



Analytical Assessment of Chaltasian Slag: Evidence of Early Copper Production in the Central Plateau of Iran

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ABSTRACT

This study reports the archaeometallurgical analyses results on six slag remains obtained from Chaltasian, Iron Age II, in the Central Plateau of Iran, excavated by Islamic Azad University, Varamin-Pishva Branch. Metallurgical studies were carried out to identify oxides, Ca-rich silicates and metallic phases in the slag material, using wavelength dispersive X-Ray fluorescence (WDXRF), followed by an analysis of one sample under the polarizing microscope: plane polarized light (PPL) and cross polarized light (XPL). Based on the analyses, it has been concluded that these six copper slag remains have a considerable amount of silica, which had been added to the smelt to increase its fluidity. Analyses showed a clinopyroxene microstructure in a glassy matrix for five samples, and a barite source, from a probable lead-zinc source in limestone, for the other sample. The absence of arsenic in these copper slags could show a paradigm shift in copper production in this space-time grid. According to the low amount of slag present on site, on the one hand, and the application of relatively advanced extraction technology on the other, this research introduces Chaltasian as an Iron Age II small copper production centre in the Central Plateau of Iran with a locally-developed copper extraction technology.

1. Introduction

The Iranian Central Plateau is located between the Southern Alborz Mountain and the Central Iranian desert and most of the fertile plains are located between the two above areas (Figure 1) (Fazeli Nashli 2013).

The study of ancient slag material plays a significant role in understanding old metallurgy. The discovery of slag remains from several sites in the Central Plateau in the 4th Millennium BC has been an important clue to prove the utilization of smelting processes. These initial developments were centred on metallurgical-rich regions on the Iranian Plateau (Pigott 2004): Tappeh Sialk, Tal-I Iblis, Tappeh Ghabristan, Arisman, Tappeh Zaqeh (Pernicka 2004a; 2004b; Nezafati, Pernicka 2006), Tepe Hissar and Hassanlo (Thornton *et al.* 2009). The existence of slag material in Chaltasian shows that the site can fall into the same category in the Early First Millennium BC. Chaltasian is an Iron Age II site, situated in the Asgariyeh Rural District, Central District of Pishva County, Tehran Province. The area of this site during the Iron Age I period was 6500 m² (Figure 2).

The first season of excavation at Tappeh Chaltasian was conducted by the Varamin-Pishva Branch of the Islamic Azad University under the supervision of Rouhollah Yousefi Zoshk from September to December 2012 (Yousefi Zoshk 2012). The Chaltasian area is located on the western edge of the city of Pishva, about 5 km away (Figure 3).

GPS coordinates for the site's centre (datum point) are N: 3519176, E: 0514135, with a height of 941 metres asl. and 5 metres above ground level. During the excavation season, two units were dug out; a 2.5 by 2.5 metre unit in the central mound and a 1.5 by 1.5 metre unit in the east mound (Figures 4 and 5) (Yousefi Zoshk 2012).

Slag is defined as the vitrified waste products of pyrotechnological practices. Generally speaking, they are composed of the glassy, solidified melt of reacted ore and gangue minerals, and fuel ash, as well as anything else which had been added to the smelt (Hauptmann 2007). Archaeometallurgy can determine old casting methods and metalworking. It can also give us access to the cultural and social norms that shaped technological practices and, perhaps, to the cognitive structures that created such norms (Smith 1965; 1978). Analytical research on ancient metals

