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CHEMICAL AND METALLURGICAL STUDY OF 'EIN ZIQ AND BE'ER RESISIM INGOTS

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Copper ingots found at two archaeological sites in the Negev – 'Ein Ziq and Be'er Resisim – were studied by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Metallographical Microscope and Scanning Electron Microscopy with Energy Dispersive Spectrometer (SEM-EDS). The results of the chemical and metallographical analysis, when compared to ores from Timna and Feinan, exploited for smelting in the Middle Bronze Age I, show similarity of the composition of the ingots to both sources. Metallographical study of the ingots reveals secondary casting of not completely oxidized copper ore.

INTRODUCTION

The existence of local copper production in Southern Israel during the Middle Bronze Age I (MB I) was indisputably established by the discovery of mines and a smelting site dating to this period at Timna (Rothenberg and Shaw 1990). Copper metallurgy was also conducted in Transjordan from Early Bronze Age with continuation on a smaller scale into MB I (Hauptmann 1991; Hauptmann *et al.* 1992). There is ample archaeological evidence of metal working technology in this region (Merkel and Dever 1989). Bar-shaped ingots, triangular in section, of unalloyed copper, distinctive for this period, were found at Lachish (Tufnell 1958), in the Central Negev – at Har Yeruham (Kochavi 1963), Be'er Resisim (Cohen and Dever 1979), and 'Ein Ziq (Cohen 1986), and in the Hebron Hills (Dever and Tadmor 1976). Seven bar-ingots from Har Yeruham and the Hebron Hills were examined metallurgically by Maddin and Stech Wheeler (1976). Greater amount of the ingots was found in 'Ein Ziq and Be'er Resisim. There were thirty-five unbroken ingots and fragments excavated at this region. Fig. 1 represents unbroken ingots having various forms of cross section: triangular, rounded and thin elongated. This paper presents the results of our studies of sixteen cleaned and fifteen uncleaned bar-ingots from 'Ein Ziq, as well as four ingots from Be'er Resisim.

BRIEF GEOLOGICAL INFORMATION

Due to geological situation at the western margin of the Wadi Arabah rift valley, the type and origin of the copper ore deposits at Timna and Feinan are similar. Therefore both districts have a comparable mineralogical and geochemical structure. According to Hauptmann (1990) and Hauptmann *et al.* (1992) in ancient Feinan two main types of ores were exploited: the first is Cambrian sandstones – Massive Brown Sandstones (MBS) with copper mineralization as malachite and primary copper sulphides: chalcocite and covellite. MBS were used for smelting mainly in the Chalcolithic period. The second is the deeper Cambrian layer – Dolomite Limestone Shale (DLS), which includes copper silicates (chrysocolla) and malachite. Copper ores from the base of DLS were used extensively later, from EB II with continuation, on a smaller scale, into MB I (Hauptmann *et al.* 1992; Hauptmann and Weisgerber 1992; Najjar *et al.* 1995).

In Timna, according to Segev and Sass (1989), Segev *et al.* (1992), copper ores occur in two stratigraphical layers: Lower Cretaceous and Cambrian. Lower Cretaceous contains Amir and Avrona Formations; ores consist of primary chalcocite and covellite and secondary malachite and (par)atacamite presented in sandstones. The latter are a result of oxidation of primary minerals. These ores were exploited in Timna from the Early Chalcolithic period (Rothenberg 1990). The deeper Cambrian layer consists of Shehoret and Timna Formations. Sandstones, presenting in Shehoret and partly in Timna Formations, contain chrysocolla, bisbeeite, malachite and minor amount of chalcocite. Timna Formation partly is represented by dolomites with djurleite, paratacamite and malachite. Jurleite is surrounded by an oxidized rim composed of dolostones. Manganese and phosphate mineralization are associated with iron oxide in the sandstones (Segev and Sass 1989; Segev *et al.* 1992).

In Timna there is only a single site, excavated in 1989–90 by Rothenberg and Shaw, ascribed to MB I (Rothenberg and Shaw 1990). It is located to the north-

