#### **ORIGINAL PAPER**



## Differential access to metal wealth from colony to capital to collapse at Phoenician and Punic Carthage: non-ferrous alloys and mineral resources from the Bir Massouda site

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#### Abstract

This article presents the first stratified archaeometric data of the earliest metallurgical assemblage of Maghrebi North Africa from the perspective of the non-ferrous alloys and minerals. From its foundations as a colony to its formation as an imperial power and subsequent decline and collapse, the Carthaginian state maintained a tradition of metallurgical production. Previous research has highlighted workshops of iron and steel manufacture at Bir Massouda and how this facilitated empire formation. The non-ferrous metals and alloys from Bir Massouda also provide information on the shifting fortunes of the Phoenician-Punic commercial endeavor in its geopolitical Mediterranean context. Following its foundation, Carthaginian non-ferrous alloys included the pure copper, tin bronze, and recycled arsenic-tin bronze alloys typical to Iron Age Mediterranean archeological deposits. At its imperial peak, Carthage maintained a relatively high diversity of alloy and mineral types, including pure copper, tin bronze, leaded tin bronze, leaded arsenical copper, and lead. Two pieces of non-ferrous slag are evidence for bronze recycling. A special cobalt-iron-copper mineral was being processed likely as a colorant for glass or other decorative pigment, and glassy copper-based debris was found adhered to a ceramic or kiln component. During its early clashes with Rome and eventual decline and collapse, the Late Punic metal procurement system was stilted, likely due to restricted access to territorial mines previously held by Carthage in the Iberian Peninsula and Sardinia, with a reversion back to an assemblage of pure copper, arsenical copper, arsenic-tin bronze, and lead.

Keywords Non-ferrous metallurgy · Archaeometallurgy · Cobalt · Glass colorants · Political economy · Wealth finance

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## Introduction

Previous research at the Carthaginian site of Bir Massouda has established the chronology for the beginning of metallurgical production in North Africa (eighth century BC, with intensive production from 650 to 425 BC; Kaufman et al. 2016). Prior to that report, there had been no reliable diachronic data for the advent of ferrous metallurgy in North Africa. The same is true for the non-ferrous metallurgy of the Maghreb, and this article presents the first diachronic archaeometallurgical data for metal and mineral acquisition and consumption in the Punic capital.

Near Eastern populations made a decisive shift from bronze to iron in the tenth century BC (Eliyahu-Behar and Yahalom-Mack 2018; Eliyahu-Behar et al. 2013; Veldhuijzen and Rehren 2007; Waldbaum 1980), but copper alloys were still retained for several applications. Due to its castability over iron, copper alloys continued to be employed for armor and helmets alongside steel weapons (Blyth 1993, 25). The Phoenicians were widely recognized through Graeco-Roman and Hebrew histories as well as Neo-Assyrian inscriptions as being the master smiths and metal traders of the era, a reputation born out in the archeological record through fine art and jewelry via precious and base metal media (Hussein and Benzel 2014; Johnston and Kaufman forthcoming; Katzenstein 1997; Markoe 1985).

With the arrival of Iron Age Phoenician settlers to North Africa in the ninth-eighth centuries BC, iron and steel technology was introduced to the local Neolithic Numidian population (Kaufman et al. 2016; Sanmartí et al. 2012). It is likely that metals and metallurgical technology were traded for food, and the presence and willingness of Phoenician traders and craftsmen to transmit this knowledge may have contributed to mutual goodwill. Extraordinarily little is known about the precontact- and contact-era Maghreb, specifically eastern Algeria and Tunisia, but it would seem that a conservative Capsian lithic production continued within a context of mixed pastoral and agricultural subsistence (Brett and Fentress 1996, 12-13). It is possible that goods such as ostrich shell-beads and painted pottery served as value items, and metals would have represented a new form of wealth finance (Earle et al. 2015; Earle 1997; Kaufman 2018; Papadopoulos and Urton 2012). There is minimal evidence that bronze goods from the Iberian Peninsula may have circulated among or been witnessed closely enough to be painted by western Maghrebi communities (Brett and Fentress 1996, 15), indicating that a distant knowledge of symbolic metal wealth was possessed by some Maghrebi communities which would have laid the groundwork for an economic common ground with Phoenicians.

Phoenician and Carthaginian trade and manufacture of alloys was predicated on their vast commercial network. The Iberian Peninsula was by far the richest provider of mineral resources in copper, tin, iron, gold, and silver (Murillo-Barroso et al. 2016; Neville 2007; Renzi and Rovira Llorens 2015), but Sardinia and Cyprus functioned as hubs for resource acquisition at various times as well (Bernardini 2008; Giardino 1995; Vonhoff 2015). From 585 to 573 BC, Tyrian power and influence in the Mediterranean was abruptly halted due to a Neo-Babylonian siege from which they never recovered. This event rippled across the Mediterranean with the restructuring of the Tyrian-indigenous mineral and maritime political economy, with evidence for destruction, abandonment, and fortification of various Punic and indigenous settlements in the Iberian Peninsula in tandem with an influx of Carthaginian material culture (González-Ruibal 2006, 128; Neville 2007, 163-167; Quinn and Vella 2014; Sanmartí 2009, 66). It is at this time that the Tyrian colony of Carthage emerged as the new economic and political node of the Phoenician-Punic world. What is sometimes called the "crisis of the 6<sup>th</sup> century BC" was in fact a golden opportunity for Carthage to put itself in the position to renegotiate alliances and commercial networks between the former Tyrian colonies,

their neighbors, and the Carthaginian state apparatus (Kaufman 2017). Carthaginian material culture increases abroad, substantial import capabilities at Carthage itself increase dramatically from across the Mediterranean (Bechtold 2008, 2010), and historical references to Carthaginian military leaders begin to be recorded (Lancel 1995; Warmington 1969). By the fourth century BC, there is consistent rural intensification across the Punic world (Van Dommelen and Gómez Bellard 2008), and Carthage reached its apex. In the third century BC, its imperial strength and subsequent overseas influence began to unravel through a series of three prolonged military confrontations with Rome over a 118year period known as the Punic Wars (First Punic War 264-241 BC, Second Punic War 218-201 BC, Third Punic War 149-146 BC). The Third Punic War terminated with the destruction of Carthage and the dismantling of the state. Despite all of this sound archeological, ceramic, and historical evidence, to date there has been no study of diachronic metallurgical trends or mineral resource import or production capabilities from the Carthaginian urban capital itself.

## Materials and methods

In addition to the ferrous metallurgy (Kaufman et al. 2016), an array of non-ferrous metals and alloys was also excavated at the site of Bir Massouda at Carthage, spanning several centuries from after the foundation of the colony until its destruction (Table 1). In this article, we report the experimental results of the non-ferrous metallurgy of Carthage over several centuries. The alloys date from the colonial foundations of Carthage, through its imperial stage, and continuing to its destruction. Non-ferrous metals were not only employed in the production of alloys, but for colorants of glass as well. Although finished glass artifacts were excavated from Bir Massouda (Docter and Sonneveld 2009; Redissi 2011, 52–53), two additional pieces of glass colorant production debris were also recovered and are reported below.

Despite clear evidence for ferrous production at Bir Massouda, archeological evidence for production of bronzes is limited. Previous analytical work was conducted by Keesmann (2001; Keesmann and Sveikauskaite 2007; Niemeyer et al. 2007; Rakob 1985) on slag, dross, and alloys from excavations under the Decumanus Maximus (Fig. 1). These metallurgical finds were likely from Phoenician-Punic (not Roman) layers, but the stratigraphy was greatly disturbed in antiquity and the chronology is not clear (Keesmann 2001). Copper slags were characterized by a high wüstite content, as well as olivine phases such as fayalite, and some delafossite crystals. Tin slag microstructure is reported without micrographs as including cassiterite crystals which could be remnant from many types of metallurgical production, including recycling of bronze, ingot melting, the cementation of tin ores

Table 1	Chronol	Chronology and material culture with verified contexts	ontexts from Bir Ma	from Bir Massouda relevant to this study			
	Early P	Early Punic period (c. 760-480 BC)					
Inventory number		Context Description	Precise stratigraphical attribution	Proposed absolute chronology of included finds including residuals	Finds' contents	Basis for chronological assignment and publications	This article
17470	1121	Street or outdoor layer with many sherds	Transitional EP/MP (c. 530–480 BC)	с. 760–480 вс	261 ceramic fragments; 1 glass fragment; 1 slagged tuyère framment	Docter/Sonneveld 2009, 126–127, Fig. 1; Docter forthcoming, cat. 110–144; Kaufman et al. 2016, Table 3	Fig. 5
38465	4460	Ochre sandy layer with many pottery and bone fragments	EP II (c. 675–530 BC)	с. 760–530 вс ( <sup>14</sup> С: 2520±40 ВР)	645 ceramic fragments; 7 fragments of 4 metal objects	Docter et al. 2008, 394-410, 415, 418, Figs. 3, 4, 5:1-4, 6:1-5, 13, cat. 24-52, Table 1; Bechtold/Docter 2010, 89; Núñez 2014. 9, 17, 19, Fiss. 1-2, 5-6	Figs. 2 and 7; Table 4
38458	4490	Gray leveling layer	EP II (c. 675–530 BC)	с. 760–480 вс	Ceramic fragments	Stratigraphy and first report in finds laboratory	Figs. 2 and 6; Table 4
40820	7466	Leveling layer with much charcoal and animal bones	Transitional EP/MP (c. 530–480 BC)	с. 760–480 вс	Ceramic fragments	Stratigraphy and first report in finds laboratory	Figs. 2 and 7; Tables 4 and 5
17466	8091	Leveling layer in which metallurgical hearth 8092 had been set	EP II (c. 675–530 BC)	с. 760–530 вс	670 ceramic fragments; 115 fragments of at least 49 slagged tuyères; slag and charcoal	Docter et al. 2003, 61, Fig. 11; Bechtold 2010, 1 0, 12, Figs. 5, 1; Kaufman et al. 2016, 37–38, 42, 45, Figs. 4–5, 11, Table 3; Docter forthcoming, cat. 23–93	Figs. 4 and 21
10191	Middle (c. 48 1093	Middle Punic period (c. 480–300 BC) 1093 Compact leveling/preparation layer below floor 1068	MP II.1 (c. 430–400 BC)	с. 650-400 вс	496 ceramic fragments;	Bechtold 2008, 86, 117; Bechtold 2010, 22, Table 5, Figs. 8, 1–2, 9, 7, 17, 2; Bechtold	Figs. 2 and 20; Tables 4 and 5
18082	C111	Comnost lavaling lavar some as 1003		080 580 560 FD ID	citatotat, stag; 1 slagged tuyère fragment; 9 fragments of metal objects 55 cosmic frommants	roundoming 1, cat. 0/0-735; Nauman et al. 2016, Table 3 Bachtold forthrowing 2 Cat. 404, 500.	× ت
28082	7111	Compact leveling layer; same as 1093 MP II. (c. 4	MP 11.1 (c. 430–400 BC)	C. 050-280 BC (EF II)	<ul> <li>ceramic fragments;</li> <li>l</li> <li>slagged tuyère</li> <li>fragment</li> </ul>	55 certainic fragments, Becntold forthcoming 2, Cat. 494–500; 1 Kaufman et al. 2016, Table 3 slagged tuyère fragment	c .gr1
30007	1104	Compact leveling layer	MP II.2 (c. 375–325 BC)	c. 480–300 BC (1 intrusive LP)	61 ceramic fragments; 1 metal object; charcoal; slag	<ul> <li>61 ceramic fragments, Bechtold 2008, 44; Bechtold/Docter</li> <li>1 metal object, 2010, 90, charcoal; slag Table 2; Bechtold forthcoming 3, cat.</li> <li>926–927</li> </ul>	Figs. 2 and 8; Table 4

