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ORIGINAL PAPER

Sourcing copper ores for production of bronzes excavated at Shuangyantang, a Western Zhou (1046–771 BC) site in Chongqing (Southwest China): evidence from lead isotope analysis

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Abstract Energy-dispersive X-ray fluorescence (EDXRF) and lead isotope analyses were applied to 12 Western Zhou (1046–771 BC) bronzes unearthed from the Shuangyantang site in Wushan County, Chongqing (southwest China), to investigate their chemical compositions and possible mineral source(s). The results showed that (1) the investigated bronzes were mostly bronzes with low, common lead and (2) their lead isotopic values almost all fall into a relatively narrow range, suggesting possibly the use of raw materials from a common copper mine. The comparison between lead isotopic values for Shuangyantang bronzes and those already published for copper mines and other bronzes produced and used about at the same times leads us to believe that the Shuangyantang bronzes probably used the same copper ores as used in bronzes from the Peng and Jin states in Shanxi Province. However, it would not be possible at this point to come up with a clear idea of where exactly these copper ores may come from. Candidate copper mines might be the Tonglvshan mines in Hubei Province, the Zhongtiaoshan mines in Shanxi Province, or the Dajing copper mines in Inner Mongolia.

Keywords Shuangyantang · EDXRF · Lead isotope · Bronze · Western Zhou

Introduction

Four lead isotopes of lead exist in nature, namely ^{204}Pb, ^{206}Pb, ^{207}Pb, and ^{208}Pb, and the ratios of them are different in metallic artifacts and natural ores. Three of them are radiogenic: ^{206}Pb, ^{207}Pb, and ^{208}Pb, which are respectively formed by the radioactive decay of ^{238}U, ^{235}U, and ^{232}Th. The isotope ^{204}Pb is stable and not radiogenic. The isotopic composition of a given reservoir today, such as the upper crust or the mantle, continuously evolves in time depending on its age and its concentration of uranium and thorium. Hence, various ore deposits may have their own specific Pb isotope signature depending on their geological origin (Gulson 1986; Gale 1989; Gulson et al. 2000; Stos Gale et al. 1997; Albarede et al. 2012; Doe 1975; Bingquan Zhu 1998; Li 2000; Binquan Zhu et al. 2001). We can therefore attempt to trace the source of metallic ores through comparison of the lead isotopic compositions of the ancient bronzes and the ores from the mines (Barnes et al. 1978; Ault et al. 1970; Begemann et al. 1989; Willem et al. 1999; Sandrine et al. 2009; Nriagu 1998).

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Application of lead isotopic to archaeology and related research in China was developed not as early as in the west. Yet, some important research had been done since Zhengyao Jin firstly carried out lead isotope study in 1980s. Among these, one of the most noticeable achievements was the discovery of the characterized high radiogenic lead (HRL, for short) of southwest China in the bronzes of Shang dynasty (~1600 BC–1046 BC) unearthed in Henan Province; considering that HRL also existed in the bronzes unearthed in Sanxingdui site and Jinsha site in southwest China, it was inferred that the lead ore of the bronzes of the Shang dynasty in the Central Plain was possibly from southwest China (Zhengyao 2008).

According to archaeological materials, potteries with the Central Plain culture characteristics of Xia-Shang period (~2100 BC–1046 BC) have been found in sites of Ba culture in east Sichuan and west Hubei; historical records, archaeological stratigraphy, and typology, together with the identification of copper ore sources of bronze wares, have provided evidence for the communication between Ba culture and the Central Plain culture in the Xia-Shang period (Qu and Naiqiang 1987; Dianzeng 2004). Historical records show that Ba State in southwest China had strong national power during the early Zhou Dynasty (~1046 BC–900 BC) and kept in contact with the Central Plain area (Zhimin 1998). However, the evidence of such cultural communication in the Western Zhou Dynasty (~1046 BC–771 BC) is absent in literature or archaeological materials. Therefore, it is important to study the copper ore source of the bronzes in the southwest region to attain more information about cultural exchange between Ba State and the Central Plain during the Western Zhou period.

