ORIGIN OF COPPER USED IN BRONZE ARTEFACTS FROM MIDDLE BRONZE AGE BURIALS IN SIDON: A SYNTHESIS FROM LEAD ISOTOPE IMPRINTS AND CHEMICAL ANALYSES

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Introduction

Burials from the Middle Bronze Age (2000-1550 BC) have been explored in Sidon by the British Museum and the Department of Antiquities since 1998 [1]. Here we present a synthesis of chemical and isotopic analyses performed on copper artefacts (named "bronzes" according to the regional archaeological terminology) found in these Sidon burials. These artefacts include weapons (daggers, knife, arrowhead, spearhead), jewels (torque, belt) and some miscellanea (pin). Our goal was to determine the geological origin of copper used to manufacture bronzes so as to provide some insights on economical exchanges. Elemental analyses allows the verification of the metal composition of the bronzes and any contamination (from soils, corrosion or secondary melting) that would affect the reliability of lead isotope imprints. Meanwhile trace and major element concentration patterns alone are not accurate enough to clearly determine the geographical origin of metal deposits used to produce bronze artefacts due to smelting heterogeneities [2]. In order to be truthful, geographic markers should remain unchanged by smelting and corrosion processes. Lead (Pb) stable isotopes (^{206}Pb, ^{207}Pb, ^{208}Pb) provide reliable imprints that can be used to distinguish ore districts possibly in operation in the Mediterranean basin during the Bronze Age [3]. Indeed, lead isotopes are end-members of the radioactive uranium (U)-thorium (Th) (^{238}U, ^{235}U, ^{232}Th) natural decay chains. The relative proportion of lead isotopes in copper (Cu), silver (Ag) and lead ores shall vary according to the age of formation of the ore body and their initial U-Th content [4]. Therefore most ore bodies will display lead isotopic imprints that can be traced in bronze artefacts and which then allow the determination of the geographic origin of the ores used to produce said artefacts. These results are expected to provide complementary insights on the economical exchanges during the Middle Bronze Age in Sidon. Bronze artefacts from burial 4, 5, 12, 13, 42, 66 and 67 have already been discussed in Véron *et al.*, (2009) and Le Roux *et al.*, (2004, 2009) according to lead ore signatures only. Here, we have revisited these findings and new artefacts (table 1) on the basis of combined lead and copper ore bodies that sometimes display different imprints.

Methods

Table 1 Artefacts from Sidon's burials with corresponding lead isotope signatures and captions used in fig. 1 to 4.

Sample	Artifact	Burial	Age	Caption	$^{206}Pb/^{207}Pb$	$^{208}Pb/^{206}Pb$
34238/1734	Knife	4	MB IIB	✚	1.1751	2.0959
34242/1744	Spearhead	5	MB IIA	✚	1.1260	2.1220
34234/1747	Axe-head	5	MB IIA	✚	1.1588	2.1031
34240/1820	Axe-head	12	MB IIA	●	1.1863	2.0822
34241/1820	Axe-head	12	MB IIA	●	1.1693	2.1133
34244/1825	Spearhead	13	MB IIA	●	1.1923	2.0746
3617/2079	Belt	42	MB IIB	◆	1.2032	2.0690
3619/2079	Belt	42	MB IIB	◆	1.2025	2.0706
3580/2079	Torque	42	MB IIB	◆	1.1860	2.0836
3544/2071	Spearhead	42	MB IIB	◆	1.1828	2.0848
3541/2068	Dagger	42	MB IIB	◆	1.1807	2.0854
2605/1846	Dagger	66	MB II	◆	1.1842	2.0815
2715/1906	Knife	67	MB IIA-B	◇	1.1852	2.0850
2916 / 1916	Spearhead	69	MB IIA-B	●	1.1823	2.0826
2989/1917	Spearhead	70 A	MB IIA	✦	1.1824	2.0860
4210/1945	Knife	70 B	MB IIA	✦	1.2083	2.0292
4056/1924	Dagger	74	MB IIA	✦	1.1869	2.0815
4140/1924A	Knife	75	MB II A	✦	1.1722	2.0927
4140/1924B	Knife	75	MB IIA	✦	1.1866	2.0816
4117/1940A	Spearhead	78	MB IIA	○	1.1900	2.0777
4117/1940B	Spearhead	78	MB IIA	○	1.1951	2.0702
4149/1940A	Axe-head	78	MB IIA	○	1.1788	2.0870
4149/1940B	Axe-head	78	MB IIA	○	1.1788	2.0871
4574/6037	Knife	100	MB IIB-C	○	1.1867	2.0820
5341/6056	Dagger	107	MB IIA-B	○	1.1994	2.0460
5363/6056	Pin	109	MB IIA-B	□	1.1694	2.0940
5320/2304	Fish hooks	Room 1	EBA	●	1.1959	2.0664

