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## FIRST DISCOVERY OF ORICHALCUM INGOTS FROM THE REMAINS OF A 6<sup>TH</sup> CENTURY BC SHIPWRECK NEAR GELA (SICILY) SEABED

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### ABSTRACT

Ingots recently recovered from the seabed near Gela, a major harbour of Sicily, reveal an unexpected side of ancient metallurgy. The ingots were found near remains of a ship and earthenware dated around the end of the VI century BC and probably coming from the eastern Mediterranean and the Aegean sea. The ingots were analysed by means of X-Ray Fluorescence spectroscopy via a portable spectrometer. Results indicate that they are mostly consist of copper and zinc although many of them have a significant amount of lead. This alloy is nowadays called brass, but in ancient time it was known as orichalcum, one of the rarest and most precious alloy along with gold and silver. Only small items of orichalcum dating before Christ were found so far. The visual examination corroborated by evaluation of dimensions and weight, are consistent with the dating hypothesis and reveals important information about the casting production. The discovery of more than twenty-two kilos of ingots is extraordinary: a first ray of light upon a forgotten technology, which involved also smelter plants (maybe more than one), a commercial network, and a number of end users, who certainly appreciated the properties of shining orichalcum: ductility, mechanical strength, durability, and value.

**KEYWORDS:** Orichalcum, ingots, X-rays fluorescence, archaeometallurgy.

## 1. INTRODUCTION

A major archaeological discovery has recently occurred in the gulf of Gela, a town on the southern coast of Sicily which was established in 689 BC by Doric colonists coming from Rodhes and Crete. A unique archaeological treasure of brass ingots was hidden in the sandy seabed few hundreds meters East of the ancient town, in an area called Bulala, where ships were moored awaiting their turn to access the city harbour. In the VI century BC, Gela was the most powerful town of blooming Sicily, and a major trading centre of the Mediterranean. It comes as no surprise that the entire gulf of Gela is an archaeologist paradise; three full shipwrecks of the Greek period have been located, and one of them has been already recovered and restored: it will be soon displayed in the Gela museum (Panvini and Benini, 2001; Vullo, 2013).

This discovery consists of 40 metal ingots which were carried by a ship that wrecked near the Gela coast. Pieces of pottery have been found nearby, which have been dated around the end of the VI century BC, and come from the Aegean Sea and the eastern Mediterranean.

Some of ingots look gold-lucent. Corrosion products, with colours (green, azure) suggesting the presence of copper, cover many of them. A wide range of copper alloys, containing tin, lead and zinc was manufactured in ancient times; their classification is determined by the relative concentrations of these elements. The lucent gold-like appearance immediately suggests an alloy containing copper and zinc similar to modern brass that, due to the estimated age of the ship, may be identified with the legendary orichalcum, a very precious alloy of ancient times.

Plato describes the ports and forts of Atlantis as covered by a rare *metal* called ὀρείχαλκος (Balouglou, 2010). The Greek word ὀρείχαλκος (ὄρος, upstream and χαλκός, Copper) is attested in several literary sources like the pseudo-Homeric *Hymn to Aphrodite*, where the birth of Aphrodite is described. Aphrodite is presented to the council of the Gods wearing divine garments decorated with *orichalcum flowers*. At the beginning of the VII century BC, Hesiod in the poem *The Shield of Heracles* describes the greaves made by *shining orichalcum as a beautiful gift of Hephaestus*.

In ancient times, the production of orichalcum was long and laborious since it was made with the cementation process (see below). The archaeological evidence of orichalcum objects is not abundant; the earliest findings are from the IV - III millennium BC from the Yangshao and Longshan periods in several sites of central and eastern China (Craddock, 1978;

Forbes, 1964). In the VIII century BC we have the orichalcum fibulas found inside the Great Tumulus at Gordion, the Phrygian capital (Young, 1981). In the Indian subcontinent, several small objects of orichalcum have been dated between the Indus Civilisation period (Harappa: 2500 - 1500 BC) and the Hellenistic Gandhara period (Taxila: IV - I century BC) (Biswas et al., 1990).