Shuangyantang site, a typical example of Ba culture, is located in Longxing village, Dachang town, Wushan County, Chongqing, China, 109° 46′ 40″ E, 31° 17′ 20″ N (Fig. 1). Archaeological survey and excavation had been done between 1957 and 2006. The survey and excavation showed that the area of the site is about 100,000 m^2, and the existing area is about 20,000 m^2. It is the largest and, meanwhile, well-preserved site of Ba culture during Western Zhou (1046 BC~771 BC) in the Three Gorges region of the Yangtze River. Potteries, bronzes, kiln sites, burning of soil, copper slags, copper ores, jades, stonewares, horn implements, and oracle bones were excavated. The typical potteries unearthed are Fu with decorative rim, pot with decorative rim, basin with decorative rim, cup with sharp bottom, and small cup (Zhan) with sharp bottom. Both the shape and decoration of these popular potteries are similar to those unearthed in Sichuan basin and western Hubei (region of Chu State) during Western Zhou period. Earth-ware Li and Dou which were prevalent in central region and Chu State unearthed in Shuangyantang site also have some features of Ba culture. Two groups of potteries unearthed in ashpit are shown in Fig. 2. Besides, hundreds of bronze relics such as arrowheads, arrows, bronze ware fragments, tapers, hairpins, hooks and accessories, and copper ores and copper slag were excavated from the same cultural layer. These bronze artifacts are small (Cultural Relics Heritage Bureau of Chongqing, Resettlement Bureau of Chongqing 2001; 2003).

