APPENDIX V

Chemical analyses of metal artefacts from Late Cypriote tombs excavated in the Limassol area, with the employment of pXRF

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Introduction

The excavations of the Late Cypriote tombs in the Limassol area brought to light a number of metal artefacts made of copper alloys, gold and silver. As the tombs date to the beginning of the Late Cypriote (they range in date from the LCI – the LCII) it was decided that it would be worthwhile to analyse the objects made of copper alloys in order to identify the types of alloys used for the different types of objects and to compare the results with those from objects dating to earlier and later periods. The analyses of the objects were carried out as part of an analytical project entitled 'A diachronic Study of Cypriote Metalwork' undertaken by Andreas Charalambous and coordinated by Vasiliki Kassianidou. The study is part of the NARNIA (New Archaeological Research Network for Integrating Approaches to ancient material studies) Project. NARNIA is a Marie Curie Initial Training Network which is funded by the FP7 and the European Union. For more information please visit the NARNIA website: http://narnia-itn.eu/.

Non - destructive analysis of metal artefacts is now possible thanks to the use of portable XRF analysers which are becoming more and more popular.

For the present study, a total of thirty-two metal artefacts were analysed. The assemblage consists of weapons (daggers and a spearhead), ornaments (rings of various sizes and pins) and small amorphous lumps of metal. Of the analysed objects there are some that stand out. The first is the dagger with a bone hilt and three rivets which are covered with gold leaf. The second is a small group of amorphous lumps which were initially thought to have been slag but were in fact found to consist almost entirely of copper metal. With the exception of a single ring made of silver (T.278/5) which was initially mistakenly identified as made of a copper alloy, none of the other objects made of precious metals were analysed.

Method of Analysis

A portable Innov-X Delta ED-XRF analyser (pXRF) was applied to the analyses and the determination of the elemental composition of metal artefacts from the excavations of Late Cypriot tombs in the Limassol area. The specific instrument, which belongs to the Archaeological Research Unit of the University of Cyprus is equipped with a 4W, 50kV tantalum anode X-Ray tube and a high performance Silicon Drift Detector (SDD). The X-rays are emitted by the miniaturized X-ray tube and the use of a collimation coin can change the diameter of the X-Ray beam (smaller than 3 mm). The measurement time for each spot analysis was 70 seconds. The final measurement value for each analysed object is the mean value of 3 to 5 measurements conducted on corrosion-free areas of each object. In some artefacts where more clean areas were detected, more than 5 measurements were taken. In the case of the metal lumps which we were allowed to sample (see discussion below) the measurements were performed on fresh-cut cross sections as well as on the surface of the objects for comparison reasons.

The analytical mode of the instrument employed for the analyses of these objects is Alloy Plus. For this mode Beam 1 (40 kV) analyses the elements Ti, V, Cr, Mn, Fe, Co, Ni, Cu, Zn, Hf, Ta, W, Re, Pt, Au, Pb, Bi, Zr, Nb, Mo, LE (light elements), Pd, Ag, Sn and Sb, while Beam 2 (10 kV) is used for the determination of Mg, Al, Si, P and S. In order to determine the concentration of arsenic (As), the objects were also analysed using the Mining mode of the instrument. The analytical data and other information concerning the experimental conditions were exported into an Excel spread sheet for further mathematical treatment. Alloy analysis utilizes a Fundamental Parameters algorithm to determine elemental concentration. This method calculates chemistry from the spectral data, without the requirement of stored fingerprints. Also, for checking the accuracy of Alloy Plus and Mining (for the case of As) modes, standard reference materials (SRMs) like CRM-875 (bronze standard) and BCR-691 (set of 5 copper alloys) were applied.

Results

The results of the analyses are presented in Table 1 and they are discussed below.

