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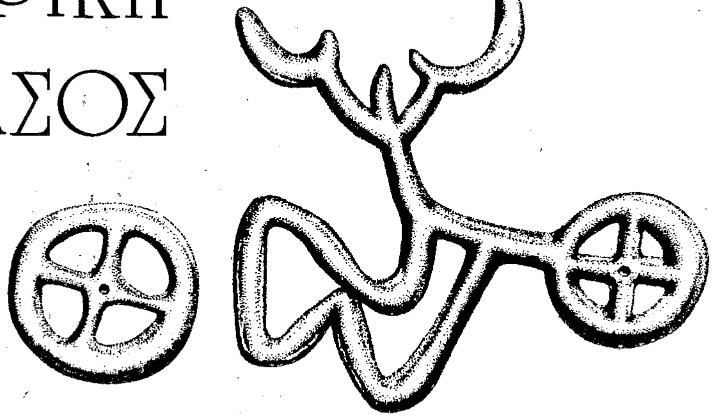

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ΠΑΡΑΡΤΗΜΑ VIIIb

LATE BRONZE AGE - EARLY IRON AGE COPPER AND IRON SLAGS FROM KASTRI AND PALIOKASTRO ON THASOS

(P l. 367)

COPPER SLAG

Copper slags together with iron slags were found in the LBA-EIA cemeteries of Kastri, namely Larnaki, Tsiganadika and Kentria. Samples for analysis were taken from the Tsiganadika graves T14 (no. D396), T18 (nos. D385, D387), T23A and T23B (nos. D383 and D384a respectively), and T24 (no. D394a), the Larnaki graves Λ2Γ and Λ5B11 (no. D399) and the Kentria grave K3A69 (Tsiore). Analytical results and dates of the samples are presented in Table 1. With the exception of Tsiganadika grave T23 which contains both copper and iron slag, the other graves contain either one or the other. Copper slags were found at another site, Paliokastro Marion. These were unstratified finds which could either date to the EIA or could be associated with the 4th-3rd c. BC levels of the same settlement (Koukouli-Chrysanthaki 1991, 679).

Analyses were carried out with the Electron Microprobe Analyser (EPMA), a Cambridge Scientific Instruments Mark V with an EDAX Link 860 x-ray microanalyser. EPMA provides the chemical composition of individual mineralogical phases as opposed to bulk chemical analysis.

Kastri copper slags (P l. 367b-e) are composed of four phases, namely, wustite (FeO) in the shape of dendrites, fayalite ($2FeO.SiO_2$) appearing as long needles, magnetite (Fe_3O_4) in the form of plates and a glassy silicate matrix exceptionally rich in barium. The composition of this phase resembles a barium silicate ($BaO.Fe_3O_4.SiO_2$) with up to 34% barium oxide in the matrix, (Table 1). The origin of this barium is barite (barium sulfate), a mineral commonly associated with the iron deposits of Thasos (Theophilopoulos 1982, 32).

In the majority of the copper slags copper occurs in both the matrix (1-7% copper oxide) and metallic inclusions (prills). The prills consist primarely of copper (Cu), iron (Fe) and sulfur (S) (D385, Table 2). The relative ratios of these three elements vary. Some of these are copper sulfide (Cu_2S), with a small amount of iron (D384a and some in D384b, Table 2). Some prills are composed of copper, arsenic antimonides or copper antimony arsenides (D384b, Table 2) with small amounts of iron and sulfur and appreciable quantities of silver and lead. These prills are characterised as speiss. Speiss, which forms insoluble phases in the slag, is a general name encompassing the alloys of metals like iron, cobalt and nickel with arsenic and antimony (Gilchrist, 1980, 232). Precious metals (gold, silver, platinum) have high affinities for speiss. This means that they will dissolve in that phase in preference to others with the exception of metallic lead.