Christopher Thornton divides orichalcum samples in six groups using the list compiled by Caley (1964): Group 1 is located around the eastern Aegean sea (first half of the 3<sup>rd</sup> millennium BC) and includes ornaments coming from Thermi (island of Lesbos) (Stos Gale, 1992; Gale, 1997). This discovery could be associated with the mines of Argenos on the northern shore of Lesbos that contain deposits of copper oxides and sulphides as well as lead and zinc sulphides (Pernicka et al., 2003). Group 2 is made by nearly 3000 artefacts from southern Mesopotamia dating from the mid/late 3<sup>rd</sup> millennium BC (Hauptmann and Pernicka, 2004). Group 3 consists of objects coming from the circus-Caspian region (including south central Asia) dating from the second half of the 3<sup>rd</sup> millennium BC (Egor'kov, 2001). Group 4 includes objects coming from the eastern Persian Gulf region dating from the late 3<sup>rd</sup>/early 2<sup>nd</sup> millennium BC (Frifelt, 1991). Group 5 objects are coming from two mid-2<sup>nd</sup> millennium BC sites in northern Mesopotamia (Ugarit and Nuzi) (Schaeffer-Forrer et al., 1982). Group 6 consists of objects coming from eastern Anatolia, northern Iraq, southern Caucasus and western Iran dating from the early 1<sup>st</sup> millennium BC (Hanfmann, 1956). Also of note are the two IX century BC Assyrian bowls from Nimrud (Hughes et al., 1988). In the Mediterranean we have to add a finger ring found in Ugarit (eastern Mediterranean coast) dating from the XIII century BC with a percentage of zinc of about 12%. The most ancient orichalcum object found in Italy is a boat-shaped fibula displayed at the British Museum (private collection) dating from the VII century BC (12,4% Zn, 85% Cu, 1,7% Sn, 0,7% Pb with traces of Fe, As and Bi) (Giardino, 2010).

Two ingots (in the middle upper side of Figure 1) are connected by a metal bridge, a spill during the casting process. In the following, the ingots will be indicated with S# (S1-S38); ingots S39 and S40 were not considered because they were out for an exhibition.

All samples have been measured and weighted. The composition of the ingots was investigated in situ by means of X-Ray fluorescence spectroscopy (XRF). This technique is non-invasive and non-destructive and can be performed by portable instruments (pXRF).



Figure 1. Ingots in the Gela Museum

## 2. METHODS

The homogeneity of the alloy composition was tested by analysing different points in several of the 5 ingots that had been polished for exposition purposes.

For this reason three points for each side of the ingots were analysed as show in Fig 2.

In the case of the unpolished ingot the same procedure was applied, by choosing points also covered by patinas.



Figure 2 Pictures of the polished ingot, stars indicate the points of analysis



Figure 3 Pictures of the unpolished ingot, the stars indicate the points of analysis

A portable XRF, spectrometer Tracer III SD Bruker AXS, was used. The measurements were performed by placing the head of the spectrometer in contact with the selected points without manipulation of the sample. The detector of the instrument is a 10 mm<sup>2</sup> silicon drift X-Flash with Peltier cooling system and a resolution of 145 eV at 100,000 cps. The source is a Rhodium Target X-Ray tube operating at 40 kV and 11 mA, with two different filters to achieve good sensitivity both at lower and higher energies (up to Ba/K-lines). Each spectrum was acquired for 30 s and a window of 3÷4 mm in diameter determined

the sampled area. This portable instrument allows the detection of elements with atomic number  $Z > 11$ . The window of the instrument was placed in contact with the sample surface. The S1PXR® Software manages data acquisition, spectral assignments and computation of concentrations trough calibration (Bruker, 2013). The approach of an empirical calibration was used for the calibration by a variant of the common linear model developed by Lukas-Tooth and Price (Drake, 2016).

Spectra of some brass standards were supported by Bruker and contained in the file Cu1.cfz, the

ranges of standard elements compositions covered the ones of the ingots.

### 3. RESULTS

#### 3.1. Elemental analysis

The homogeneity of the composition was checked in several ingots by analysing different points for each of them. In particular, for the polished ingot S33 the average %w/w concentrations and standard deviations on 9 points are: [Cu] =  $80.2 \pm 0.2$ , [Zn] =  $16.3 \pm 0.1$ , [Pb] =  $2.2 \pm 0.2$ . For an unpolished ingot (S37) we obtained [Cu] =  $75.4 \pm 0.4$ , [Zn] =  $19.9 \pm 0.2$  and [Pb] =  $4.8 \pm 0.6$ . The standard deviations are low, implying a homogeneous composition for all ingots; the unpolished sample with the patina has somehow larger standard deviations but it indicates that, under all circumstances, XRF yields reliable composition data for the most abundant elements. Therefore, the subsequent analyses upon the other samples were performed on a single spot without polishing. Of course, the relative errors with trace elements are much larger, and these elements will be ignored in the following.