All artefacts were treated with mechanical (brushing, scalpel scrapping) and chemical (corrosion inhibitor and Benzotriazole stabilizer) procedures by Isabelle Skaf. Artefact subsamples were acid leached (HCl-HNO_3) and rinsed (MilliQ water) in order to remove soil and corrosion residues. Trace and major elements were measured by ICP-OES (Varian Vista-MPX Iris Intrepid 2 -Thermo Electron) after concentrated acid digestion (HNO_3) and are presented in table 2 (concentrations are in %, u.l. is used for concentrations "under detection limit"). Aliquots for stable lead isotope analyses were purified on an AG1X8 anionic exchange resin [5]. Isotopic ratios ($^{206}Pb/^{207}Pb$, $^{208}Pb/^{206}Pb$, Table 1) were determined by Multi-Collector-Inductively Coupled Plasma Mass Spectrometry (MC-ICPMS) (ISOPROBE, UQAM, Montréal) at GEOTOP. Calibration and mass fractionation were corrected with concurrent analyses of thallium and the SRM981 NIST standard. Standard deviation for lead ratios is 0.01%.
Different aliquots (A and B) were analyzed for three samples 4140/1924 (burial 75), 4117/1940 and 4149/1940 (burial 78). The latest aliquots are in perfect isotopic agreement, while the two others show significant discrepancies (table 1). This could result from either the artefact heterogeneity (ore mixing or secondary smelting) and/or soil

corrosion contamination. The calcium (Ca) enrichment of aliquot 4140/1924A (Ca 29%, table 2a) could suggest soil contamination that could be invoked to explain the isotopic discrepancy between aliquots 4140/1924B ($^{206}Pb/^{207}Pb$ = 1.1866) and 4140/1924A ($^{206}Pb/^{207}Pb$ = 1.722) (table 1), in spite of acid cleaning of the samples. Aliquot 4117/1940B ($^{206}Pb/^{207}Pb$ = 1.1951) is enriched in silver

Sample	Burial	Cu (%)	Ag(%)	As (%)	Au (%)	Fe (%)	Pb(%)	Sb(%)	Sn(%)	Zn(%)	Al(%)	total % ("Metals")
2916 / 1916	69	72.95			n.d.	1.29						74.24
2989/1917	70 A	87.13	1.4		n.d.	0.54	traces				0.84	89.89
4210/1945	70 B	72.62	1.4		n.d.	0.06	traces		20.59			94.64
4056/1924	74	94.98			n.d.	0.36	traces				0.20	95.55
4140/1924A	75	64.29	0.6	0.233	n.d.	1.31	traces				1.62	68.03
4140/1924B	75	95.17			n.d.							95.17
4117/1940A	78	88.21	3.5	0.408	n.d.	0.83					0.36	93.27
4117/1940B	78	96.69		0.048	n.d.	0.05					0.05	96.83
4149/1940	78	96.31			n.d.		0.2081					96.52
4149/1940	78	95.37	0.2		n.d.		1.15015					96.70
4574/6037	100											
5341/6056	107	95.36		1.486	n.d.	2.05	traces				0.13	99.03
5363/6056	109	94.81			n.d.				0.6		0.86	96.27
5320/2304	Room 1	92.60		0.220	n.d.	1.37					1.42	95.61

Table 2 a Chemical composition of the bulk artefact including elements brought by soil contamination. *% of major and minor elements compared to the sum of these elements.*

(Ag) as compared to aliquot 4117/1940A ($^{206}Pb/^{207}Pb$ = 1.1900). We also observe a silver enrichment for 4140/1924A (0.6%, table 2a) as compared to 4140/1924B. These could be explained by artefact heterogeneities due to the mixing of ores of different origin and shall be discussed on the basis of isotope systematics.

