مطالعات فنی دو شی آلیاژ مس متعلق به دوره مفرغ ایران

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چکیده
در این پژوهش، مطالعات فنی با استفاده از روش‌های آزمایشگاهی دو شیء ساخته‌شده از آلیاژ مس از ناحیه شمال ایران شامل یک خنجر متعلق به تورنگ‌تپه و یک حلقه (بازوبند) متعلق به تپه حصار از دوره مس‌سنگی جدید و دوره مفرغ انجام شده است. در این مطالعه از روش‌های آنالیز شیمیایی و میکروسکوپی شامل آنالیز پلاسمای جفت شده القایی–طیف سنجی نشر نوری (ICP-OES)، میکروسکوپی الکترونی روبشی مجهز به طیف سنجی پراش انرژی پرتو ایکس (SEM-EDS) و متالوگرافی استفاده شده است. نتایج مطالعات نشان داد که خنجر از آلیاژ برنز قلعی ساخته شده؛ درحالی‌که حلقه از مس ناخالص یا مس آلیاژنشده با میزان کم ارسنیک ساخته شده است. روش ساخت و شکل‌دهی هر دو شیء شامل چرخه‌های متناوب کار سرد و تابکاری است با این تفاوت که میزان تابکاری برای از بین بردن مغزه‌دار شدن اولیه در حلقه مسی کافی نبوده است. این امر موجب ایجاد ساختار روی‌هم افتاده به صورت نوارهای تقریبا موازی باقی‌مانده بر روی ساختار کارشده و بازتبلوریافته است که در ریزساختار اچ شده نمونه قابل مشاهده است. از سوی دیگر، آخال‌های غیرفلزی در ریزساختار هر دو شیء قابل مشاهده هستند. ترکیب شیمیایی این آخال‌ها در ریزساختار خنجر برنزی شامل سولفیدهای مس و در ریزساختار حلقه مسی شامل اکسیدهای مس است. این امر بیانگر تفاوت در ترکیب سنگ معدن‌های استفاده شده برای تولید این دو شیء است. در نتیجه، می‌توان فرض کرد که دو شیء مورد مطالعه با دو فرایند متفاوت تولید شده‌اند اگرچه هر دو فرایند در دوره مفرغ در ایران معمول بوده‌اند.

واژگان کلیدی: فلزگری باستانی، تورنگ‌تپه، تپه حصار، برنز قلعی، مس آلیاژنشده.

https://doi.org/10.22034/JINM.2021.252916

Technical Studies on Two Copper-Based Objects from the Bronze Age of Iran

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Abstract
A multianalytical study was undertaken on two copper-based objects from northern Iran that are dated to the Late Chalcolithic and Bronze Age including an arm ring from Tappeh Hissar and a dagger from Tureng Tepe. The study was performed by using chemical and microscopic methods including inductively coupled plasma-optical emission spectroscopy (ICP-OES), scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDS) and optical microscopy (metallography). The results showed that the dagger has been made of tin bronze alloy while the arm ring is made of impure copper with a low amount of arsenic. Both objects are shaped and manufactured by cycles of cold-working and annealing although it has not been enough in the arm ring to remove all coring occurred during the casting. This is recognizable by the fact that super-imposed microstructure in the form of partially parallel banded "ghost" microstructure is visible in the etched cross section of this object. The non-metallic inclusions are scattered in the microstructure of the objects. They can be identified as sulphidic copper inclusions in the dagger and oxidic copper inclusions in the arm ring. This shows the use of different copper ores to produce these objects. Consequently, it can be assumed that two objects were manufactured and produced by two different processes even though both were common production and working techniques during the Late Chalcolithic and Bronze Age on the Iranian Plateau.

Keywords: Archaeometallurgy, Tureng Tepe, Tappeh Hissar, Tin bronze, Unalloyed copper.