Results for the 38 ingots are reported in Table 1. The copper/zinc weight ratio is reported in the same table.

*Table 1. XRF compositions and copper on zinc weight ratio.*

	% w/w			[Cu]/[Zn]
	Cu	Zn	Pb	
S01	76	20	2.9	3.7
S02	79	14	4.3	5.7
S03	70	26	1.6	2.7
S04	75	18	5.7	4.1
S05	75	21	2.0	3.6
S06	77	20	3.3	3.9
S07	78	19	2.9	4.1
S08	76	21	3.2	3.7
S09	78	16	4.6	4.8
S10	70	26	2.7	2.7
S11	78	17	3.7	4.5
S12	75	17	6.8	4.4
S13	76	18	2.6	4.2
S14	70	26	1.8	2.7
S15	79	17	4.0	4.7
S16	80	17	3.7	4.8
S17	75	18	4.8	4.1
S18	79	16	3.5	4.8
S19	71	26	2.0	2.8
S20	79	17	2.6	4.5
S21	77	20	1.9	3.8
S22	73	20	4.8	3.7
S23	77	21	2.4	3.7
S24*	79	16	5.1	4.9
S25	79	17	1.0	4.6
S26	77	19	3.2	4.1
S27	75	18	6.4	4.1

S28	74	18	5.2	4.1
S29	70	25	3.1	2.8
S30	82	14	1.7	5.7
S31	74	22	1.6	3.3
S32	75	17	6.0	4.4
S33	80	16	1.8	4.9
S34	81	14	3.5	5.7
S35*	78	16	5.5	4.9
S36	81	17	1.5	4.8
S37	75	20	4.8	3.8
S38	82	15	1.0	5.5

\*The compositions of the two ingots connected by a metal bridge (S24 and S35) are the same (The figures are rounded to the last significant digit).

#### 3.2. Visual examination

The shape, section and the surface morphology of the ingots suggest a casting mono-valve mould production. No immediate evidence suggests the use of different production techniques, but two main forms can be recognized: one cigar-like with a triangular section, the other flatter and wider with an extended elliptical shape and a semi-ellipsoidal section (see Figure 1). The ingots have a variety of colours, shapes, dimensions and weights. The surfaces are mostly grey-black or reddish but occasionally the patina has azure-blue or green tones due to the oxidation products. In two ingots the patina is green.

Most ingots have sharp contours and a rough upper surface - i.e., the surface that was exposed to air during casting - while a few have jagged contours. The surfaces that were in contact with the mould during casting are more regular, smooth and flat, but they indicate that a rough refractory material was used as mould. Probably, the moulds were simply pits in the ground near the furnace; each pit was used just once.

Ripples are often present on the upper surface exposed to air during cooling. These ripples are due to a rapid solidification of the melted alloy, whose temperature was substantially above its melting temperature. The black crusts seen between the ripples are due to foams containing lighter impurities that separated during the fusion. The presence of relatively abundant impurities suggests that the ingots were obtained with a single casting, or primary production process, i.e., the often used procedure of recasting to improve purity was not applied to these ingots. Some of the impurities, included within the metal, created cavities. Details of ripples, black crusts, impurities and cavities can be observed in the details of ingots S23 and S30 reported in Figure 4.

The poor defined shape of this set of ingots is very unusual by considering the high value of the objects, it is worth to underline that to our knowledge is the first time that orichalcum is found in form of ingot.

Regarding other metal ingots found in shipwrecks, their shape is better defined and they are more regular in dimension, e.g. the lead roman ingots which also contain the brand of the producer (Tisseyre et al., 2008) or the copper ingots with the arms shape very useful for the transport (Galili et al., 1986).

The lengths, widths and weights of 38 ingots were measured; results are reported in Table 2. The lengths and the widths range from 15 to 40 cm, and from 2 to 6 cm respectively. The weights range between few hectograms to more than 1 kg.

Table 2. Lengths, widths and weights of the 38 ingots.