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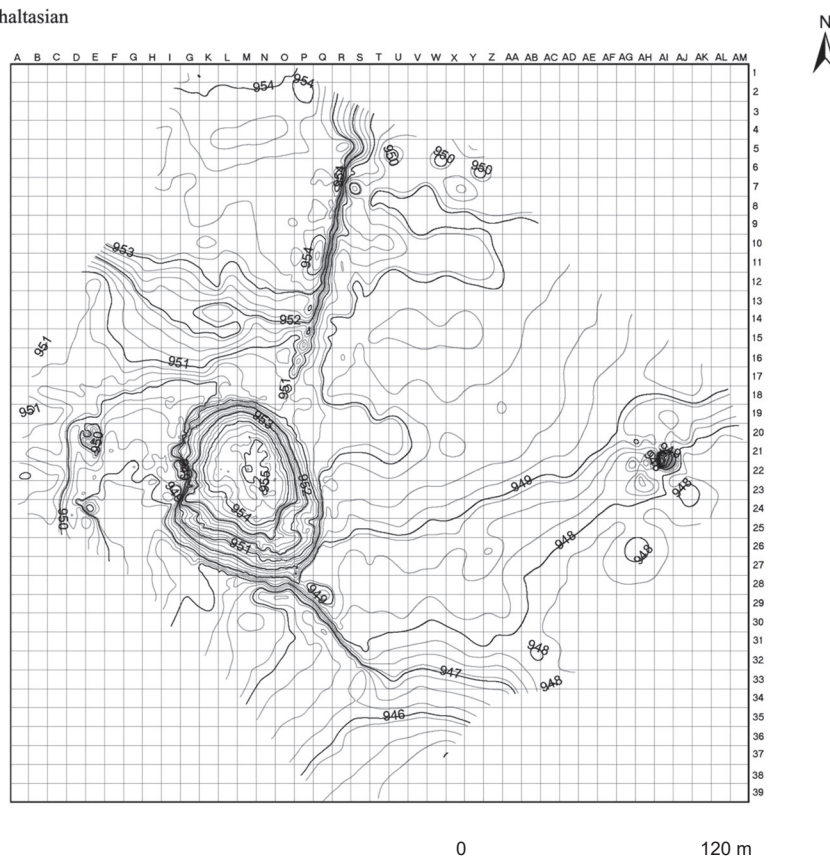


Figure 1. Map of Iran.



Figure 2. Aerial Photo, Chaltasian (Yousefi Zoshk 2012)

Figure 3. Topographical Map, Chaltasian Tepe Chaltasian (Yousefi Zoshk 2012)



is currently a common technique to reach an understanding of the specialization in alloy production. Elemental analyses are a type of characterization research in archaeometry. The comparative assessment of chemical composition can lead to the determination of metal manufacturing processes. Archaeologists, therefore, prefer to employ chemical and physical techniques to identify both the elemental composition and production technology (Kashani *et al.* 2013a). In this work, six slag remains have been analyzed to obtain information about the quantitative elemental composition of the slag material and its mineral resources.

2. Theoretical background

The production and use of copper and its alloys on the Iranian Plateau might have been started in the Neolithic site of “Ali Kosh” in the south west of Iran, where a rolled bead of native copper was found (Moorey 1969; Pigott 1999a; Thornton 2009). The bead from “Ali Kosh” has been dated to between the 8th and 7th Millennium BC (Hole 2000; Thornton 2009). It has been further specified that copper extraction technology on the Iranian Plateau had local developments during the Bronze Age (Dyson, Voigt 1989; Oudbashi *et al.*



Figure 4. Chaltasian, central mound (Yousefi Zoshk 2012).



Figure 5. Chaltasian, eastern mound 2 (Yousefi Zoshk 2012).

2012). Consequently, at that time, there was a vast variety of local copper production centres in several regions in west and central Iran (Thornton 2009). Archaeometallurgical studies on the copper production centres in Iran have been reported for several different areas, such as: Lurestan, Kerman, Yazd, Khuzestan, Sistan and the northern part of the Central Iranian Desert (Pigott *et al.* 1982; Hezarkhani, Keesmann 1996; Emami 2005; Keesmann 2005; Thornton 2009; Roustaei 2012). The technological development of metallurgy extended through the Central Desert, west and southwest of the Zagros Range (in some part of today's Iraq) to the north and northeast of Iran (Mueller Karpe 1990). This route might be identified as the copper path, which still exists in large parts of the South and Central Iranian Plateau (Emami 2005; Frame 2010). The association of the mines with archaeological settlements has always been of great interest. It is an important criterion for the identification of ancient metallurgical activities and might also be the main clue for interpretation of analytical results.

The diversity in the applied technologies used in copper-based alloy production is noticeable through the presence of lead in bronze artefacts from “Malian” (Pigott *et al.* 2003) or arsenic-bearing copper from “Teppeh Yahya”, the arsenical coppers from “Talmessi” and “Messkani”, close to “Anarak” (Pigott 1999b; Chegini 2004), and the arsenic-bearing copper artefacts from the Late Chalcolithic Meymanatabad, Central Iranian Plateau (Kashani *et al.* 2013a). Analytical studies of several cupreous artefacts from the Middle Bronze Age (3000 – 2000 BC) revealed that the main element is copper with variable amounts of Zn, Pb, As and Sn (Thornton *et al.* 2002; Thornton, Ehlers 2003). This diversity in elemental constituents reflects the dissimilarity in the metallurgical process and in the fabrication of objects, and raises the question of the source of economic ores. Recent analyses on slag remains from Arisman proved the application of arsenic in the smelting process (Kashani *et al.* 2013a; Rehren *et al.* 2012). Arisman is located close to the important site of Sialk