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Introduction

As a matter of the fact, the first evidence for copper-based metallurgy can already be observed during the Neolithic period (ca. seventh millennium BC). Afterwards it was further developed by using arsenical copper and tin bronze during the Chalcolithic and Bronze Age in the Iranian Plateau (Oudbashi et al. 2012; Pigott, 2004; Thornton, 2009). It is worth noting that the early evidence of tin bronze (Cu-Sn) metallurgy emerged in the western Iran (Luristan) while it was spread to the central and southern parts of the Plateau in later times from third to first millennium BC (Fleming et al. 2005; Helwing, 2013). Available information is obtained based on few scientific and analytical studies performed on copper alloy objects dated to prehistoric period of Iran.

The evaluation of the published information about the emergence and spread of copper-based metallurgy in the prehistoric Iranian Plateau suggests that it is necessary to extend the systematic analytical and archaeological research. It may result in a better recognition of archaeometallurgical activities and remains during the a specific time, especially from the Bronze Age to the Iron Age, a long period in which tin bronze metallurgy was formed and established. Early dated proofs for the usage of tin bronze were discovered in the Early and Middle Bronze Age levels of various archaeological sites such as Kalleh Nisar, Bani Surmeh, Susa, Deh Dumen and Godin Tepe. There, beside many arsenical copper/arsenical bronze artefacts the number of tin bronze objects (with low tin concentrations) is very limited (Fleming et al. 2005; Nezafati, 2006; Oudbashi et al. 2016; Flame, 2010). At the end of the third millennium BC the metallurgical situation in specific regions of Iran changes and arsenical copper/arsenical bronze is regularly replaced by tin bronze. As example one can point out the metallic remains found in Susa or Malyan (De Ryck et al. 2003; Pigott et al. 2003). Generally speaking, (with some exceptions), tin bronze became the commonplace metal during the Late Bronze Age in most parts of the Iranian Plateau (Pigott, 2004; Oudbashi et al. 2012; Oudbashi, 2019). Nevertheless, tin bronze is rare in the north-eastern and eastern Iran even during the Late Bronze Age as is demonstrated by the metallic remains from various important archaeological sites such as in Tepe Yahya (Tappeh Yahya), Tepe Hissar (Tappeh Hissar), Shahr-i Sokhta and Shahdad (Thornton et al. 2002; Thornton et al. 2004; Meier, 2011; Hauptmann et al. 2003). In fact, the Bronze Age, which begins in the late of the fourth/early third millennium BC on the Iranian Plateau, may be considered as the era during that the change from extensive use of arsenical copper (arsenical bronze) metallurgy to the emerging and developing of tin bronze metallurgy can be observed in this specific region (Oudbashi et al. 2012; Overlaet, 2004). Only few tin bronze objects are detectable among the copper-based artefacts in Iranian Plateau from early Bronze Age of Luristan (Fleming et al, 2005).

The present paper aims to focus on the metallurgical analysis of two copper-based objects found in northern Iran to characterize the manufacturing and metallurgical processes used to produce these objects.

Materials and Methods

Two copper-based objects from the Iran National Museum were selected for analytical studies. The objects include a dagger from Tureng Tepe (Gorgan Plain) and a bracelet or arm ring from Tappeh Hissar (north-central Iran). (Fig. 1). The first is recorded in the museum as No. 2250 is dated back to Tappeh Hissar IA level (Late Chalcolithic/Early Bronze Age, ca. fourth millennium BC) and the second is recorded as No. H 76-52 is dated back to Tureng Tepe IIIC level (Late Bronze Age, ca. second millennium BC). The objects were named as TUT-IA-070 and HES-IA-071, respectively (Figure 1). The dating of these objects is undertaken based on their information recorded in the museum database.