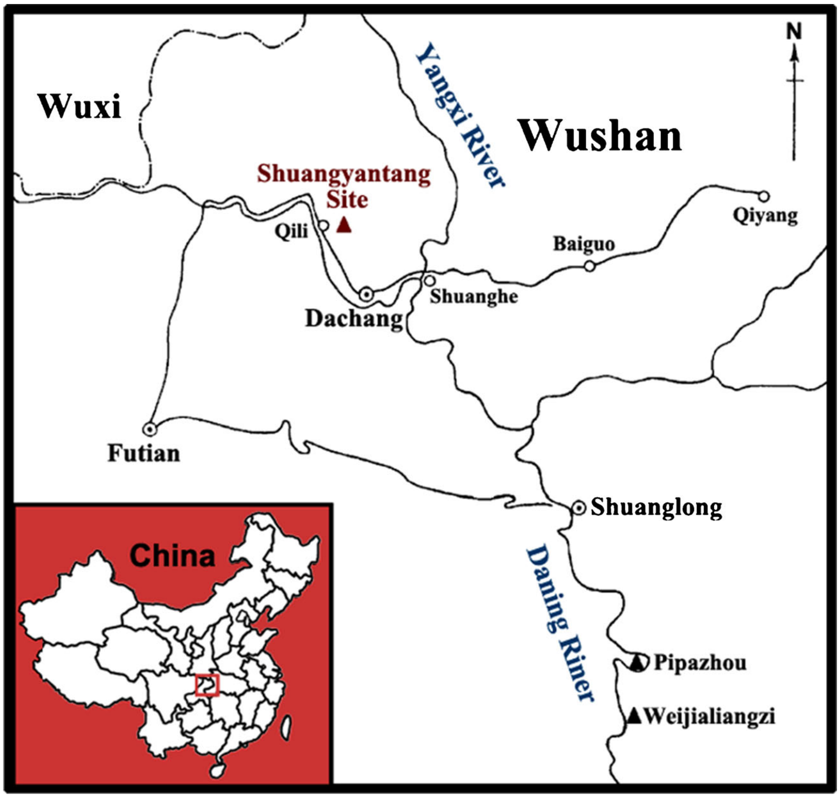

Fig. 1 A sketch map of Shuangyantang site

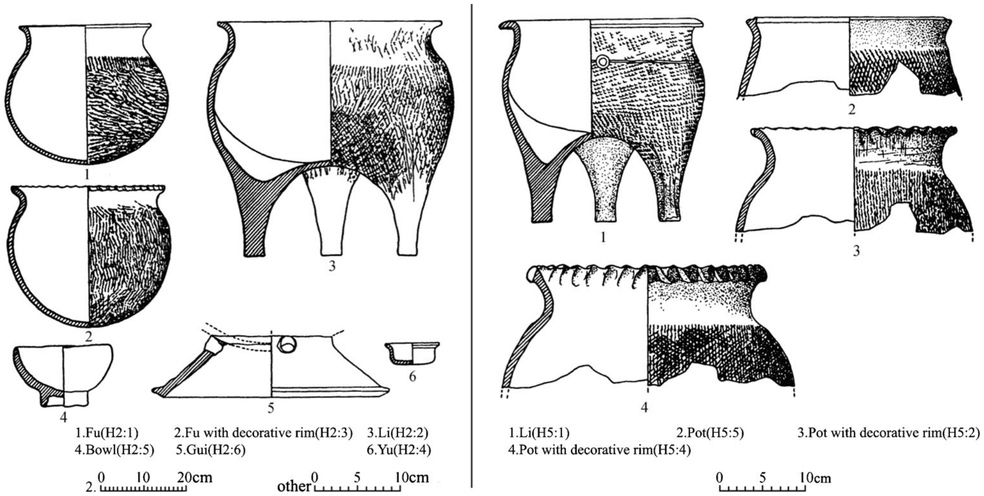

Fig. 2 Two groups of potteries unearthed in ashpits H2 and H5 at Shuangyantang site

Wushan County is located in the geographical area where cultural communication between Ba State and Chu State may occur. So, Shuangyantang site is important for the study of relationship between Ba State and Chu State. The bronze relics, copper ores, and copper slag unearthed at Shuangyantang site were representative in the archaeological materials of Ba State, so we can explore the source of copper ores of Ba State during the Western Zhou period and therefore provide technological support for the study of cultural communication between Ba State and Chu or Central Plain by analyzing the bronzes, slags, and ores unearthed at Shuangyantang site as much as possible.

Materials and methods

Materials

One casting slag sample, three bronze arrowheads, seven bronze fragments, and one copper ore (Fig. 3) were analyzed. Basic information of these samples is listed in Table 1. Those 12 western Zhou samples were unearthed, respectively, in different grid of T340, T342, T351, T356, T365, T385, T425, T437, T442, and T472 at Shuangyantang site. The grid distribution map is shown in Fig. 4. All of these samples were collected from the same cultural layer coexisting with the same potteries as Fig. 2, which belongs to western Zhou period. (See Table 2.)

Energy-dispersive X-ray fluorescence

Chemical component compositional analysis was carried out on an Eagle III energy-dispersive X-ray fluorescence analyzer by American company EDAX. The X-ray spectrometer is equipped with an Rh target and operated in the beryllium window. Test condition was as follows: beam spot size 300 μm, working voltage 40 kV, working current 150 μa, and vacuum light path. The collected data were analyzed through Vision 32 software system.

Inductively coupled plasma mass spectrometry

Lead isotope analysis was carried out at School of Archaeology and Museology, Peking University in Beijing. Firstly, tiny bronze pieces of 2 mg were chipped off and then dissolved in 3 ml of HCl and 1 ml of HNO_3. Later, the clear solution was leached and diluted with deionized water into 10 ml. The solutions were then measured to detect the lead contents by ICP-AES (PHD, Leeman Labs Inc., CA, USA). According to the results which represented the lead contents, the solutions were diluted to 1000 ppb. The thallium (T1) standard SRM997 was added to the solutions. Lead isotope analyses

