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Portable laser ablation sheds light on Early Bronze Age gold treasures in the old world: New insights from Troy, Poliochni, and related finds

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ABSTRACT

The paper focuses on the archaeometric analyses of the gold objects from the famous so-called ‘treasures’ of Troy and Poliochni on the island of Lemnos. Altogether 61 Early Bronze Age (EBA) gold objects dating between 2500 and 2000 BCE were investigated in this study. They were primarily sampled with a portable laser ablation (pLA) unit in the National Archaeological Museum in Athens and analyzed with ICP-MS at the Curt-Engelhorn-Centre Archaeometry (CEZA) in Mannheim. The main advantage of this approach is the possibility to obtain samples on site without the necessity of transport. It is a minimally invasive method that leaves no visible damage on the objects. As an additional advantage there are no restrictions on the size of the objects under study. The central goal of the study was to obtain high-quality compositional analyses of gold objects from the sites Troy and Poliochni to investigate, if the typological similarity is paralleled by the elemental composition of the gold, including the trace elements. This would suggest not only similar procurement of the metal across these sites but also potential exchange of objects and/or specialist workers. In a second step, the results were compared with data from contemporary gold objects from Ur in southern Mesopotamia, from where LA-ICP-MS data have recently become available. Finally, a survey of such data for gold objects from gold-rich regions is used to narrow down the possible origin of Early Bronze Age gold in the Old World.

1. Introduction

The famous gold treasures of the Early Bronze Age settlements of Troy in western Anatolia have been well known since their discovery during Heinrich Schliemann’s excavations in the 19th century (Schliemann 1881). Chronologically, the treasures are mainly associated with the phases of Troy II–III (EBA 2–3), dating to the second half of the third millennium BCE. The enormous number of high-quality gold objects is striking, especially since the practice of hoarding prestige objects is not previously known in the Aegean and in western Anatolia. This new cultural phenomenon is strongly linked with the formation of new socio-politically and hierarchically organized societies and the first ’proto-urban’ centers around 2600 BCE. The practice of hoarding gold treasures is not only evident in centers like Troy, but also in other settlements, such as in Poliochni (Bernabò-Brea 1976; cf. Cultraro 1999, 2007). This site is located on the island of Lemnos and is situated in close proximity to Troy, approximately 50 km from the western Anatolian coast. It remains unclear who the owners of these precious objects were, why the treasures were stored, if the objects were local productions, and not least from where the gold originally derived.

Since Schliemann’s time, the origin of the prehistoric gold remains an unsolved enigma in archaeology and archaeometallurgy (Schliemann 1881, 278–295; 1884, 56–75). In western Anatolia, numerous gold deposits are known, of which – according to ancient tradition – the Astyra mine was mined by the Trojans during the Trojan War (Strabo, geography 14,5,28; cf. Pernicka et al. 1984; Pernicka et al. 2003). An

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overview of western Anatolian gold deposits was recently compiled by Cahill et al. (2020) and Late Bronze Age gold mining is attested in northern Greece and southern Bulgaria (Vavelidis and Andreou 2008; Popov and Jockenhövel 2010; Popov et al. 2017), while there are no known gold deposits in the southern Aegean. Moreover, distinct objects found among the assemblages indicate far-reaching communication and trade networks in the Aegean and beyond.

Archaeometric studies of gold objects generally focus on several questions, such as the development of metallurgical processes, the geological origin of the raw material, workshop identification, as well as goldsmithing techniques. Specific claims of provenance are still a matter of debate. The systematic analysis of trace elements in archaeological gold artefacts began with the studies of Axel Hartmann (1970, 1982), who analyzed some 4000 archaeological gold objects in view of classification and provenance with atomic emission spectrometry (AES). Later, several elemental and isotopic approaches had been tested with more sensitive methods (Gondonneau et al. 1996; Junk and Pernicka 2003; Bendall et al. 2009; Ehser et al. 2011; Standish et al. 2015; Baron et al. 2019). Presently, investigations on material classification as well as provenance of gold artefacts are mainly based on the analysis of major, minor, and trace elements, including the platinum group elements (PGE) (Guerra et al. 1999; Pernicka 2014).

Nowadays, the combination of laser ablation with inductively coupled plasma mass spectrometry (LA-ICP-MS) is the most widely used technique for the analysis of archaeological gold objects. Initial problems with quantification (Grigorova et al. 1998) have largely been overcome (Kovacs et al. 2009). However, conventional LA-ICP-MS techniques require either a tiny metal shaving (as it has been the case in Hartmann's time) or the transport of the objects to the laboratory. Both options often turn out as highly problematic from a conservation point of view and/or due to insurance policy and political reasons as a result of their high intrinsic value and uniqueness. Furthermore, due to the limited dimensions of most ablation cells, stationary laser ablation setups often exclude larger objects from minimally-invasive scientific analyses.

Therefore, LA-ICP-MS as well as other established, yet laboratory-based approaches like proton-induced X-ray emission (PIXE; Constantinescu et al. 2008) or synchrotron-based X-ray fluorescence analysis (Radtke et al. 2017) cannot be used to investigate a large number of gold objects currently stored in museums. Instead, a minimally invasive or non-invasive mobile method for sampling in the respective museums has to be applied for a comprehensive investigation.

Given that workshop analyses and provenance studies of gold have to include the quantification of trace elements, established mobile analytical methods such as the non-destructive portable X-ray fluorescence (e.g., Hauptmann et al. 2018) cannot be applied, as they are not sensitive enough and, accordingly, do not allow quantification of most trace elements. Besides, with X-rays the depth of information in gold is only in the order of 10 μm and may thus not provide the composition of the original alloy due to surface effects (Blakelock 2016). In contrast, the information depth of laser ablation is in the order of 200 μm and thus less affected by such effects. While it is possible to apply laser-induced breakdown spectroscopy to quantify PGE (Rifai et al. 2020; Mohamed et al. 2021), there seems to be no portable version available (pLIBS; Fortes and Laserna 2010) that quantifies PGE in gold artefacts.

However, in the last decade, a portable laser ablation device (pLA) has become available for field micro-sampling of elemental and isotopic analyses of archaeological objects, including gold (Glaus et al. 2012, 2013; Born et al. 2015; Käser 2015; Burger et al. 2017; Knaf et al. 2017, 2021; Seman et al. 2021; Merkel et al. 2022). Considering the above-mentioned constraints regarding the analysis of gold artefacts, pLA devices seem to be the best choice as a virtually non-destructive, on-site sampling method for archaeological gold in order to determine their elemental and, potentially, even isotopic composition. This method allows for minimally invasive sampling, i.e. not visible to the naked eye, on site or in the museum, with subsequent analysis of the collected material in the laboratory. This approach combines both the versatility of a mobile system, which can easily be transported worldwide, with the most sensitive method for elemental analysis (ICP-MS). In contrast to the stationary methods mentioned above, the portable laser ablation setup does not impose any limitations with regard to the analyzed artefact's dimensions, as there is no sample chamber needed in its setup.

The present study focuses on the application of the pLA-ICP-MS method for sampling and analyzing precious gold objects from the Aegean Early Bronze Age. Specifically, the gold objects of the so-called 'treasures' of Troy and Poliochni on Lemnos offer the opportunity for material classification and the possibility to answer open questions regarding their production, the origin of the used gold, and to uncover regional and supra-regional contacts which are essential for understanding the socio-cultural developments in the 3rd millennium BCE.

2. Materials and methods

2.1. Samples

In total, 84 analyses were performed on 61 gold objects from Troy and Poliochni (Figs. 1 and 2; Table 1) dating to the Early Bronze Age 2–3 period.

Most of the gold objects from Poliochni (n = 25) and Troy (n = 20)

Fig. 1. Analyzed gold objects from Troy (photos: J. Huber, C. Schwall; Sch 6007/1–15 after Born et al. 2009, 22, Fig. 2; 2014, 123, Fig. 2). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Table 1
Details of the analyzed gold objects from Troy and Poliochni.