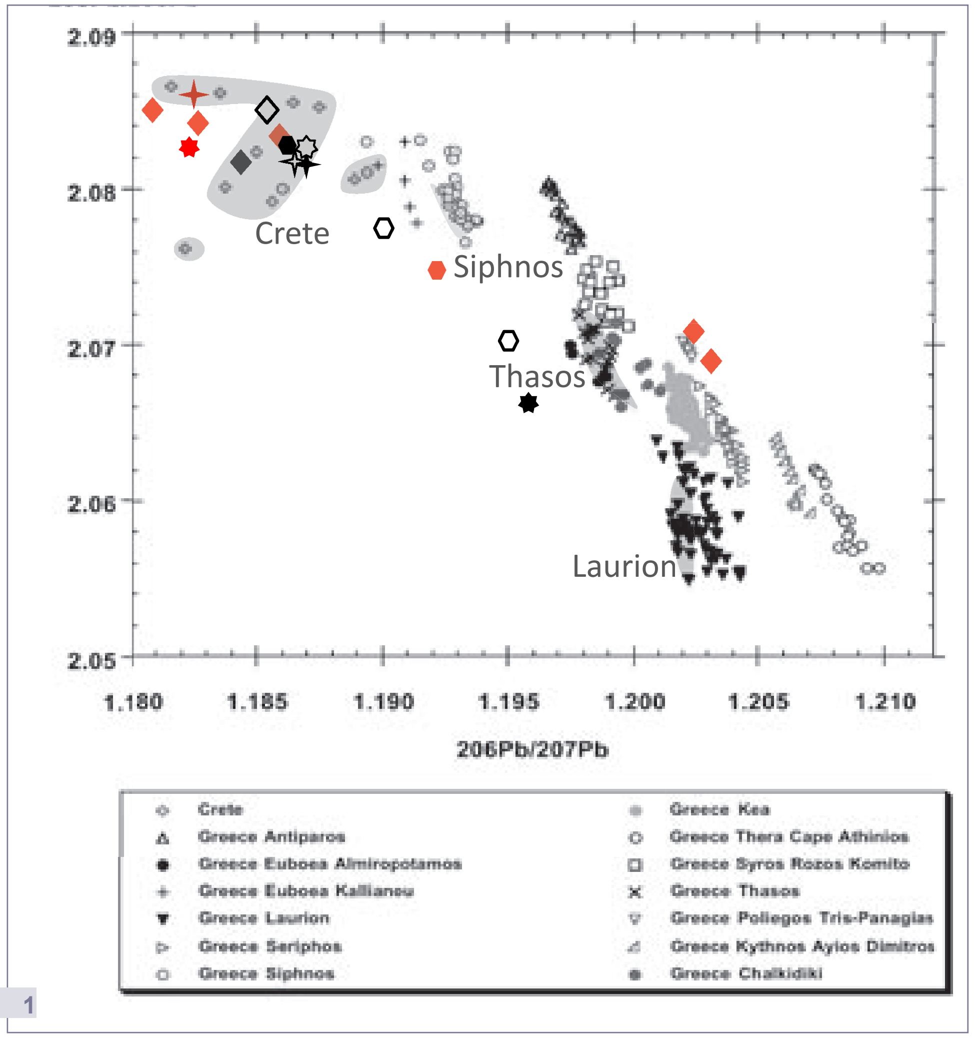
Chemical composition

The composition of the new samples is given in table 2 a, including minor and trace elements allowing the identification of corrosion and soil inclusions (see above), which will be further used in the lead isotope analyses.
Table 2 b shows a synthesis of metal compositions found in Sidon's artefacts [6] including iron and aluminium as possible tracers of soil inclusion and corrosion. The new results confirm previous measurements (table 2 b), which show that most of the artefacts are made of copper without intentional alloying. Only one sample (4210/1945) is a very high tin bronze, whereas other samples are low tin bronzes (2605/1846, 2715/1906, 5363/6056). There is not a clear chronological pattern based on metal composition. Arsenic is present in 5 samples, again without any clear pattern. There is no clear selection of alloy by function as hypothesized by Philip *et al.*, (1991) for weapons from EBA in Palestine.