A very small fragment of each object was cut by a jeweller's saw and about 0.5 g of each object was drilled by a micro-drill for analytical studies. The separated fragments were mounted in epoxy resin, ground and polished with abrasive paper and diamond paste and prepared for microscopic analysis. The drilled fil-

Fig. 1. Two copper-based objects from Iran National Museum, analysed in this study.
The location of sampling is marked by arrow.

ings were dissolved in diluted acid (aqua regia) and prepared for chemical analysis.

For chemical analysis, inductively coupled plasma- optical emission spectroscopy (ICP-OES) was performed to characterize the major, minor and trace elements in the composition of the metal objects. The prepared solution was measured by (ICP-OES) method. This analysis was undertaken by an ICP-OES Varian 735 model in Zarazma Mineral Studies Company, Tehran. The detection limit of the method was 0.001 weight percent (wt.%).

Microscopic studies were performed on the objects so as to understand the microstructural features as well as the manufacturing processes applied for shaping copper-based objects. The mounted cross sections were studied by metallography using a Zeiss reflected and transmitted light microscope model Primotech before and after etching with alcoholic $FeCl_3$ solution. Furthermore, the microscopic observation in high magnification and chemical microanalyses of samples were obtained from cross sections of the objects by scanning electron microscopy coupled with energy dispersive X-ray spectroscopy (SEM-EDS). The cross sections were coated with carbon and then inserted into the SEM housed at the VIAS, University Vienna. The analysis was conducted by Zeiss EVO 60 XVP with an EDS system produced by Oxford Instruments (INCA 400) with specifics as accelerating voltage: 20 kV, working distance of 9.5 mm, beam current 100μA, dead time between 30 and 40%. The stability of the beam current was verified by cyclical measurements of a cobalt standard, various BAM-Bundesanstalt für Materialprüfung and in-house standards and reference materials were additionally used. All results were normalized to 100% and are given as mass percentage. THE SEM-EDS system is located at the VIAS, University of Vienna.

Results and Discussion

The results of chemical composition of the samples are presented in Table 1. The composition of sample TUT-IA-070 shows that this dagger is made of copper-tin alloy with about 5.8 wt.% of tin. Arsenic is measured as 0.017 % and lead is determined as 0.653 wt.% respectively. Also, sample HES-IA-071 is a copper object with about one percent of arsenic. Lead and tin are measured as trace elements (< 0.1 %).

The chemical composition of two objects is completely different and the dagger (TUT-IA-070) can be considered as a tin bronze while the second one (HES-IA-071) consists of impure copper (or unalloyed copper) with significant amounts of arsenic (about 1 wt.%).

The metallographic study on the cross section of the two samples before etching showed the single phase matrix of the alpha solid solution including the presence of grey inclusions that are scattered in the microstructure of samples. The size of these inclusions differs in the two objects and are significantly larger in the arm ring (HES-IA-071). The shape of these inclusions are variable and rounded in many cases (Fig. 2a and 2c).

The etched microstructure of the dagger shows equiaxed grains including the worked and annealed grains with twin bands. It shows that the dagger is manufactured by repetitive cycles of thermomechanical operations including cold-working and annealing or hot-working. The twin bands are straight and no slip lines are present in the grains. The microstructure of the arm ring from Tappeh Hissar also shows the presence of twinned and equi-axed grains.

	Ag	**Al**	**AS**	**Ba**	**Bi**	**Ca**	**Cd**	**Cu**	**Fe**	**K**	**Mg**
TUT-IA-070	0.085	0.001	0.017	n.d.	0.031	0.015	n.d.	2.827	0.063	0.004	0.004
HES-IA-071	0.152	0.002	0.989	0.001	0.031	0.019	0.003	8.294	0.007	0.001	0.005
	Na	**Ni**	**Pb**	**S**	**Sb**	**Sc**	**Si**	**Sn**	**Ti**	**V**	**Zn**
TUT-IA-070	0.089	0.007	0.653	0.246	n.d.	0.001	0.034	5.814	0.001	0.001	0.105
HES-IA-071	0.084	0.005	0.083	0.093	0.064	0.001	0.032	0.017	0.001	0.001	0.113

Table 1. The results of ICP-OES analysis of the chemical composition of two objects in wt.%. n. d. =under the detection limit of the analytical device.