Site (n)	Sample Source		
	Inv.-no.	Description	Reference(s)
Troy (n = 35, Fig. 1)	*National Archaeological Museum Athens (Greece)*		
	Π 4331A	Necklace of flat-winged disc beads (n = 5)	Demakopoulou 1990, 152, Fig. 8a (bottom); Tsivilika 2003a, 264, no. 168b
	Π 4331B	Necklace of serrated wheel-shaped beads (n = 6)	Demakopoulou 1990, 152, Fig. 8d (second from top); Tsivilika 2003a, 264, no. 168c
	Π 4331C	Necklace of barrel-shaped beads and flattened wire loops (n = 7)	Demakopoulou 1990, 152, Fig. 8e (top); Tsivilika 2003a, 264, no. 168d
	Π 4332	Crescent-shaped earring (n = 1)	Demakopoulou 1990, 150, Fig. 6 (right); Tsivilika 2003c, 267, no. 172 (right)
	Π 4333	Hair ring (n = 1)	Demakopoulou 1990, 149, Fig. 5 (left); Tsivilika 2003b, 267, no. 171 (right)
	Museum of Prehistory and Early History, Berlin (Germany)		
	Sch 6007/1–15	Tutuli (n = 15)	Born et al. 2009, 22, Fig. 2; 2014, 123, Fig. 2
Poliochni (n = 26, Fig. 2)	*National Archaeological Museum Athens (Greece)*		
	Π 7161.1	Hair ring (n = 1)	Bernabò-Brea 1976, pl. 246.18; Papazoglou-Manioudaki 2003a, 271, Fig. 175 (left)
	Π 7162		Bernabò-Brea 1976, pl. 246.15
	Π 7163		Bernabò-Brea 1976, pl. 246.1
	Π 7165		Bernabò-Brea 1976, pl. 246.12
	Π 7166		Bernabò-Brea 1976, pl. 246.14
	Π 7167.1–2	Pair of hair rings (n = 2)	(1): Bernabò-Brea 1976, pl. 246.20; (2): Bernabò-Brea 1976, pl. 246.22
	Π 7168	Chain of hair rings (n = 1)	Bernabò-Brea 1976, pl. 246.6
	Π 7170	Hair ring, type Manika (n = 1)	Bernabò-Brea 1976, pl. 246.23
	Π 7171	Torque (n = 1)	Bernabò-Brea 1976, pl. 246.25
	Π 7172.1–5	Chain of tutuli (n = 5)	Bernabò-Brea 1976, pl. 250.1–16
	Π 7173	Perforated bar (n = 1)	Bernabò-Brea 1976, pl. 250 (bottom); 252.11
	Π 7185	Pin with double spiral and ram application (n = 1)	Bernabò-Brea 1976, pl. 240
	Π 7186B–C	Earring with poppy capsule pendant (n = 2)	Bernabò-Brea 1976, pl. 245b–c; Papazoglou-Manioudaki 2003b, 272, Fig. 176 (left = 7186C)
	Π 7187	Chain of flat-winged disc beads (n = 6)	Bernabò-Brea 1976, pl. 247c
	SAM archive (Germany)		
	Π 7161.2 (Au 4358)	Hair ring (n = 1)	Bernabò-Brea 1976, pl. 246, 19; Papazoglou-Manioudaki 2003a, 271 no. 175 (right); Hartmann 1982, pl. 103.Au 4358

were sampled in the National Archaeological Museum in Athens with a portable laser ablation setup (in total n = 45) and analyzed afterwards with ICP mass spectrometry (see below). Additional data from objects analyzed with laboratory-based LA-ICP-MS (method after Kovacs et al. 2009) was incorporated in this study: 15 tutuli from Troy (Museum of Prehistory and Early History in Berlin; Born et al. 2009, 2014) and one hair ring from Poliochni (SAM (Studien zu den Anfängen der Metallurgie) archive: Au 4358; National Archaeological Museum Athens: Π 7161.2) were analyzed with this method.

2.2. Portable laser ablation

The pLA setup (Fig. 3) consists of a diode pumped solid state laser (Wedge HB 532, BrightSolutions S.r.l., Cura Carpignano, PV, Italy) which produces pulsed green light (wavelength $\lambda = 532$ nm). The energy of the photons emitted, as well as the emission frequency, can be adjusted (max. pulse energy: 1.05 mJ; repetition rate: single shot to 2 kHz) via an external control unit. The laser is coupled via a lens mounted on an xy-stage (LM05XY/M, Thorlabs, Newton, NJ, USA) with an optical glass fiber (Ocean Optics QP450-2-XSR). The fiber is coupled with the ablation unit, in which the laser light is focused by two lenses onto the sample surface, resulting in an ablation diameter of 120 μm (Glaus 2013).

In order to find a suitable ablation position, a monochromatic CCD camera (Chameleon, CMLN-13S2M-CS, FLIR Integrated Imaging Solutions (formerly: Point Grey Research), Richmond, BC, Canada) is mounted on top of the ablation head. This allows for the observation of ca. 1 mm^2 magnified surface area, which is illuminated with an LED ring mounted inside the ablation head (cf. Glaus 2013). Due to its high spatial resolution, the portable laser ablation method is therefore ideally suited for the analysis of small structures.

The focused laser beam evaporates and sputters off a small amount of the material. The ablated material is collected by suction with an oil-free membrane pump through a pre-cleaned silicon tube (Rotilabo®-silicon tube, Carl Roth GmbH + Co. Kg, Karlsruhe, Germany), which is attached to the ablation head's end piece (see Fig. 3), and is subsequently deposited on hydrophilic polycarbonate membrane filters (Merck HTTP01300; pore size 0.4 μm; thickness: 10 μm; diameter: 13 mm; Merck KGaA, Darmstadt, Germany) mounted on separate filter-wheels (6 filters per wheel). In order to minimize possible cross-contamination during the ablation process, the silicon tube is replaced by a new one after each sampling process. At the same time, the ablation head's end piece is dismantled and thoroughly cleaned by wiping with a soft tissue and isopropanol.

The ablation frequency is set to $\nu = 100$ Hz, a pulse duration of 1 ns, a total ablation time of 60 s per sampled spot, and a pulse energy of 0.6 mJ leading to an energy density of about 3.4 J cm^{-2} (depending on the quality and stability of the coupling between the laser exit and the optical fiber via the xy-stage, the fiber attenuation, and the optical losses inside the ablation head) in order to obtain enough material for the subsequent analyses in the laboratory.

For chemical analysis this method is combined with inductively coupled plasma mass spectrometry (ICP-MS) as a separate step in the laboratory: After sampling the filters are stored separately in cleaned and sealed containers that can be transported to the laboratories of CEZA. In order to dispatch the ablated material from the membrane filters, a wet chemistry protocol, which is described in detail in the supplementary material no. 1, has been developed. Subsequently,

Fig. 2. Analyzed gold objects from Poliochni (photos: C. Schwall). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

