogeneous nature of their materials and achieved success with it by adapting thermo-mechanical treatments. They knew to select the medium- and high-carbon steel, which is harder than pure iron, for improved tool production. They could apply effective heat treatments by stabilizing the temperatures in their smithing hearths. They were also aware of refractory materials, and the advantages they offered in furnace construction or as fluxes. Kinet's ironsmiths thus belonged to a broad community of contemporary craftsmen with shared technological knowledge.

This paper has also demonstrated the successful application of its combined analytical procedures to secure information about ancient manufacturing technologies from iron-bearing materials. On the strength of the partial results presented here, continuing research on the large sample of metallurgical remains recovered from Kinet Höyük can be expected to expand archaeological documentation for a region that was well supplied in resources and shipping networks, and is therefore critical to the study of ancient metals.

## Acknowledgements

This project is supported by the Heinrich Winkelman Post-doctoral Scholarship funded by the Deutsches Bergbau Museum. We would like to thank the wonderful Materialskunde Labor team of the Deutsches Bergbau-Museum for their great efforts in sample preparation, chemical analysis and XRD. We would also like to thank the two anonymous reviewers for their comments.

## Notes

- 1 While cold-worked 10% tin-bronze, the hardest version of Bronze, has a hardness of 228 Brinell, the hardness of wrought iron measures 100 Brinell, and worked mild steel measures 246 Brinell (Snodgrass, 2000, p.215). Quenching increases hardness to over 600 Brinell.
- 2 Normalizing is a heat treatment to make the structure uniform. As the result of the treatment, grain sizes are refined. The temperature needs to be ca. 50 °C over A3 (the upper critical temperature: for the structure seen in Figure 6, A3 is around 800 °C), where austenite is the stable form. After keeping the object for the sufficient time at this temperature, the object is air-cooled.
- 3 Annealing is done at temperatures similar to normalizing, but they differ significantly in their cooling rates. In the

case of normalizing, the object is taken from the furnace for cooling, whereas annealing involves leaving the object in the furnace to be cooled at a much slower rate.

- 4 Cast iron micro-structures were observed in the preliminary analysis of a discarded bloom during the on-going project on ironworking at Sirkeli Höyük.
- 5 Samples KT15662 ('02 EH 276 L. 587), KT 15985('02 EH 273 L. 621) and KT17007 ('02 EH 331 L. 745) belong to the earliest phase of the Period 8 compound. KT16287 ('02 EH 54 L. 660) and KT16622 ('02 EH 299 L. 695) were reused in the compound's later-phase masonry, but can reasonably be attributed to its early phase also.
- 6 In addition to the Kinet samples of Iron Age and medieval iron objects, smithing slags and basalt fragments, a Late Bronze tuyere (KT26282) was also examined. Because of its early date, it is not included in this paper's discussion of its analysis and results. The chemical composition of the tuyere's clay, according to ICP-MS analysis, consisted of 16.2 % MgO, 4.67 % CaO and 51.7 % SiO<sub>2</sub>. XRD analysis demonstrates the presence of magnesium- and calcium-bearing minerals such as forsterite, diopside, and dolomite. Petrographic imagery shows crushed sea shells as temper in the clay.

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