Paliokastro copper slags are different in composition from those of the cemeteries at Kastri. The absence of barium is notable as is the near absence of sulfur in the copper prills. The prills are primarily copper with some iron and occasionally antimony. The matrix is mainly fayalitic with copper ranging from 1-4% CuO and less than 1% CuO in the magnetite. The majority of iron oxides in these samples is magnetite rather than wustite (P l. 367f-g). No copper is present in the matrix of sample Pal. Th. 1 (Table 1) and, had it not been for the presence of metallic prills containing copper and iron, the slag would have been classified as iron slag. This observation serves as a word of caution when analysing very early material, since the samples available are usually very small in size and number and cannot provide an accurate picture of either furnace conditions or more important the extractive processes involved.

TABLE 1. *Microprobe analyses of copper and iron slag from Kastri (LBA-EIA) and Paliokastro (undated), Thasos: non-metallic phases.*

	Sample No.	Museum No.	Date	Phase	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	FeO	BaO	CuO	As_2O_3	Sb_2O_3
KASTRI	IRON SLAG																		
	Λ2Γ			wustite	0.00	0.00	0.37	0.54	0.00	0.00	0.00	0.17	0.92	0.26	92.47	0.00	0.00	0.00	—
				fayalite	0.00	3.47	0.00	29.81	0.00	0.00	0.00	1.97	0.28	0.96	59.85	0.00	0.00	0.00	—
				fayalite	0.00	0.00	0.00	30.44	0.00	0.00	0.00	1.82	0.35	0.86	62.01	0.00	0.00	0.00	—
				matrix	0.00	0.00	20.80	33.66	0.72	0.00	3.54	12.61	0.83	0.00	27.25	0.00	0.00	0.00	—
	D399	Λ5B11	EIA	matrix	0.00	0.14	3.96	33.16	0.53	0.00	1.33	11.68	0.27	0.54	47.25	0.00	0.00	0.00	—
				'faya'	0.00	0.00	1.92	27.21	0.40	0.00	0.00	11.01	0.00	0.32	50.89	0.00	0.00	0.00	—
				matrix	0.00	0.00	8.29	36.51	0.94	0.00	2.82	10.18	0.42	0.43	37.36	0.00	0.00	1.05	—
	D383	T23A	EIA	wustite	0.00	1.06	9.89	0.48	0.00	0.00	0.00	0.29	3.23	0.70	80.35	—	—	—	—
				matrix	0.00	0.69	8.95	30.00	0.38	0.00	0.39	13.81	6.07	0.97	27.28	—	—	—	—
	COPPER SLAG																		
	D394a	T24	EIA	matrix	0.00	11.35	0.21	34.69	0.00	0.00	0.00	28.94	0.00	1.36	22.53	0.00	0.00	0.00	—
				matrix	0.00	1.05	10.54	48.43	0.56	0.00	2.73	11.51	0.36	0.25	12.49	3.89	4.36	0.93	—
				matrix	0.00	1.03	9.61	50.76	0.00	0.00	2.69	10.24	0.46	0.22	12.66	3.56	3.69	0.87	—
	D384a	T23B	EIA	matrix	0.00	0.00	11.75	36.77	0.00	0.00	2.08	3.27	0.00	0.26	11.64	31.09	2.82	0.00	—
				matrix	0.00	4.67	2.45	47.18	0.00	0.00	0.32	20.79	0.00	0.76	19.47	4.40	0.00	0.00	—
				matrix	0.00	0.83	1.77	32.45	0.00	0.00	0.81	5.43	0.00	1.28	23.11	29.20	1.71	1.10	—
	D396	T14	EIA	matrix	0.00	0.00	5.95	36.24	0.28	0.00	2.14	11.78	0.48	0.91	25.37	13.35	1.43	0.00	—
				matrix	0.00	6.07	0.00	33.11	0.00	0.00	0.34	22.99	0.00	1.50	31.30	1.86	0.35	0.00	—
				matrix	0.00	2.11	10.23	37.86	0.00	0.00	2.55	11.76	0.00	0.78	19.55	15.73	0.39	0.00	—
	D385	T18AΠ106	LBA-EIA	matrix	0.00	3.29	2.07	31.99	0.27	0.00	0.34	25.34	0.00	1.25	31.17	2.91	0.00	0.00	—
				wustite	0.00	0.61	1.37	0.28	0.00	0.00	0.00	0.36	1.56	0.52	83.78	0.47	0.00	0.00	—
	D384b	T23B	EIA	matrix	0.00	0.00	9.98	32.09	0.00	0.77	1.44	14.72	0.00	0.85	21.36	16.99	0.73	0.00	—
				matrix	0.00	0.00	8.39	29.77	0.00	3.55	2.56	17.60	0.00	0.78	29.25	8.19	0.00	0.00	—
	Tsiore	K3A69		matrix	0.86	0.59	3.39	32.67	0.71	0.00	0.64	11.74	0.00	0.00	20.23	29.83	0.00	0.00	—
	D387	T18Π3	LBA-EIA	matrix	0.63	0.46	3.72	30.56	0.84	0.00	0.85	8.18	0.00	0.00	19.55	33.66	0.00	0.00	—