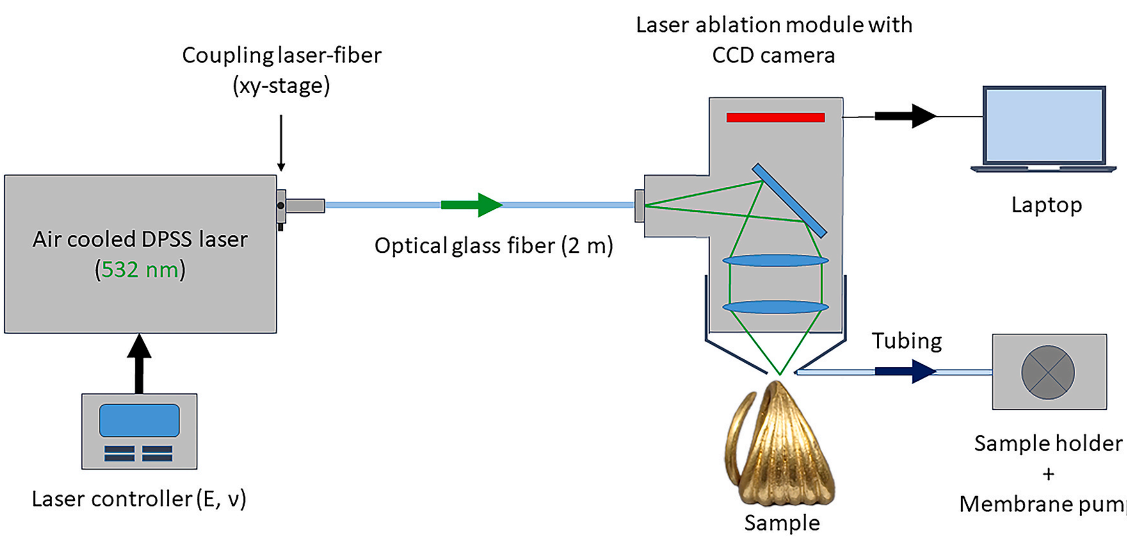

Fig. 3. The portable laser ablation setup with a sample from Poliochni (photo: Π 7161.1/C. Schwall).

aliquots of the sample solution are diluted for elemental analyses performed with Q-ICP-MS (ICAPQ with CCT (collision cell technology), Thermo Fisher Scientific Inc., MA, USA).

Scanning electron microscope (EVO MA 25, Carl Zeiss Microscopy GmbH, Jena, Germany with an EDX analysis system QUANTAX, Bruker, Billerica, MA, USA) images from an ablation spot (gold standard, NA-2 [NA-Au-31, Aurubis AG, Hamburg, Germany]) as well as of the lateral view of an ablation hole (pure gold ingot) are shown in Fig. 4. The latter crater resulted from focusing the laser light for the same duration and with the same power as above on the edge of a rectangular gold ingot. The induced craters in gold are cone-shaped (cf. Fig. 4). Cone-shaped ablation craters were also reported by Russo et al. (2002) and Knaf et al. (2017). Repeated sampling processes result in identical ablation craters with the same diameter (ca. 120 μm) and depth (ca. 180 μm). Given the ablation crater dimensions shown in Fig. 4, the total ablated amount of gold per ablation spot is in the order of ca. 13 μg.

Longer ablation times do not result in considerably deeper ablation holes. While, on the other hand, shorter ablation times cause less deep

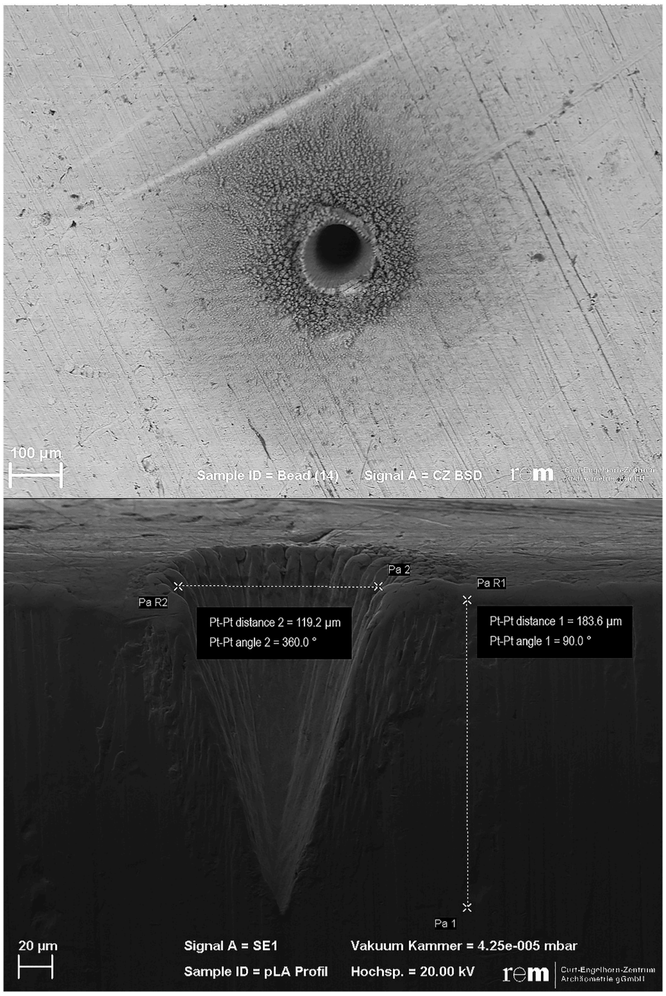

Fig. 4. Top: Electron microscope image of an ablation crater with debris on a gold object before cleaning the surface; Bottom: Electron microscope image of an ablation crater (lateral view).

ablation holes and a reduced crater diameter which means that the ablated sample mass is also reduced. When sampling material from very thin objects like foils, sample duration may be reduced in order to avoid penetrating the object/foil, which might contaminate the sample due to ablation of the underlying material (e.g., bronze).

During the ablation process, some of the ablated debris accumulates around the ablation crater (Fig. 4). This material is a representative part of the ablated material and can easily be removed from the surface using a soft tissue. Thereafter, the small indentation induced on the object's surface cannot be recognized with the naked eye. On that account this innovative method can be characterized as minimally invasive.

3. Results

3.1. General characteristics of the analyzed Early Bronze Age gold objects

All analytical results are summarized in supplement no. 2. The artefacts from Troy and Poliochni revealed a wide range of silver contents (3–45%; Fig. 5) which is typical for native gold (Jones and Fleischer 1969; Chapman et al. 2021). While most of the analyzed objects show silver concentrations between 10 and 45%, one flat-winged disc bead (Π 4331A.1) and all analyzed parts of the crescent-shaped earring with granulation (Π 4332.1–4) from Troy show significantly lower silver concentrations of around 5%.

The objects have comparatively low copper concentrations

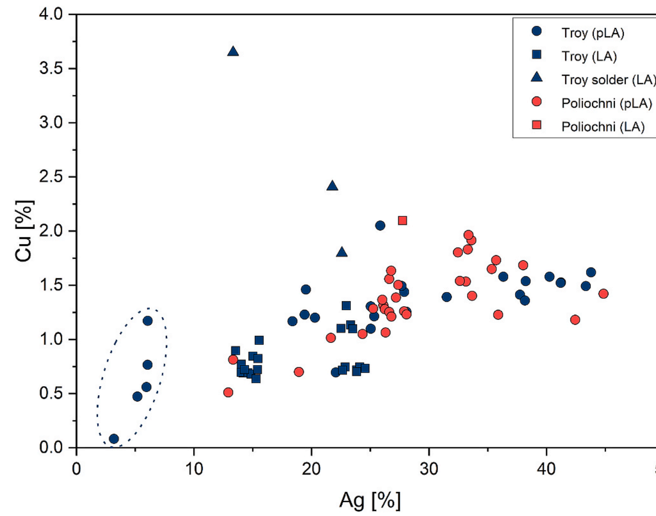

Fig. 5. Diagram of the silver (Ag) and copper (Cu) concentrations of the samples from Troy (blue symbols, number of analyses: n = 52) and Poliochni (red symbols, number of analyses: n = 32), analyzed with pLA-ICP-MS (circles) or LA-ICP-MS (rectangles). Triangles indicate areas with reaction solder; the dotted ellipse indicates samples from Troy with low silver concentrations. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

(0.08–2%). However, as it is the case for most prehistoric gold objects, the copper contents of the majority of the objects from Troy and Poliochni are higher than in native gold, which typically contains less than 0.1% copper (Pernicka 2014). This raises the question of intentional alloying with copper, for instance in order to alter the color of the gold. Visible changes in the color of the alloy can only be observed above several percent copper (German et al. 1980). As the slightly elevated copper contents for two of the 15 tutuli from Troy (Sch 6007/1 and Sch 6007/5) are due to the application of reaction solder for the granulation (Born et al. 2009, 2014), intentional alloying of natural gold with copper cannot be attested.