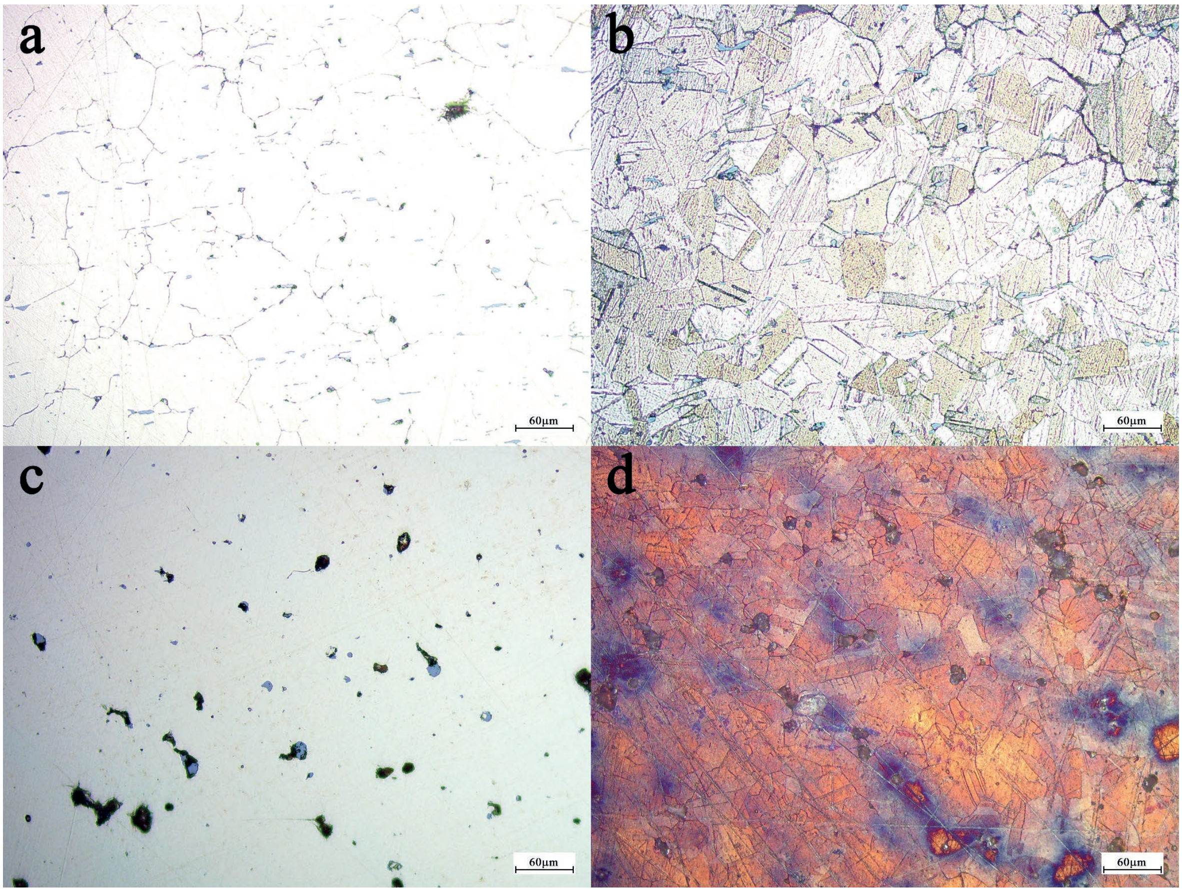

Fig. 2. Metallographic micrograph of the cross section of two objects: a) microstructure of the dagger (TUT-IA-070) before etching, b) microstructure of the dagger after etching, c) microstructure of the arm ring (HES-IA-071) before etching and d) microstructure of the arm ring after etching. Etchant: alcoholic $FeCl_3$ solution, magnifications: 200x.