	Sample No.	Museum No.	Date	Phase	Na_2O	MgO	Al_2O_3	SiO_2	P_2O_5	SO_3	K_2O	CaO	TiO_2	MnO	FeO	BaO	CuO	As_2O_3	Sb_2O_3
PALIOKASTRO	COPPER SLAG																		
	Pal. Thas. 1	—	undated	wustite	0.00	0.47	3.10	5.46	0.00	0.00	0.15	2.28	1.55	0.69	83.01	0.00	0.00	0.00	—
				wustite	0.00	0.60	2.97	7.71	0.00	0.00	0.38	3.43	1.74	0.93	78.95	0.00	0.00	0.00	—
				matrix	0.00	0.44	4.38	34.93	0.00	0.00	2.33	13.51	3.67	1.68	34.12	0.00	0.00	0.00	—
	Pal. Thas. 1	—	undated	'faya'	0.00	0.48	1.56	45.62	0.44	0.00	0.20	10.76	0.24	0.00	40.04	0.00	1.36	0.00	—
				'faya'	0.00	0.40	1.44	43.59	0.40	0.00	0.17	8.80	0.21	0.00	44.18	0.00	1.29	0.00	—
				wustite	0.00	0.00	0.58	0.50	0.00	0.00	0.00	0.00	0.41	0.00	93.19	0.00	0.51	0.00	—
				wustite	0.00	0.00	0.63	0.87	0.00	0.00	0.00	0.18	0.53	0.00	93.40	0.00	0.33	0.00	—
	Pal. Thas. 2	—	undated	fayalite	0.00	0.56	1.73	35.43	0.31	0.00	0.13	6.53	0.31	0.19	48.30	0.00	4.33	0.00	—
				'faya'	0.00	0.75	1.17	39.87	0.00	0.00	0.18	6.88	0.30	0.00	44.89	0.00	2.87	0.00	—
				'faya'	0.00	0.29	1.48	40.44	0.28	0.00	0.31	5.31	0.22	0.00	51.43	0.00	0.72	0.00	—
				wustite	0.00	0.00	0.56	0.92	0.00	0.00	0.00	0.13	0.44	0.00	92.19	0.00	0.63	0.00	—
	Thas. PK	—	undated	fayalite	0.00	1.09	2.29	44.54	0.00	0.00	0.89	6.33	0.92	0.36	40.95	0.00	0.88	0.00	1.11
				'faya'	0.00	0.51	2.73	40.31	0.00	0.00	0.61	8.55	1.84	0.00	33.68	0.00	1.12	0.00	0.73

'Faya' refers to phases which resemble the fayalite composition.
All concentrations in weight %.
— = not detected.
0.00 = concentration in that element approximates zero.