Exploitable gold deposits are either primary or secondary in nature. Primary gold deposits have commonly formed as veins, lodes or massive (sulfide) ore lenses by chemical precipitation from ascending hot fluids whereas secondary gold deposits originate from the deep weathering and oxidation of pre-existing primary gold ores. The erosion of such (micro)nuggets and the fluviatile transport in creeks and rivers leads to the accumulation of heavy minerals and gold nuggets in trap sites where they form placer deposits. Since the tin mineral cassiterite (SnO_2) is also highly resistant to weathering and has a high specific gravity, it can also occur in such placer deposits. Accordingly, elevated concentrations of tin are a strong indicator of the use of alluvial gold instead of primary gold (Hartmann 1982). While the co-occurrence of gold and cassiterite is not uncommon in placer deposits, it is extremely rare in primary deposits (Velazques 2014; cf. Schmiderer et al. 2007). Gold recovered in this manner usually consists of very small particles (<0.1 mm diameter), so it had to be melted in a crucible for further processing. In this process, the cassiterite can be reduced to the metal under low-oxygen conditions and thus be taken up by the gold (Gumprich 2004). Such conditions are quite likely since the gold dust in the crucible was probably covered with glowing charcoal. Copper and lead could also enter the gold in a similar manner, since copper and lead minerals also have a high specific gravity and can enter the heavy mineral concentrate. In this way, tin was found in almost all samples at concentrations between ca. 10 and 1900 mg/kg), which suggests that we are primarily dealing with alluvial gold.

This assumption is supported by the presence of palladium (Pd) and platinum (Pt) in the objects from Troy and Poliochni, since both elements are not known to be associated with primary gold. Other than gold, platinum-group elements (PGE) are usually associated with mafic/

ultramafic host rocks. Platinum-group minerals (PGM), such as isoferroplatinum-tetraferroplatinum or osmium-iridium-ruthenium crystals, were liberated from their host rocks by weathering followed by purely physical enrichment of the most resistant PGM grains in placers, similar to gold (Cabri et al. 2022). While platinum and palladium are soluble in gold and can thus be incorporated on melting alluvial gold, the remaining four platinum-group elements (Rhodium (Rh), Ruthenium (Ru), Osmium (Os), and Iridium (Ir)) are not and are thus frequently found as discrete osmium-iridium-ruthenium inclusions in ancient gold objects. It is evident that platinum minerals are much more rare than gold particles in fluvial concentrates and it is, therefore, difficult to obtain a representative sample of native gold that can be directly be compared with an object. It is furthermore to be expected that the absolute concentrations of platinum and palladium in gold can be variable from the same source, because a few grains of PGM can make a large difference. Therefore, it is also necessary to consider the ratios of platinum and palladium, which may show less variation within a geological source.

According to the trace element patterns, the objects from Troy and Poliochni are quite similar (Fig. 6), especially for palladium and platinum. Particularly remarkable are the high concentrations of platinum and palladium in the Early Bronze Age gold from Troy and Poliochni with platinum concentrations ranging up to 0.2% (Fig. 7). Elevated platinum concentrations had already been observed in gold objects from Poliochni that were analyzed as part of the SAM project (Hartmann 1982).

In addition to the concentrations also the platinum/palladium (Pt/Pd)-ratios have proven to be useful for classification due to their stability during different metallurgical processes (see above). They show two distinct maxima, roughly around 10–15 and 30–40 (cf. Fig. 8 right), which may indicate two different geological sources. Lower ratios are also clearly associated with lower platinum (cf. Fig. 8 left) as well as palladium concentrations. Furthermore, lower Pt/Pd-ratios are tied to a much smaller scatter range concerning the silver content (between ca. 13 and 28%) and are also associated with lower absolute concentrations of antimony, lead, and bismuth.

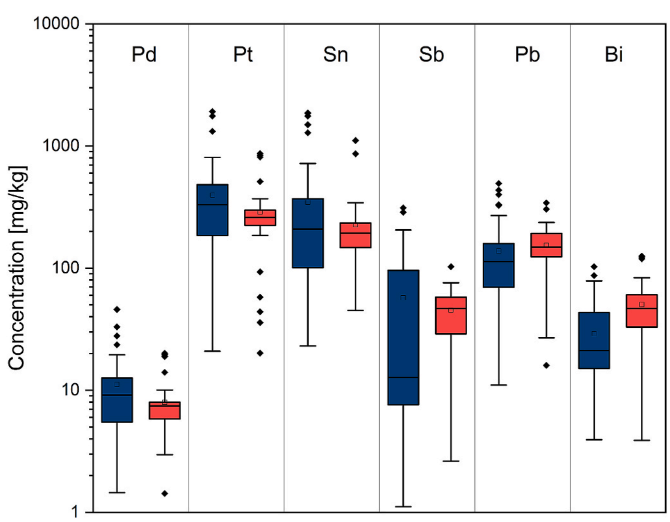

Fig. 6. Box plots of the palladium (Pd), platinum (Pt), tin (Sn), antimony (Sb), lead (Pb), and bismuth (Bi) concentrations in the analyzed gold objects from Troy (blue boxes, number of analyses: n = 52) and Poliochni (red boxes, number of analyses: n = 32) as boxes (25th and 75th percentile) and whiskers (boundaries based on the 1.5 interquartile range) with outliers (black diamonds). Horizontal lines indicate the median value, whereas the mean value is indicated by open squares. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

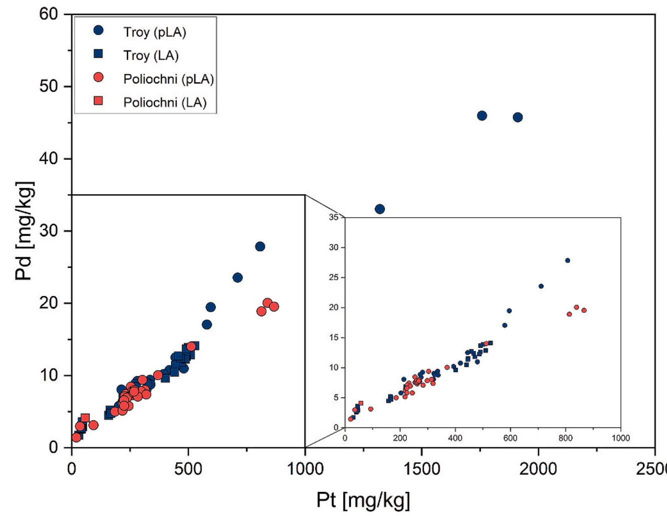

Fig. 7. Diagram of the platinum (Pt) and palladium (Pd) concentrations of the analyzed samples from Troy (blue symbols, number of analyses: n = 52) and Poliochni (red symbols, number of analyses: n = 32) analyzed with pLA-ICP-MS (circles) or LA-ICP-MS (rectangles); the insert shows objects with lower Pt and Pd concentrations. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3.2. Classification

Since most of the gold objects from Troy and Poliochni are clearly related in typological, morphological, and stylistic sense (i.e., earrings, pendants, hair rings, torques, beads, and tutuli: Sazcı 2007, 137–353; Bernabò-Brea 1976, pl. 240–252; cf. Maxwell-Hyslop 1971, 48–60), it is no surprise that most of them consist of gold of similar composition. However, three tutuli from Troy (Berlin tutuli Sch 6007/1, Sch 6007/6, and Sch 6007/12) (in total, n = 7 analyses), two out of the three tubular beads from Troy (Π 4331C.2 and Π 4331C.3) (n = 2), two hair rings from Poliochni (Π 7161.2 and Π 7170) (n = 3) have significantly lower platinum as well as palladium contents than the other objects. Their Pt/Pd-ratios are also lower and cluster around 15, suggesting the exploitation of, at least, one more natural gold source, as discussed above.