Nevertheless, a different feature is visible in the microstructure of this object. Under the microscope the presence of the super-imposed or banded "ghost" microstructure is visible. It includes some light and dark parallel (partially) bands that are imposed over the worked and annealed microstructure. These parallel bands, known as banded "ghost" microstructure, are the remnant of coring occurred during casting of the molten metal that has not been removed completely by thermo mechanical operations (cold-working and annealing) (Dungworth, 2013; Dungworth, 2002; Scott, 1991). Another interesting aspect is, that the grain size is larger in the dagger of Tureng Tepe, showing less times of thermomechanical cycles undertaken on this object in comparison with the arm-ring. In fact, it is worth noting that both objects are manufactured by the same processes including working and annealing processes but the times of the thermo mechanical cycles and the temperature of the annealing were different during the production process.

SEM-BSE micrographs of partially etched cross section of the two objects show the presence of numerous inclusions and phases scattered in the copper matrix. Also, intergranular corrosion is visible in both objects. Two main inclusions are visible in the SEM-BSE micrographs:

- The dark grey inclusions (phase A) that are in different shapes (as also was observed in metallographic images). The size of these inclusions are different in the two objects.
- The bright inclusions or phases (phase B) that are only visible in the cross section of the dagger (TUT-IA-070) and are circular in shape. This phase was not observed in OM micrographs.

Some phases were analysed by SEM-EDS method to identify the chemical composition of them. Results of this analysis are presented in Table 2. The results of EDS analysis of the dark grey inclusions (A) shows that from chemical point of view they are different in two objects. The main elements in the dark grey inclusions of TUT-IA-070 are copper and sulphur. Copper is measured more than 70 wt.% while sulphur is measured at about 20 wt.% in five analysed phases in the cross section of this object. In contrast, the dark grey inclusions of the arm ring (HES-IA-071) have very low amount of sulphur and the main constituents are copper (about 65 to 75 wt.%), chlorine (about 16 to 25 wt.%) and oxygen (about 6 to 8 wt.%). Based on the analytical data, it can be concluded that the dark grey inclusions of the dagger are copper sulphides with low concentrations of iron (in two cases) while the inclusions present in other object are made of copper oxides. Determining chlorine in high concentration in these inclusions may be due to corrosion attacks occurred in the partial etching of the sections before SEM analysis.

The non-metallic (oxidic or sulphidic) inclusions are commonplace in the microstructure of the ancient copper-based objects of Iran. These are remnants of the copper ores used to produce metallic copper (e.g. matte remnants or products) that are not completely transformed to metal during smelting process. These inclusions were segregated from the metallic matrix during cooling/solidification of melt because they are not soluble in the copper solid solution. Thus, they are scattered as segregated non-metallic sulphidic inclusions in the metallic matrix. The concentration of sulphur in the composition of two objects is different as it is measured as 0.246 wt.% in the dagger in which sulphidic inclusions are available while it has been measured 0.093 in the arm ring (Table 1).

The EDS analysis of the bright phases (B) of the dagger shows that the main element is Pb. It has been measured at about 71 wt.%. These are segregated Pb globules that are not dissolved in the copper matrix due to immiscibility of lead in copper (Scott 1991). The measurements were rounded.