Sample	Length (cm)	Widht (cm)	Weight (g)	Sample	Length (cm)	Widht (cm)	Weight (g)
S1	28.7	2.7	580	S20	28.3	2.4	482
S2	30.7	2.6	641	S21	31.1	2.7	563
S3	29.0	3.1	476	S22	28.8	6.7	1307
S4	28.8	3.2	615	S23	28.2	2.5	460
S5	23.6	4.1	810	S24	23.4	2.5	~390
S6	27.0	2.8	391	S25	23.4	3.1	511
S7	26.4	3.3	397	S26	27.0	2.4	440
S8	23.2	4.3	305	S27	31.7	2.5	530
S9	30.3	2.2	545	S28	27.4	6.1	264
S10	28.1	2.7	545	S29	30.2	3.4	670
S11	31.1	2.5	509	S30	31.5	3.0	820
S12	27.6	2.8	413	S31	24.9	2.8	421
S13	17.7	3.4	275	S32	17.6	6.3	909
S14	30.2	2.4	436	S33	32.1	3.6	1339
S15	31.5	2.7	568	S34	33.8	3.1	1052
S16	17.2	2.4	254	S35	24.5	2.5	~390
S17	20.4	5.9	983	S36	23.0	3.5	532
S18	31.0	2.8	573	S37	29.0	2.5	495
S19	27.8	2.3	382	S38	30.0	4.8	1155

Figures are rounded to the last significant digit.

Half of their overall weight was assigned to the S24 and S35 connected ingots.



Figure 4. Details: of ripples ● and black crusts ▲(S23); impurities ◆ and cavities ■(S30)

#### 4. DISCUSSION

The elemental compositions of all ingots, with [Zn] < 26% w, are within the range of modern brass and of the ancient objects made of orichalcum. The lead concentrations range from 1.1% to 6.8%w, implying a voluntary addition of the metal, very certain for the higher concentrations. The presence of lead is useful when a large casting, as in a statue, is performed since it lowers the melting point of the alloy at the price of reduced mechanical properties. The

copper/zinc weight ratio distribution is reported in Figure 5.

The wide distribution of the Cu/Zn (w/w) ratio may indicate that the manufacturer had not a preferential value of composition, or a reliable way to control it; alternatively, the ingots came from different furnaces, possibly using raw minerals from different geographical areas.

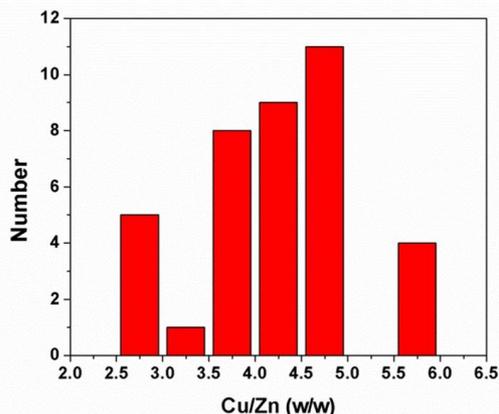


Figure 5. Copper and zinc weight ratio distribution

The so called cementation process was applied to the production of brass from the antiquity up to the XVI century AD. The zinc content (in general, lower than 26% w/w) is compatible with the brass production using the cementation process. In fact, it has been claimed that it is impossible to attain a Zn concentration  $> 32$  w/w with the cementation process; this limit, also known as the magical limit, helps discriminating between modern and ancient brass (Bourgarit and Bauchau, 2010).

The cementation process consists of a series of chemical reactions in a “sealed” crucible at temperatures of about  $1000 \pm 100$  °C. Firstly, the Zn contained in zinc ores such as sphalerite ( $ZnS_2$ ) or

smithsonite ( $ZnCO_3$ ) is reduced by heating; then, the Zn vapor partly diffuses into the Cu to form the Cu-Zn alloy. The temperature should be maintained between 917 °C (Zn vapors) and 1083 °C (melting point of Cu).

The development of such a complex process implies a deep knowledge of the raw materials, and the ability to set the conditions required to obtain the alloy with the desired properties; furthermore, the delicate range of the high temperatures needed and the use of sealed crucibles require major technological developments. But even the most skilled artisan would have had hard time in controlling the diffusion of Zn vapors into the molten Cu.

A study of the elemental composition of more than 1200 copper alloy objects from northern Britain archeological sites finds a Zn content below 23% w/w, which indicates a poor cementation technology (Dungworth, 1997). The average Zn content in the artefacts classified as brass was 18% w/w. However artefacts dated from the XV century BC made of a copper alloy containing about 20% w/w of zinc were discovered in southeastern Iran (site of Tepe Yahya) indicating the availability of this technology long before the Greek and Roman era (Thornton *et al.*, 2002).

