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Studies in Honor of James D. Muhly

edited by

Philip P. Betancourt and Susan C. Ferrence



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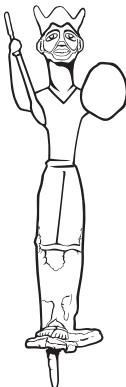
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List of Abbreviations

Abbreviations for periodicals in the bibliographies of the individual articles follow the conventions of the *American Journal of Archaeology* 111.1 (2007), pp. 14–34.

AKR	excavation number, Akrotiri, Thera	LChal	Late Chalcolithic
cm	centimeter	LH	Late Helladic
dia.	diameter	LM	Late Minoan
EBA	Early Bronze Age	m	meter
EC	Early Cycladic	MBA	Middle Bronze Age
EChal	Early Chalcolithic	MC	Midlle Cycladic
ED-XRF	emission dispersive X-ray fluorescence	MChal	Middle Chalcolithic
EH	Early Helladic	MH	Middle Helladic
EM	Early Minoan	MM	Middle Minoan
gr	gram	NCSR	National Center for Scientific Research “Demokritos”
h.	height	NM	National Archaeological Museum of Greece
HM	Herakleion Archaeological Museum	NMD	Neolithic Museum, Diros, Mani
HNM	Hagios Nikolaos Archaeological Museum	pers. comm.	personal communication
L.	length	pers. obs.	personal observation
LBA	Late Bronze Age	pres.	preserved
LC	Late Cycladic or Late Cypriot		



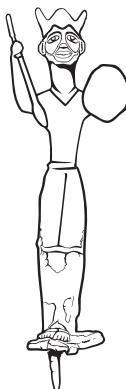
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METALLURGY: UNDERSTANDING HOW, LEARNING WHY

SEM/EDX	scanning electron microscopy and energy dispersive microanalyses	wt.	weight
SM	Siteia Archaeological Museum	XRD	X-ray diffractometry
th.	thickness	th.	thickness
w.	width	XRF	X-ray fluorescence spectrometry

C H A P T E R

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Reconstructing Early Cretan Metallurgy: Analytical Evidence from Kephala Petras, Siteia

Mihalis Catapotis, Yannis Bassiakos, and Yiannis Papadatos

Introduction

Recent excavations at Kephala Petras in eastern Crete (Papadatos 2008; Papadatos et al., forthcoming) brought to light new evidence for copper-smelting activities in Crete, dating at least to the earliest part of the Early Minoan (EM) I period or possibly to the end of the Final Neolithic (FN) period (Papadatos 2007a). The finds, typical of early copper-smelting processes, include two pieces of copper ore, six slag fragments, and four pieces of iron ore. To these probably are to be added a small number of deformed clay fragments clearly subjected to high temperatures, which might represent refractory material used in the metallurgical process.

The importance of these finds, despite their small quantity, lies in the fact that they are among the earliest direct evidence for copper-smelting activities

on Crete. Evidence of similar date is extremely rare both within Crete (Betancourt 2006, 2007) and in the rest of the Aegean (Nakou 1995; Muhly 2002, 2006; Papadatos 2007a). For this reason, an analytical project was undertaken by the Laboratory of Archaeometry of the National Center for Scientific Research (NCSR) “Demokritos” for the technological study of the metallurgical activities evidenced at Kephala Petras. The project combined microscopic analysis, chemical analysis, and phase microanalysis of ore and slag samples in an effort to elucidate the major technological aspects of the smelting process. This paper presents the analytical results from the study of the metallurgical finds and discusses their implications for our understanding of the early metallurgical technology in this area.

Raw Materials

Direct evidence for the nature of the raw materials used in the smelting process at Kephala Petras is provided by the two small pieces of copper ore found at the site. Microscopic examination of the ore samples indicated that they consist of secondary minerals of copper and iron oxides in a siliceous matrix, containing only a small amount of residual pyrite and chalcopyrite (Fig. 8.1), which is also reflected in the low levels of sulfur in their bulk composition (Table 8.1). That the smelted ores were of the oxidized type is further corroborated by the low levels of sulfur and the limited presence of matte inclusions in all slag samples.