The results of the multianalytical studies on two copper alloy objects from two important Bronze Age sites from northern Iran shows the variability of the copper-based metallurgy during that period. The dagger from Tureng Tepe is made of a binary tin bronze alloy while the arm ring from Tappeh Hissar is manufactured by an impure copper with about 1 % of arsenic. Both materials were used during the 3rd and 2nd millennium BC as raw materials to produce different objects. In fact, the metallurgy of copper alloys in the Bronze Age (ca. 3000-1500 BC) includes application of different materials from impure (unalloyed) copper, arsenical copper/bronze as well as tin bronze. It is worth noting that the third and second millennium BC can be considered as transition from copper metallurgy (impure copper and arsenical copper) to tin bronze in the Iranian Plateau. On the other hand, the copper sulphide inclusion and lead globules are commonplace in the microstructure of the

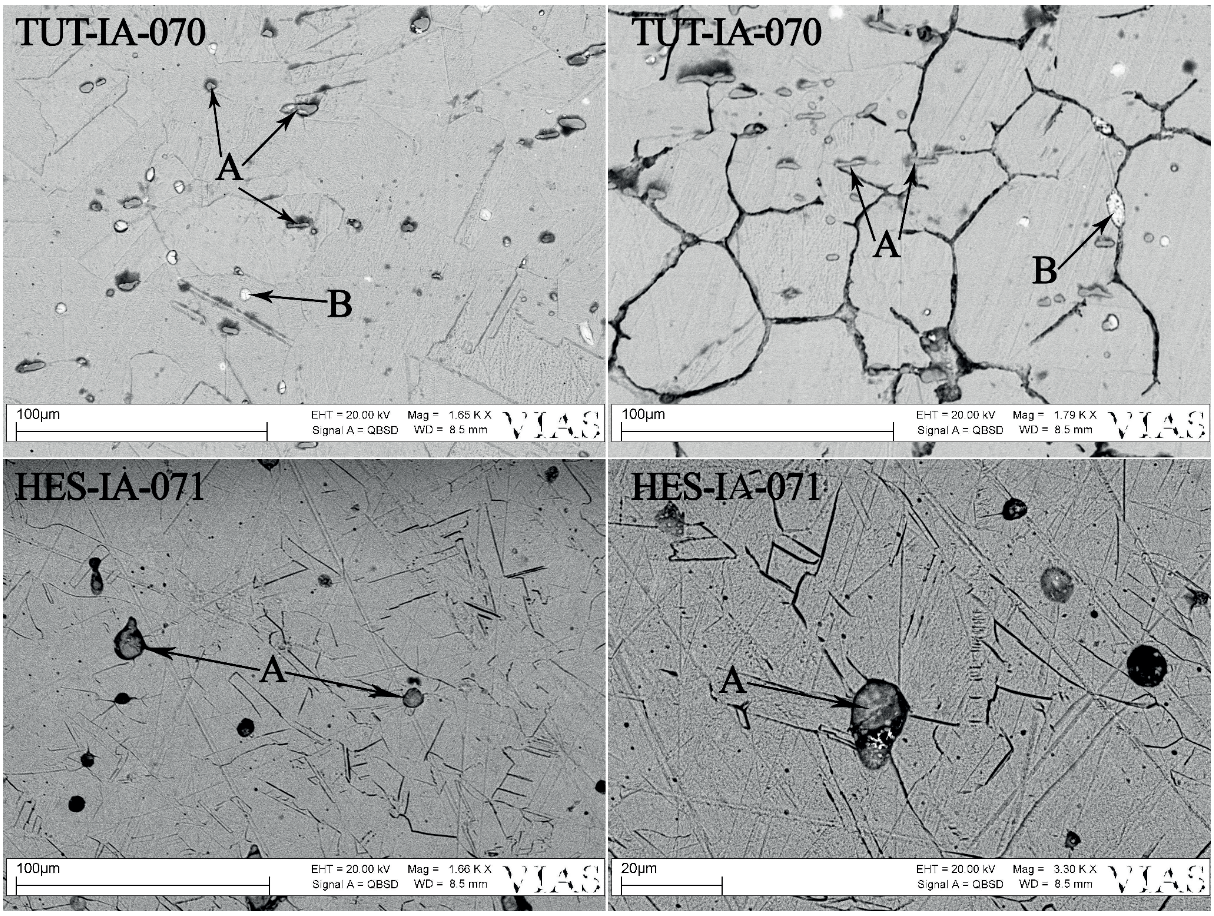

Fig. 3. SEM-BSE micrograph of the cross section of two objects showing presence of two main types of inclusions in the microstructure of objects, the dark grey inclusions (A) and the bright phases (B).