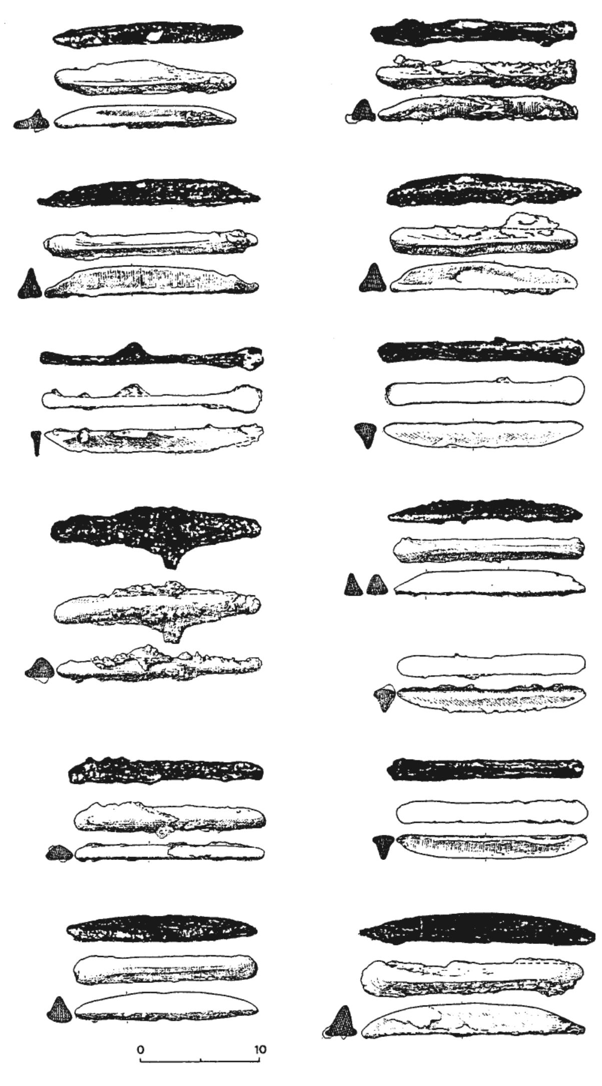

Fig. 1. Well preserved copper ingots from 'Ein Ziq and Be'er Resisim. Note various form of cross section.

east of Timna Valley (Site 149), and its archaeo-metallurgy is presently under study. Ancient mines were discovered nearby (Site 250), where the copper veinlets within the dolomites are widely spread. In these veinlets the main copper minerals are malachite, bisbeeite, chrysocolla and atacamite.

ANALYTICAL PROCEDURE AND SAMPLE PREPARATION

Major, minor and trace elements were determined by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES). The microstructure of polished and etched sections was studied using a metallographic microscope. Inclusions in the samples and in their peripheral margins were analyzed by Scanning Electron microscopy employed with Energy Dispersive X-ray Spectrometer (SEM-EDS).

Initially the samples were sectioned. A slice from each ingot was cut off, mounted in resin and polished. Aqueous ferric chloride solution was used for etching. An additional piece from each ingot was cleaned in acetone, and drillings were taken for chemical analysis. After acid dissolution, fifteen major, minor, and trace elements were determined using a procedure described in Segal *et al.* (1994). The limits of detection (LOD) for defined elements are listed in Table 1.

RESULTS AND DISCUSSION

1. Chemical analysis

The chemical composition of the ingots is given in Tables 2 and 3. Elements with content below the LOD are omitted. In addition to ICP-AES analyses, the copper, lead, and iron contents were determined using SEM-EDS, and the data compared well with those obtained by ICP. Silicon and sulphur were determined only by SEM-EDS. The data indicate that for all ingots lead and iron contents are significant varying widely in range 0.20–3.8% and 0.02–2.84% respectively. Arsenic, nickel and cobalt occur in all ingots as a trace elements; some samples contain traces of silver. Although in Timna and Feinan ores there are small amounts of lead, arsenic and silver; these elements are present in ingots. Explanation of that gave Merkel (1990) noted that remelting and refining of the black copper lead to the concentration of lead, arsenic and silver in the metal, not in the slag. Ingot 18 contains 0.3% P. Table 4 presents the medians for the ingots from 'Ein Ziq compared with the medians for other Bronze Age copper objects from Timna (Leese *et al.* 1986) and Feinan (Hauptmann *et al.* 1992). A compar-

Table 1. Limits of detection of determined elements (in solution).

Element	Wavelength, nm	L.O.D., ug/l
Sn	189.980	349.0
As	193.699	31.0
Mo	202.030	10.0
Zn	206.200	2.6
Pb	220.353	22.0
Co	228.616	5.7
Cd	226.502	1.7
Ni	231.604	11.0
Au	242.795	3.5
Mn	257.610	0.7
Fe	259.940	8.3
Cr	267.716	2.0
Cu	324.754	0.7
Ag	338.289	1.2
Sb	206.833	17.0

Table 2. Chemical composition of copper ingots from the Central Neguev, % wt.