The smelting of oxidized ores for the production of copper was standard throughout the southern Aegean during the EBA, as shown by several studies of smelting sites on Seriphos, Keros, Kea, Kythnos, and Crete (Gale et al. 1985; Bassiakos and Doumas 1998; Papastamataki 1998; Bassiakos and Catapotis 2006; Bassiakos and Philaniotou 2007; Catapotis and Bassiakos 2007; Georgakopoulou 2007). The principle of the process was the conversion of copper oxides to metallic copper by creating a reducing atmosphere in the furnace (Newton and Wilson 1942, 151). Copper is a fairly noble metal, and its oxides could be relatively easily reduced. The major challenge was rather the removal of the “earthy” component of the ore (i.e., the gangue minerals) by creating a fluid slag which could allow the separation of the reduced metal. This involved firstly the manipulation of the composition of the charge and secondly the attainment of sufficiently high temperatures during the process.

Unfortunately, the evidence is too limited to provide a quantifiable picture of the gangue minerals of the ores smelted at the site. Bulk analysis of the two copper ore samples proved one of them to be iron rich and the other rather siliceous (Table 8.1). This simply could reflect minor variations among different pieces collected from the same ore deposit, given that such deposits are always heterogeneous bodies. It is equally possible, however, that copper ores brought to Kephala Petras for smelting (if indeed more than one smelting event took place there) did not always have the same mineralogical composition. Whether this implies the use of ores from different sources is open to further analysis. It remains,

however, an intriguing possibility, given that lead isotope analysis of slags from the other copper-smelting site on Crete, namely Chrysokamino, revealed that ores from various copper deposits were probably smelted at the site (Stos-Gale and Gale 2006, 313–316).

Whatever the case, the chemical composition of the slag from the site reveals that some care was probably taken to ensure that the composition of the charge would enable the formation of a liquid slag during the smelt. Indeed, as seen in Figure 8.2, the chemical composition of all five slags falls into an area of the $\text{FeO}_x\text{-SiO}_2\text{-CaO-Al}_2\text{O}_3$ phase diagram characterized by relatively low liquidus temperatures in the range of 1250°C. Moreover, it is notable that the samples form a fairly tight compositional cluster, which contrasts with the variability reflected in the two copper ores. Although we should not dismiss the possibility that all slag samples come from a single smelt, this consistency might suggest that smelters at Kephala Petras exerted some control over the composition of the smelting charge.

This control could have been achieved through a range of possible beneficiation and fluxing techniques. As far as beneficiation is concerned, the small size of the copper ore fragments (1.6–2.8 cm) and the large concentrations of copper oxides clearly visible in their surface (see also Table 8.1) may suggest that some sort of beneficiation process was taking place prior to smelting, involving the crushing of the ore and the selection of the richest pieces for the smelt. As for the possibility of fluxing, the pieces of iron ore found at the site may be illuminating. Of the four iron ores discovered during the excavations, only one has been analyzed so far. Arguably, its chemical composition (Table 8.1) does not seem ideal for the fluxing of either siliceous or ferrous copper-ores. Nonetheless, the fact that it contains no traces of copper, coupled with its distinctively high levels of alumina, suggests that this piece of ore probably came from a different deposit from that of the copper ores found at the site. Moreover, that the iron ore was found in the same context with a piece of siliceous copper ore suggests that it might have been somehow related to the metallurgical activities at the site. Put together, this evidence may raise the possibility that this piece of iron ore is an example

Find number	Type	Percent (%)									
		FeO	CuO	SiO ₂	Al ₂ O ₃	MgO	CaO	K ₂ O	TiO ₂	BaO	SO ₃
KP 03/473	copper ore	48	39	6.5	0.4	0.5	0.4	0.1	—	—	5.9
KP 03/1156	copper ore	16	48	34	—	0.3	0.4	0.1	—	—	1.7
KP 03/855	iron ore	36	—	35	27	—	0.4	0.3	1.4	—	—
KP 03/1119	slag	48	2.2	32	5.4	1.3	7.5	1.2	—	3.2	0.3
KP 03/230	slag	50	2.9	35	4.2	1.2	6.4	0.7	—	—	0.3
KP 03/244	slag	50	1.3	32	4.6	1.3	9.0	1.1	0.2	—	—
KP 03/759	slag	27	3.8	45	9.4	1.8	9.0	1.7	—	1.8	—
KP 03/194	slag	43	1.6	37	5.7	1.8	9.0	1.2	0.5	—	—

Table 8.1. Chemical composition from area scans of ore and slag samples from Kephala Petras using scanning electron microscopy–energy dispersive spectroscopy (SEM-EDS) (multiple scans; normalized data).