IRON SLAG

The first evidence for iron exploitation on Thasos comes form the analysis of three pieces of iron slag from two graves at Larnaki and one at Tsiganadika (Table 1). These cemeteries date to phase IIB2-IIB3 of the EIA (900-700 BC). Iron itself makes its appearance on Thasos (Kastri) earlier, during IIA-IB (1050 BC) in the form of a bimetallic knife with an iron blade and a cast bronze handle (Koukouli-Chrysanthaki 1991, 683). Analysis of the bronze handle by N.H. Gale showed that the copper was not local but imported possibly from Cyprus, since its lead isotope composition fell within the Cypriot or western Asia Minor field (Koukouli-Chrysanthaki 1991, 680 and 683). This suggests a corresponding origin for the iron blade.

The iron slag from Larnaki (Λ2Γ, Table 1) contains all three commonly occurring phases, i.e. wustite, fayalite and a glassy matrix. The composition of the latter resembles that of mellilite, a potassium-calcium-iron-aluminum silicate, common in most of the iron slags analysed from Macedonia (Photos et al., 1986 and 1987). The second sample from Larnaki (D399, Table 1 and P l. 367a) also contains the same phases, together with metallic iron prills free of slag inclusions. Arsenic has been detected in its matrix suggesting that the ore source may have been that at Kokoti reported by Theophilopoulos (1982, 51). However, it is difficult to draw conclusions on the basis of one sample only.

The third piece of iron slag is from the cemetery at Tsiganadika (D383, Table 1). It contains a relatively higher titanium content in the wustite and the matrix compared to the other two, but no other mineralogical phases are evident which may support an alternative source of iron ore.

DISCUSSION

The picture emerging from the analyses of the LBA-EIA copper slags from Kastri and the undated slags from Paliokastro suggests the use of at least two types of copper oxide ores at these or later periods on the island. On the one hand, there are the copper minerals malachite and azurite, which occur within the main barite-rich iron deposits of Thasos in places like Mavrolakas and Koumaria-Platania. The slags that derive from the smelting of these ores contain sulfur in the metallic prills. On the other hand, there are the copper oxide ores in the vicinity of Paliokastro, the slags of which contain no barium oxide. The copper prills contain no sulfur, only small amounts of iron and antimony. The role of barite in the production of the Kastri copper slags is of particular importance for two reasons: a) it is the origin of the sulfur in the metallic phases of the slag, thus directing us to the source of the copper ore and b) it has helped decrease the viscosity of the slag making it free-running at lower temperatures than if it had been absent. Barium sulfate decomposes at high temperatures to barium oxide, sulfur dioxide and oxygen: $BaSO_4 \rightarrow BaO + SO_2 + \frac{1}{2}O_2$

The reaction can be promoted by localised reducing conditions and the presence of a high SiO_2 content with which barium forms a stable silicate (Rosenqvist 1983, 232). In the present case, a barium silicate glass matrix is formed incorporating iron, alumina and calcium oxides (matrix in Table 1 and P l. 367d, e). In the process of barite dissociation, SO_2 has reacted with copper resulting in the formation of Cu_2S or matte prills present in the Kastri copper slag. Cu_2S is formed in preference to CuS which dissociates at c. 1120° C. The reaction of SO_2 with Cu, releasing O_2 in the process, implies that the origin of the copper sulfide prills need not necessarily be attributed to a sulfidic copper ore, as is usually assumed. It is clear from the present discussion that an alternative source of sulfur, such as barium sulfate, should also be investigated.

Viscosity can be estimated by the viscosity index which is the ratio of the sum total of the basic oxides (CaO, MgO, FeO, BaO etc.) over the sum total of the acidic oxides (SiO_2, Al_2O_3) (Bachmann 1980). The lower the viscosity index, the higher the viscosity of the slag, meaning that the viscosity decreases with increasing amount of basic oxides (Rosenqvist 1983, 312). Thus, it is probable that the copper slags at Kastri were more fluid than those of Paliokastro given the higher total amount of basic oxides (BaO and CaO). As a result, the slag metal separation would be better in the Kastri slags in comparison with those from Paliokastro.