Description	Lab. #	As	Ag	Pb	Ni	Co	Zn	Fe	Cu	Mn	Si	S
Be'er Resisim												
3214-387/111	9	0.044	0.004	2.26	0.065	0.058	0.048	0.48	96.90	0.0074		0.5
3004-365	10	0.038		0.62	0.033	0.006		0.15	98.72	0.0003		0.5
F-110-32	13	0.071		0.34	0.081	0.069		0.33	98.94	0.0006		1.1
3214-385	14	0.045		1.28	0.074	0.051	0.045	0.46	98.03	0.0011		0.5
'Ein Ziq												
85-779 78-443/11	17	0.110	0.006	0.95	0.072	0.042	0.076	2.30	96.20	0.0026		
85-812 78-443/10	8	0.021		0.54	0.065	0.098	0.150	1.80	95.04	0.0014	2.4	0.5
85-813 78-443/1	16	0.052	0.002	0.40	0.046	0.011	0.023	0.29	98.16	0.0004		0.6
85-814 71-338/1	15	0.053	0.001	1.70	0.045	0.006		0.02	97.60	0.0001	0.1	0.2
85-815 78-443/2	1	0.060	0.005	0.80	0.061	0.074	0.064	1.10	95.38	0.0014	0.6	2.2
85-816 78-443/7	6	0.060	0.002	0.20	0.019	0.004	0.005	0.30	97.90	0.0002		1.2
85-817 78-443/3	4	0.075	0.003	0.30	0.030	0.010	0.012	0.52	96.50	0.0006	0.3	1.7
85-818 78-443/9	5	0.052		0.70	0.105	0.062	0.056	1.70	94.80	0.0010		1.3
85-819 78-443/6	3	0.022		1.10	0.051	0.033	0.048	0.29	95.90	0.0015	0.4	1.3
85-820 78-443/12	7	0.084	0.002	1.20	0.074	0.089	0.068	2.40	95.94	0.0058	0.5	0.9
85-821 78-443/8	12	0.027	0.012	0.49	0.068	0.015	0.017	0.45	96.60	0.0012	0.8	0.5
85-822 78-443/4	2	0.072	0.004	0.80	0.057	0.033	0.025	1.48	97.51	0.0007	0.1	0.8
85-823 78-443/5	11	0.056		0.62	0.064	0.086	0.140	1.83	96.40	0.0030	0.4	0.9
61-293	18	0.072	0.007	0.26	0.054	0.053	0.121	2.50	95.54	0.0007	0.5	1.7
26-170	19	0.074		0.43	0.054	0.058	0.117	2.76	94.85	0.0008		0.5
84-446	20	0.029		0.81	0.048	0.037	0.022	0.73	98.49	0.0019		0.6

Note: Si and S were checked in SEM-EDS.

ison of these data with those cited in the literature for Timna copper objects shows that the MB I ingots from ‘Ein Ziq differ from the Timna Bronze Age copper objects. They are similar, however, to those from the Feinan region.

In order to ascertain the provenance of the ingots in the present study, we compared the composition of ingots, copper ores and slags from the Timna – MB I archaeological sites 149 and 250 (chemical data were provided by B. Rothenberg) with those from Feinan. The Co/Ni and Sb/As abundance ratios may be useful for this purpose. The fields of the Co/Ni and Sb/As ratios in copper ores from Timna and Feinan were defined by Hauptmann *et al.* (1992). Fig. 2 shows that the ratios for ores and slags are located in the DLS field. Thus it can be concluded that, with respect to these

Table 3. Chemical composition of uncleaned ingots form the Central Neguev, % wt.

#	As	Ag	Pb	Ni	Co	Zn	Fe	Cu	Mn
'Ein Ziq									
1	0.028	0.002	1.44	0.039	0.014	0.020	0.96	97.30	0.0038
2	0.022	0.002	0.62	0.044	0.018	0.021	0.11	98.45	0.0009
3	0.062		0.56	0.026	0.006	0.010	0.22	98.50	0.0003
4	0.042	0.002	0.34	0.031	0.001	0.017	0.31	98.50	0.0001
5	0.060		0.57	0.067	0.099	0.105	2.40	96.60	0.0033
6	0.055	0.006	0.48	0.125	0.098	0.043	2.04	96.54	0.0033
7	0.091	0.006	0.32	0.059	0.048	0.039	1.82	97.20	0.0020
8	0.051		0.66	0.057	0.090	0.126	2.84	96.12	0.0020
9	0.052		3.80	0.071	0.073	0.180	1.98	92.90	0.0170
10	0.086	0.006	0.34	0.091	0.035	0.029	1.35	97.81	0.0017
11	0.091	0.008	0.51	0.045	0.010	0.006	0.45	96.50	0.0004
12	0.054	0.005	1.35	0.055	0.054	0.031	0.63	96.84	0.0009
13	0.034		0.52	0.046	0.026	0.022	0.32	97.40	0.0016
14	0.054		2.02	0.085	0.119	0.095	2.76	93.81	0.0015
15	0.018		0.51	0.041	0.016	0.030	0.18	98.42	0.0002