Artefacts made of copper alloys

Compositional analysis showed that twenty-two of the metal artefacts from the Limassol area tombs which were studied are made of copper alloys. Twenty of them have a tin (Sn) concentration ranging between 2.2 and 14.2% and can therefore be classified as bronzes. The earliest objects of bronze from Cyprus date to the Philia period (Webb *et al.* 2006, 274), but the alloy takes quite some time to become established. Thus, even until the Middle Cypriote III (1725-1600 BC), only about half of the objects produced, were made of bronze or tin-arsenic-copper alloys. Furthermore, the smiths seem to reserve this new alloy only for certain types of the limited repertoire of metal objects (Weinstein Balthazar 1990, 431). At the beginning tin-bronze was used for riveted knives and toggle pins, while in the Middle Cypriote III period shaft hole axes were also made of this alloy. Hook tang weapons, scrapers, awls, tweezers and pins were still made of arsenical bronze until the end of the Middle Bronze Age. In the Late Bronze Age when Cyprus begins to export large quantities of copper, tin becomes widely available and the use of arsenical copper is abandoned (Kassianidou 2003, 111-112). Nevertheless, as an imported raw material in Cyprus as elsewhere in the eastern Mediterranean tin was more expensive than copper.

Two artefacts, the spearhead from Tomb 11 (T.11/10) and a ring from Tomb 8 (T.8/25), were found to have a very low concentration of tin (0.2 and 0.3 respectively). Such a low concentration is highly unlikely to have been the result of a deliberate addition of tin to copper in order to produce bronze. On the other hand Cypriote copper ores do not contain tin and thus tin would not have been present in the raw copper (Muhly 1985, 277). Its presence may be explained by the melting together of copper metal with a small quantity of bronze scrap (Weinstein Balthazar 1990, 72-73).

If one looks at the concentration of tin in relation to the type of object and the archaeological context it becomes clear that daggers from Tomb 322 and Tomb 323 all have a tin concentration higher than 8%. According to the archaeological evidence Tomb 322 and Tomb 323 date to the LC IIA:2 and LC IIB respectively. Indeed the object with the highest tin concentration (14.2%) is the dagger with a bone hilt and rivets that are covered with a thin sheet of gold (T.322/23). Clearly this object was one of great value as it stands apart from the others both by the choice of alloy used to produce the blade and by the combination of materials used to assemble it (although we cannot be sure that the other daggers did not also have gold plated rivets).

The daggers from 621-IV and 621-V, which according to the archaeological evidence date to the LC IIA, have a tin concentration of 6.9% and 6.5% respectively. The lowest concentration of tin among the weapons of this assemblage is observed in the dagger from Tomb 8 (T.8/29) and the sword from Tomb 11 (T.11/10). They contain 2.2% and 0.2% of tin respectively. Both tombs have been dated to the LC I or LC II based on the typology of the ceramics and are thus earlier than the tombs with daggers of high tin concentration discussed above. According to Catling (1964, 110) the hooked tanged weapons, which first appear in the Early Cypriote, do not survive after the LC I. The sword should, therefore, be dated to the LC I. This early date may explain its chemical composition and low tin content which is similar to the chemical composition of objects of this type dating to the end of the MC III (Weinstein Balthazar 1990, 308 and Table 73 on 321).

As far as the rings are concerned, there is some variability in the concentration of tin. Even within the same tomb, for example Tomb 8, the tin concentration of the rings ranges from 0.3% to 9.5%. The choice of alloy in this type of object would have had less to do with its mechanical properties and more with the colour of the alloy. A bronze with a tin content of 9.5% has a colour very similar to gold and thus the alloy may have been chosen to imitate the precious metal.

Most artefacts have no or an extremely low arsenic (As) content. Arsenic is present only in ten objects and the concentration ranges 0.03% to 0.4%. The arsenic level is very low and it is again highly unlikely that the specific element was added deliberately. Its presence may be the result of smelting polymetallic copper ores which contained small amounts of arsenic. Such ore deposits can be found at Laxia tou Mavrou and Pevkos which are located in the area of the Limassol Forest (Gass *et al.* 1994, 183-184) (see Fig. 1). These deposits would have played an important role in the earliest phase of the Bronze Age for the production of arsenical bronzes, as they are the only arsenical ores on the island (Swiny 1982, 71). The fact that the objects that contain arsenic also contain significant levels of tin (for example T.322/21 and T.323/10) clearly indicates the melting of copper and tin together with scrap made of arsenical copper (Weinstein Balthazar 1990, 78).