TABLE 2. *Metallic prills in copper slag from Kastri (EBA-EIA) and Paliokastro (undated).*

Sample No.	Cu	Fe	S	Sb	As	Ag	Pb
D384a	76.77	1.95	29.09	0.00	0.00	0.00	1.15
D384b	68.76	7.11	23.33	0.00	1.21	0.00	0.00
	69.78	5.66	23.22	0.00	0.00	0.00	0.00
	58.13	12.69	27.64	0.00	0.00	0.00	0.00
	51.55	0.93	2.00	25.34	20.18	1.20	0.00
	14.69	0.98	0.50	54.22	11.63	2.02	2.03
	5.99	0.67	1.08	78.28	5.54	3.06	4.05
D385	34.84	29.23	44.89	—	—	—	—
	25.35	36.21	32.05	—	—	—	—
Pal. Thas. 1	96.90	3.20	0.22	—	—	—	—
	95.68	4.14	0.37	—	—	—	—
Thas. PK	97.14	3.50	—	2.19	—	—	—
	97.33	4.20	—	2.02	—	—	—

In each sample one or more metallic prills have been analysed.
All concentrations in weight %.
— = not detected.
0.00 = concentration in that element approximates zero.

Kastri and Paliokastro copper slags are clearly metallurgical process slags, but are they the result of smelting copper ore in a furnace or melting copper in a crucible? This distinction is not always easy to make. In an effort to establish distinguishing criteria between the two types of process slags, Tylecote (1980, 203) suggested that small amounts of iron oxide associated with high silica, calcium oxide and alumina with relatively high copper (8-40% Cu) contents in the slag are a sound criterion for slags melted in a crucible. Cooke and Nielsen (1978) suggested that crucible melting slags usually have a low iron content and a high ($SiO_2 + Al_2O_3$) to (FeO-MnO) ratio compared to smelting slags. In addition, the crucible melting slags have only a small amount of crystalline phases in comparison again with smelting slags.

The presence of barium in the Kastri copper slags rules out the possibility that they are crucible melting slags since barium would have been removed at the smelting stage. The Paliokastro slags have high iron contents and copper prills with sufficient impurities for them also to be considered smelting slags. But it is difficult to decide conclusively on such sparse evidence. Thus, the Kastri slags are considered to be the product of smelting and the Paliokastro slags most probably the same (Table 3).

The identification of iron sources for the provenance of early iron artefacts is often a rather difficult task unless tracer elements like titanium, aluminium, vanadium, chromium are detected in the slag inclusions of the artefacts or the slags themselves. Thasos slags are exceptional because titanium has been detected in both iron smelting slags and artefacts. Titanium-rich slags have been located at the south side of the island but date to the Late Byzantine-Early Ottoman period. On the other hand, titanium was found in the slag inclusions of artefacts dating to the Hellenistic period. The source of this titanium is the titanium-rich magnetite sands collectable in small amounts, but can be subjected to considerable enrichment, on the south shores of the island (Photos et al. 1986).

The Kastri iron slags contain no tracer elements which could lead us to a particular source of iron ore on the island. This can mean two things: a) that these are simply smithing slags, the result of shaping an iron lump into a useful object by eliminating all residual traces of slag or b) that an ordinary iron oxide ore like hematite, free of any characteristic elements was used.

TABLE 3. *Summary of differences between Kastri and Paliokastro copper slags.*

Kastri LBA-EIA	**Paliokastro** Undated
Copper carbonate minerals associated with barite-rich iron ore	Copper carbonate minerals
Sulfur present in matte prills	Sulfur absent
Speiss inclusions	Copper prills, 4% Fe
Copper in the silicate matrix	No copper in matrix
Presence of wustite suggests sufficiently reducing conditions in the furnace	Presence of magnetite suggests less reducing conditions in the furnace
Smelting slag	Probably smelting slag

In either case no more can be said on the type of metallurgical activity which produced the iron slags of Kastri apart from the very important observation that iron was indeed being worked on the island at that early date.