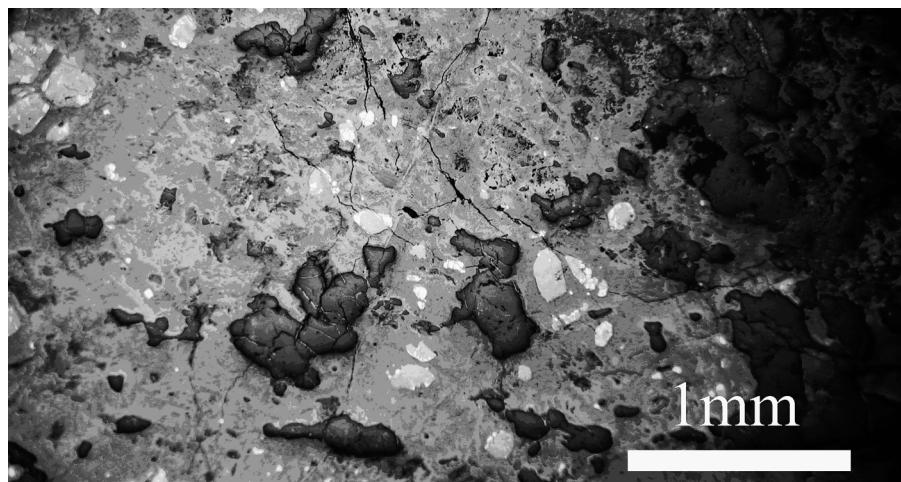


Figure 8.1. Copper-ore sample KP 03/1156. Note the residual sulphide minerals seen as white in the micro-photograph (optical microscope; cross-polarized light [XPL]).

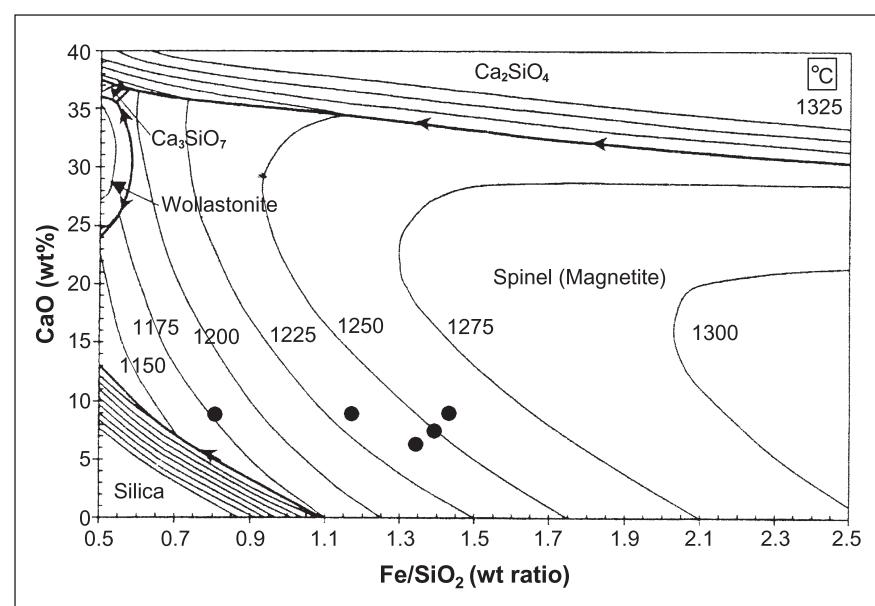


Figure 8.2. Reduced chemical composition of slag samples from Kephala Petras plotted on the Fe/SiO₂–CaO (+7% Al₂O₃) phase diagram (diagram after Kongoli and Yazawa 2001, fig. 11).