Sample	Phase	O	S	Cl	Si	Fe	Cu	As	Pb
TUT-IA-070	Grey Inclusions (A)	2.7	19	0.6	–	–	78	–	–
		2.7	19	0.8	–	–	78	–	–
		2.2	19	0.7	0.3	–	78	–	–
		3.5	20	0.5	–	1.6	75	–	–
		3.5	21	0.5	–	1.8	74	–	–
	Bright Phases (B)	4.1	–	22	–	–	3.4	–	71
		6.7	–	14	1.2	–	8.8	–	69
HES-IA-071	Grey Inclusions (A)	6	0.8	21	–	–	72	–	–
		7.5	0.9	25	–	–	66	1.0	–
		7.7	0.5	16	–	–	76	–	–

Table 2. The results of SEM-EDS analysis of different types of inclusions present in the SEM-BSE micrographs of the objects, in wt.%.

prehistoric copper and bronze objects from Iran but the oxidic inclusions are less observed than the sulphidic ones, although are observed in some objects from northern and north-eastern Iran (Oudbashi et al, 2020; Oudbashi et al, 2021). The grain microstructure of the samples shows different volume of thermomechanical operation to shape these objects leading to form recrystallized grains with twin bands, although some remnants of primary coring is retained as super-imposed microstructure in the arm ring of Tappeh Hissar. Accordingly, the metallurgical aspects observed in both objects have been commonplace during the Bronze Age of the Iranian Plateau.

Conclusion

The study of metallic objects from the prehistoric period of the Iranian Plateau can contribute fundamental knowledge to understanding of ancient metallurgy. Due to long history of metallurgy and innovations used by ancient metalworkers to produce numerous objects much – but widely unstudied – metal artefacts are present. The Bronze Age (ca. 3000-1500 BC) is a very important period for the research on copper base metallurgy in the Plateau because it can be named as the transition stage from copper/arsenical copper to tin bronze metallurgy. In this study, two objects excavated at two Bronze Age sites including a dagger from Tureng Tepe (Gorgan Plain) and an arm ring from Tappeh Hissar (north-central Iran) were selected and studied by multianalytical methods. Both objects belong to the Iran National Museum. The results showed that the dagger from Tureng Tepe has been made of tin bronze with about 5 wt.% of tin. This object is shaped by cycles of working and annealing and there are numerous dark grey and bright phases scattered in the alpha solid solution matrix that are copper sulphide inclusions and lead globules, respectively. The arm ring from Tappeh Hissar is produced by impure copper with low concentrations of arsenic. It is shaped with comparable technology like the dagger but with lower volume of thermo mechanical process leading to retain the primary coring from the casting process. It can be recognized from the banded "ghost" structure that has been observed also in other copper-based objects from the Bronze Age. The dark grey inclusions scattered in the copper matrix of this object are copper oxide inclusions. The different non-metallic inclusions in these objects are due to using different copper resources (copper ores) to obtain raw materials to produce copper for metallurgical activities. Consequently, the metallurgical features observed in these objects fall in line with the available data from the copper base metallurgy in the Bronze Age of Iran.

Acknowledgements

The authors want to thank to Dr. Jebrael Nokandeh and Yousef Hassanzadeh from Iran National Museum for their supports to perform this study. Special gratitude goes to the Kharazmi University-Ministry of Science, Research and Technology (MSRT) of I.R. Iran and Österreichischer Austauschdienst (OeAD) of Austria under IMPULSE (Iran-Austria) joint program for the financial support of this research (title: The emergence and spread of tin bronze alloying in prehistoric Iran – an interdisciplinary approach).

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