Lead Isotope systematics

Lead isotope ratios are shown in table 1, and are compared to copper and lead ore body imprints from Crete, Greece (fig. 1), Turkey (fig. 2), Oman, Sardinia, Spain, Italy (fig. 3) and Cyprus, Egypt, Southern Levant, Iran-Iraq, Syria (fig. 4). Because of the copper content of the artefacts, available copper ores were represented along with lead ore signatures for each region (grey or red shaded areas in figures with corresponding location). Both copper and lead ore imprints generally overlap except for the Taurus region in Turkey (fig. 2) and for Egyptian ores (fig. 3). The geographic origin of copper found in bronze artefacts, based on lead isotope imprints, is presented in table 3 where each possible ore body origin is marked with crosses (one to three depending on increasing matching imprints between the artefact and the ore signatures in figures 1 to 4). The three most common origins are Cyprus, Oman and Crete (Tab. 3). No other obvious geographic source is evidenced for these artefacts, except for one sample likely originating from the Southern Levant region in burial 5 (34234/1747). While Feinan and Timna (Southern Levant) have been extensively mined for copper since the middle of the third millennium BC [7] and throughout Antiquity, almost none of these ores are found in Sidon. With the exception of burial 5 (34234/1747), the Southern Levant ores do not appear to contribute much to ore sources in Sidon artefacts during the Middle Bronze Age. The two artefacts found in burial 5 have a different origin as fingerprinted by Pb isotopes. This confirms the essential role of this burial in Near Eastern archaeology, where the structure of this burial

Mg(%)	Mn(%)	Na(%)	P(%)	S(%)	Si(%)	Ca(%)
1.47					11.37	12.91
1.07			0.43	0.12	1.41	7.09
0.21			1.34		0.88	2.92
0.78					1.16	2.51
1.09			0.35	0.11	0.84	29.57
0.62					2.16	2.05
0.31					3.52	2.90
0.51			0.20	0.05	0.19	2.21
0.63					0.77	2.09
0.53			0.27		0.80	1.71
0.07			0.12	0.32	0.03	0.42
0.75					1.07	1.91
0.62			0.35		0.39	3.03



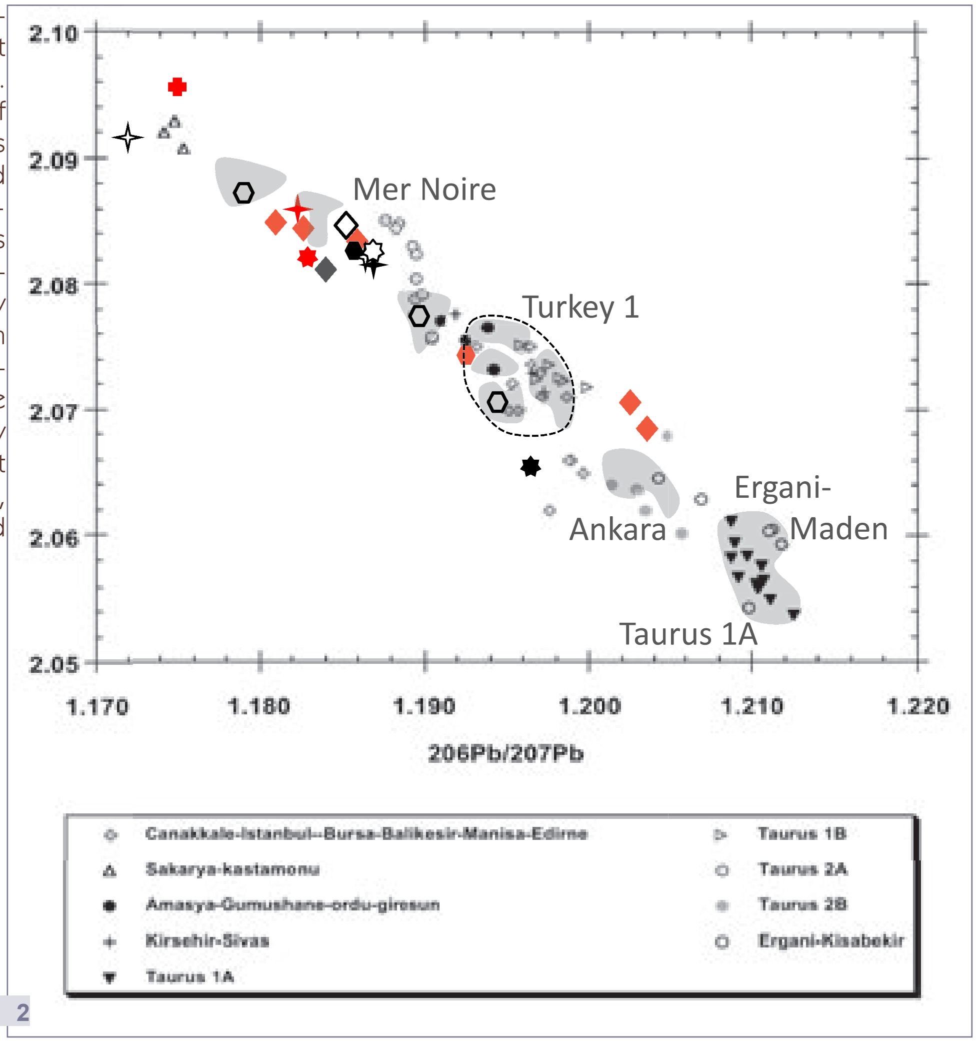
1 Comparison of isotopic imprint ($^{208}Pb/^{206}Pb$ vs. $^{206}Pb/^{207}Pb$ ratios) of burial bronze artefacts to those of Greece and Crete lead (see symbols and corresponding locations in caption under figure) and copper (grey shaded areas with corresponding location within figure) ore deposits.