Table 1	Table 1 (continued)	(p					
	Early P	Early Punic period (c. 760–480 BC)					
Inventory number		Context Description	Precise stratigraphical attribution	Proposed absolute chronology of included finds including residuals	Finds' contents	Basis for chronological assignment and publications	This article
38237	1107	Compact leveling layer	MP II.1 (c. 425–400 BC)	с. 480–400 вс	<ul> <li>575 ceramic fragments; 7 fragments of metal objects;</li> <li>2 fragments wall plaster;</li> <li>2 fragments of pavenent;</li> </ul>	Bechtold 2008, 44; Bechtold 2010, 14, 19, 22, 30, Table 5, Figs. 7, 7, 10, 6, 17, 1, 17, 4; Bechtold/Docter 2010, 90, tab, 2; Bechtold forthcoming 1, cat. 749–788	Fig. 3; Table 3
38597 38599 42777	2420	Black grayish primary destruction layer	MP II.2 (c. 340–320 BC)	c. 500–325 Bc (Carthaginian coin 370–340 Bc)	slag; charcoal 973 ceramic fragments, 8 fragments of wall plaster; 2 pavement fragments; 1 grinding stone; 1 coin;	Bechtold 2008, 44; Bechtold 2010, 19, 23-24, 29-31, 33, Table 7, Figs. 10, 7, 15, 1-2, 17, 3, 18-19, 20, 1; Bechtold/Docter 2010, 90, Table 2; Redissi 2011, 36, Fig. 5, cat. 9; Bechtold forthcoming 4; Frey-Kupper forthcoming	Figs. 2, 3, 10, and 11; Tables 3 and 4
45010	2504	Sandy layered level	MP II.2 (c. 330/20-300 BC)	c. 400–300 Bc (coin 370/360–340/330 Bc)	<ul> <li>I glass ocad</li> <li>158 ceramic fragments;</li> <li>9 fragments of wall plaster;</li> <li>1 pavement</li> <li>1 glass bead;</li> <li>1 piece of slag</li> </ul>	Bechtold 2008, 44; Bechtold 2010, 23–24, 27, Table 7, Figs. 14, 6, Figs. 14, 8, Bechtold/Docter 2010, 90, Table 2; Redissi 2011, 54–55, Fig. 18, cat. 23; Bechtold forthcoming 4; Frey-Kupper forthcoming; Kaufinan et al. 2016,	Figs. 2 and 19; Table 4
38441	4440	Dark brown fill below pavement 4427 MP II.2 (c. 35	MP II.2 (c. 350–325 BC)	с. 530–300 вс	415 ceramic fragments	able 4 Bechtold 2008, 44; Bechtold 2010, 23–24, Table 7; Bechtold/Docter 2010, 90,	Figs. 2 and 9; Table 4
38448	4444		MP II.1 (c. 430–400 BC)	с. 530-400 вс	506 ceramic fragments	1able 2; Bechtold forthcoming 5         Bechtold 2008, 44; Bechtold 2010, 34,         Fig. 21A, 1; Bechtold forthcoming	Figs. 2, 17, and 18; Tables 4 and 5
40812	7452	Preparation layer for floor 7441 of bathroom	MP II (c. 410–350 BC)	с. 530–350 вс	29 ceramic fragments	29 ceramic fragments Docter et al. 2006, 49; Maraoui Telmini 2011, 58, Fig. 10; Maraoui Telmini 2012,	Fig. 2, Table 4

	Early P	Early Punic period (c. 760–480 BC)					
Inventory number	Contex	Inventory Context Description number	Precise stratigraphical attribution	Proposed absolute chronology of included finds including residuals	Finds' contents	Basis for chronological assignment and publications	This article
						24–25, 28, 42–43, 127–129, Figs. 9–10, 127–128, cat. 134–135	
	Late Pu	Late Punic period (c. 300–146 BC)					
20176	1068	1068 Late Punic pavement	LР II (с. 200–146 вс)	c. 200–146 BC	Ceramic fragments	Stratigraphy and first report in finds laboratory	Fig. 3; Table 3
33573	1295	Preparation layer of floor 1291	LP II (c. 200–146 BC)	c. 300–146 BC	Ceramic fragments; 2 glass wasters	, 52–53, Fig. 16, cat. 20–21; iy and first report in finds labo	Figs. 2 and 13; - Table 4
38439	4438	Pale leveling layer	LP II (c. 200–146 BC)	c. 200–146 BC	22 ceramic fragments; 1 terracotta fragment	22 ceramic fragments; Stratigraphy and first report in finds 1 terracotta laboratory fraement	Figs. 2 and 12; Tables 4 and 5
33531	7271	Brown to orange sandy fill with large LP II and small stones (c.	<ul> <li>LP II</li> <li>(c. 200–146 BC)</li> </ul>	c. 300–146 BC :)	Ceramic fragments	Stratigraphy and first report in finds laboratory	Figs. 2, 14, and 15; Table 4
33539	7288	Brown loose sandy layer	LP II (c. 200–146 BC)	c. 300–146 BC ;)	Ceramic fragments	Stratigraphy and first report in finds laboratory	Figs. 2 and 16; Table 4

Table 1 (continued)

in molten copper, or co-smelting of ores (Keesmann 2001, 96–98; Rademakers et al. 2018; Valério et al. 2013). At the Middle-Late Punic metallurgical zone on the slopes of the Byrsa (late fifth–late third centuries BC), Tylecote (1982, 273) surmised that limited copper alloy processing was being conducted in the ferrous furnaces, but that what minimal physical evidence he did find for this was further hampered by the "highly saline and corrosive conditions" of the excavated sediments which likely mineralized copper compounds but left the iron working debris. Indeed, the materials from Bir Massouda are often severely corroded, but archaeometric analysis was employed in order to attempt reconstruction of alloys and alloy classes as much as the materials could allow.

The materials considered in this study were excavated from 2000 to 2005 in the Bir Massouda area of Carthage (Table 1, Fig. 1; excavation extent of 1500 m<sup>2</sup>; alternately spelled Bir Messaouda). The excavations were executed by the University of Amsterdam, the Archaeological Department of Ghent University, and the Tunisian Institut National du Patrimoine (INP; for additional excavation information, cf. Bechtold 2008; Docter 2002–2003, 2007, 2008, 2009, 2010; Docter and Bechtold 2011; Docter et al. 2006; Docter et al. 2003; Docter et al. 2002; Maraoui Telmini et al. 2014;

Maraoui Telmini 2012). The materials were assigned periods based upon the field reports and chronological interpretations of the contexts provided by Roald Docter. Radiocarbon sequences have established the first absolute dates at Carthage (Docter et al. 2008; Docter et al. 2005), and relative chronologies were clarified by recent excavations (Bechtold and Docter 2011; Bechtold 2010). The ferrous metallurgical activities of Bir Massouda begin in the eighth century but the furnaces are most intensely operated from ca. 650–425 BC (Kaufman et al. 2016). At the end of the fifth century BC, the metallurgical zone activities were halted and a residential quarter was constructed.

Alloys were selected for analysis based on whether or not they came from a sound chronological context, and the samples taken from these contexts are analyzed and reported here. All of the artifacts were collected or had representative samples taken from the archives of the Department of Archaeology of Ghent University, Belgium, where they have been stored since being temporarily exported from Tunisia. All archaeometallurgical artifacts were divided by chronological phase, i.e., Early Punic (EP, 760–480 BC, often referred to as the Phoenician or Archaic period), Middle Punic (MP, 480–300 BC), and Late Punic (LP, 300–146 BC, these latter two traditionally referred to as broadly the Punic period), as well as more specific

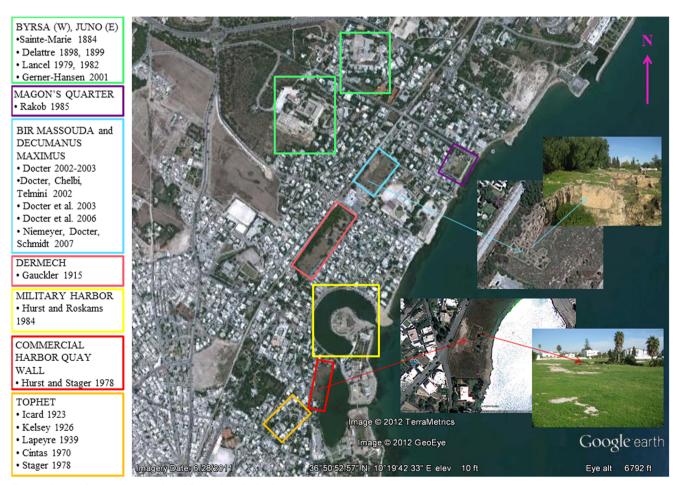


Fig. 1 History of excavations at Phoenician and Punic Carthage

temporal ranges determined by pottery, radiocarbon, or numismatic dating in order to determine the extent of the entire corpus of metallurgical material culture remains.