parameters, Timna Formation ores are similar to those from Feinan DLS and we cannot discriminate between these two ore sources. Fig. 2 also shows that the above-mentioned ratios, obtained in the present study for the ingots, are also located in the same region, suggesting that the ingots were manufactured either from MB I Timna or Feinan DLS ores. We must note that the data on researches of the ceramic artifacts from these MB I sites from the Negev speak in favour of Feinan because they were related to their equivalents in Transjordan (Goren *et al.* forthcoming). Contrary to that the lead isotope ratios of an ingot from 'Ein Ziq, determined by Stos-Gale from the Isotrace Laboratory at Oxford, 'was fully consistent with its origin from Timna ores' (Stos-Gale 1991). It is unclear, however, which copper ores were employed for defining lead isotope ratios field: ores from the Amir-Avrona or Timna Formations. This aspect will be examined in greater detail in the future.

2. Metallography

2.1 Crust

The next step in our research was the metallurgical study of the ingots and their crust. Roman (1990) studied Timna ingots and noted that the metallographic examination of the crust can reveal important information on provenance and smelting technique. Almost all our ingots evince a thin upper and, in several cases, a lower crust. The upper crust is more porous than the metal and comprises several layers (fig. 3); ingot 17 was exceptional in lacking crust. The external layer

Table 4. Medians of several elements in Bronze Age copper objects from Timna (Leese *et al.* 1986), Feinan (Hauptmann *et al.* 1992) and from the Central Negev (this study).

Element	Timna	Feinan	Central Negev
Ag	0.016	0.001	0.002
As	0.068	0.033	0.054
Co	0.002	0.030	0.042
Ni	0.031	0.025	0.047
Zn	0.008	0.011	0.031
Pb	0.303	0.290	0.620

consists of iron-free copper oxide (cuprite), which was underlain by lead layer, and at last copper metal itself. Fig. 4 shows how the lead penetrates between the copper grains and intrudes to the surface, being partly or completely absent inside (compare figs. 4 and 5). This phenomenon apparently occurred during slow solidification due to low melting point of lead; it solidifies at the end of the congealing process. Lead layer in the crust occurs only in ingots with triangular and round form of cross section. Contrary to that in ingots having thin elongated section all the lead is dispersed throughout the ingot (fig. 6) and it is no lead layer in the crust. This is the result of rapid solidification due to thin form of mould.

The porous crust is filled with slag material of variable composition, e.g., various silicates in ingots 2 and 3; barite and silver inclusions occur in the crust of ingot 9. Relatively large inclusions of wustite and iron phosphide, probably remnants of slags, and apatite are located between the external Cu-oxide and internal layers in ingots 7, 11 and 18. The presence of manganese in iron-rich slag-like material and arsenic in lead-rich globular inclusions is noteworthy. The thin lower crust, when is present, contains quartz inclusions, a small amount of copper oxide and globular lead-copper inclusions.

It was mentioned previously that the ores used for smelting in MB I were either from the eastern region of Timna (Site 250) or from Feinan, located in Transjordan to the northeast of Timna. Both ores contained chrysocolla, malachite and a small amount of chalcocite, a product of incompletely oxidized ore. Manganese minerals occur in these ores together with barite, silver and apatite. The composition of the slag remnants