(though not representative) of minerals that were added to the charge, when necessary, to adjust its composition in order to ensure the formation of a

liquid slag. In other words, although further research is necessary, it seems possible that fluxes were used in the smelting process at Kephala Petras.

Smelting Conditions

To seek evidence for the temperature and redox conditions that prevailed during the smelting process at Kephala Petras, the mineralogy and texture of the slag were examined. Macroscopic and microscopic examination of five samples indicated that although the slag is not fully liquified it contains only a few pieces of unreacted raw materials, such as copper ore and, more commonly, silica (Fig. 8.3). This suggests that the maximum temperatures attained during the smelt were close to, though not necessarily above, the liquidus temperature of the slag, which has been estimated to be about 1250°C.

That the maximum smelting temperature was close to the liquidus temperature of the slag is also demonstrated by the notable levels of copper in the slag. More specifically, the Cu weight-% in all five samples is above 1%, reaching a maximum of 3% in the more siliceous slag (Table 8.1). This suggests that the smelting temperature was not high enough to enable the production of a fluid slag. The slag remained instead fairly viscous, leading to the entrainment of metallic prills and thus increasing copper losses.

Evidence for the redox conditions during the smelt is offered by the oxidation state of copper and iron as well as the composition of metal-rich prills in the slag. The virtual absence of primary (i.e., not post-depositional) cuprite and the copper content of the slag (1%–3%) suggests that the partial pressure of oxygen (pO_2) did not exceed the 10^{-5} atm. On the other hand, the low levels of iron in the copper prills (0.4%–3.0%) and matte inclusions (1.1%–4.0%) (Table 8.2), coupled with the predominance of magnetite (against wustite) in all slag samples, argue against pO_2 values below the 10^{-11} atm. Overall, the mineralogy of the slag samples from Kephala Petras points to moderate redox conditions, with the pO_2 ranging from 10^{-6} to 10^{-11} atm.

What is, perhaps, more important is that the mineralogy of the slag samples reflects significant variability in the redox conditions prevailing during the smelt. Although the small size of the sample does not allow firm conclusions, the co-existence of delafossite, magnetite, and wustite in a single slag sample (Fig. 8.4) strongly suggests that the smelting conditions were far from stable. Similar conclusions can be drawn from the highly variable levels of iron in copper prills (Table 8.2). Considering also the small number and size of the slag pieces found at the site and the absence of any evidence for a major metallurgical installation, this variability could be taken to imply that the smelting process was taking place inside a small ceramic container, possibly a bowl-shaped crucible, or inside a small hearth, lined with refractory clay.

We could consider, at this point, the deformed clay fragments found at the site, whose state of vitrification suggests that they have been subjected to high temperatures (Papadatos 2007a, figs. 10.7, 10.8). Although none of the sherds bears traces of copper or slag, given the absence of any fire-destruction layers at the site, it is very likely that these burned ceramics may relate to a pyrotechnological process. If this is indeed the case, it is possible that they are the remains of small ceramic vessels or hearths used as crucibles for copper smelting. This is reinforced further by petrographic analysis, which shows that they are made of a semicoarse fabric containing much organic temper and rare inclusions of quartz and quartzite (E. Nodarou, pers. comm.)—a fabric not used for any other classes of pottery at the site. Moreover, fabrics of this type with organic temper were very common for the manufacture of refractory material used for metallurgical purposes in Early Minoan Crete (Doonan, Day, and Dimopoulou-Rethemiotaki 2007, 104–105).

Find number	Phase	Percent (%)		
		Cu	Fe	S
KP 03/1119	matte inclusion	77	4.0	19
KP 03/1119	matte inclusion	81	1.1	18
KP 03/1119	copper prill	99	0.5	0.3
KP 03/1119	copper prill	98	1.9	0.2
KP 03/230	copper prill	97	3.0	0.4
KP 03/244	copper prill	98	1.6	0.2
KP 03/759	copper prill	99	0.5	0.6
KP 03/194	copper prill	99	0.4	0.2

Table 8.2. Chemical composition of metallic inclusions in slag samples from Kephala Petras determined by SEM-EDS (normalized data).