Artifacts are labeled by locus followed by artifact number, for example locus 2420 artifact number 38599. The state of preservation of artifact forms is not as good as that of the alloys, restricting more detailed potential discussions surrounding typology and use but permitting reconstruction of alloy preferences and material procurement limitations. Some alloys found in a Punic septic pit were not available for analysis, and do not come from a context specific enough for this investigation (Docter et al. 2006; Maraoui Telmini 2011). Coins, mostly small copper ones (578 excavated in total, 255 from 2000 to 2001 excavations (BM00/01) and 323 from BM02-BM-05), were excluded from analysis as they represent a distinct material type with particular economic forces that impact alloy choice and cannot readily be included in a comparison of non-monetary alloys.

The selected non-ferrous metals consist of 17 copper alloys (n = 14) and lead pieces (n = 3) (Figs. 2 and 3). Other metalliferous remains may point toward glass colorant production, or alloy experimentation. These include one piece of cobaltrich material which may be a glass colorant or pigment (Fig. 2), one artifact of glassy copper colorant material adhering to a ceramic kiln or other structural debris (Fig. 4; see Rehren et al. 2010, Fig. 5 for a comparison), and two pieces of slagged ceramics associated with bronze recycling (Fig. 5; Kaufman et al. 2016, Table 3).

The alloys with remnant metal and corrosion materials were subjected to standard metallurgical representative sampling practices: both before and after sampling, artifacts were photographed in order to document the original piece and the subsequent change. Artifacts were secured by a padded vice, and a jeweler's saw was used to take "V" shaped samples and/ or edge sections. Cross sections were also sometimes removed. Sampled artifacts were stored in Eppendorf vials or conservation paper. The samples were thereafter exported to the Cotsen Institute of Archaeology laboratories where they were prepared and analyzed, with research also conducted at the Molecular and Nano Archaeology Laboratory (MNA), and the GCI Conservation Institute Laboratories at the Getty Villa. Samples were mounted in a two-part epoxy resin, ground with 240 then 600 PSA backed grit, followed by polishing and finishing with monocrystalline diamond suspension and/or non-crystallizing colloidal silica suspension of 6, 1, and/or 0.02 µm.



**Fig. 2** Non-ferrous alloys and minerals from Bir Massouda. Top row left to right: 4460 38465, 7466 40820, 4490 38458; second row left to right: 2420 38599, 4440 38441, 1093 10191; third row left to right: 1104

30007, 2420 38597, 4444 38448, 2504 45010, 7452 40812; fourth row left to right: 4438 38439, 1295 33573, 7271 33531, 7288 33539

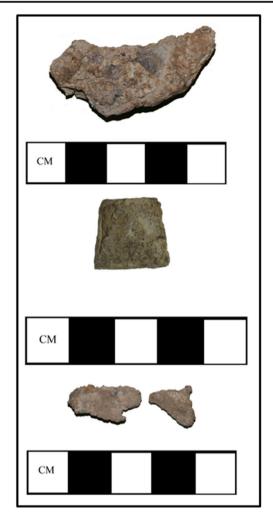


Fig. 3 Lead artifacts from Bir Massouda. From top to bottom:  $1107\ 38237, 2420\ 42777, 1068\ 20176$ 

Archaeol Anthropol Sci (2019) 11:4075-4101

## Instruments and settings

Analytical methods employed were portable X-ray fluorescence spectroscopy (pXRF), variable pressure scanning electron microscopy coupled with energy X-ray dispersive spectroscopy (VPSEM-EDS), and metallography using reflected light optical microscopes.

## X-ray fluorescence spectroscopy (XRF)

Following the methodology of Kaufman et al. (2016: 41), elemental analysis was conducted using a Thermo Niton XL3t GOLDD+ handheld XRF equipped with a silver anode tube and a large silicon drift detector (SDD) operating at a maximum voltage of 50 kV and current of 200 µA with a resolution better than 160 eV and producing an average spot diameter of about 8 mm. The glassy colorant adhering to the ceramic (8091 17466) was analyzed in "Mining" mode which uses fundamental parameters calibration iterative algorithm and manufacturer-set internal calibrations to convert X-ray counts into concentrations. For the lead alloys and Getty bronze standards (published by Heginbotham et al. 2010), "General Metals" mode was used, with 120 s duration (maximum time 121 s), divided into three parameters for detection of elements, 60 s high, 30 s low, and 30 s light. One spot was taken with a toggle spot (3 mm spot) and one spot taken without (8 mm spot). Results are reported to a resolution of 0.1 wt%, where detected concentrations of > 0.05 wt% were rounded up to 0.1 wt%.

## Metallography and polarized light microscopy (PLM)

Metallographic polarized light microscopy was conducted with a Nikon Epiphot-TME Metallograph microscope, as well



**Fig. 4** Copper-based glass colorant debris from Bir Massouda, artifact 8091 17466

as a Leica DMRM. Settings used included reflected optical light, polarized light, and dark field. Micrographs were mostly taken with a 14-MP eyepiece digital camera UCMOS series microscope camera, with the ToupView 3.2 image software, but also in some cases with a Nikon digital camera D3000. These methods were employed in order to discern the microstructure of the alloys and corrosion products. It was conducted on all of the alloys and corrosion products excluding lead which was analyzed using only pXRF.

Etching was conducted to accentuate microstructural features of the alloys. Ferric chloride (FeCl<sub>3</sub>) was used for noncolor etching. Color etching was executed by first pre-etching the mounted samples in a 10% aqueous solution of ammonium peroxydisulfate, 98%. The color tint etching was then obtained by soaking the mounted samples in a bath of a saturated solution of sodium thiosulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>), in distilled water to which a few grains of sodium metabisulfate (Na<sub>2</sub>S<sub>2</sub>O<sub>5</sub>) were added, a variation of Klemm's Reagent II, following Scott (2010). Following color etching from the various acid baths, samples were repolished for conservation purposes in order to inhibit accelerated corrosion.

# Scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS)

SEM with energy dispersive EDS was performed on alloys and corrosion, and on the bronze standards (non-Getty, Table 2). The instrument located at the Molecular and Nano

4083

Archaeology (MNA) Laboratory in the School of Engineering and Applied Science at UCLA is a FEI Nova™ NanoSEM 230 scanning electron microscope with field emission gun (FEG) and variable pressure capabilities, equipped with a Thermo Scientific NORAN System 7 X-ray Energy Dispersive Spectrometer (EDS). For the alloys and corrosion, and bronze standards, a gaseous analytical detector (GAD) detector in variable pressure was used for the detection of backscattered electrons (BSE). Accelerating voltage was kept to 15 keV, usually a spot of size of 5 was employed, chamber pressure was set at 50 mPa, and working distance around 8 mm. Aperture settings were adjusted in order to increase peak intensities. Five average spots were taken at × 1500 magnification for the alloys and cobalt mineral, for a total analyzed area of  $0.043 \text{ mm}^2$  per specimen. Although a relatively small area, the consistent methodology allows for comparisons to be made between the alloys. Accelerating voltage was 10 keV for one of the spots of the five averages of 2504 45010.

Efforts were made to capture only metal spots on the alloys, but often corrosion was unavoidable. For artifacts that were completely corroded, the alloy average should be close to the original barring leaching into the soil matrix which is discussed below in particular cases. Corrosion can significantly alter chemical composition. Whatever the case, the amount of tin and arsenic represented are real values representing the availability of these mineral resources, if not representative of the original alloy, while also conclusively allowing for determination of alloy classes. Clearly, for quantitative

**Fig. 5** Non-ferrous recycling slag from Bir Massouda. Left: unmounted artifact 1121 17470 Note visible metallic prill. Center: mounted artifact 1112 38082. Right: Cross section of 1121 17470 with analysis resulting in lower micrograph with partially oxidized circular bronze prills in a glassy fayalite matrix. In both of the circular prills, the bright white areas contain metallic copper, whereas the darker bulk zones contain oxidized copper-tin dross or slag