Sample	Burial	Cu (%)	Ag(%)	As (%)	Au (%)	Fe (%)	Ni(%)	Pb(%)	Sn(%)	Zn(%)	Al(%)
34238/1734	4	97.15	ul	0.2	ul	2.11	0.02	0.12	0.12	0.02	0.25
34242/1744	5	97.24	ul	0.03	ul	0.23	0	2.28	0.11	0.01	0.11
34234/1747	5	97.16	ul	0.04	ul	1.3	0.01	0.1	0.5	0.03	0.88
34240/1820	12	93.66	ul	0.02	ul	2.76	0.01	0.66	1.39	0.02	1.49
34241/1820	12	98.31	ul	0.03	ul	0.35	0	0.03	1.16	0	0.11
34244/1825	13	94.49	0.17	0.11	ul	0.86	0	0.2	3.78	0.04	0.35
3617/2079	42										
3619/2079	42										
3580/2079	42										
3544/2071	42										
3541/2068	42										
2605/1846	66	94.63	ul	0.06	ul	0	0.14	0.01	0.23	ul	4.93
2715/1906	67	91.86	0.01	0.42	0	0.01	0.34	0.01	0.54	ul	6.7
2916 / 1916	69	98.26			n.d.	1.74					
2989/1917	70 A	96.93	1.54		n.d.	0.60		traces			0.933
4210/1945	70 B	76.74	1.44		n.d.	0.07		traces	21.76		
4056/1924	74	99.41			n.d.	0.38		traces			0.210
4140/1924A	75	94.50	0.86	0.34	n.d.	1.92		traces			2.379
4140/1924B	75	100			n.d.						
4117/1940A	78	94.58	3.70	0.44	n.d.	0.89					0.390
4117/1940B	78	99.85		0.05	n.d.	0.05					0.049
4149/1940A	78	99.78			n.d.			0.22			
4149/1940B	78	98.63	0.18		n.d.			1.19			
4574/6037	100				n.d.						
5341/6056	107	96.30		1.50	n.d.	2.07		traces			0.129
5363/6056	109	98.48			n.d.				0.62		0.896
5320/2304	Room 1	96.85		0.23	n.d.	1.43					1.485