IRON FROM COPPER?

The presence of iron in copper prills as observed in the Paliokastro samples, is a common enough phenomenon in Early Bronze Age objects from the East Mediterranean reported by a number of investigators. Jones (1980) has reported iron contents in bronze objects from the rich EIA cemeteries at Lefkandi in Euboea. The same investigator, in his analysis of pieces of copper/bronze from Servia (Ridley and Wardle 1979), again reported iron (4% Fe) in the copper. Both sets of analyses were done non-destructively with XRF energy dispersive analysis, so it is possible that his results may have been influenced by surface segregation phenomena. Cooke and Aschenbrenner (1975) suggested that in copper smelting, excess iron oxide which was not taken up by the slag under localised reducing conditions would reduce to metallic iron. As the copper reduces simultaneously, metallic iron (up to 4%) would dissolve into the copper. The presence of metallic prills with this composition in slag from Paliokastro (Table 2) corroborates this observation.

The co-existence of metallic copper and iron brings to mind an important issue, namely the presence of iron ore in a copper smelting furnace and the possible evolution of iron metallurgy from copper metallurgy (Wertime 1973; Charles 1980; Pigott 1982).

It has frequently been reported in the literature that iron oxides were added intentionally as flux during the smelting of copper ores in order to produce a fluid fayalitic slag in combination with the silica in the copper ore. However, it now seems clear that copper minerals were often found in association with iron ores. This implies that iron ores made their way into the copper smelting furnace accidentally as charge rather than as intentional additions. Pigott (1982, 21) has presented an eloquent photograph of a malachite "pocket" in an iron ore body, while Merkel (1983) showed that the charge in the Chalcolithic furnaces in Timna, Israel, could contain up to 20% iron in the copper ore, in addition to the iron-rich nodules which were intentionally added as flux and which contained up to 43% iron. Flux is the material (iron oxides in the case of copper smelting) that helps reduce the temperature at which the slag is free running. Thus, slag and metal are separated with the lowest energy input possible.

Evidence for copper association with iron ores in mines exploited in antiquity occurs in the classical texts (Strabo, X, I, 9) refers to a double mine of both iron and copper found above the city of Chalkis in Euboea. What Strabo's reference implies is that by late antiquity, mines would be worked for more than one metal, if the potential was there.

The Kastri slags provide the first material evidence of what has for some time been speculated, namely the exploitation of copper ore out of iron deposits in the LBA and EIA. The presence of barium, a unique tracer element in the provenance of the Thasos main iron ore bodies, clearly suggest that the origin of the copper in the LBA-EIA were deposits like that of Mavrolakas, in which copper carbonates are found in small amounts even today (Theophilopoulos 1982, 58). Far from being an intentional addition, hematite/limonite with associated barite entered the furnace accidentally. It is very likely that metallic prills or small lumps were formed as a result of localised, strongly reducing condition. Early bronze-smiths may have noticed them but were probably not in a position to do much with them, at least initially. The main reason for their lack of success is that they would have tried to work the iron either by melting it as copper or lead or by cold-working and annealing it like native copper or gold. But pure iron with only traces of carbon (wrought iron) melts at temperatures (c. 1550° C) which would be unattainable in the existing hearths and furnaces. In addition, iron would be forged only within a limited range of temperatures according to the impurities incorporated in its matrix. In short, any effort to shape it would have resulted in fracture or no change at all.

In summary, the analytical data suggest that the early bronze-smiths on Thasos may have been led to the smelting of iron ores through their continuous experimentation with copper ores given that copper minerals were extracted from the main barite-rich iron deposits. This interpretation argues in favour of an independent development if iron metallurgy on Thasos as opposed to a diffusion of technological know-how from other sources. This suggestion would have been even more securely argued had there been available iron slag samples from the EIA cemeteries at Kastri which like their copper counterparts also contained barite. This analytical work is still in progress and six more slag samples from the same context are still pending analysis.

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