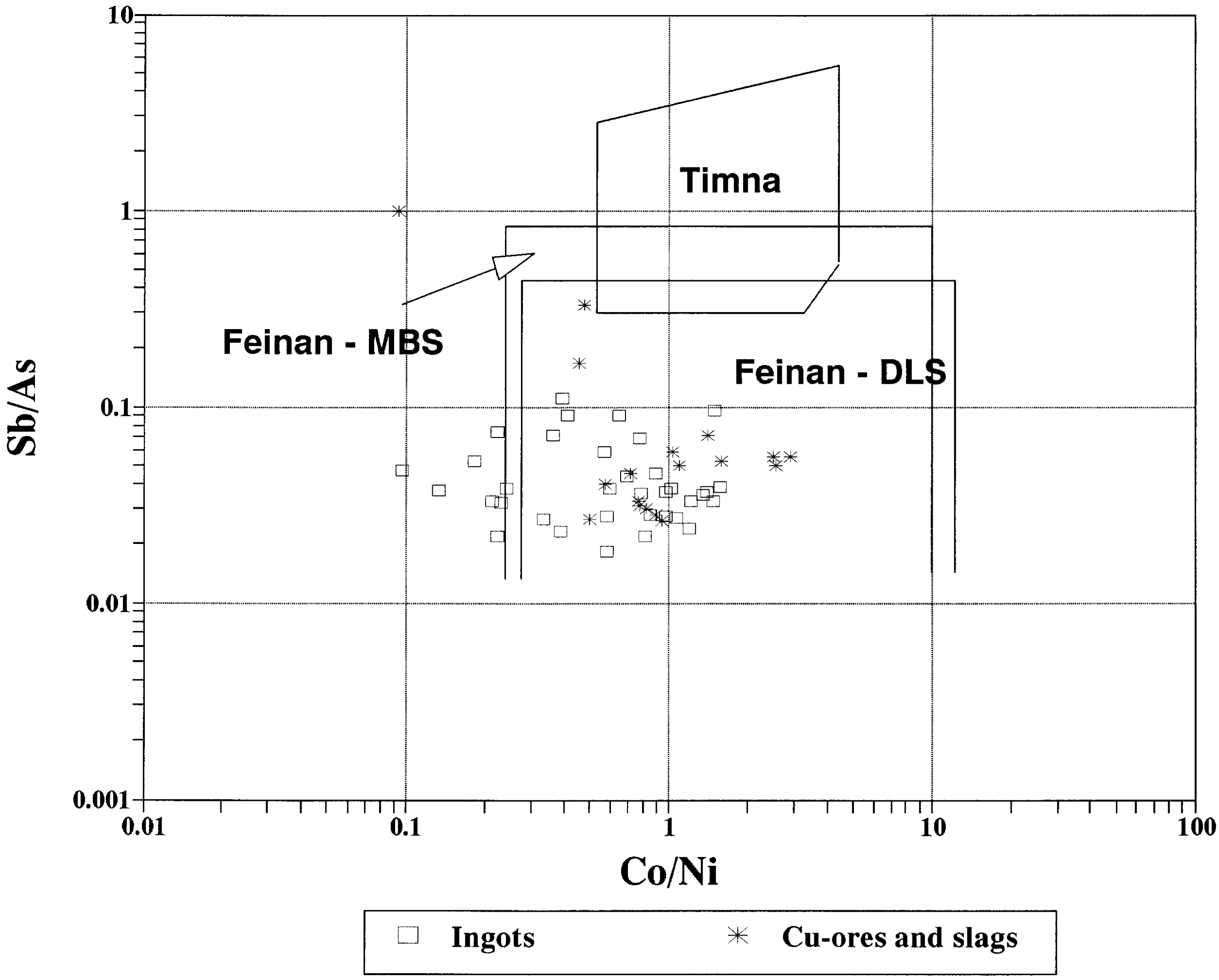

Fig. 2. Comparison of the Co/Ni and Sb/As ratios in ingots from the Negev and in Cu-ores and slags from Timna sites 149 and 250 (this study) with the ore fields as defined by Hauptmann *et al.* (1992).

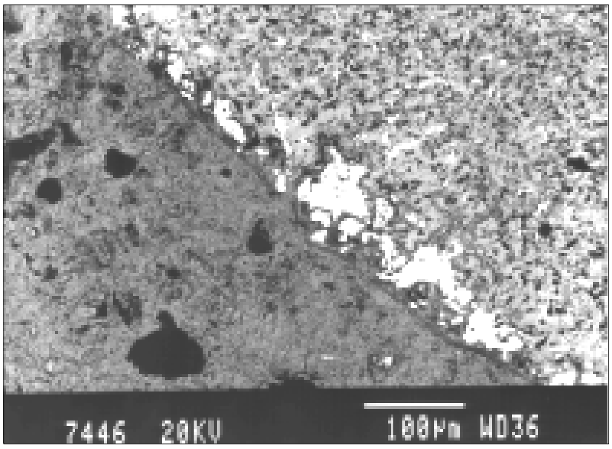

Fig. 3. Typical upper crust structure: external gray layer is copper oxide, white – lead and internal light gray layer is copper. Dark gray and black inclusions are remnants of the slag (BEI).

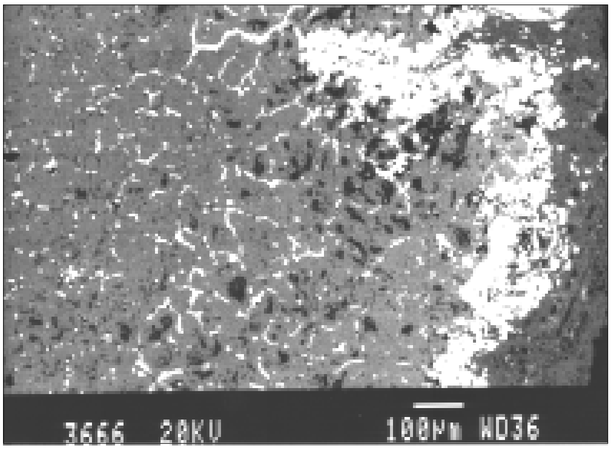

Fig. 4. Penetration of lead (white) through the copper grains from middle part of the ingot to the surface (Ingot 13, for example, BEI).

in the crust suggests that the ingots were produced by smelting this type of ore, followed by casting in a mould.