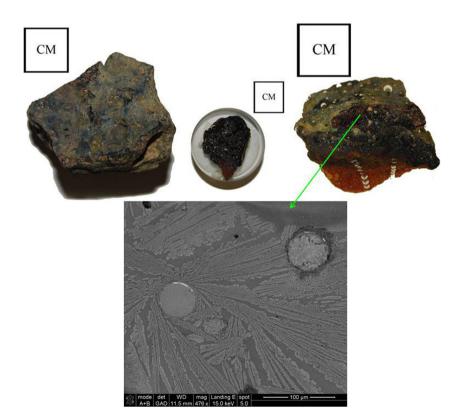


Table 2 EDS as	nalysis c	of bronz	ze stano	dards														
BNF C71.34-3	Al	Si	Р	S	Cr	Mn	Fe	Со	Ni	Cu	Zn	As	Zr	Ag	Sn	Sb	Pb	Total
Standard	0.0	0.0	0.0	0.2	0.0	0.1	0.3	0.0	0.0	86.9	1.6	0.2	0.0	0.0	8.2	0.1	2.5	100.0
EDS	0.0	0.0	0.0	0.4	0.0	0.1	0.3	0.0	0.2	84.5	1.9	0.1	0.0	0.0	9.4	0.5	2.6	100.0
CTIF B32	Al	Si	Р	S	Cr	Mn	Fe	Со	Ni	Cu	Zn	As	Zr	Ag	Sn	Sb	Pb	Total
Standard	0.1	0.1	0.0	0.0	0.0	0.0	0.1	0.0	1.5	74.9	1.2	0.0	0.0	0.0	5.9	0.1	16.1	100.0
EDS	0.1	0.0	0.2	0.1	0.0	0.0	0.1	0.0	1.4	72.3	0.7	0.0	0.0	0.1	7.9	0.3	16.9	100.0
CTIF UE15	Al	Si	Р	S	Cr*	Mn	Fe	Co	Ni	Cu	Zn	As	Zr	Ag	Sn	Sb	Pb	Total
Standard	0.0	0.1	0.3	0.0	0.0	0.0	0.2	0.0	0.2	87.1	0.2	0.1	0.0	0.0	10.8	0.6	0.5	100.0
EDS	0.1	0.0	0.1	0.1	0.0	0.0	0.1	0.0	0.4	86.3	0.0	0.1	0.0	0.1	11.2	0.8	0.6	100.0
CTIF UE53	Al	Si	Р	S	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Zr	Ag	Sn	Sb	Pb	Total
Standard	0.0	0.0	0.0	0.0	0.0	0.0	0.2	0.0	1.0	84.0	3.5	0.0	0.0	0.0	4.8	0.5	6.0	100.0
EDS	0.1	0.0	0.0	0.1	0.0	0.0	0.1	0.0	1.1	84.9	2.5	0.0	0.0	0.0	5.9	0.7	4.6	100.0
CTIF UZ51	Al	Si	Р	S	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Zr	Ag	Sn	Sb	Pb	Total
Standard	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.2	83.4	14.5	0.1	0.0	0.0	1.5	0.0	0.2	100.0
EDS	0.0	0.0	0.0	0.0	0.0	0.0	0.1	0.0	0.2	80.9	16.6	0.0	0.0	0.0	1.9	0.1	0.1	100.0
IARM 94B	Al	Si	Р	S	Cr	Mn	Fe	Co	Ni	Cu	Zn	As	Zr	Ag	Sn	Sb	Pb	Total
Standard	10.8	0.0	0.0	0.0	0.0	0.1	4.0	0.0	4.3	80.6	0.1	0.0	0.0	0.0	0.0	0.0	0.0	100.0
EDS	9.2	0.0	0.0	0.0	0.0	0.1	4.4	0.0	4.8	80.9	0.5	0.0	0.0	0.0	0.0	0.0	0.0	100.0
MBH 32X SN3	Al	Si	Р	S	Cr	Mn	Fe	Со	Ni	Cu	Zn	As	Zr	Ag	Sn	Sb	Pb	Total
Standard	0.0	0.1	0.0	0.1	0.0	0.7	0.0	0.0	0.6	81.0	0.4	0.0	0.0	0.0	15.1	0.5	0.3	98.8
EDS	0.0	0.0	0.0	0.1	0.0	0.0	0.1	0.0	0.5	78.6	0.9	0.0	0.0	0.1	18.9	0.5	0.3	99.9
CTIF UE13	Al	Si	Р	S	Cr	Mn	Fe	Со	Ni	Cu	Zn	As	Zr	Ag	Sn	Sb	Pb	Total
Standard	0.0	0.2	0.2	0.3	0.0	0.0	0.5	0.0	0.1	83.9	0.6	0.2	0.0	0.0	13.7	0.3	0.2	100.2
EDS	0.1	0.2	0.1	0.1	0.0	0.0	0.5	0.0	0.2	81.8	0.2	0.4	0.0	0.1	15.6	0.5	0.2	100.0

Only two spots were taken for Cr on this sample

## Table 3 pXRF analysis Bir Massouda lead artifacts and lead-rich standards

Middle Punic lead, pX	RF										
Artifact	Dates BC	Spots	Pb	Fe	Cu	Sn	Bi	Мо	Total		
1107 38237	425-400	2	99.6	0.1	0.1	_	0.1	_	99.9		
2420 42777	340-320	6	96.1	0.6	-	0.1	0.1	0.1	97.0		
Late Punic lead, pXRF	7										
Artifact	Dates BC	Spots	Pb	Fe	Cu	Sn	Bi	Mo	Total		
1068 20176	200-146	4	98.9	0.4	-	0.4	0.1	0.2	99.9		
A - Chinese coin (unkt	nown date)										
Element	Fe	Ni	Cu	Zn	As	Ag	Sn	Sb	Pb	Bi	Total
Getty standard	0.6	< 0.35	71.0	< 0.79	0.5	< 0.15	4.1	0.2	24.0	< 0.12	101.2
pXRF	0.7	0.1	71.5	-	-	-	3.8	0.2	21.2	0.1	96.8
pXRF (3 mm spot)	0.7	0.1	72.9	-	-	-	4.0	0.2	20.6	0.1	97.9
J - CTIF B32 (CRM)											
Element	Fe	Ni	Cu	Zn	As	Ag	Sn	Sb	Pb	Bi	Total
Getty standard	0.1	1.5	74.9	1.2	0.0	-	5.9	0.1	16.1	-	99.6
pXRF	0.1	1.5	74.6	1.1	-	-	5.3	0.1	15.3	0.0	97.9
pXRF (3 mm spot)	0.1	1.5	74.8	1.0	—	_	5.4	0.1	15.4	0.0	98.3

reconstruction of copper-based metals and alloys, it would be ideal to analyze only uncorroded samples. However, this is a research component beyond the control of archeologists, and we hold that attempts should be made to study the pieces given the known constraints. Oxygen, carbon, chlorine, calcium, potassium, aluminum, and phosphorus were excluded from the alloy readings as these mostly represent postdepositional inclusions, but all detected elements were included for the cobalt mineral 1093 10191 except carbon which accounts for the totals being well below 100 wt%.

This study gauges average tin and arsenic contents in order to identify metal consumption practices and possible trade routes, so a brief explanation is warranted on the assignment of alloy typologies. It is necessary to separate leaded from non-leaded alloys for this tally because the mechanical properties of leaded bronzes are vastly different from unleaded bronzes. Indeed, tin bronze and arsenical coppers are so similar that they are essentially "interchangeable" (Budd and Ottaway 1991; Lechtman 2007, 335). Therefore, a meaningful comparison of tin and arsenic content is only justifiable across like artifacts, "like" here meaning mechanical properties. The only questionable artifact is 7466 40820, which is here not considered a leaded tin bronze due as its lead content of 5 wt% which is the lower limit for intentionally alloyed leaded bronzes (Scott 2010, 174). It is therefore unclear if this lead was intentionally added, although it would aid in reducing viscosity during casting. Arsenical coppers are arbitrarily considered as those alloys with over 0.5 wt% As.

#### Analysis of standards

A number of standards were employed to approximate margins of error for both the pXRF and SEM-EDS. Experimental analysis was undertaken on 10 copper alloy standards to establish general margins of error for the unknown archeological samples (Table 2). Although not pure lead standards, two lead-rich alloys from Heginbotham et al. (2010) were analyzed using pXRF to report instrument accuracy to a resolution of 0.1 wt% (Table 3). For EDS, five or ten areas on each standard were analyzed at either  $\times$  1500x or  $\times$  3000 magnification, respectively, and the

 Table 4
 EDS analysis of non-ferrous copper alloys and minerals from Bir Massouda

Early Punic b	ronze, EDS													
Artifact	Dates BC	Spots	Cu	Sn	As	Fe	Pb	S	Ag	Total				
4490 38458	675–530	4	88.9	10.6	_	_	_	0.5	_	100.0				
4460 38465	675–530	5	96.1	_	0.3	0.3	_	3.3	-	100.0				
7466 40820	530-480	5	76.7	16.4	1.8	_	5.0	_	0.1	100.0				
Average				9.0	0.7									
Middle Punic	bronze, EDS													
Artifact	Dates BC	Spots	Cu	Sn	As	Fe	Pb	S	Ag	Total				
7452 40812	410-350	5	84.4	13.6	0.3	0.3	_	1.3	_	100.0				
1104 30007	375-325	5	97.1	1.9	0.3	0.3	0.3	0.1	_	100.0				
4440 38441	350-325	5	99.9	_	_	_	0.1	_	-	100.0				
2420 38597	340-320	5	97.9	_	0.7	0.1	_	1.4	-	100.0				
2420 38599	340-320	5	98.8	_	0.6	0.6	_	0.1	-	100.0				
Average				3.1	0.4									
Late Punic bro	onze, EDS													
Artifact	Dates BC	Spots	Cu	Sn	As	Fe	Pb	S	Ag	Total				
4438 38439	200-146	5	90.8	5.4	0.7	0.6	1.5	1.0	-	100.0				
1295 33573	200-146	5	99.4	_	0.1	0.4	_	0.1	-	100.0				
7271 33531	200-146	5	98.9	_	0.8	_	_	0.3	_	100.0				
7288 33539	200-146	5	97.3	_	1.3	0.4	_	0.9	-	100.0				
Average				1.4	0.7									
Middle Punic	leaded bronze	, EDS												
Artifact	Dates BC	Spots	Pb	Sn	As	Cu	S	Ni	Total					
4444 38448	430-400	5	33.4	_	23.9	41.8	0.8	0.1	100.0					
2504 45010	330-300	5	20.3	6.7	0.3	72.7	_	_	100.0					
Middle Punic	cobalt-rich ma	aterial, ED	S											
Artifact	Dates BC	Spots	CoO	FeO	CuO	$Al_2O_3$	$SiO_2$	K <sub>2</sub> O	CaO	Cl	ZnO	$ZrO_2$	$P_2O_5$	Total
1093 10191	430–400	5	21.0	54.1	8.0	1.0	7.0	0.2	1.7	1.0	0.1	0.0	0.1	94.2

results averaged. The alloy reading in wt% was then compared against the known standard alloy wt%. Arsenic content was not significantly high enough in any of the standards to be useful to establish an instrumental margin of error. Results are rounded and reported with a resolution of 0.1 wt%.

## Results

Based on the experimental data, the non-ferrous artifacts from Bir Massouda can be divided into alloys and corrosion products (n = 17; Table 4; Figs. 6, 7, 8, 9, 10, 11, 12, 13, 14, 15, 16, 17, 18, and 19), non-ferrous recycling slag (n = 2; Fig. 5; Kaufman et al. 2016, Table 3 artifacts 1112 38082 and 1121 17470, a cobalt-rich mineral or pigment likely used as a glass colorant (n = 1; Figs. 2 and 20), and copper-based glass metal colorants (n = 1; Figs. 4 and 21). Although this is not a relatively large assemblage, it does represent the entirety of the precisely dated Phoenician and Punic non-ferrous metallurgical assemblage and can therefore provide a basis for what may hopefully be greater amounts of materials recovered in future excavations.