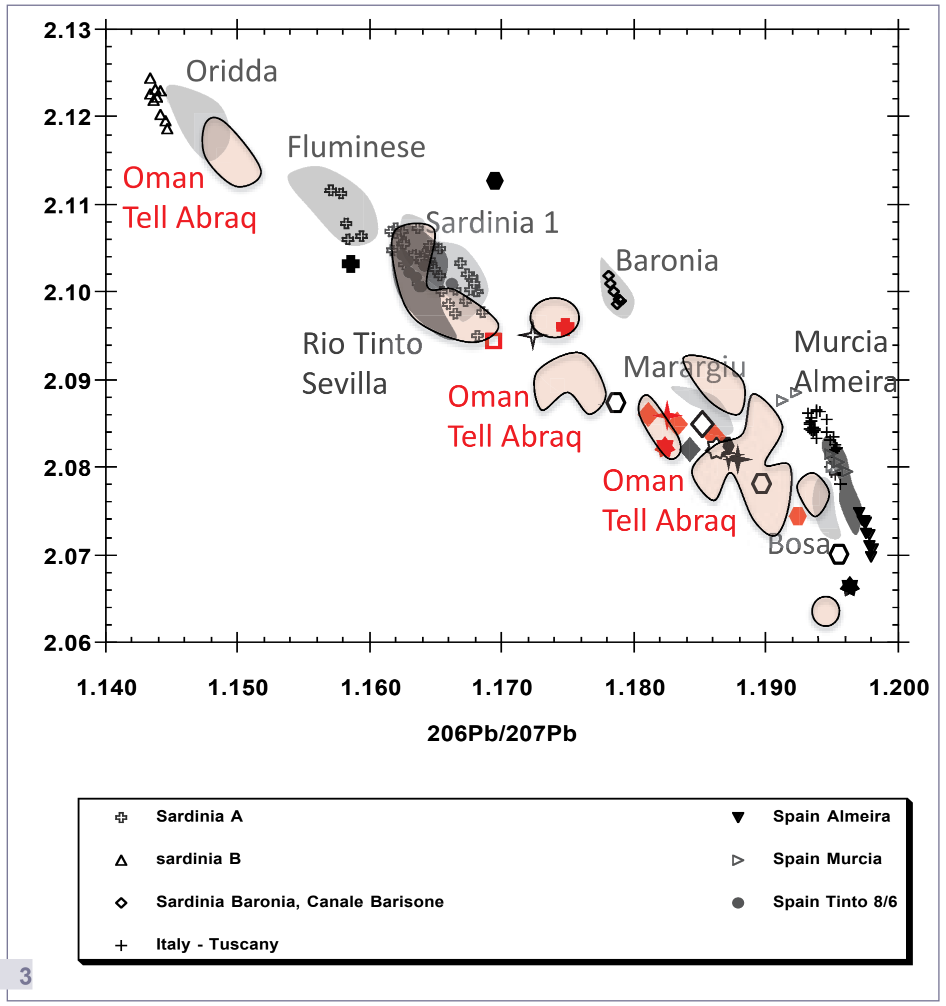
Table 2 b Synthesis of metal composition of bronze artefacts found in Sidon's burial (this study, A. Le Roux *et al.*, 2004, 2009). *% of major and minor elements compared to the sum of " metals" to allow comparison with previous results.*

was linked to others from Middle Euphrate, Oman and Mesopotamia [8]. The destination for the immense quantities of copper mined in the Southern Levant still needs to be elucidated. The silver enrichment in artefact 4117/1940A (table 2b) is associated with a Turkish origin (table 3). Another artefact from burial 78 (4149/1940) also displays an isotope imprint from Turkey. These results strengthen the possible and quite unique Turkish origin for the ores used in burial 78 artefacts. Such Turkish origin was also evidenced for silver artefacts found in burial 27 [9]. Because of isotope imprint overlap, some ore sources could not be identified (Tab. 3). This is particularly true for artefacts 34240/1820 (burial 12), 4056/1924 (burial 74) and 4574/6037 (burial 100). "Other" is noted when no ore body imprint could match the artefact (Tab. 3). Trace element analysis can help resolve some of these source uncertainties. Indeed, the four artefacts for which no geographic ore imprint could be determined are characterized by specific content of tin (21.76% and 1.16% for 4210/1945 and 34241/1820, table 2b), lead (2.28% for 34242/1744, table 2b) and iron (2.07% for 5341/6056, table 2b). The latest evokes a corrosion problem while the others indicate either an alloy or metal recycling.
Like for the chemical composition of artefacts, there is no clear pattern between provenance determined by lead isotope analyses and

2 Comparison of isotopic imprint ($^{208}Pb/^{206}Pb$ vs. $^{206}Pb/^{207}Pb$ ratios) of burial bronze artefacts to those of Turkey lead (see symbols and corresponding locations in caption under figure) and copper (grey shaded areas with corresponding location within figure) ore deposits. The "Turkey 1" copper ore imprint includes Sudan, Ankara, Sivas and Taurus 1B.