Lead (Pb) has been detected in fifteen objects, ranging from 0.02 to 0.64%, while in two cases the amount of lead is much higher, 1.9 and 6.2%, respectively. Both objects with the higher lead concentrations are pins (621-VI/7 and 621-VI/24a). Lead is not present in Cypriote copper ores (Constantinou 1982, 15) so its presence in these artefacts can only be interpreted as a deliberate addition. Lead is a common additive to bronze, improving fluidity and cast ability, but it reduces the alloy's hardness and toughness, when added in a higher amount than a few per cent (Craddock 1977).

Iron (Fe) is found in all objects in the range 0.05 to 1.6%. The iron found in artefacts made of copper alloys is almost certainly residual from the smelting process: it is either coming from the smelting of sulphidic ores which contain iron or from the addition of iron oxides as flux in the smelting charge (Ingo *et al.* 2006; Ashkenazi *et al.* 2012). Cypriote copper ores are sulphidic and, therefore, the presence of small concentrations of iron in the objects is not unexpected. Interestingly the highest concentration (1.6%) of iron was detected in two objects (T.8/25 and T.11/10) which have almost no tin and are early in date (LC I or LC II).

Amorphous lumps of copper metal

Among the studied objects were nine samples of amorphous copper lumps. The lumps come from four different features from the same site, namely Enaerios: 621-V, 621-VI, 621-VII and LM 1328. They were found in small concentrations within each of these contexts and they were initially identified as pieces of slag. Unlike the finished objects, we asked and were granted permission to sample some of the lumps which were covered by corrosion products and soil, in order to achieve better analytical results. Sectioning of the lumps immediately revealed that they were in fact made of metal and not slag (Fig. 2). They are, therefore, extremely important as they are some of the earliest examples of raw metal from Cyprus.

It is well known that in the Late Bronze Age copper was traded in the form of ingots which had a plano-convex, an oval or an oxhide shape (these are best illustrated in the cargo of the Uluburun ship, which dates to the end of the 14th century BC (Pulak 2008, 307-310)). The latter are the most popular and the ones which are usually connected with Cyprus (for a detailed discussion see Kassianidou 2009) but the earliest examples of oxhide ingots from the island date to the 14th century BC (Kassianidou 2009, 58). We know that Cypriote copper had been traded in the form of oxhide ingots already since LC I because of the results of Lead Isotope Analysis on complete and fragmentary oxhide ingots from the LM IIB sites of Mochlos, Zakros and Gournia which found them to be consistent with a Cypriote provenance (Stos Gale 2011, 223). We do not know the type of ingot used in the Middle Bronze Age during which Cyprus began to export Cypriot copper.

According to the archaeological context, these lumps, which cannot be called ingots or fragments of ingots as they are very small and do not seem to have been part of a shaped object, date to the LC IIA and LC IIB. Thus they are contemporary with the earliest oxhide ingots from Cyprus, which were found in Enkomi and Maroni, and are some of the only known and earliest examples of raw copper from the island.

The results of the chemical analyses are presented in Table 2. For each lump we present both the results for the analysis undertaken on the clean section as well as on the outer surface. As noted above most of the copper lumps are highly oxidized on the surface, so the variation between the chemical composition of the section and the surface is considerable.

Four of the lumps are made of almost pure copper (99.3-99.9% Cu). The rest have a significant concentration of iron (4.2-6.2% Fe). Indeed, one sample (621-VII/12b) was found to consist of 15.1% Fe. The high iron amount of the specific sample can be justified from the fact that iron inclusions are dispersed throughout the mass (Fig. 3). The analysis of the darker areas that are located at the periphery of the copper lump, shows an even higher amount of iron (65% Fe – 35% Cu), while the analysis of the surface gives 91.8% iron and only 8.1% copper.