Part of the samples analyzed belong to stylistically identical objects. This offers the opportunity to obtain insights into the workflow of the Early Bronze Age workshops as well as their production technology. A detailed discussion will be published elsewhere but a few selected examples may be discussed here.

3.2.1. Flat-winged disc beads (Troy, Poliochni)

Two necklaces with typologically identical, flat-winged disc beads from Troy (Π 4331A.1–5, n = 5 analyses) and Poliochni (Π 7187.1–6, n = 7 analyses) were analyzed (cf. Figs. 1, 2, and 9; Table 1). The relatively large variations regarding the major components can possibly be explained by the intention to combine differently colored beads in one chain/necklace (cf. Fig. 9). However, it is not certain that the present arrangements of the necklaces reflect the original ones. The elemental pattern clearly shows at least five different material groups with platinum concentrations between 200 and almost 2000 mg/kg as indicated in Fig. 10. Since their Pt/Pd-ratios are more or less constant, they may derive from the same gold source.

It is particularly interesting that one bead from Troy (Π 4331A.3) consists of a very similar material as three beads from Poliochni (Π 7187.1, 5, 6) (cf. Fig. 10). It could be interpreted that the two necklaces were actually made in the same workshop. Overall, the analytical results of the flat-winged disc beads provide evidence that at least a part of the typologically similar objects were serially produced, possibly even in the same workshop, which may have been located in Troy or Poliochni.

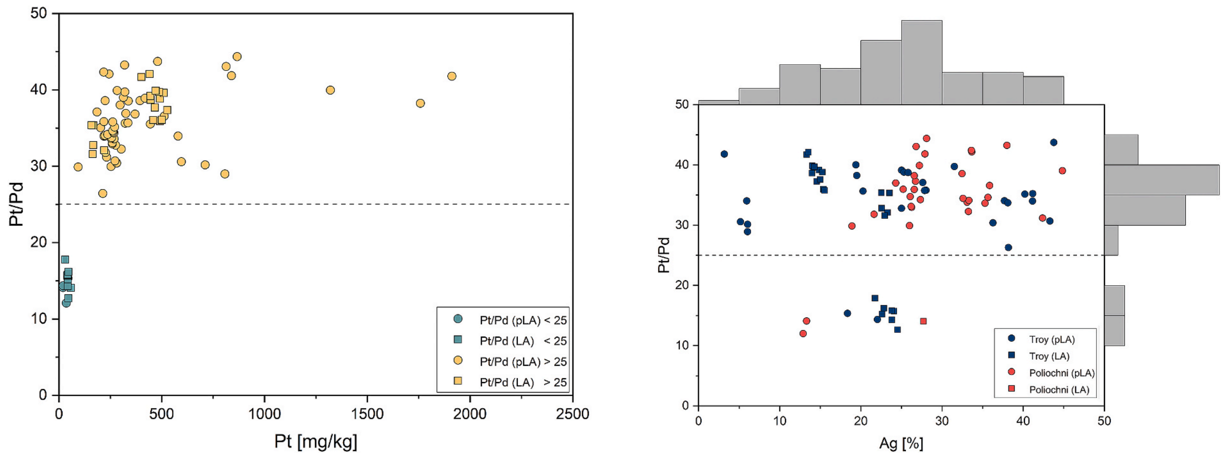

Fig. 8. Left: Diagram of the platinum concentrations and the Pt/Pd-ratios of the analyzed objects, analyzed with pLA-ICP-MS (circles) or LA-ICP-MS (rectangles). The ratios below 25 (n = 12) are marked green, those above 25 (n = 72) orange. The boundary between lower and higher ratios is indicated by the dashed line. Right: histogram of the silver (Ag) concentrations and the Pt/Pd-ratios of the same samples. Again, objects analyzed with pLA-ICP-MS are represented by circles, those analyzed with LA-ICP-MS are represented with rectangles. Objects from Troy and Poliochni are represented by the colors blue and red, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

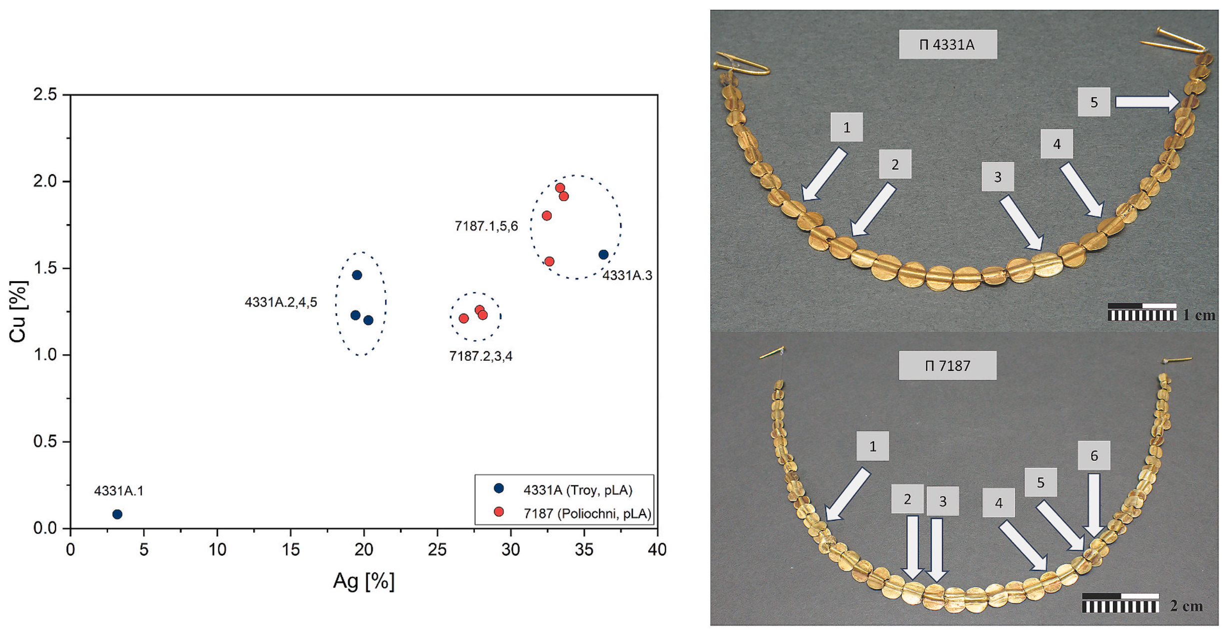

Fig. 9. Diagram of the silver (Ag) and the copper (Cu) concentrations of five flat-winged disc beads from Troy (Π 4331A) and Poliochni (Π 7187) (on the left). The beads from Troy (n = 5 analyses) and Poliochni (n = 7 analyses) were all analyzed with pLA-ICP-MS and are represented by blue and red circles, respectively. The ellipses indicate possible material groups and are merely drawn to aid the eye. The sampled beads are shown on the right (photos: J. Huber, C. Schwall). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

3.2.2. Tutuli (Troy, Poliochni)

Five out of 16 tutuli from Poliochni (Π 7172.1–5) (n = 7 analyses) were analyzed with pLA-ICP-MS. Furthermore, the so far unpublished results of 15 tutuli from Troy (Sch 6007/1–15) (n = 25 analyses), which have been investigated previously with LA-ICP-MS (Born et al. 2009, 2014), will be discussed for comparison (cf. Fig. 1; Table 1). From a typological point of view, the tutuli from both sites are identical with the exception that those from Troy are decorated with granulation. Excluding the analyses that were performed on the solder, at least four distinct compositional groups can be identified regarding the silver and the platinum as well as the palladium concentrations: Two subgroups with samples from Troy and one from Poliochni are generally very similar, especially considering their palladium, platinum, and silver concentrations and Pt/Pd-ratios (Fig. 11 left and right; indicated by the ellipses). Therefore, this gold may derive from the same geological source. The third group of Trojan tutuli have significantly lower Pt/Pd-ratios around 15 suggesting another geological source.