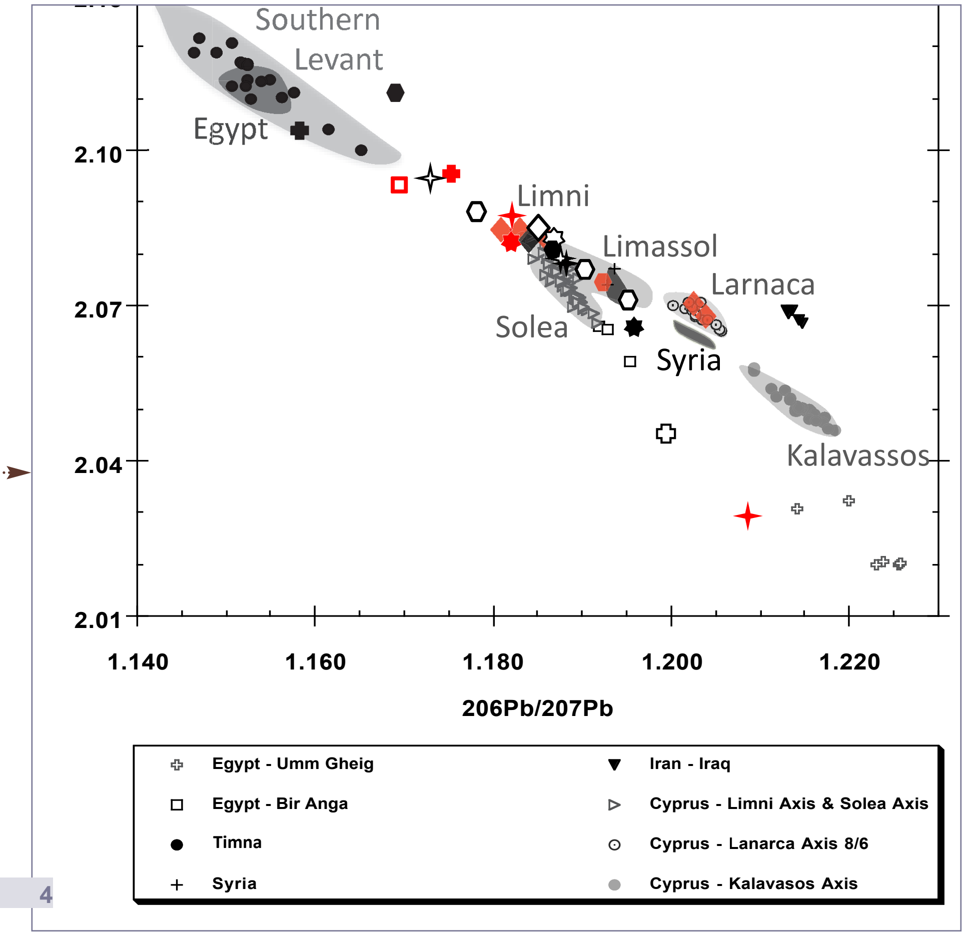


3 Comparison of isotopic imprint ($^{208}Pb/^{206}Pb$ vs. $^{206}Pb/^{207}Pb$ ratios) of burial bronze artefacts to those of Oman-Tell Abraq, Sardinia, Spain and Italy lead (see symbols and corresponding locations in caption under figure) and copper (grey and red shaded areas with corresponding location within figure) ore deposits. Lead ore imprints from Sardinia A and B include regions from: (A) Sulcis, Iglisiente, Barbagia, Solas, Arburese, Fluminense and (B) Iglisiente, Masua Oridda, S. Giovani. Sardinia 1 copper ores embrace Arburese, Barbagia and Sulcis.



chronology of the burials, site or object function. Copper provenance appears mainly to come from the Oman region, Cyprus, and, to a lesser extent, Crete whereas copper use from other closer sources like Syria or the Southern Levant seems scarce. If the Oman region and Cyprus are well-known sources for copper ores, use of copper from Crete is not often reported and should be further considered using an additional lead isotope (^{204}Pb) as well as additional data on ores and artefacts.