(3rd Millennium BC). This site cannot be considered as the only metallurgical evidence in the northeastern Iranian Desert. Based on previous literature, arsenical copper had an important role on the Iranian Plateau during the 3rd Millennium BC (De Ryck *et al.* 2005). We should take into account that arsenical copper had also been produced by adding arsenical iron (*i.e.* Speiss) to the copper matte (Marechal 1985); a recent publication about the appearance of tin bronze in Eurasia deals with this question – the existence of different copper alloys as accidental metallurgy or even more experimental metallurgy (Radivojevic *et al.* 2013). The disappearance of the arsenic-copper alloy in some parts of the Iranian Plateau at the end of the second Millennium BC suggests that this alloy was a cultural alloy and its production must have had an old tradition of preference for these objects and was not purely accidental. One could claim that the beginning of metallurgy for making a well-known prescribed arsenical alloy was accidental, but through to the end of the second Millennium BC this became a tradition in some parts of the Iranian Plateau. The comparatively advanced technologies of melting and smelting copper in Iran were established during the Chalcolithic period in Tal-e Iblis (5500–3200 BC), as crucibles and slags have been excavated there (Pigott 1999a; Frame 2012). For this period, the smelting of copper ores can be identified through the appearance of impurities in the extracted metal, such as, for example, arsenic, and this gives early evidence of the use of arsenical copper (Frame 2012). From an archaeometallurgical point of view, there are several scientific reports of a metallurgical interest for the south central Iranian desert (Hezarkhani, Keesmann 1996; Matthews, Fazeli 2004; Momenzadeh 2004; Pernicka 2004; Pigott 1999a; 2004b; Schreiner 2003). However, despite several researchers having carried out geo-archaeological surveys on the North Iranian Plateau, some of the ancient mines located in the middle of the desert still remain relatively unknown. Furthermore, their importance can be supposed due to the accessible route to some of the key

archaeological sites and to the ancient mines in the northern part of the central desert (Thornton *et al.* 2009; Roustaei 2012).

3. Material and methods

In this work, six pieces of slag have been analyzed to obtain information about their quantitative elemental composition and minerals. Considering the historical and/or artefactual significance of each artefact, it seems logical to use, as far as possible, non-destructive analytical methods (Guerra 1998; Kashani *et al.* 2013a; 2013b; Sodaei, Kashani 2013) (Figure 6).

However, slag, which is defined as a glass-like, leftover by-product, does not belong to the category of archaeological artefacts. Therefore, it was decided to prepare small samples in order to reduce the damage to the historical material. The samples were taken by scalpel from the metal cores of marginal parts of the slag. Thereafter, they were cleaned and powdered to 200 mesh in disc form. Two devices were used to characterize these slag samples. First, XRF, a well-established method in archaeometry, was applied. Elemental analyses of the selected slag samples were carried out by wavelength dispersive X-Ray fluorescence (WDXRF), model Philips PW 2404, with a detection limit of ± 1 ppm in *Tarbiat Modarres University*, Tehran, Iran. The tube's high voltage was 40 kV with a tube current of 30 mA. The samples were also studied under a polarizing microscope (Olympus BX41 Trinocular Pol) equipped with a digital Olympus 7.1 Mp C-7070 digital camera, in the metallography laboratory located in the Cultural Heritage, Handicrafts and Tourism Organization of Iran. In this case, samples were analyzed under plane polarized light (PPL) and cross polarized light (XPL). The technique has a long history of use in a range of polarizing microscopy applications, from optical crystallography and petrography to fibre analysis (Rochow, Tucker 1994). It is also applied in chemistry for distinguishing between isomorphs and polymorphs.

However, the technique is only useful for those samples with sufficient optical differences (Stoiber, Morse 1994).