Overall, this case demonstrates once again an impressively close relationship between Troy and Poliochni not only when comparing the objects from a typological point of view, but also in terms of their almost identical chemical compositions. The production in one workshop, possibly by different goldsmiths (cf. Born et al. 2009, 24; 2014, 121), seems to also reasonable in the case of the tutuli.

Both the analyses of the flat-winged disc beads as well as those of the tutuli demonstrate the significant insights obtained by chemical serial analyses of typological and technological identical objects. It seems that extensive investigations on a larger number of objects can possibly lead to completely different insights into the serial production of the Early

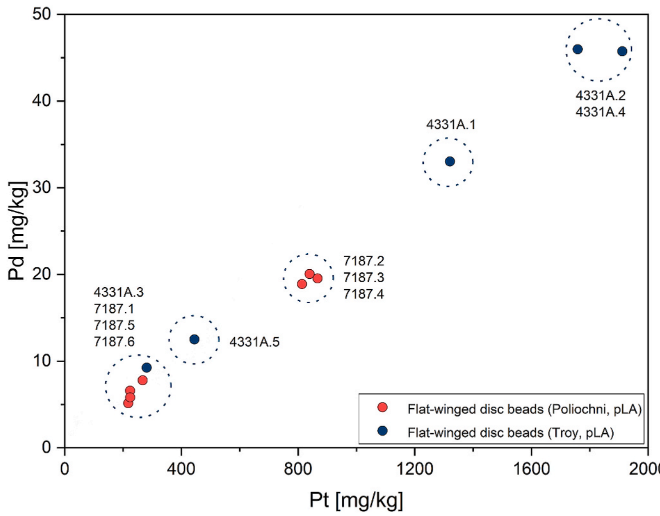

Fig. 10. Diagram of the platinum (Pt) and the palladium (Pd) concentrations of the same flat-winged disc beads from Troy (Π 4331A) and Poliochni (Π 7187) as in Fig. 9. The beads from Troy (n = 5 analyses) and Poliochni (n = 7 analyses) were all analyzed with pLA-ICP-MS and are represented by blue and red circles, respectively. The dotted ellipses indicate possible material groups and are merely drawn to aid the eye. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Bronze Age goldsmiths rather than isolated analyses of only a few objects.

Besides the tutuli and the flat-winged disc beads, the crescent-shaped earring with granulations provide evidence for serial rather than mass production by the goldsmiths, which is impressively illustrated by the different parts of the earring (Π 4332) made of the same stock of gold.

4. Discussion

4.1. Comparison with the Early Bronze Age gold from Ur

There is a long-lasting debate if and to what extent the gold found in the Royal Graves in Ur in Mesopotamia is related to the gold objects found in Troy (cf. Maxwell-Hyslop 1971, 57–61; McCallum 2015). As there are no natural gold deposits in Mesopotamia, it was indeed suggested earlier that the gold from Ur may derive from western Anatolia (Young 1972, 9). Meanwhile, much more evidence has become available that shows that there was a high degree of contact in the Old World between the Aegean and the Indus valley in the third millennium BCE. During the Early Bronze Age, the whole region is characterized by the administrative use of seals (Maran 1998, 232–240; Maran and Kostoula 2014), the use of canonical types of weights for different geographical regions that were partly spread over wide areas (cf. Rahmstorf 2016, 30, Fig. 2.3), and by the use of prestigious materials like lapis lazuli and carnelian (cf. Rahmstorf 2015, 162–163, Figs. 9–10; Ludvik et al. 2015). Particularly striking is the distribution of distinct jewelry items: flat-winged disc beads (cf. Maxwell-Hyslop 1971, 34–37; Aruz 2003; Rahmstorf 2011, 149; Döpper and Schmidt 2013, 33; Schwall et al. in press), and quadruple spiral motifs (Fig. 12; cf. Maxwell-Hyslop 1971, 34–37; Huot et al. 1980; Aruz 2003; Rahmstorf 2011, 147; Schwall et al. in press) which were also found at Troy and Poliochni (Sazcı 2007, 174–175, 193; Bernabò-Brea 1976, pl. 247c). These objects were primarily made of gold and their presence indicates an extended trade and communication network reaching from the Aegean to the Indus region with multiple junctions on waterways and caravan routes (cf. Horejs et al. 2020, 52, Fig. 35b; Schwall et al. in press).

Despite some variation in shape of the flat-winged disc beads, and the quadruple spiral motifs of beads, on pendants, and in rings, the use of these distinct objects over such a vast region indicates a kind of 'specific type of jewelry' used by elites. These individuals would thus have been participating in a far-reaching communication network by which goods and administrative practices as well as prestige objects and precious raw materials were distributed (Schwall et al. in press). Regarding the question of a possible connection between Troy/the Troad and Ur, pendants with an attached quadruple spiral are of special significance (Fig. 12). Four adornments, assigned to the Troad region, presumably Troy, were purchased by the Penn Museum in Philadelphia, and were recently returned to Turkey (Bass 1966, 37; 1970, pl. 86.20–23; Velioğlu et al. 2013). Remarkably, nearly identical gold objects are known from the Royal Tombs of Ur, Iraq (Woolley 1934, pls. 134.U.9656; 220; cf. Armbruster 2016, 130, Fig. 14). Based on the similarities of the pendants' composition and the motifs, the objects from the Troad were considered to be imitations of the Mesopotamian objects or even imports (cf. Bass 1966, 37; cf. Maxwell-Hyslop 1971, 35; Hauptmann et al. 2018, 101). It is now possible to recognize the striking typological relationships between Troy, Poliochni, and Ur with a comparison of their chemical compositions (Jansen et al. 2016a/b, 2018, 2021; Jansen 2019, Table 10.28–10.30).

Regarding the major, minor, and trace elemental concentrations and their ranges, the results of this study and the one of Jansen (2019) are quite comparable with only minor differences, for instance the lack of

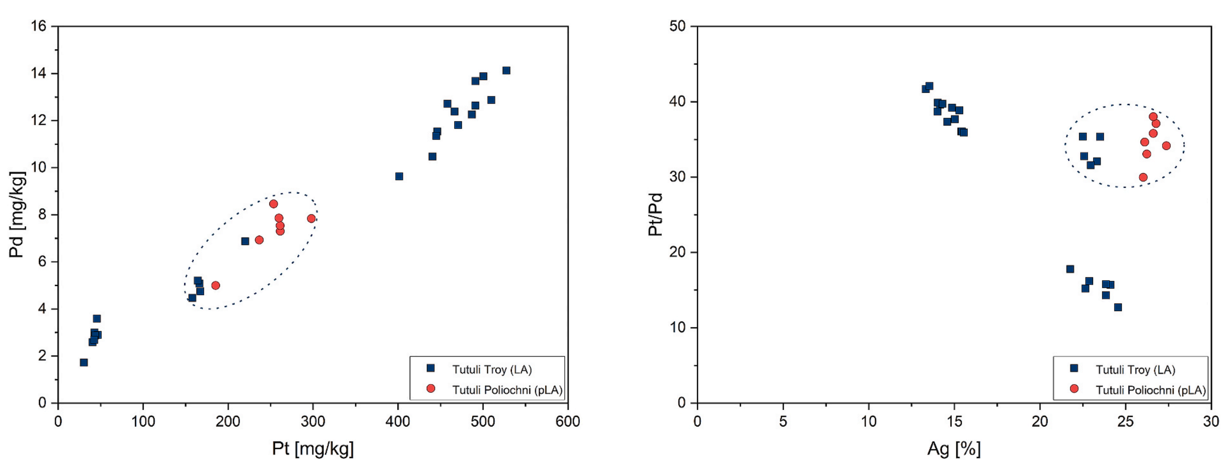

Fig. 11. Diagram of the platinum (Pt) and the palladium (Pd) concentrations (left) and of the silver (Ag) concentrations and the Pt/Pd-ratios (right) of the tutuli from Troy (Sch 6007/1–15) (n = 25 analyses) and Poliochni (Π 7172.1–5) (n = 7 analyses). The tutuli from Troy were analyzed with LA-ICP-MS and those from Poliochni with pLA-ICP-MS and are represented by blue rectangles and red circles, respectively. The dotted ellipses indicate possible material groups and are merely drawn to aid the eye. (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