The distributions of lengths, widths and weights are reported in Figure 6.

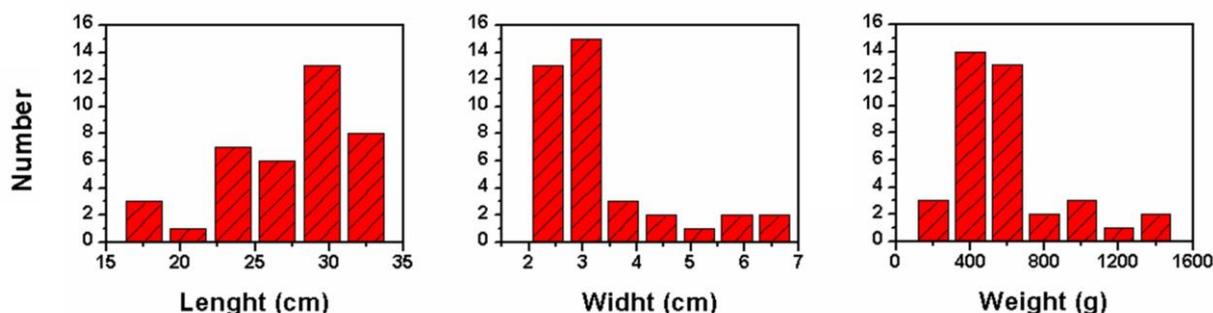


Figure 6. Ingots lengths (a), widths (b) and weights (c) distributions

All the distributions are wide with an asymmetric shape; the most probable length is shifted towards higher values while the most probable width and weight are shifted towards lower values. More than a half of the samples have widths and weights in the range  $2.0 \div 3.5$  cm and  $350 \div 650$  g, respectively. Probably, the artisan-clients using the ingots favoured cigar-shaped ingots, easier to turn into larger and thinner ornaments by cold forging. Accordingly, the lengths distribution is larger, and no preferential

value can be assigned. The morphological analysis reveals a poor moulding and casting technology. These findings, and the pottery retrieved at the site of the wreck dated from the end of the VI century BC, suggest production of ingots by oriental plants within a Greek-dominated Mediterranean sea-trade.

In ancient Greece, the brass was known as orichalcum, a precious material that had a commercial value second only to that of gold and silver. The discovery of orichalcum ingots is important both for the

unheard amount and because it adds this alloy to the list of metals which were commonly traded as ingots in the Mediterranean: lead, tin, bronze, copper and iron.

## 5. CONCLUSION

We report a first analysis of antique ingots, recovered from the sea near Gela. The site of the finding, and the pottery recovered nearby, are consistent with an origin going back to the VI century BC, when Gela ruled most of Sicily. The major components of the ingots are copper, zinc and lead, as for the modern-day brass; the zinc content is compatible with the composition range of orichalcum, the most precious alloy of ancient times.

Ingots morphology suggests a casting mono-valve mold production. The mold surfaces are poorly defined, implying the use of a rough refractory material. The presence of ripples on the surfaces exposed to the air during the cooling process, due to a rapid solidification of the melted metal, indicates that the temperature was near the melting temperature of the alloy. The presence of numerous impurities among the ripples suggests a primary production of the alloy, with no melting/refining steps, and a rudimentary casting technique. Despite this, the technology involved in the production of orichalcum is quite complex and it comes as a surprise that the trade of

this alloy was so articulated that hundreds of miles separated the sites of “industrial production” of ingots from the end users.

The discovery of a huge amount of orichalcum ingots in the sea of Gela in the area of a VI century BC wreck gives a great contribution to the knowledge of ancient Mediterranean sea routes and trade. It opens new scenarios of historical research related with the role of Gela, a rich Mediterranean trade and cultural crossroad, and with the amount and nature of the material, not comparable with that of the small artefacts assigned to the pre-Roman period. It is reasonable to believe that we will be able to have more insight on the sources of the base materials through an accurate characterization of the trace elements, to have more hints about the location of the plants, and a better understanding of the steps involved in the production of the ingots and of the technological tradition that goes with it. In any case, we have now to reconsider the possibility that the walls, or the portion of wall surrounding the main gateway of a town Atlantis like, could have been covered with orichalcum; with a proper supply of Gela-like ingots and enough slaves, any metal worker at the time of Plato would have easily achieved what Plato was speaking about.

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