4. Results and discussion

Based on the results of the XRF analysis, all the studied samples are mixtures of silica (silicon dioxide) and metal oxides: Al_2O_3 , K_2O , Cl , CaO , Ti_2O_3 , MnO , Fe_2O_3 , Cu_2O , Na_2O , MgO , ZnO , SO_3 , PbO , SrO , and BaO (Table 1). Surprisingly, no traces of arsenic was detected in the XRF results. The slag samples are quite distinct in their glassy appearance, high density and sharp edges. A high amount of silica (silicon dioxide) is noticeable in all six samples (Table 1). This high percentage (37.93–44.64%) could not be accidental or unintentional. Silica was added to the smelt in order to improve the slag's fluidity. Silica is usually found in nature as quartz. However, in many parts of the world, silica is the major constituent of sand (Iler 1979). The percentages of calcium oxide in the first five samples are relatively high. Therefore, one can claim that in the case of these five slag samples, which in their composition have a close similarity, a calcium-rich silicate had been applied. Regarding these slag samples' compositions, this silicate might be a metamorphic and igneous rock in the pyroxenes group. In this group, the chemical composition shows a high amount of SiO_2 and FeO , MgO , and MnO . However, pyroxenes show a variable composition with different proportions of elements by weight (Emami 2014). Major elements in the samples are Si, Al, Fe, Mg and Ca, and minor elements are Na, K, Mn, Ti and Zn. Trace elements are considered as indicators of orogeny and deposits formation. Through the chemical analyses of the clinopyroxene slag material (samples 1 to 5), it has been verified that with increasing content of CaO and MgO , the sum of SiO_2 and Al_2O_3 also increased. In addition, the amount of Fe_2O_3 depends largely on the sum of SiO_2 and Al_2O_3 .

To have a better view, sample 1 was also studied under a petrographic microscope. Microscopic analysis of polished

Figure 6. Slag samples found at the site.



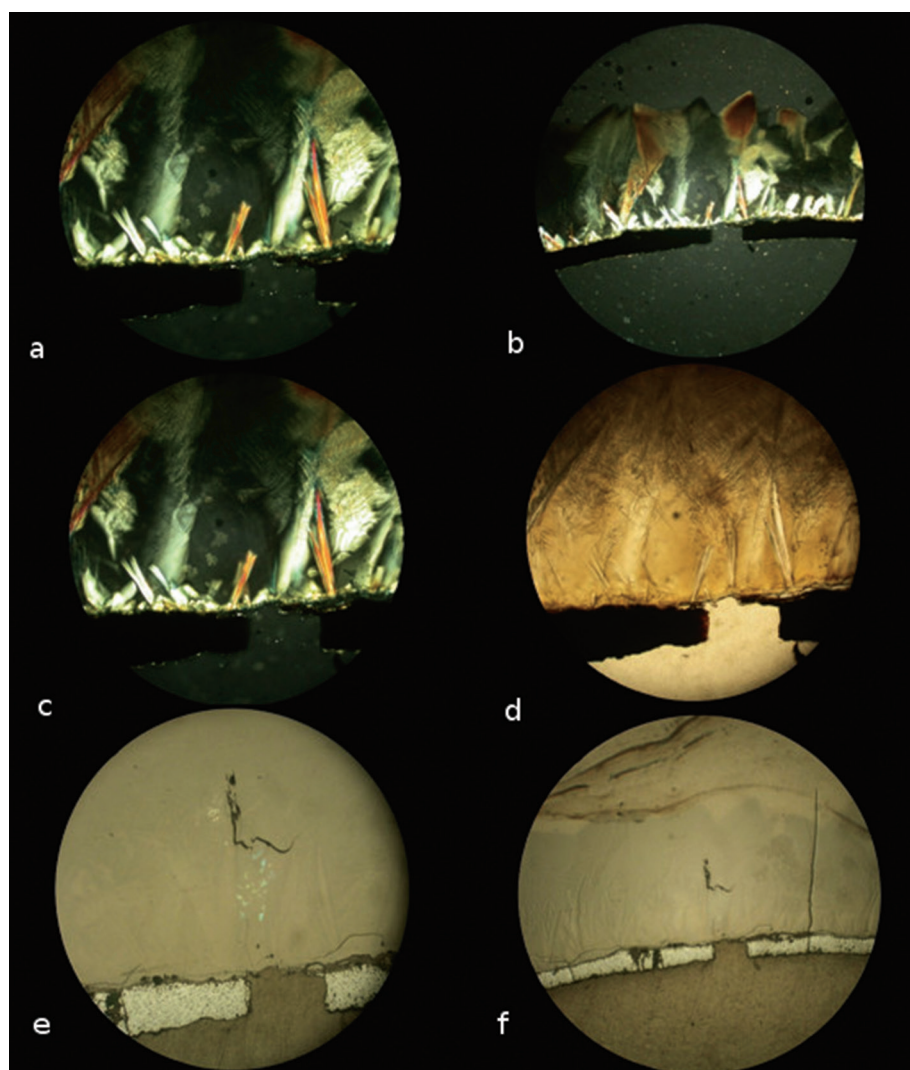


Figure 7. a) Plane polarized light (PPL) (10x), Pyroxene microstructure in sample 1. b) Plane polarized light (PPL) (10x), Pyroxene microstructure in sample 1. c) Cross polarized light (XPL) (10x), Pyroxene microstructure in sample 1. d) Plane polarized light (PPL), sample 1, 10X. e) Petrographic microscope, Sample 1, 10X. f) Petrographic microscope, Sample 1, 4X.