In six of the lumps a small but significant quantity of sulphur, ranging from 0.5-1.1% was also detected. As noted above Cypriote ores are sulphidic and the most common copper ore is chalcopyrite which consists of copper, iron and sulphur. These lumps of metal are clearly the end result of copper production through a multi-stepped process which involves roasting and smelting of the ores to matte, re-roasting and resmelting of the matte to black copper (Craddock 1995, 149). The black copper which would still contain some iron and sulphur (not unlike some of the examples we analysed) would then have been re-melted to remove residual iron and slag inclusion thus resulting in a pure metal (such as the samples which have a concentration of over 99% copper) which would have been cast into ingots (Craddock 1995, 150). The source of this metal must be one of the many copper mines that are located around Limassol (Fig. 1). The mining district of Kalavasos which includes the third largest concentration of copper ore deposits on the island (Gass *et al.* 1994, 187) is located some 25 kilometres to the north east while the ore deposits of Laxia tou Mavrou are located some 18 kilometres to the north east. The discovery of these artefacts within these deposits is intriguing and it is hard to know if they were deliberately deposited there. Their presence, however, is a clear indication of metallurgical activity (either the refining of metal or the casting of objects) in this area.

Artefact made of silver

One of the rings (T.278/5) that was thought to have been made of bronze was found upon analysis to be made of silver. The results of the chemical analysis are presented in Table 3. The amount of gold is relatively high, 5.1% which may suggest that the object was made of the natural alloy of silver and gold known as electrum (Craddock 1995, 111). The object also contains 3.7% copper which must have been added deliberately, probably to harden the metal. Lead, tin and iron were also detected in low concentrations of below 1%. The presence of the latter two elements may be the result of adding scrap metal rather than pure copper to the alloy. Previous analyses of silver artefacts from Pyla-*Kokkinokremos* also included cases with high gold contents (1.4 - 4.4% Au) (Gale and Stos-Gale 1984).

Silver first appears in the Early Cypriote III on the island but silver objects are relatively few throughout the Middle Cypriote and even the Late Cypriote I (Åström 1972, 565). It is on sites of the Late Cypriote II, such as Tomb 278 that more silver artefacts are found, but their numbers remain low (Åström 1972, 565). Anatolia possesses numerous important argentiferous ore deposits that were extensively exploited in Antiquity (Bayburtoğlu and Yildirim 2008, 43-45; Moorey 1994, 235). Some of the best known are those of Sardis where the river Pactolus had significant deposits of electrum (Ramage 2000, 14).

Conclusions

The chemical analysis of this small assemblage of metal artefacts from the area of Limassol has brought to light some interesting results. Through the analysis the gradual increase of tin in the alloy used to produce weapons through time was clearly detected. As time passed and Cyprus becomes more and more involved in long distance trade, tin becomes more widely available on the island. Analysis also detected that what were originally believed to have been lumps of slag were in fact lumps of raw metal which are an extremely rare find from Cyprus. Finally the analysis of a ring made of silver identified the possible use of electrum, a natural alloy of silver and gold which is quite common in Anatolia.

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Table 1. Chemical analyses of artefacts made of copper alloys