Fig. 12. Distribution of the quadruple spiral motifs and the location of gold sources. (map: OeAW-OeAI/C. Schwall, M. Börner; quadruple spirals: Ur – Collins 2003, 130, no. 78; Legend – Højlund 2007, 75, Fig. 131; Troad – Born 2016, 467). (For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

material with less than 15% silver (Ag) in Ur. Trace element ranges described by Jansen (2019) match almost perfectly to those presented in this study (e.g., tin (Sn), palladium (Pd), platinum (Pt), and lead (Pb), see Figs. S3b and c, supplementary material no. 1). This clearly distinguishes both datasets from the gold that was commonly distributed in the Early Bronze Age in central Europe, which is generally characterized by very low contents of palladium (Pd), platinum (Pt), lead (Pb), and bismuth (Bi) (e.g., Ehser et al. 2011; Lockhoff and Pernicka 2014; see Figs. S3b and c, supplementary material no. 1). However, besides similar concentration ranges for Troy, Poliochni, and Ur, slight differences in elemental ratios were observed for palladium (Pd) and rhodium (Rh), which are commonly known to be stable during metallurgical processes and alteration effects. Analytically, both element concentrations are prone to interferences due to copper argide formation during ICP-MS analysis that need to be carefully considered in such a comparison. The application of a correction model to this data (see supplementary material no. 1) evidently results in a remarkable agreement between the gold objects from Ur on the one hand and Troy and Poliochni on the other (Fig. 13a).

Generally, the comparability of LA-ICP-MS data from different laboratories is a crucial point in archaeometric and geological gold studies (e.g., Gauert et al. 2016; Milidragovic et al. 2016; Blet-Lemarquand et al. 2019) and is therefore discussed along with the correction model in the supplementary material no. 1. As Jansen already pointed out (Jansen 2019), three Early Bronze Age objects from Ebla, dating to 2400–2300 BCE, also show a remarkably close chemical composition to the objects from Ur, suggesting the usage of the same gold source (Fig. 13a).

4.2. *Possible provenance of the gold from Troy and Poliochni*

Gold mainly occurs as a metal in nature, creating the impression that there should be not much change in composition between the geological occurrence and the archaeological artefact. However, there are many obstacles to a convincing provenance determination of gold with geochemical methods (Pernicka 2014). For the chemical characterization and differentiation especially of alluvial gold, the most serious problem is the fact that most gold that occurs in nature does so in the form of small flakes or micro nuggets. Thus, to produce a gold object of several grams, several thousands of these micro nuggets have to be melted together, which homogenizes the composition so that it may not resemble the composition of any individual grain. Accordingly, ancient gold objects consist of the melting product of a heavy mineral mixture with native gold as major component, but possibly supplemented by such minerals containing copper, tin, palladium, and platinum (see above). As a result, it is difficult, if not impossible, to directly compare fine-grained alluvial gold with archaeological gold artefacts.

Gold has only one stable isotope so that isotope analysis is only possible of the other minor and trace elements in the gold. Of these the isotope ratios of lead, copper, silver, and osmium have been tested, but so far with limited success. The reason for this is that lead and copper, for example, may have been added intentionally, which would indicate the provenance of these metals and not of the gold. Furthermore, lead concentrations are usually rather low so that the sample size required for analysis is often prohibitive. The chemical compositions and the osmium isotope ratios of PGM inclusions in gold appeared promising for provenancing gold. However, this proved difficult by the findings that both geochemical parameters are quite variable between different inclusions of the same object (Meeks and Tite 1980; Junk and Pernicka 2003). Using this approach, Jansen et al. (2016b, 22; cf. Jansen et al. 2018, 134 and Jansen et al. 2021, 288) have suggested that “Samti in Afghanistan and Takab in Iran were particularly intriguing potential sources for the gold artefacts from Ur”. However, Lydian gold coins, which also frequently contain PGM inclusions (Young 1972), show osmium isotope ratios that are almost identical to those of the gold from Ur (Junk and Pernicka 2003). Since it is rather unlikely that the Lydians obtained the gold from Afghanistan rather than from their own rich regional

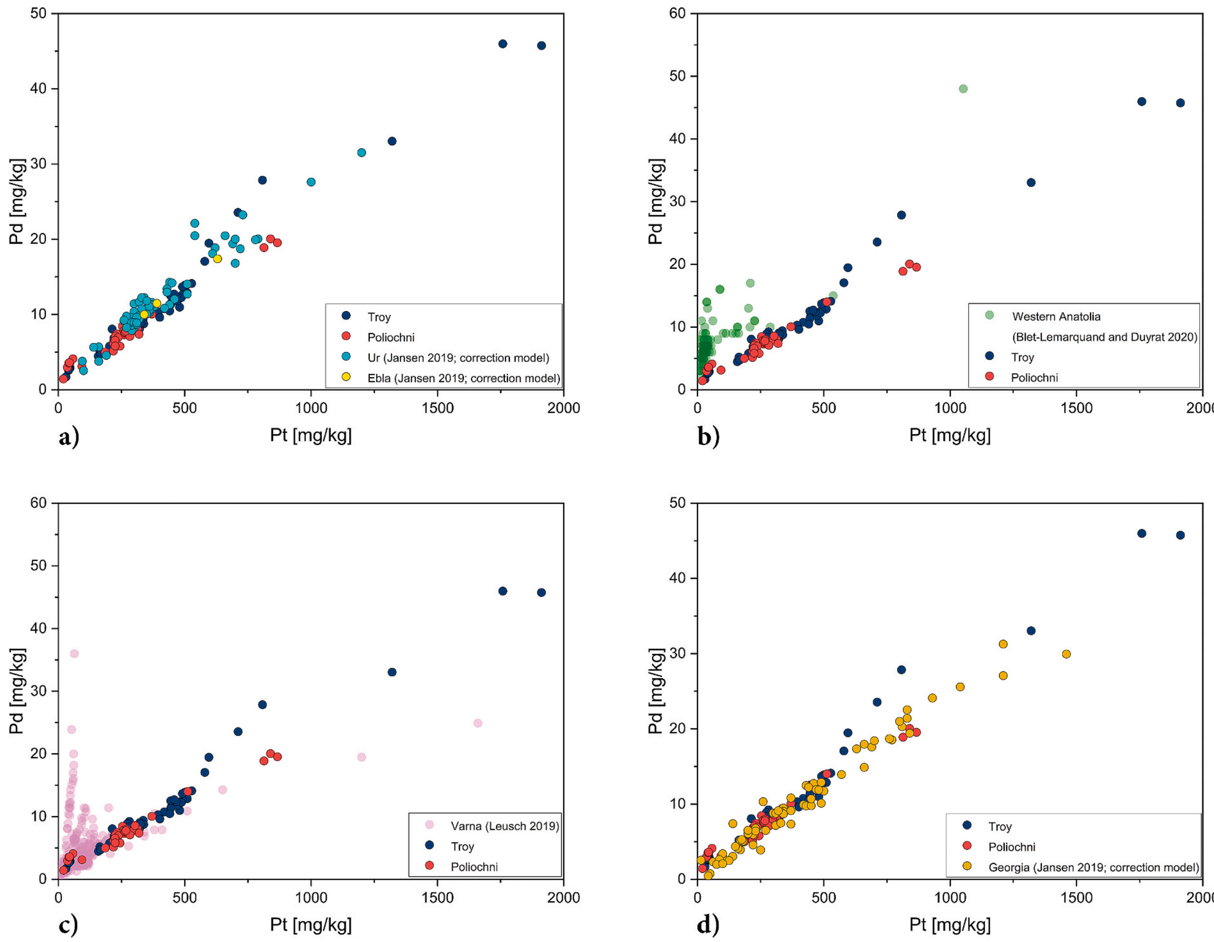

Fig. 13. (a) Comparison of the platinum (Pt) and the palladium (Pd) concentrations of gold objects from Troy, Poliochni, Ur, and Ebla (the data was partially modified according to a correction model, see supplementary material no. 1 for details). (b–d) The diagrams show the comparison of the data of this paper with gold objects from regions which could be possible natural gold sources: (b) Western Anatolia/Lydian electrum coinage, (c) Chalcolithic gold from the Varna cemetery, and (d) Early and Middle Bronze Age gold objects from the southern Caucasus/Georgia. Expanded versions of the lower left corner of Fig. 13b and c are to be found in Fig. S4 in the supplementary material no. 1.(For interpretation of the references to color in this figure legend, the reader is referred to the Web version of this article.)