## Non-leaded copper alloys

#### Early Punic (800-480 BC)

Two tin bronzes date broadly to the Early Punic (800–480 BC) and represent the earliest tin bronze artifacts from Carthage. Fibula 4490 38458 is a tin bronze and demonstrates Liesegang phenomenon (Fig. 6; Scott 1985). The artifact is completely corroded. A tin rich internal core reaches around 90 wt% tin,

with lead, silver, iron, and arsenic inclusions (not including oxygen, as discussed above). The core was excluded from the average, as the high tin content poses a risk of skewing the average tin content. Therefore, the most conservative estimate for tin is at the exclusion of the core (4 spot average instead of 5), rendering 10.63 wt% tin. Counting the core would result in an average tin content of 26.55 wt%. This is therefore, qualitatively speaking, a true (10–14 wt%) or high tin bronze.

Artifact 7466 40820 is a ternary high tin arsenical bronze artifact with silver and lead impurities, excavated from a workshop context rich in charcoal and bones. The alloy was mostly corroded but some metal remained (Fig. 7i–iii). Strain lines are observed in the corrosion, but the preserved bulk metal ( $\alpha + \delta$  eutectoid) was unaffected by the working. There are no observable grains - the object was cast but not annealed. The five average spots were taken only from the metal components, and were relatively low in tin content compared to the outer corrosion layers. Outer corrosion layers begin to include elevated levels of calcium and phosphorus, and high levels of tin and lead, demonstrating the leaching out of these outer metallic layers into the sediment matrix, perhaps aided by inverse segregation during the casting (Table 5). Lead is particularly present in the outermost corrosion layer, from leaching.

Pure copper artifact 4460 38465 is completely corroded, coming from what may be a domestic Early Punic context (Núñez 2014). Some of the broken segments, circular in nature, suggest a fibula. The circular morphology is also attested in the corroded circular core (Fig. 7iv). The artifact was lumped together with a ferrous chunk of corrosion as well. The sulfur content is high—unlikely from the original cast but more probably from post-depositional interactions with the sediment.

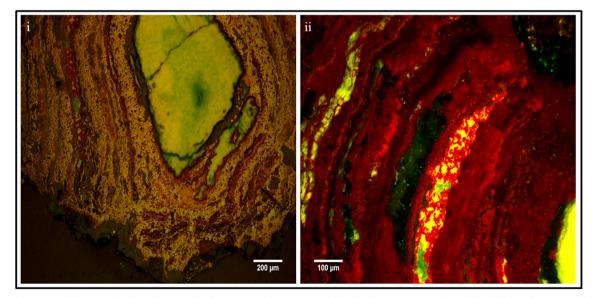
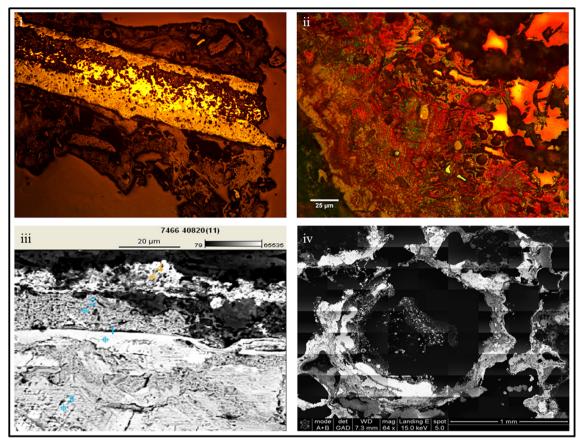


Fig. 6 Artifact 4490 38458. i) Optical micrograph of Liesegang phenomenon with tin rich green core; ii) polarized light of laminated Liesegang corrosion structure



**Fig. 7** i) Unetched optical micrograph of outer and inner corrosion layers, metal preserved in core [7466 40820]; ii) etched with ferric chloride, strain lines visible in corrosion but not metal [7466 40820]; iii)

## corrosion layers spot analysis (Table 5) [7466 40820]; iv) GAD montage of central corroded area and circular casting [4460 38465]

#### Middle Punic (480–300 BC)

Artifact 7452 40812 is a corroded tin bronze artifact with a well-maintained shape, although its original purpose is unclear (Fig. 2). It was excavated from a basin preparation layer. The tin content is deviated across the spots, meaning that throughout the five averaged spots the content fluctuates from  $\sim 6-22$  wt%. The average yields a good tin bronze.

Low tin bronze nail 1104 30007 was excavated from a compact leveling layer and had metal preserved to a high degree. The minor constituents of arsenic, iron, and lead are likely due to recycling activities, and silver impurities were also recovered likely remnant from the ore. Recrystallized grains and proliferation of twins indicate repeated episodes of working and annealing, but some coring remains. Slip planes and elongated grains show that the final act was annealing, not working. There is good patination as evidenced from the unetched samples, with limited, superficial intergranular corrosion (Fig. 8).

The impressively pure copper artifact 4440 38441 is heavily attacked by corrosion but still maintains a type of swirling pattern that remains from the original casting, perhaps evidence of rapid cooling in the mold, if not from cold work (Fig. 9). The artifact was heavily worked with some annealing, and the deformed slip planes, elongated grains, and grain directionality with bent twin lines are evidence of the final stage in the manufacture being cold-worked to shape. The green blotches throughout the bulk metal include various elements such as lead, iron, sulfur, and selenium, coupling of the latter two common in Ancient Near Eastern bronzes (Rehren 1991).

Copper alloy stake or peg 2420 38597 (same locus 2420 as 38599 below) was found in the same context as a Carthaginian coin (dated 370–340 BC) and has a low arsenic content which may be the result of recycling. The threepronged pure copper oxide central corrosion area may be an intentional feature of this unique artifact (Fig. 10). Since the corrosion is pure copper and the alloy is distinctly different, with arsenic and sulfur content, it could be that a pure copper metal suspension core was placed in the cast (White and Hamilton 2014, 818–819), but at present it seems unlikely and this issue is unresolved. The elongated swirling grain microstructure that abuts the central core shows that the molten alloy froze against the core, again perhaps retained through rapid cooling. Equiaxed grains show excellent production technique with many annealing

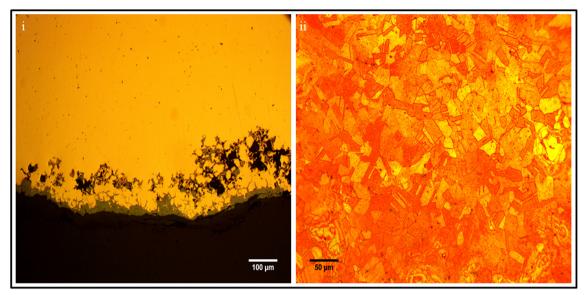


Fig. 8 Artifact 1104 30007. i) Unetched optical micrograph showing patination and superficial intergranular corrosion; ii) color etched optical micrograph of bulk with equiaxed grains, slip planes, and twins

and working events, despite the substantial sulfur content. Impurities are iron sulfides, tellurium, and selenium.

Hook or brooch 2420 38599 is also the result of fine copper smithing, also with minimal arsenic content and

iron sulfide impurities like the other alloy found in the same locus. There are many twins with some elongated grains, but many are equiaxed showing repeated working and annealing (Fig. 11).

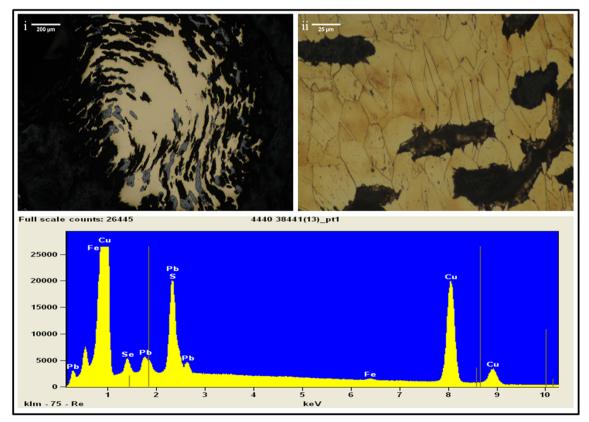


Fig. 9 Artifact 4440 38441. i) Optical micrograph of circular microstructure; ii) etched with ferric chloride showing elongation, directionality, slip planes, and impurities; iii) spectrum of impurity with lead, sulfur, iron, and selenium

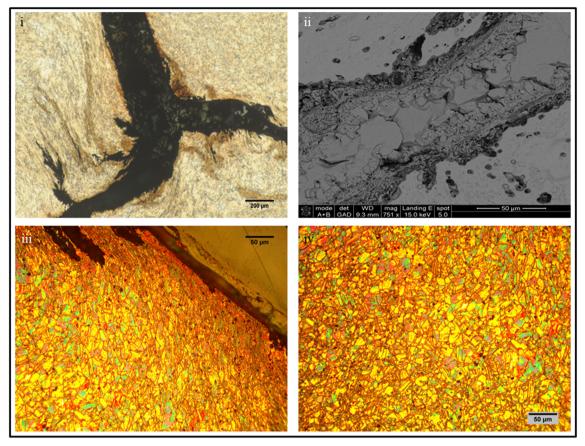


Fig. 10 Artifact 2420 38597. i) Optical micrograph of corrosion core, etched with ferric chloride; ii) BSE micrograph of the central core, internal band is pure copper oxide, external sides abutting the alloy

#### Late Punic (300–146 BC)

The typology of tin bronze artifact 4438 38439 is unidentified, but the alloy and microstructure is rather complex and anomalous. It was probably cast in an open mold due to the evident porosity (Fig. 12). It was heavily coldworked as evidenced by strain lines found throughout.

have some iron and sulfur; iii) color etched micrograph of swirling microstructure of the surface; iv) color etched micrograph of equiaxed grains with strain lines and working twins