sections can provide information about their petrographic features, original ore, added gangue materials, fuels used, and conditions within the smelt. A thin and polished section was prepared from sample 1 and thinned down to 30 μm . Studying sample 1 under the petrographic microscope revealed a clinopyroxene microstructure in a glassy matrix (Figures 7a, 7b and 7c). The glassy texture in Figures 7a through 7c demonstrates the over-abundance of crushed, partially-reacted quartz and feldspar gangue (so-called “free silica”) in the smelt. In these Figures, the dark colours are associated with Fe-rich varieties, while titan augite is more distinctly coloured from brown/pink to violet. In Figures 7a

to 7c, a dark layer shows Fe_2O_3 , while this layer is shown in a white colour in Figures 7d to 7f. Table 1 shows a considerable amount of Fe_2O_3 in the samples (8.24–13.51%). The high percentage of Fe illustrates that the copper was mostly extracted, while Fe and small amounts of Cu, as parts of the waste product, remained in the slag. Hence, all the samples under study are copper slag samples. Depending on their composition, these copper slag samples could be molten at about 1300°C.

XRF analysis proved the presence of lead (Pb), zinc (Zn) and barium in sample 6. Based on the strong correlation between lead and barium, a lead ore source with barite

Table 1. X-Ray Fluorescence (XRF) – results.

Sample	Na_2O	MgO	Al_2O_3	SiO_2	Cl	K_2O	CaO	Ti_2O_3	Fe_2O_3	Cu_2O	ZnO	SO_3	SrO	BaO	PbO	MnO
1	1.55	2.55	7.78	44.64	0.25	2.60	26.25	0.56	9.41	3.16	0.14	0	0	0	0	1.11
2	2.61	2.32	8.11	44.35	0.47	2.64	26.53	0.76	9.11	3.05	0	0	0	0	0	0.05
3	3.76	2.45	7.59	43.04	1.95	3.91	24.47	0.82	8.24	2.95	0	0	0	0	0	0.82
4	3.91	2.51	7.71	41.08	1.92	3.92	23.58	0.89	8.9	3.95	0	0.84	0	0	0	0.79
5	4.89	2.18	7.86	41.58	1.94	2.85	24.32	0.96	8.84	3.20	0	0	0.49	0	0	0.89
6	3.87	0	0	37.93	0.58	2.78	7.99	0.59	13.51	4.62	16.46	0	0	5.15	6.35	0.17

(BaSO₄) could be suggested for this sample. In fact, barite commonly occurs in lead-zinc veins in limestones (Rubin 1997). Barite is a mineral consisting of barium sulfate. It is generally white or colourless, and is the main source of barium (Hanor 2000).

5. Conclusion

This study shows that all the samples under study are from copper slag material. In copper production, Fe was not extracted. This element together with the remaining Cu, Si, Al, Mg, and Ca are major elements in these samples of copper waste products. The minor elements found are Na, K, Mn, Ti and Zn. The considerable amount of silica, which can be seen in the XRF results and with the petrographic microscope, indicates the intentional process of adding silica to the smelt in order to increase fluidity. The XRF results, together with the photos taken under the plane polarized light (PPL) and cross polarized light (XPL) microscope, prove that the first five samples are calcium-rich silicates with a clinopyroxene microstructure in a glassy matrix. Based on the XRF results, sample 6 differs from the other five samples: regarding its composition, this sample has a probable lead-zinc ore source with barite (BaSO₄). The adding of arsenic to copper, which was common during the Chalcolithic Period and Bronze Age in this region, was not happening here in Chaltasian. This could show a paradigm shift in copper production in Iron Age II in this part of the Central Iranian Plateau. The slag material under study, as waste products of pyro-technological practices, can provide sufficient evidence for an Iron Age II copper production and extraction at this site. The application of different techniques, as discussed in this study, makes it possible to elucidate knowledge of the copper-extracting technologies that metalworkers of that time possessed. However, based on the low quantities of slag material present at Chaltasian, this production did not occur on a large scale during the Early First Millennium BC. Therefore, during this period of time, one can say that Chaltasian fell into the category of small copper production centres in the Central Plateau of Iran.

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