4 Comparison of isotopic imprint ($^{208}Pb/^{206}Pb$ vs. $^{206}Pb/^{207}Pb$ ratios) of burial bronze artefacts to those of Cyprus, Syria, Iran-Iraq, Egypt and Southern Levant lead (see symbols and corresponding locations in caption under figure) and copper (grey shaded areas with corresponding location within figure) ore deposits. The Southern Levant copper field includes ores from Feinan, Timna and Wadi Faynan. Data for figures 1, 2, 3 and 4 are from L. Barnes *et al.*, 1974; F. Begemann *et al.*, 2001, 2010; F. Cattin, 2008; V. E. Chamberlain and N. H. Gale, 1980; N. H. Gale *et al.*, 1981, 1988; 1996; B. Hamelin *et al.*, 1988; A. Hauptmann, 1992; Y. Hirao *et al.*, 1995; M. Hunt-Ortiz *et al.*, 2003; V. Koppel 1997; E. Pernicka *et al.*, 1984, 1990; G. Philip, 1991; G. Philip *et al.*, 2003; C. Pomies *et al.*, 1998; J. F. Santos Zaldegui *et al.*, 2004; E. V. Sayre *et al.*, 1992, 2001; B. Scaife, 1997; T. C. Seeliger *et al.*, 1985; E. T. C. Spooner and N. H. Gale, 1982; Z. A. Stos-Gale and N. H. Gale, 1981, 1992; Z. A. Stos-Gale *et al.*, 1981, 1984, 1985, 1986, 1990, 1995, 1996, 1997; I. G. Swainbank *et al.*, 1982; M. Vavelidis *et al.*, 1985; G. A. Wagner *et al.*, 1985, 1986, 2003; L. R. Weeks, 1999; K. A. Yener *et al.*, 1991.



Sample	Burial	Oman Tell Abraq	Cyprus	Crete	Turkey	Southern Levant	Sardinia	Other *
34238/1734	4	**XXX**						
34242/1744	5							X
34234/1747	5					**XXX**		
34240/1820	12	**XXX**	**XXX**	**XXX**				
34241/1820	12							X
34244/1825	13	XX	**XXX**		X			
3617/2079	42		**XXX**					
3619/2079	42		**XXX**					
3580/2079	42	**XX**		**XXX**			X	
3544/2071	42	XX		X	X			
3541/2068	42	**XXX**	XX	X			X	
2605/1846	66		X	**XXX**			X	
2715/1906	67	X	X	**XXX**				
2916 / 1916	69	**XXX**		X	X			
2989/1917	70 A	**XXX**		**XXX**	X			
4210/1945	70 B							X
4056/1924	74	**XXX**	**XXX**	**XXX**				
4140/1924A	75	**XXX**						
4140/1924B	75	XX	**XXX**	**XXX**				
4117/1940A	78	**XXX**	**XXX**		**XXX**			
4117/1940B	78				**XXX**	X		
4149/1940A	78				**XXX**			
4149/1940B	78				**XXX**			
4574/6037	100	**XXX**	**XXX**	**XXX**				
5341/6056	107							X
5363/6056	109	XX					X	
5320/2304	Room 1	XX	XX					

Table 3 Geographic origins for copper used in bronze artefacts found in Sidon's burials based on lead isotope imprints (see figures 1 to 4).

NOTES

1 C. Doumet-Serhal, 2003, 2007.

2 W. R. Griffitts *et al.*, 1972, H. W. Cattling and R. E. Jones, 1977; J. D. Muhly, 1977.

3 R. H. Brill and J. M. Wrampler, 1965; C. C. Patterson, 1971; N. H. Gale and Z. A. Stos-Gale, 1982; N. H. Gale, 2001.

4 R. B. Doe, 1970.

5 G. Manhès *et al.*, 1978.

6 G. Le Roux *et al.*, 2004, 2009 and this study.

7 A. Hauptmann 2000; G. Weisgerber 2006; F. Begemann, *et al.*, 2010.

8 G. Gernez, 2007.

9 A. Véron & G. Le Roux, 2004.

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