There are some small twins and equiaxed grains indicating that it was annealed, recrystallized, and cold-worked. Differential recrystallization occurred in the different colored areas. The lighter areas were most affected by the strain and contain the twins and recrystallized grains, whereas the darker areas have less microstructural complexity. In the optical micrographs, the darker phases

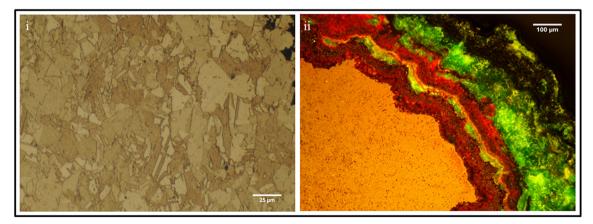
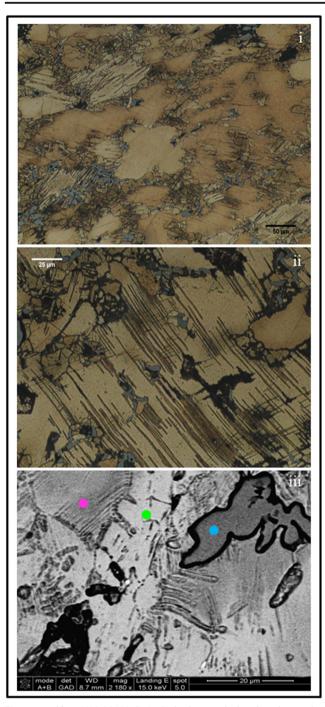


Fig. 11 Artifact 2420 38599. i) Twins, elongated grains, recrystallized grains, etched with ferric chloride; ii) polarized micrograph of bulk alloy and patina layers



**Fig. 12** Artifact 4438 38439. i) Optical micrograph showing phase color differentiation, green sulfidic zinc impurities, and strain lines; ii) strain lines concentrated in lighter, tin depleted zones; iii) alloy spot analysis (Table 5)

proved to be tin rich (on SEM backscattered mode these are the lighter areas), the lighter areas tin poor (darker phases in the SEM), and the green blotches rich in zinc and sulfur. Some lead impurities are present as well.

Artifact 1295 33573 is an incredibly pure copper (Table 4) tool or accoutrement of some kind, excavated from pavement or floor bedding of the Late Punic period. The minimal

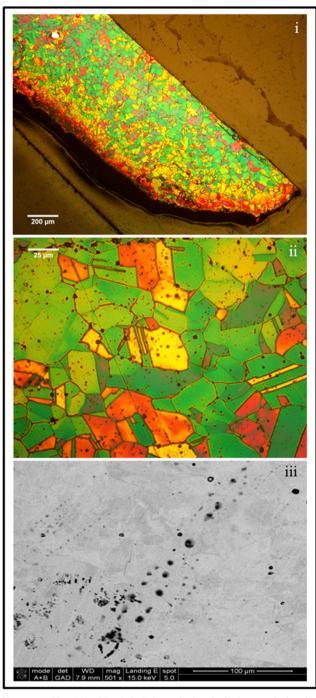
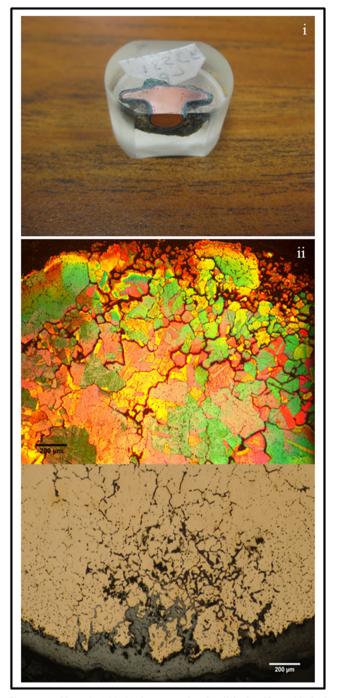


Fig. 13 Artifact 1295 33573. i) Color etched optical micrograph showing mostly recrystallized grains; ii) twins, slip plans, recrystallized grains; iii) Ca-K-S-Fe slag stringers

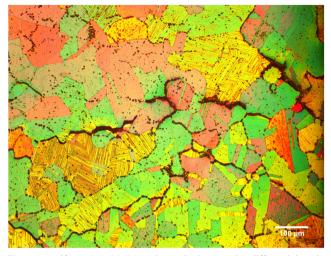
inclusions testify to a very successful smelt. Twins and equiaxed grains are evidence of annealing and cold-working (Fig. 13).

Artifact 7271 33531 is a clenched nail with round head and has a low arsenic content that may be the result of recycling, but can be considered an arsenical copper due to the absence of other alloying elements, excluding sulfur which is an impurity. It could be that a low arsenic content was used for



**Fig. 14** Artifact 7271 33531. i) Mounted and polished artifact with dual cross sections in nail head and shank, with visible stress corrosion cracks on top of head; ii) mirror image of half of the shaft, color etched top and unetched bottom, intergranular corrosion and differential grain size seen throughout alloy

clenched nails to make them hard enough for use but not too brittle for repeated loosening and clenching. The other clenched nail in the corpus (7288 33539) has 1.3 wt% arsenical content. Stress corrosion cracks in the head of the nail attest to its use lifetime. Some zones display equiaxed grain and twins indicate annealing and hammering, whereas others



**Fig. 15** Artifact 7271 33531, color etched. Note the differential grain size, and differential strain microstructure in this nail shank

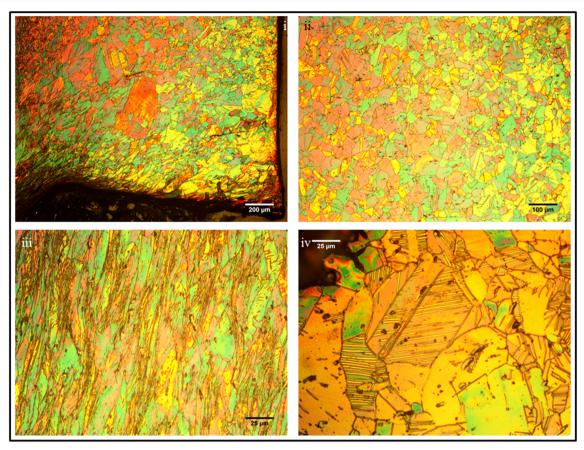
show differential grain size (Fig. 14). Grain boundaries are attacked by intergranular corrosion, especially in the shank. The shank also possesses geometric microstructure on some grains, possibly due to hard hammering of the nail head, visible in Fig. 15. The artifact was excavated from a domestic fill.

Arsenical copper clenched nail 7288 33539 was also mounted in a like fashion with doubly exposed cross sections of the head and shank. The artifact exhibits the swirling microstructure that is characteristic of the cast alloys in this corpus. Heavy strain lines are present close to top of head, indicating hammering either in production or in use lifetime. The swirling and directionality of grains with relatively low twinning indicates annealing followed by cold-working. This nail also has regions with well-preserved strain lines differentially distributed throughout the grain, similar to 7271 33531 (Fig. 16). It was excavated from a layer immediately below what is likely the destruction stratum of Carthage. These last two artifacts, along with 4438 38439, show that certain grains, and perhaps phases of different compositions, will be more resistant to strain than others.

## Leaded copper alloys

#### Middle Punic (480–300 BC)

Leaded arsenical bronze 4444 38448 has multiple phases which reflect varying compositions of the copper, lead, and arsenic components. It is fully corroded and has been subjected to preferential corrosion heterogenization. Another possibility is that this is an ore or intermediate product. The exaggerated laminated corrosion structure that remains would not have been intentional or present in the alloy. Rather, this could represent the phases segregating themselves in the thousands of years of deposition, although the high lead content would not have been soluble and some degree of heterogeneity



**Fig. 16** Artifact 7288 33539, color etched. i) Interface of the head and shank where strain lines are most intense; ii) equiaxed grains of shank, protected from heavy strains and working but still demonstrating

annealing twins; iii) directionality of grains and strain lines near top of the head; iv) strain lines aligning in slip planes in the head

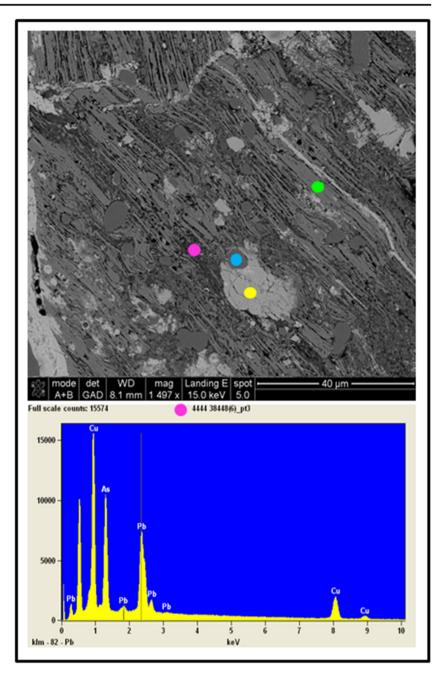
certainly would be found in the metal alloy. The darker phases are arsenic rich, whereas the lighter phases are lead rich, while some phases such as that marked by the pink dot (far left) have almost equal levels of copper, arsenic, and lead (Fig. 17, Table 5). Some phases are also copper sulfides with low arsenic content. Other areas boast lead veins, and nickel impurities (Fig. 18, Table 5). Definitive reconstruction of the original alloy is hampered by the usual changes that the composition undergoes due to corrosion, in addition to the interesting segregated nature of this particular corrosion. Still, averages were taken over all various zones in order to render an alloy determination as accurately as possible.

Leaded tin bronze 2504 45010 was cast poorly which resulted in a great deal of porosity (Fig. 19). There is heavy dendritic coring. Corrosion has attacked the alloy which is often connected to heavy working, evident here by the strain lines. Indeed, the corrosion in some cases has stimulated grain differentiation or stress corrosion cracking (SCC). There are also silver impurities associated with sulfur impurities. The artifact was excavated from a context along with a Carthaginian coin (dated from 370/360–340/330 BC), and pottery dated from 330 to 300 BC.