2.2 Metal

Backscattered electron image (BEI) of the samples revealed granual structure even without etching, because of presence of various inclusions between the grains; after etching all the samples exhibited an equiaxial grain structure with some random elongation clearer (fig. 7). The grain boundaries are decorated with copper sulphides, copper-iron sulphides and lead inclusions (fig. 8). Metallic lead also occurs within the copper grains and copper sulphide inclusions as small spherules (fig. 9). Occasionally lead is associated with arsenic. Presence of rare lead sulphide inclusions indicates that some galena occurred in the ores. Remnants of copper sulphide show that the oxidized copper ore, from which the ingots were originally produced, did not undergo complete oxidation.

In the case of three ingots (ingots 2, 5 and 6), the ferric chloride etchant was not effective. When etched with sodium dichromate, large grains covered by numerous spherical and splatter inclusions were observed. In ingots 2 and 5 it was copper sulphides containing black iron oxide inclusions (figs. 10 and 11). The shape and composition of these clusters in ingot 6 is typical of a Cu_2O-Cu eutectic microstructure (figs. 12 and 13). Four of the ingots (7, 11, 18 and 19) contained iron phosphide inclusions and elemental phosphorus in the copper matrix. Iron was probably present in the copper ores as hematite, or iron ore was added to the copper ore as a flux. Microhardness of the ingots varied in the range of 58–106 HV.

Equiaxial grain structure, shrinkage, exhibiting on the uppermost surface of the ingots, their smooth surface provide evidence that the metal was cast and that the moulds were carefully prepared and well insulated. Both by the chemical composition and by the structure ingots from ‘Ein Ziq and Be’er Resisim are similar to those from Har Yeruham, Hebron Hills (Maddin and Stech Wheeler 1976) and from Har Sayyad

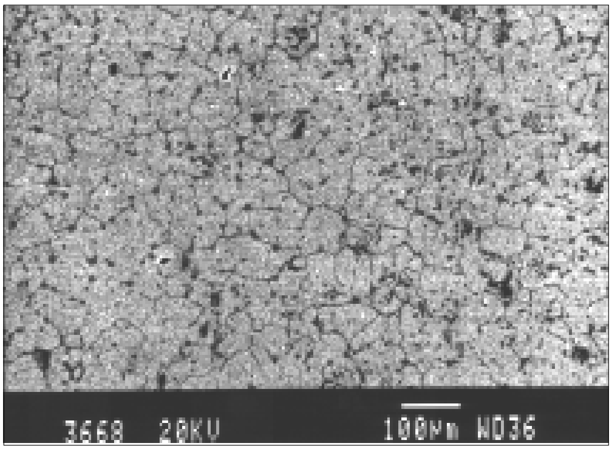

Fig. 5. Structure of the middle part of Ingot 13 with wide triangular section showing absence of lead inside of it (BEI).

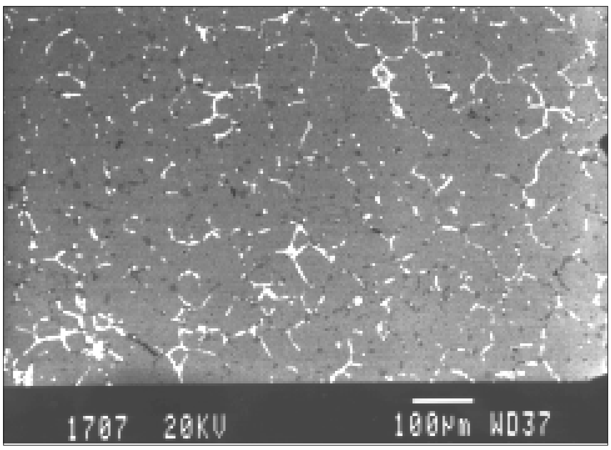

Fig. 6. Structure of ingots with thin elongated form of section. Lead is distributed throughout the ingot (BEI).

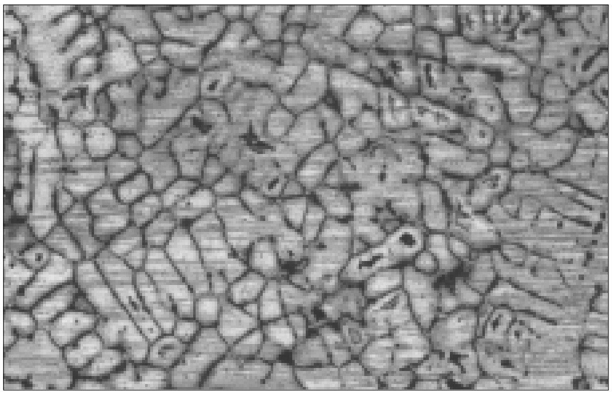

Fig. 7. Equi-axed grain structure of ingots showing that they were cast in the mould, metallographic microscope, x125.