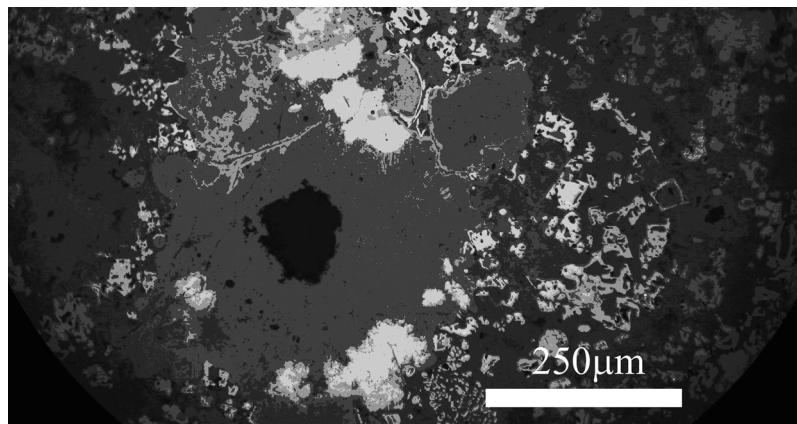


Figure 8.3. Slag sample KP 03/230 containing piece of unreacted copper-ore (dark inclusion at the center) surrounded by magnetite skeletons (optical microscope with plain-polarized light [PPL]).

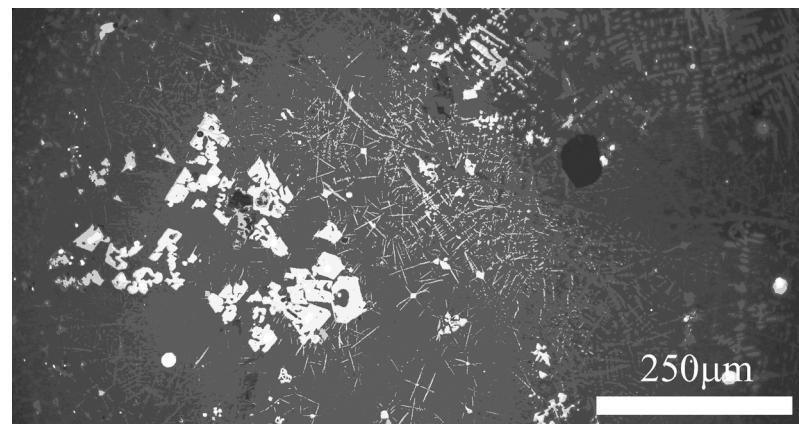


Figure 8.4. Slag sample KP 03/1119 that features the co-presence of delafossite laths (center), magnetite skeletons (left), and wustite dendrites (right) (optical microscope with PPL).

Product

Analysis of the metallic phases embedded in the slag samples suggests that the product of the smelting activities evidenced at Kephala Petras was metallic copper containing low levels of iron and possibly some copper-rich matte inclusions. Arsenic and other impurities were below the detection limit of the analytical instrument, as was the case with the two copper ore samples from the site. Such a product is compatible with the technology reconstructed at the site, i.e., with the use of oxidized copper ores containing only small amounts of residual sulfides and the attainment of moderate redox conditions during the smelt. A final refining stage would have been necessary in order to remove iron and other impurities before using the metal for the manufacture of artifacts.

This new evidence strengthens the view that the predominance of arsenical copper in the metalwork

of the Early Bronze Age (EBA) southern Aegean should not be attributed to a widespread practice of smelting arsenic-rich raw materials. Indeed, as previous studies have shown, most copper-smelting sites in that area produced arsenic-free copper (Bassiakos and Doumas 1998; Papastamataki 1998; Bassiakos and Philaniotou 2007). Arsenical-copper smelting is only evidenced at the sites of Chrysokamino on Crete (Catapotis and Bassiakos 2007); Daskaleio-Kavos on Keros, where arsenic-free copper and arsenical copper were produced by two distinct processes (Georgakopoulou 2007); and Skouries on Kythnos, where a small number of slag samples were found to contain arsenic-rich copper prills (Gale et al. 1985; contra Bassiakos and Philaniotou 2007, 51–52).