Sample	Artefact	Composition (wt% ± std)				
		Cu	Sn	Pb	As	Fe
T.8/24	Ring	91.3 ± 0.6	8 ± 0.3	0.2 ± 0.1	n.d	0.5 ± 0.02
T. 8/25	Ring	97.9 ± 0.4	0.3 ± 0.1	n.d	0.03 ± 0.01	1.6 ± 0.3
T.8/26	Ring	89.9 ± 0.9	9.6 ± 0.6	n.d	n.d	0.5 ± 0.1
T.8/27	Hair-ring	92.8 ± 0.6	6.8 ± 0.5	n.d	n.d	0.3 ± 0.1
T.8/29	Dagger	95.9 ± 0.5	2.2 ± 0.2	0.6 ± 0.1	0.4 ± 0.1	0.9 ± 0.1
T.11/10	Sword or Spearhead	98.1 ± 0.7	0.2 ± 0.02	n.d	0.04 ± 0.01	1.6 ± 0.2
T.322/14	Dagger	86.3 ± 1.1	12.9 ± 0.8	0.5 ± 0.1	0.2 ± 0.04	0.3 ± 0.1
T.322/21	Dagger	87 ± 1.3	12.4 ± 0.9	0.1 ± 0.02	0.3 ± 0.1	0.2 ± 0.1
T.322/22	Dagger	90.4 ± 0.8	9.4 ± 0.5	0.02 ± 0.01	n.d	0.1 ± 0.03
T.322/23	Dagger with bone hilt	85.5 ± 0.9	14.2 ± 0.8	n.d	0.04 ± 0.01	0.2 ± 0.04
T.322/24	Dagger	86.9 ± 0.7	11.7 ± 0.4	0.5 ± 0.1	0.2 ± 0.02	0.7 ± 0.2
T.323/6	Dagger	89 ± 0.5	10.4 ± 0.3	0.07 ± 0.01	0.2 ± 0.03	0.3 ± 0.03
T.323/7	Dagger	91.1 ± 0.4	8.4 ± 0.3	0.2 ± 0.1	n.d	0.2 ± 0.1
T.323/10	Dagger	91.2 ± 0.8	8.1 ± 0.4	0.2 ± 0.03	0.3 ± 0.1	0.2 ± 0.02
621-IV/10	Ring	94.1 ± 0.6	5.4 ± 0.2	0.05 ± 0.01	n.d	0.4 ± 0.1
621-IV/11	Small ring	94.5 ± 0.6	4.9 ± 0.3	n.d	n.d	0.6 ± 0.1
621-IV/16	Dagger	92.6 ± 0.9	6.9 ± 0.6	n.d	n.d	0.3 ± 0.1
621-V/5	Dagger	93.3 ± 0.5	6.5 ± 0.3	0.1 ± 0.01	0.02 ± 0.01	0.05 ± 0.01
621-VI/7	Pin	87.4 ± 0.8	6 ± 0.5	6.2 ± 0.2	n.d	0.3 ± 0.1
621-VI/17	Ring	93.2 ± 0.7	6.5 ± 0.4	0.04 ± 0.01	n.d	0.2 ± 0.03
621-VI/24a	Pin	91.5 ± 0.8	6.3 ± 0.5	1.9 ± 0.3	n.d	0.2 ± 0.1
621-VI/24	bPin	92.1 ± 0.6	6.8 ± 0.4	0.6 ± 0.1	n.d	0.5 ± 0.1

Table 2. Chemical analyses of amorphous metal lumps

Sample	Area of analysis	Composition (wt% ± std)					
		Cu	Fe	S	As	Pb	Mn
621-V/15.1	Cross section	94.5 ± 0.2	5 ± 0.1	0.5 ± 0.05	n.d	n.d	n.d
	Surface	87.8 ± 0.6	12.2 ± 0.5	n.d	n.d	n.d	n.d
621-V/15.2	Cross section	99.6 ± 0.2	0.3 ± 0.1	n.d	0.02 ± 0.01	n.d	0.01 ± 0.005
	Surface	99.4 ± 0.2	0.6 ± 0.1	n.d	0.02 ± 0.01	n.d	n.d
621-V/15.3	Cross section	99.9 ± 0.1	0.1 ± 0.05	n.d	n.d	n.d	n.d
	Surface	99.3 ± 0.1	0.6 ± 0.1	n.d	0.02 ± 0.01	n.d	n.d
621-V/15.4	Cross section	92.7 ± 0.6	6.2 ± 0.4	1.1 ± 0.2	n.d	n.d	n.d
	Surface	91.6 ± 0.9	8.4 ± 0.7	n.d	n.d	n.d	n.d
621-V/15.5	Cross section	94.2 ± 0.5	4.9 ± 0.3	0.9 ± 0.2	n.d	n.d	n.d
	Surface	90.7 ± 0.6	9.2 ± 0.5	n.d	n.d	n.d	0.03 ± 0.01
621-VI/18-1	Cross section	95.3 ± 0.5	4.2 ± 0.4	0.5 ± 0.04	n.d	n.d	n.d
	Surface	84 ± 0.9	15.5 ± 0.8	n.d	n.d	0.03 ± 0.01	0.02 ±0.01
621-VII/12a	Cross section	99.7 ± 0.1	0.2 ± 0.05	n.d	0.02 ± 0.01	n.d	n.d
	Surface	99.2 ± 0.2	0.7 ± 0.1	n.d	0.03 ± 0.01	n.d	n.d
621-VII/12b	Cross section	84.5 ± 0.7	15.1 ± 0.6	0.4 ± 0.04	n.d	n.d	n.d
	Surface	8.1 ± 0.3	91.8 ± 0.4	n.d	n.d	n.d	0.06 ± 0.01
LM 1328/9	Cross section	99.5 ± 0.1	0.04 ± 0.01	0.4 ± 0.0	30.02 ± 0.01	n.d	n.d
	Surface	98.8 ± 0.2	0.9 ± 0.1	n.d	0.05 ± 0.01	0.1 ±0.05	n.d