#### Lead

## Middle Punic (480-300 BC)

Pure lead artifact 1107 38237 was excavated from a compact leveling layer. The typology of the artifact is unknown, but the piece attests to an incredibly high degree of lead refinement technology in the Middle Punic - not surprising as this metal was essentially used as the standard weighing device for commerce as seen in lead weight 2420 42777 (Table 3). Interestingly, weight 2420 42777 is the least pure of the lead artifacts. The weight weighs 18.0 g. It is a six-sided artifact, so one spot was taken on each face. Post-depositional formation processes may have affected the alloy, as silicon, phosphorus, and aluminum had to be excluded from the alloy reading. Only one side had these impurities, and the other six were all at least 98.5 wt% lead. The iron content may be attributed to this as well-but not the tin content which is either an intentional addition, or is the result of recycling. Less likely is that tin leached out of the coin also excavated from this context (dated 370-340 BC). Also possible is the occurrence of tin trace elements in a lead mineralization, and the combination of these **Fig. 17** Artifact 4444 38448. Phase spot analysis and spectrum of pink dot (far left) of ternary alloy with equal parts copper, lead, and arsenic (Table 5)



two metals is attested in other prehistoric and ancient lead artifacts (Pernicka et al. 1982, Table 2; Pernicka et al. 1980, Table 4; Tylecote 1962, Table 39). It might also be mentioned that Valério et al. (2003, 332–333) attributed a Phoenician lead weight of the same typology with a high tin content as a way to deceive customers, but the tin content of the Bir Massouda weight is too low for such an effect.

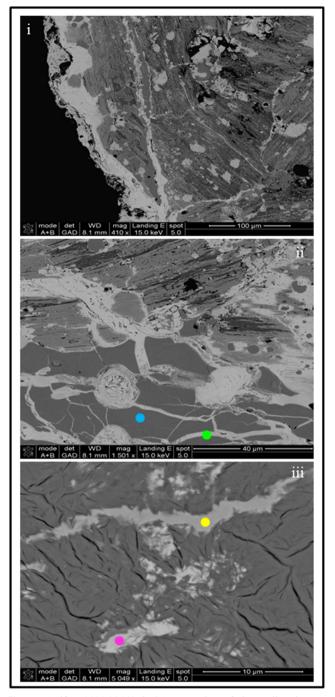
## Late Punic (300-146 BC)

Lead artifact 1068 20176, of unidentified typology excavated from a floor context, also has tin impurities. This is also likely

the result of smelting lead in a mixed furnace also used for bronze, as in the case of the weight above with tin content, and artifact 1107 38237 with attested copper content. For all of the lead artifacts, silver was below the detection limit of the XRF instrument used, not allowing us to comment on the relationship between lead and silver.

## **Cobalt-rich material**

Middle Punic artifact 1093 10191 is a mineralized material without analogies known to the authors, although based upon known uses of cobalt in antiquity it is most likely a glass



**Fig. 18** Artifact 4444 38448. i) GAD overview of phases; ii) vein spot analysis (the green dot (right) on this figure is larger than the actual spot analysis, which captured just the white vein; Table 5); iii) nickel impurities spot analysis (Table 5)

colorant (Tables 4 and 5; Freestone 2008, 93; Henderson 1985; Rehren 2001). Phases of metallic copper prills (~ 90 wt% Cu) indicate that this piece is not an ore, but rather a colorant or pigment at some intermediary stage of its *chaîne opératoire* that has been cooled slowly from a molten state (Fig. 20). The copper prills are not redeposited copper but instead indicate that some copper reduction occurred during

the manufacturing process of the mineral or glass colorant waste. Another less likely possibility is that this piece is a bead or other decorative item abandoned or lost during the production process, or some other final product but too mineralized to discern its original appearance. Blue colorant for Carthaginian terracotta figurines was found to be the copperbased Cuprorivaite (CaCuSi4O10, or Egyptian blue, Karmous et al. 2005), but the blue colorant for Punic glass is otherwise unstudied. The artifact was recovered from a find-rich compacting level below a floor. It was deposited toward the end of the last third of the fifth century BC (430–400), al-though much residual material of the seventh and sixth centuries BC had been included.

#### **Glass colorant debris**

Early Punic artifact 8091 17466 (Fig. 4) represents glass colorant production during the early centuries of the colony. The context from which it comes is a leveling layer in which the metallurgical furnace was set, and furthermore locus 8091 produced the majority of ferrous tuyères from Carthage (Kaufman et al. 2016). Copper, lead, and maybe antimony peaks observed in the pXRF spectra (Fig. 21) would have been intended as colorants for the glass. Copper and iron (from slag) are known to have been used as colorants in other examples of Carthaginian glass (Eremin et al. 2012).

#### Discussion

# Temporally variable alloy diversity as a function of political access

The diversity of copper- and lead-base alloys at Carthage correlates with the archeological and historical data regarding resource procurement, consumption, and trade. During the earlier stages of the Early Punic period, at the height of metallurgical activity, there are just three copper alloys. They are the standard copper alloys that can be expected at most archeological sites of the Iron Age—pure or slightly leaded copper, with some tin and arsenic. Along with the two nonferrous slag pieces, the glass copper colorant debris shows that occasional or incidental non-ferrous alloy and glass production was practiced in Bir Massouda alongside the centralized and intensive ferrous workshop.

By the time that the metallurgical precinct was being phased out of Bir Massouda in the fifth century until right through the end of the Middle Punic (300 BC), the diversity of alloys is greatly increased (Fig. 22). Of ten artifacts, seven are distinctly different alloy types. This corresponds archeologically with the residential levels of the site, and historically from the imperial peak of Carthage abroad. There is evidence in this period of what is likely production of cobalt-rich materials, probably for

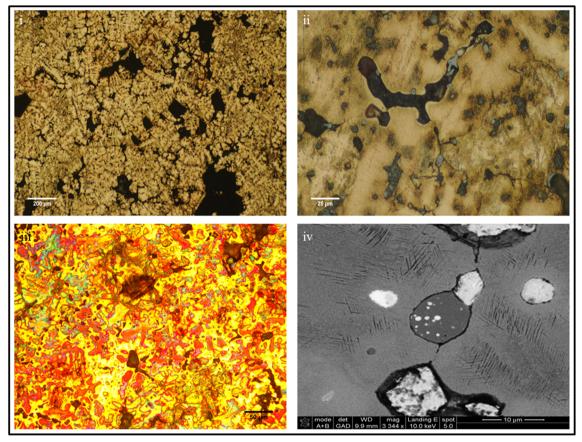


Fig. 19 Artifact 2504 45010. i) Ferric chloride etched optical micrograph showing dendritic coring; ii) strain lines and lead impurities; iii) color etched showing non-granular dendritic microstructure; iv) GAD showing silver-rich white speckles on a sulfidic impurity, also containing copper and arsenic

glass colorant use—the high cobalt, iron, and copper content of the artifact is unlikely to occur naturally meaning that this unidentified object was intentionally subjected to pyrotechnological manipulation. Whatever it may have been, there is no doubt that it is a luxury item. Beautiful blue Carthaginian glass is well known in the Mediterranean, although to our knowledge no other analytical research has been conducted on colorants used. Of the 17 artifacts, four of them (nearly a quarter of all alloy specimens), date to the wellbracketed 30-year period at the end of the fourth century BC

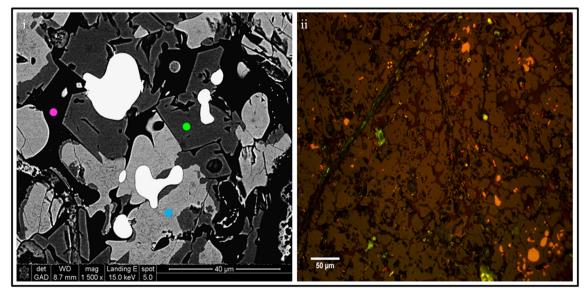


Fig. 20 Artifact 1093 10191. i) EDS phase spot analysis (Table 5); ii) dark field micrograph of cobalt mineral with orange metallic copper prills

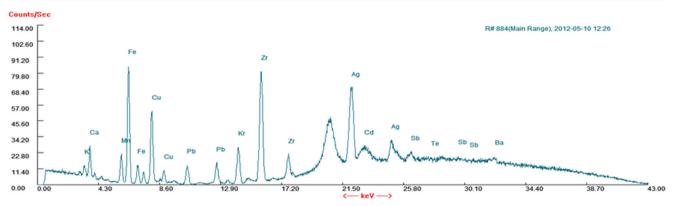


Fig. 21 Artifact 8091 17466. Spectrum of glass colorant showing copper and lead peaks

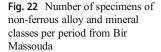
(330–300 BC). This period immediately follows the final fall of Tyre to Alexander the Great (332 BC), placing Carthage as the undisputed leader of the Phoenician and Punic World. It is also the final period where it can be said that Carthage was one of the uncontested empires of the Mediterranean. The military contests officially began with Rome in 264 BC, and it is at this time that the alloy diversity at Carthage is stilted.