resources, it seems that also osmium isotope ratios of PGM in ancient gold are much less distinct for a certain deposit or mineralized region and thus much less useful for provenance aspects than originally assumed. This is corroborated by the observation that most PGMs in chromitites from ophiolites all over the world yield $^{187}Os/^{188}Os$ ratios between 0.12 and 0.13 with a median of 0.12 (Cabri et al. 2022), exactly the range observed in PGMs in gold objects from Ur and in Lydian electrum coinage. Overall, this means that we are still largely left with the trace element pattern as a means to narrow down the possible provenance of ancient gold, specifically the concentrations of platinum and palladium as well as the possibility of analyzing other trace elements.

To overcome the problems described above it seems more promising to assume ancient artefacts as representative for the kind of gold that was available in a certain region irrespective of their dating, as long as it can be reasonably assumed that they do not consist of recycled gold and are not made from gold from distant sources. The major regions in the Old World with natural gold mineralization (cf. Fig. 12) are from west to east Transylvania in Romania, southern Bulgaria and northern Greece, western Anatolia around Izmir, the southern Caucasus with northwestern Iran, the gold-tin belt stretching from Turkmenistan to the Altai region and northeastern Afghanistan (cf. Muhly 2015). Furthermore, there are numerous gold mineralizations in the Eastern Desert of Egypt and the Sudan as well as in western Saudi Arabia.

Of these regions trace element data of ancient gold objects are only available from southeastern Europe (Leusch 2019), from western Anatolia (Blet-Lemarquand and Duyrat 2020), and from Georgia (Jansen 2019). The analyzed objects chronologically span several millennia; however, they are mainly regarded as compositionally representative of the regional alluvial gold.

The major problem in the interpretation of the analyses of the Lydian electrum coinage (Ramage and Craddock 2000) is the possibility of alloying gold with silver (Cahill et al. 2020; Blet-Lemarquand and Duyrat 2020). The addition of silver to natural or to purified gold would alter the trace element concentrations (especially those of lead and bismuth). However, since silver was produced from argentiferous lead ores, the addition of such silver is not expected to change the ratio of platinum and palladium to any degree. Accordingly, these two elements can still be used for comparison with gold from Troy and Poliochni. In addition, since we are dealing with the earliest coinage we assume possible recycling to be negligible. From Fig. 13b it seems that western Anatolian gold sources as represented by the electrum coinage can be excluded for the majority of the gold objects from the Early Bronze objects from the northeast Aegean and Ur.

In a similar way, we can compare our results with the Chalcolithic gold from Varna on the Bulgarian Black Sea coast (Leusch et al. 2015, 2016). This shows that the regional gold also contains platinum other than assumed by Hartmann (1982), who suggested an origin from the southern Caucasus instead. Figs. 13c and S4 (supplementary material no. 1) show a partial overlap of the gold from Varna with gold objects from the northeast Aegean, which suggests that one of the sources that provided gold to Varna in the fifth millennium BCE could also have contributed to gold objects from Troy and Poliochni in the third millennium BCE. However, especially the objects from Varna with platinum concentrations <100 mg/kg seem to follow several trend lines that are primarily associated with lower Pt/Pd-ratios. One of these trend lines may be related with those Early Bronze Age objects from Troy and Poliochni with lower ratios (cf. Fig. 8 left). Nevertheless, objects from Troy and Poliochni with platinum concentrations >200 mg/kg differ from most of the Varna gold. In any case, regarding the gold supply of the sites of Troy and Poliochni in the third millennium BCE, the Varna gold source seems to be of minor significance.

Finally, there is also comparable data available from Early and Middle Bronze Age gold objects from Georgia (Jansen 2019). If we use the same correction model for the palladium concentrations as above, we find a remarkably good match of the platinum and palladium concentrations with the gold from Troy, Poliochni, and Ur (Fig. 13d). Jansen (2019) concluded that the large variations of some trace elements like antimony and bismuth in gold objects from Georgia compared with those from Ur would indicate that the two regions followed different procurement strategies of gold: While Ur seemed to rely on a single gold source, there may have been several in use in Georgia. He furthermore suggested that silver may have been added to gold, at least in the second millennium BCE, which would have also affected the concentrations of lead and bismuth. However, antimony and bismuth are not reliable indicators of gold provenance and in addition their large variation ranges cover the smaller ranges of Troy, Poliochni, and Ur. The more significant trace elements platinum, palladium, and tin have a striking overlap of their interquartile ranges in objects from the three sites and from Georgia suggesting a common origin. Since western Anatolian and southeastern European gold sources have largely been excluded, the southern Caucasus presently remains the best match as the source for the majority of the gold objects from the three sites and Ebla. In order to substantiate this line of argumentation more analyses of Early Bronze Age gold and of the heavy mineral concentrates of rivers in these regions are required. First indications for this can be seen in the Ushkiani project in Armenia (Kunze et al. 2013; Kunze et al. in preparation).

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: The authors report financial support was provided by the Gerda Henkel Foundation. The authors report financial support was provided by the Austrian Academy of Sciences.

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5. Conclusions

The innovative pLA-ICP-MS method has been successfully implemented for the analysis of Bronze Age gold objects. By applying this technique, it was possible to perform quasi non-destructive on-site sampling in museums for the first time. Access to spectacular gold masterpieces of the Aegean Bronze Age, which is usually restricted, was permitted. The analyses yielded a large number of significant results regarding the composition, processing, and possible origin of the gold. This innovative approach, therefore, illustrates the enormous potential of interdisciplinary research between the natural sciences and the humanities. Important insights into the use of gold during the Bronze Age period have been achieved:

- Secondary (alluvial) gold deposits were exploited for the production of the analyzed objects from Troy and Poliochni.
- Most of the gold objects from Troy and Poliochni are likely to derive from one geological source. The comparison with the gold from Ur and Ebla evidently shows that gold that is almost completely chemically identical was used for their production, suggesting far reaching supply networks in the Early Bronze Age in the Old World. However, the geological source remains unknown and further analyses on natural gold as well as on representative gold objects are needed in the future.
- Detailed evaluation of gold with low platinum contents suggested that a second gold source may have contributed to the gold objects from Troy and Poliochni to a minor degree. This second source may be sought in southeastern Europe as the comparison with fifth millennium BCE gold from Varna indicated.
- Regarding the production technology of the gold objects, it has been shown that different gold batches were used for similar finds from same contexts (i.e., flat-winged disc beads and tutuli). This leads to the assumption that a serial production was practiced in these workshops in the Early Bronze Age.
- Regarding a future sampling strategy of gold objects, the presented results underline the necessity of performing not just one analysis of complex composite objects but as many as possible.

In the course of our project, we primarily studied Late Bronze Age gold from the Mycenaean culture. These results will be presented in a separate monograph.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jas.2022.105694.

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