(Segal *et al.* in preparation). Unfortunately, there were found no moulds at these sites, so it seems to be that the ingots were manufactured in the areas closed to ore sources, e.g., it were workshops of wide profile – from smelting of raw metal to casting in the moulds. Were these workshops dispersed or located at one settlement is still a question. But it is clear that the source and the technology of manufacturing of all ingots were the same.

CONCLUSIONS

1. There is no difference in either chemical composition or metallography between the ingots from Be'er Resisim and 'Ein Ziq. Moreover they are similar to those from the other sites in the region.

2. The analysis of the overall chemical composition indicates that the copper ore used in the primary smelting did not originate in Timna's Amir-Avrona Formation, which was exploited from the Chalcolithic Period. The MB I smelters derived their ore from the Timna Formation that are similar to Feinan DLS; these ores are present at both Timna and Feinan. We did not find any differences in the content of trace elements between these two sources.

3. The presence of small amounts of copper sulphide (chalcocite) and copper iron sulphide indicates that the copper ores used for the smelting were incompletely oxidized. The iron content varies from one ingot to another. It occurs in the copper sulphides, as well as in the copper matrix, and sometimes as separate iron oxide and phosphide inclusions. Either charge contained hematite veins in the copper ores, providing a self-fluxing raw material; or that iron ores located nearby were added to copper ores as a flux.

4. Lead is insoluble in copper, therefore it is dispersed inhomogeneously throughout the metal. Lead inclusions are located mainly in the grain boundaries as metal. Occasionally, lead is present as sulphide and copper lead sulphide. Remnants of the latter indicate that the copper ore contained lead sulphide (galena). In the copper sulphide surrounding, it presents as small metallic spherules.

5. All the ingots are covered by a crust, which appears mainly on their upper surface. There are several layers on the ingot surface: an external layer of copper oxide; a middle layer of metallic lead, which rose to the surface in the solidification process; an innermost layer of copper containing various inclusions. Lead layer occurs only in triangular and rounded ingots; in the ingots having thin elongated form of cross section lead is dispersed over the ingot volume. The slag remnants in the crust show that the black copper used for casting was insufficiently clean, i.e., it did not undergo refining. The study of the crust composition was useful for ascertaining the metal's provenance and pointed out two possible ore sources – Timna and Feinan.

6. An equiaxial grain structure and minor shrinkage on the upper surface of the ingots show that they were cast in well insulated moulds of various form of section.

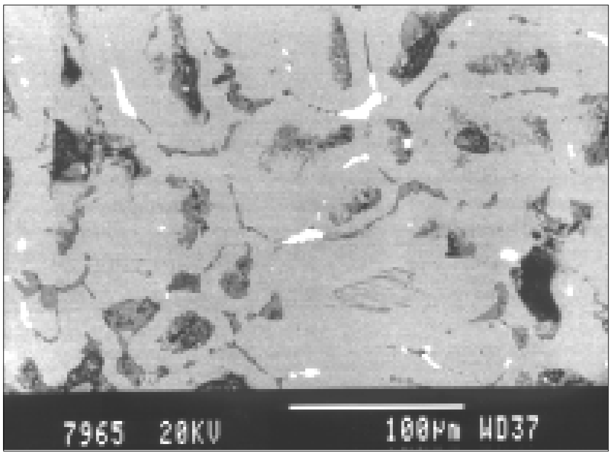

Fig. 8. Same as fig. 7 showing presence of copper sulphide (gray) and lead (white) inclusions in the grain boundaries (BEI).

Acknowledgments

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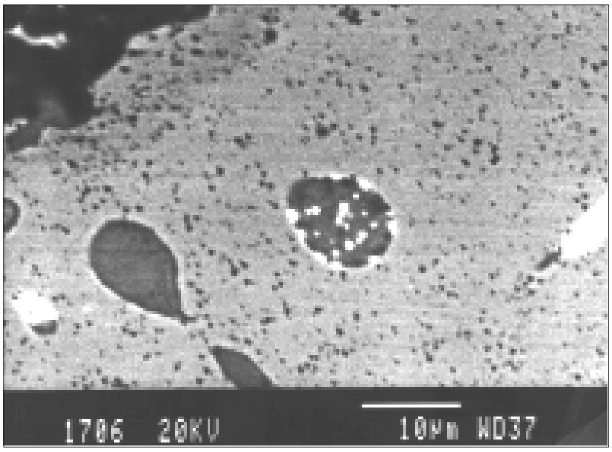

Fig. 9. Complicated structure of copper sulphide inclusions with numerous small lead spherules inside (BEI).