Kephala Petras in the Context of Early Cretan Metallurgy

Kephala Petras and Chrysokamino

Despite the limited available evidence, it would be worth putting the results of our analysis within the wider context of early Cretan metallurgy. Interesting conclusions can be drawn from a technological comparison of the evidence from Kephala Petras with its sole counterpart on Crete, the copper-smelting site of Chrysokamino. As demonstrated in Table 8.3, the copper-smelting technologies employed at Kephala Petras and Chrysokamino bear similarities but also significant differences.

At both sites, oxidized copper ores were used, and the composition of the charge seems to have been controlled, to a lesser or greater extent, through some combination of ore selection, beneficiation, and/or fluxing. What differentiated Chrysokamino, however, was the use of arsenic-rich raw materials, which enabled the repeated production of arsenical copper (Catapotis and Bassiakos 2007).

The smelting process at Chrysokamino was conducted in furnaces consisting of a perforated conical shaft (a technology also evidenced in the western Cyclades and Attica) laid on top of a clay-lined

pit, which probably enabled the smelting of tens of kilograms of ore in every single smelt. At Kephala Petras there is no direct evidence for the type of vessel used in the smelting process. However, the size, texture and mineralogy of the analyzed slag samples seem to suggest the use of a small clay-lined hearth or a crucible, which would have enabled the smelting of some hundreds of grams of ore in each smelting episode. The difference in the capacity of the smelting installations and the quantity of the metallurgical debris from the two sites may reflect the difference between regular processing of copper ores at an activity-specific location (Chrysokamino) on the one hand, and occasional small-scale smelts conducted very close to or within the borders of a settlement (Kephala Petras) on the other.

At Chrysokamino, oxygen was supplied into the system in two ways. First, wind penetrated through the numerous perforations on the shaft, enabling the early combustion of the fuel and the efficient preheating of the charge (Catapotis, Pryce, and Bassiakos 2008). At the lower levels of the furnace air was supplied by pot-bellows, evidence of which has been reported from the site (Betancourt 2006,

Smelting Processes	Chrysokamino	Kephala Petras
Copper ores	Oxidized	Oxidized
Beneficiation	Yes?	Yes?
Fluxing	Iron ore?	Iron-ore?
Arsenical ingredient	Yes	—
Smelting vessel	Perforated shaft furnace	Crucible?
Air supply	Wind + pot-bellows	?
Furnace temperature	≈1300°C	<1250°C
Redox conditions	Moderate	Variable
Slag processing	Crushing, pulverizing(?)	?
Copper losses	1%	1%–3%

Table 8.3. Comparison of technical aspects of the smelting processes at Kephala Petras and Chrysokamino (Catapotis and Bassiakos 2007).

125–132). The temperature and redox atmosphere thus created inside the furnace were sufficient to ensure the reduction of most copper-oxide to its metallic state, the formation of a fluid slag, and a good separation of the two formed phases. In the case of Kephala Petras, the manner of air-supply in the process remains uncertain, but smelting conditions appear to have been variable, and the temperatures attained were not sufficient to lower the viscosity of the slag, resulting in a less successful slag-metal separation compared to Chrysokamino. Nonetheless, it should be noted that copper losses in the slag from Kephala Petras (1%–3% in analyzed samples) are still of the same order of magnitude as those of Chrysokamino and other smelting sites in the Aegean (Catapotis and Bassiakos 2007, fig. 4.7).

Assigning a chronological significance to the differences evidenced at the two sites, though tempting, is not without problems. Direct metallurgical evidence at Kephala Petras (ores and slag) comes from secondary deposits, containing FN and EM IA pottery, which were sealed by the floors of an EM IA building. Only the burned clay fragments come from undisturbed pure FN deposits, but their association with metallurgy, though possible, has not yet been firmly established. Thus, the current evidence cannot provide a precise dating for metallurgy at Kephala Petras; it can only set a terminus ante quem to the EM IA period (Papadatos 2007a).

Establishing the chronology of the metallurgical activities at Chrysokamino presents greater difficulties. The metallurgical evidence derives from an extensive slag heap, which constituted a disturbed, unstratified deposit, containing a mixture of FN, EM I–II, and mostly EM III–MM IA pottery (Betancourt 2006, 68–71). Only a small area of this extensive deposit was found sealed by the successive floors of an EM III–MM IA hut. Therefore, the EM III–MM IA floor is the secure terminus ante quem for the dating of the slag heap. Based on this evidence, it is possible that metallurgical activity started as early as the FN (Betancourt 2006, 179), but the main period of use seems to be EM III (Muhly 2006, 155). Further support comes also from the pot bellows, which can be securely dated solely to the EM III–MM IA period (Betancourt 2006, 126).