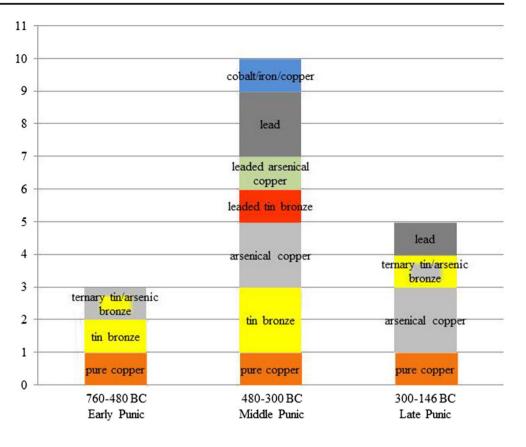
Keeping in mind the limitations of small sample size, the patterns that emerge from the diversity of alloy constituents and types at Carthage can inform future work regarding Phoenician metallurgy and mineral trade. In the Early Punic period, when Phoenician trade interests in the Central and Western Mediterranean were at their most unrivaled peak, tin was readily available. This access to tin did not remain

 Table 5
 EDS spot analysis of selected artifacts

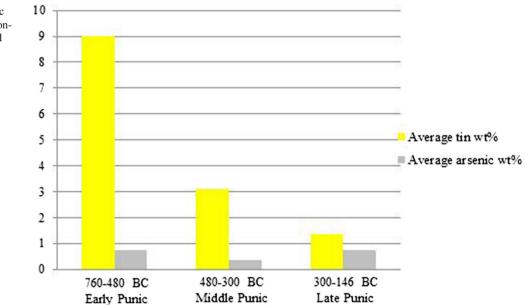
4096

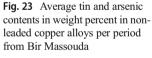
Figure 7iii														
Artifact 7466 40820														
Spot	Cu	Sn	As	Fe	Pb	Ca	Р	Total						
1	46.1	30.0	4.8	_	18.7	_	0.4	99.6						
2	76.1	0.6	0.6	_	18.9	0.1	0.4	96.8						
3	38.4	43.0	2.9	0.8	14.4	_	0.5	100.0						
4	30.6	-	1.1	0.5	55.3	6.3	6.2	100.0						
Figure 12iii														
Artifact 4438 38439														
Spot	Cu	Sn	As	Fe	S	Zn	Total							
Blue (right)	73.2	1.5	0.6	1.7	19.4	3.6	100.0							
Green (center)	90.4	8.4	0.8	_	0.4	_	100.0							
Pink (left)	94.2	2.7	1.0	0.4	1.7	_	100.0							
Figure 17														
Artifact 4444 38448														
Spot	Pb	As	Cu	S	Total									
Blue (center)	-	2.8	75.5	21.7	100.0									
Green (top)	2.1	38.2	59.5	0.2	100.0									
Pink (left)	34.5	33.8	31.7	_	100.0									
Yellow (bottom)	80.4	1.8	6.5	11.2	99.9									
Figure 18ii	00.4	1.0	0.5	11.2	,,,,									
Artifact 4444 38448														
Spot	Pb	As	Cu	S	Total									
Blue (left)	FU —	As 1.9	74.9	23.2	100.0									
Green (right)	_ 67.5	20.3	12.2	-	100.0									
Figure 18iii	07.5	20.5	12.2	—	100.0									
ç														
Artifact 4444 38448	DI.	A -	<b>C</b>	Ni	T-4-1									
Spot	Pb 51.1	As 31.5	Cu 16.3		Total									
Pink (left)				1.2	100.1									
Yellow (right)	37.0	37.0	24.1	2.0	100.1									
Figure 20i														
Artifact 1093 10,191	0.0		<b>a c</b>	41.0	0.0	W O	<b>G</b> 0	CI	7.0	D O		NG	14.6	
Spot	CoO	FeO	CuO	$Al_2O_3$	SiO <sub>2</sub>	K <sub>2</sub> O	CaO	Cl	ZnO	$P_2O_5$	MgO	NaO	MnO	Total
Blue (bottom)	36.4	58.5	1.5	0.6	0.9	_	-	0.1	0.9	_	0.5	_	_	99.4
Green (right)	15.4	71.4	1.3	2.1	1.0	_	0.2	0.1	—	-	-	-	-	91.5
Pink (left)	13.2	26.8	2.5	2.0	36.3	1.5	12.5	_	_	0.5	0.5	3.0	0.2	99.0





stable as the colony of Carthage transitioned into its own imperial center. Tin, as an essential and tactical component of bronzes used for naval fittings, armor, bridles for cavalry, and weapons for infantry, was in great shortage at Carthage during the three Punic Wars. The limited evidence for third century bronzes may indicate both a shortage and that surplus bronze items were requisitioned for military campaigns abroad. Considering only alloys of similar material properties, namely arsenical and tin bronzes, there was an 85% drop in average tin content from the Early Punic to Late Punic (from 9 to 1.35 wt%; Fig. 23). Considering this same group again, there was a reduction in average tin content of 56% from the Middle to Late Punic (3.1 to 1.35 wt%). If the tin content of the softer leaded bronzes is also included, the average tin content from





the Middle to Late Punic (3.18 wt%) stays about the same as it is reduced by 58%. Removing 7466 40820 from the nonleaded alloy list to the leaded alloy list would alter the average tin and arsenic content. The tin average of the Early Punic would then be 3.5 wt%, about the same as during the imperial Middle Punic period. Even in this case, the available average Late Punic tin content would still be less than half of the previous periods, which further supports the argument that Carthage was afflicted by a resource deficit in its final century.

## Local production and outsourcing

Conclusive provenance of the mines and smelting and casting locations of these particular non-ferrous alloys from Bir Massouda is an open question, but archaeometallurgical and epigraphic data indicate a localized production. Certainly for the Late Punic based on historical evidence which commonly refers to the ground and naval forces of the Carthaginian state, much metal production must have been outsourced even as some local production continued—the quantities were massive.

There is evidence of local production from the non-ferrous slag pieces from Bir Massouda, and on the slopes of Byrsa with metal production including some copper base alloys (Tylecote 1982). Casting molds were recovered from Late Punic Bir Massouda, but only for coins, not other artifact types (Docter 2005; Frey-Kupper 2009). Further evidence for local production at Carthage comes from epigraphy. Carthaginian copper alloy specialists and casters are attested through several inscriptions. The city had a rich tradition of bronze casters and metalworkers, in addition to ironsmiths, forgers, master smiths, and goldsmiths (Kaufman 2014: 110–125 and citations therein). It is therefore not surprising to see the high level of quality and control that these metallurgists employed in their trade.

Above, the microstructural characteristics found by Keesmann (2001) and the interpretations of Tylecote (1982) were discussed. Experimental results of the two non-ferrous slag pieces from Bir Massouda further confirm Tylecote's conclusion and Keesmann's microstructural observations of what may be contemporary bronze slags (Fig. 5; Kaufman et al. 2016, Fig. 10). The Bir Massouda non-ferrous slags do point toward recycling practices. Whether or not any number of these bronzes were produced at Carthage or imported, in both cases they reflect the reach of the Carthaginian state as it relates to mineral resources as the raw materials had to be imported from further afield such as Sardinia or the Iberian Peninsula. Furthermore, Carthaginian outsourcing metal production sites such as at Tharros in Sardinia and Lixus in Morocco (Attanasio et al. 2001; González-Ruibal 2006) show that the alloys could easily have been produced at other production sites across imperial territories, with raw mineral resources obtained from Cyprus, the Levant, or elsewhere in

Africa via their extensive maritime and commercial network. Future isotopic research on the Bir Massouda finds may help refine the geographical sources of the raw materials.

## Conclusions

Applying archeological, archaeometric, historical, and epigraphic perspectives and methods to diachronic datasets allows for sound interpretations to be proposed on long-term changes in the political economy of ancient cultures. Despite the small sample size in this study, representing the entirety of well-dated excavated non-ferrous materials from the Carthaginian urban capital, the diversity of alloy types reveals Carthaginian tastes and may inform contemporary assemblages within a Mediterranean context. The activities of Carthaginian smiths and maritime traders are recorded through the changes in alloy and mineral resource consumption recovered from the archeological record.

The diversity of alloy types corresponds well with the shifting fortunes of the Carthaginian state. Historical and archeological records attest to the rise and later slipping control of Carthage within the urban center and abroad. These data converge with archaeometallurgical evidence demonstrating a sharp increase in Carthaginian mineral exploitation capabilities after the sixth century BC ascent of Carthage over its Tyrian parent state including an array of non-ferrous alloy types, cobalt, and lead. The reorientation of trade, settlement structure, and mineral rights seen in the sixth century BC Iberian Peninsula is corollary with an influx of Carthaginian imports, and the beneficial access gained by the Carthaginian state to these territorial mines is also seen through the nonferrous assemblage at Bir Massouda. High quality non-ferrous alloys and minerals were produced at the capital by skilled smiths and artisans who memorialized their activities on ritual inscriptions. The imperial peak of the Middle Punic period is followed by a loss of mineral access and reversion to a basic alloy assemblage in the third century BC onwards. This shows that a tin shortage existed in the capital during the Punic Wars.

Lancel (1998, 182) surmised that following the Second Punic War, "Carthage had lost the exploitation of Spanish mines, and very probably control of the tin route, as well." Recent preliminary research shows that Roman access to silver, limited before the Second Punic War to occurrences in the Aegean, expanded abruptly into silver mines on the Iberian Peninsula after 209 BC (Westner et al. 2017). Livy reports that immediately following the Second Punic War, silver tribute that the Carthaginians brought to Rome was assayed and found to be debased, comprised of 25% base metal (*Ab Urbe Condita*, 32.2.1-3). Mineral wealth passed from Carthaginian to Roman hands, both in terms of access to mines and ownership of metals. These multiple lines of archaeometric and historical evidence, of which the decreasing tin content from Bir Massouda is one, show clear evidence for shortages and the eventual irreparable loss of valuable, strategic wealth finance and military hardware for the Carthaginian state—one aspect of its decline and ultimate demise.

However, as can be expected from such societal trauma as the loss of the Second Punic War and Roman seizure of the entirety of its territorial holdings outside of urban Carthage, the decline of the Carthaginian state was not steady but rather characterized by an increase in economic volatility (Hoyos 2015; Wolff 1986, 240–243). The Carthaginian mercantile elite salvaged enough of its network to offer the full 50-year war indemnity that Carthage owed to Rome-10,000 Euboic talents = 260,000 kg of silver—after a mere 10 years (Lancel 1998, 182). Rome refused. Reputedly, Carthage supplied 400,000 bushels of wheat to Rome in 200 BC, and again offered 500,000 bushels for free in 191 BC, surpluses that alarmed Cato the Elder sufficiently to eventually call for the destruction of the city (Ab Urbe Condita 31.19.2; 36.4.9; Lancel 1998, 183). Even if we are to reduce Livy's numbers considerably, such an enormous potential to acquire silver and supply grain when under embargo, albeit marked by apparently extreme surplus commodity volatility and uncertainty, provides a window into the capacities of the state before the loss to Rome in the Second Punic War.

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