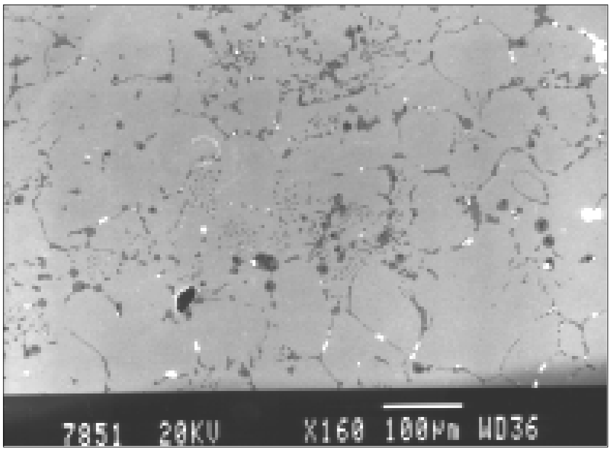

Fig. 10. Etched surface of ingots 2 and 5 revealing presence of black iron oxide inclusions within copper sulphide. Rare white droplets are lead (BEI).

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Rudolph Cohen was born in 1939 in Italy. He completed his PhD in 1986 at the Hebrew University of Jerusalem on the subject: The Settlement of the Central Negev in the Light of Archaeology and Literary Sources during the 4th–1st Millennia BCE. *His main excavations are the 'Central Negev Highlands Project', Kadesh-Barnea Horvat, 'Ein Ziq and 'En Hazeva. His specialities are concerned with the Middle Bronze Age I, the Iron Age, the Roman Period, and the Nabatean Period. His current position is the Deputy Director of the Israel Antiquities Authority.*

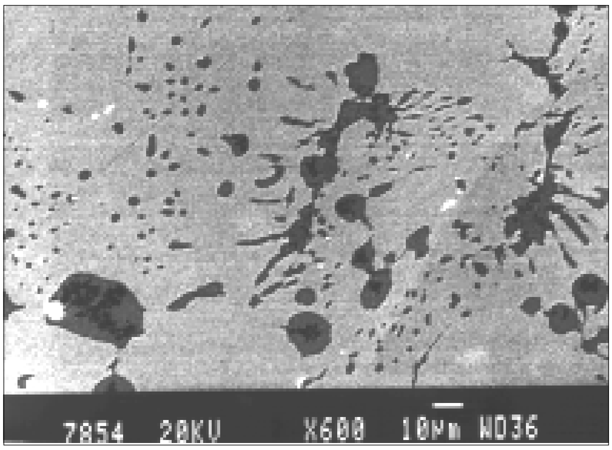

Fig. 11. Enlargement of fig. 10.

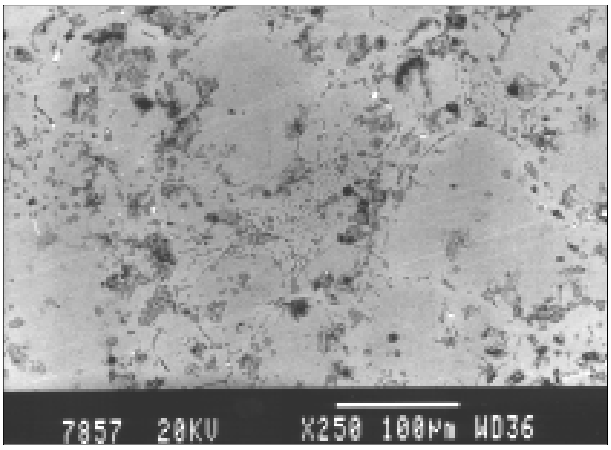

Fig. 12. Surface of Ingot 6 showing Cu_2O-Cu eutectic structure (BEI).

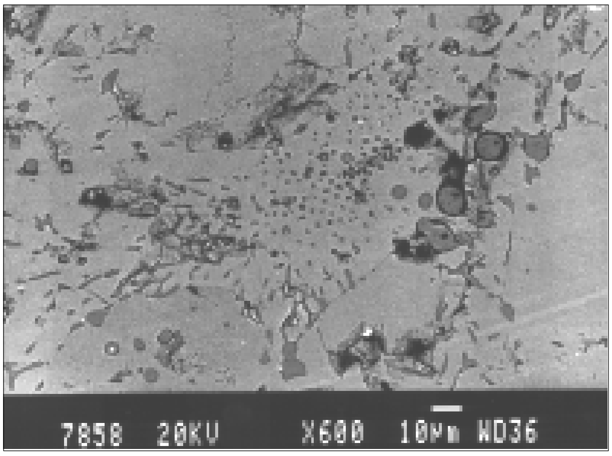

Fig. 13. Enlargement of fig. 12.

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