Table 3. Chemical analysis of silver artefact

Sample	Artefact	Composition (wt% ± std)					
		Ag	Au	Cu	Sn	Pb	Fe
T.278/5	Ring	89.4 ± 0.6	5.1 ± 0.2	3.7 ± 0.2	0.7 ± 0.1	0.8 ± 0.1	0.3 ± 0.1

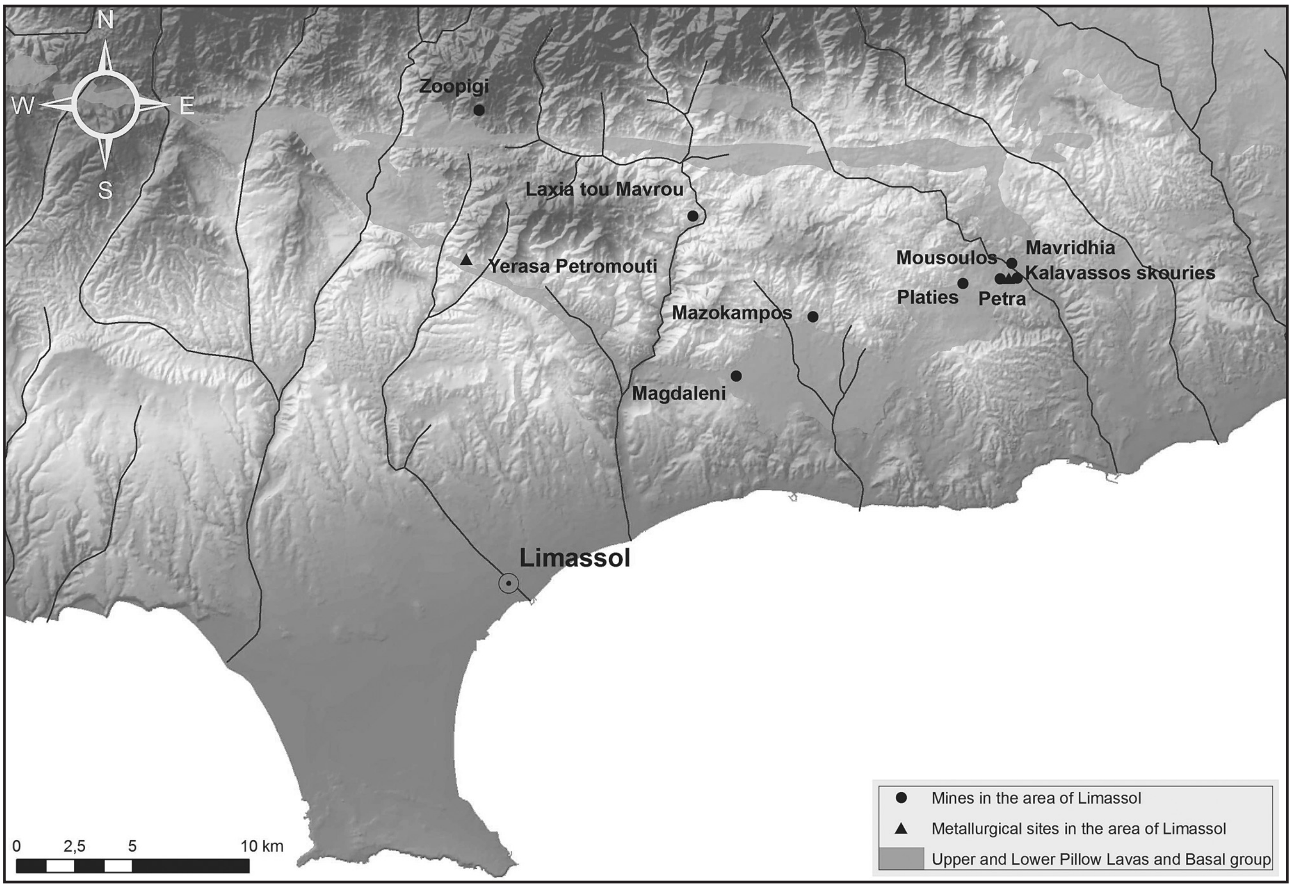
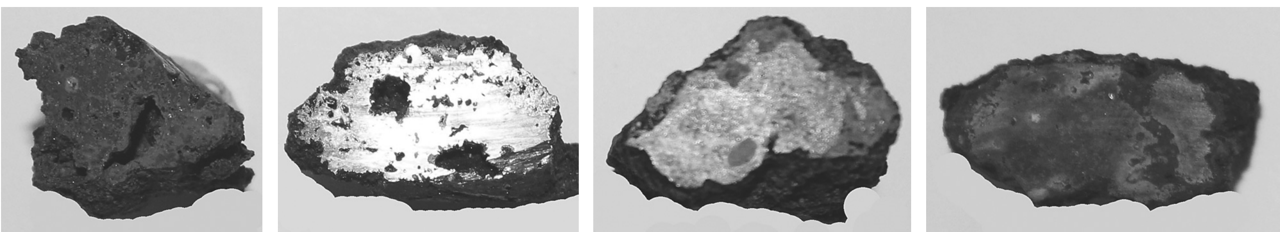
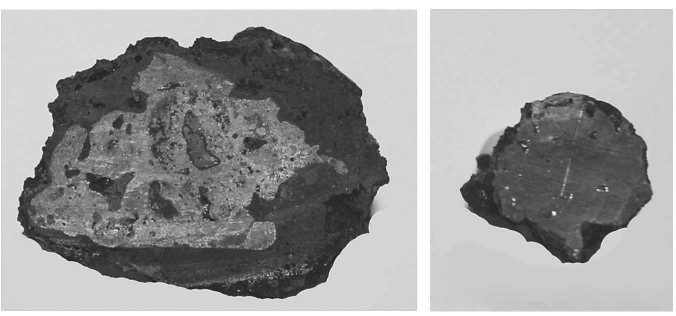


Fig. 1 Map of the Limassol area indicating the location of copper mines and metallurgical sites

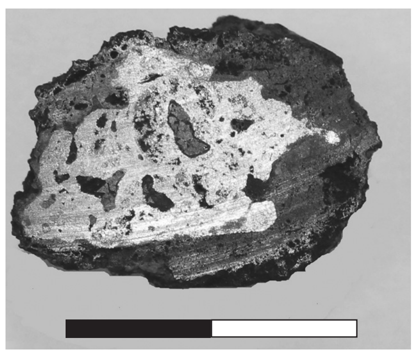


621-V/15.2 **621-V/15.3** **621-V/15.4** **621-VI/18.1**



621-VII/12b **LM 1328/9**

Fig. 2 Section of the analyzed lumps revealing their metallic nature



621-VII/12b

Fig. 3 Section of sample 621-VIII/2b, where the areas with high iron content (black colour) can be seen at the periphery of copper lump