Whatever the case, the fact that two copper-smelting sites located on the northern shores of eastern Crete demonstrate such different smelting techniques is indicative of the technological variability that characterized early metallurgical practices in the southern Aegean during the 3rd millennium B.C. Similar variability has been also noticed in the Cyclades, sometimes within the same island, e.g., Kythnos (Bassiakos and Philaniotou 2007) and Seriphos (Georgakopoulou 2005) or even the same settlement, e.g., Kavos North on Keros (Georgakopoulou 2004, 2007).

Kephala Petras and the Kampos Group Metallurgical Sites

One of the most important features of the early part of the EBA in Crete is the appearance on the northern shore of sites with strong Cycladic affiliations, namely Poros-Katsambas, Hagia Photia, and Gournes (Davaras and Betancourt 2004; Galanaki 2006; Wilson, Day, and Dimopoulou-Rethemiotaki 2008). Known also as the Kampos Group in the EBA Cyclades, they date to the EM IB-IIA period. Recent approaches associate these sites with the emergence of nodal points controlling the inflow of exotic materials and techniques of Cycladic origin, including metals and metallurgy, to Crete (Day, Wilson, and Kiriati 1998; Broodbank 2000, 300–304; Papadatos 2007b; Wilson, Day, and Dimopoulou-Rethemiotaki 2008).

Two sites, Poros-Katsambas and Hagia Photia, have yielded metallurgical evidence. At Poros-Katsambas, a rich metalworking assemblage has been discovered, including crucibles, molds, tuyères, copper ingots, arsenic-bearing minerals, casting spills, melting slag, and dross (Doonan, Day, and Dimopoulou-Rethemiotaki 2007). At Hagia Photia

the material comes from two different contexts. The cemetery has yielded copper-based artifacts and two complete crucibles, which, according to preliminary analyses, were used for the melting of copper (Betancourt and Muhly 2007, 150). At the adjacent settlement, nine chisel molds have been discovered in an EM IB-IIA deposit underlying the Middle Minoan strata (Tsipopoulou 2007).

What is important to note is that at both sites the evidence relates to metalworking, not to the production of metal from its ores. In other words, the evidence suggests that metal from the Cyclades and/or other sources, reached the “Kampos Group” sites in Crete in its ready-made form. The evidence from Kephala Petras shows, instead, that copper ore was brought to the settlement in order to be smelted for the extraction of the metal. Whether this should be taken to reflect a different mode of metal procurement (Papadatos 2007a, 163–165) in action on Crete before the Kampos Group phase remains to be addressed by future research. In any case, this contrast reinforces the picture of variability in the spatial arrangement of metallurgical activities evidenced in the archaeological record of the Early Bronze Age in the southern Aegean (Catapotis 2007).

Conclusions

Despite the limited amount of metallurgical evidence from Kephala Petras, it was possible to reconstruct a copper-smelting technology that is compatible with what is known so far about early smelting techniques in the southern Aegean. The process involved the smelting of oxidized ores, possibly mixed with a flux, inside a small clay container where fairly high temperatures and moderate redox conditions were attained. This enabled the production of copper with low levels of iron that were probably removed in a final refining stage, not attested so far at Kephala.

Although the evidence from the site seems to point to small-scale smelting activities, it proves

beyond doubt that copper production was taking place on Crete before the “Kampos Group” period. Therefore, the paucity of metal objects and their absence from tombs of the FN-EM IA period, such as Pseira (Betancourt and Davaras, eds., 2003), Partira (Mortzos 1972), and Hagios Nikolaos-Palaikastro (Tod 1902–1903), should not be viewed as evidence for the non-practicing of metallurgy on Crete during that time. Rather, it is indicative of different depositional practices, which have been convincingly associated with the changing role of metal in the southern Aegean societies during the EB I-II period (Nakou 1995; Papadatos 2007a).

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