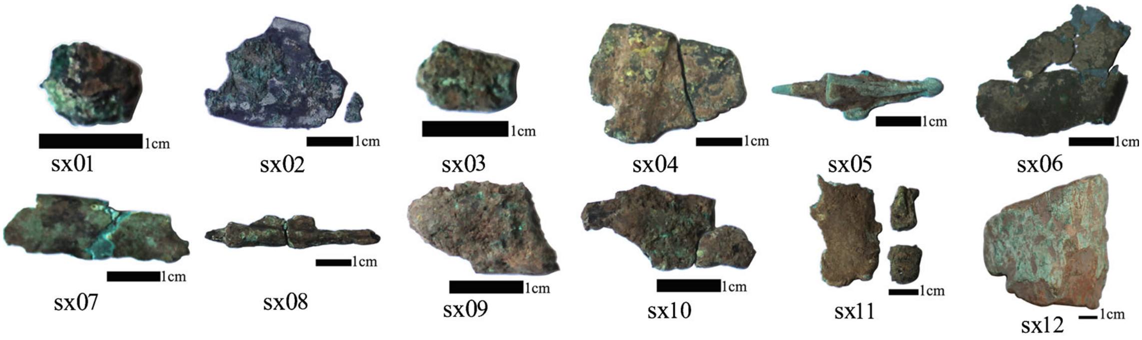

Fig. 3 Photographs of bronze from the Shuangyantang site

Table 1 The information of bronze samples unearthed at Shuangyantang site

Sample number	Archaeological number	Name	Sample number	Archaeological number	Name
SX01	T351-④:11	Casting slag	SX07	T340-④:7	Bronze fragment
SX02	T365-④:3	Bronze fragment	SX08	T385-⑤B:1	Arrowhead
SX03	T342-④:17	Bronze fragment	SX09	T472-⑤C:7	Arrowhead
SX04	T351-④:8	Bronze fragment	SX10	T425-⑤C:15	Bronze fragment
SX05	T356-④:1	Arrowhead	SX11	T437-⑤B:38	Bronze fragment
SX06	T340-④:10	Bronze fragment	SX12	T442-⑤C:7	Copper ore

were performed by a MC-ICP-MS (VG AXIOM, Thermo-Elemental Inc., Winford, England). The spectrometer was a double-focusing magnetic sector instrument equipped with an array of ten variable Faraday collectors. And, it had a further fixed Faraday and an electron multiplier detector. Based on repeated analyses of SRM981, the overall analytical 2σ error for all lead isotope ratios was less than 0.004 % (Table 3). Pb isotopic compositions of ore, slag, and the bronzes unearthed at Shuangyantang site are listed in Table 3.

Identification of the copper ore sources

Characteristics of Pb isotope of the bronzes unearthed at Shuangyantang site

The energy-dispersive X-ray fluorescence (EDXRF) result showed ten samples with lead under 4 %, approximately 83 % of the total, and nine samples with lead under 2 %, approximately 75 % of the total, which suggested that most of the bronzes and copper slag unearthed at Shuangyantang site were the low-lead bronze.

From the previous research, we know that the obvious differences between the smelting slag and the casting slag are as the follows: Smelting slags are essentially ferrous silicates, high in iron and low in nonferrous metals. So, the content of iron was very high and the content of copper was very low in smelting slags. On the other hand, crucible slags tend to be high in nonferrous metal and wood ash, but low in iron. So, the content of iron was very low and the content of nonferrous metal was higher in the casting slags (R. F. Tylecote 1992; Luo 2007). Base on these, we can confirm that the SX1 is a casting slag sample.

The result showed that ^{206}Pb/^{204}Pb ratios ranged from 17.752 to 18.632 and ^{207}Pb/^{204}Pb ratios were between 15.255 and 15.733, ^{208}Pb/^{204}Pb between 37.559 and 39.018, ^{207}Pb/^{206}Pb between 0.8443 and 0.8768, and ^{208}Pb/^{206}Pb between 2.0939 and 2.1386, which indicated that the lead in

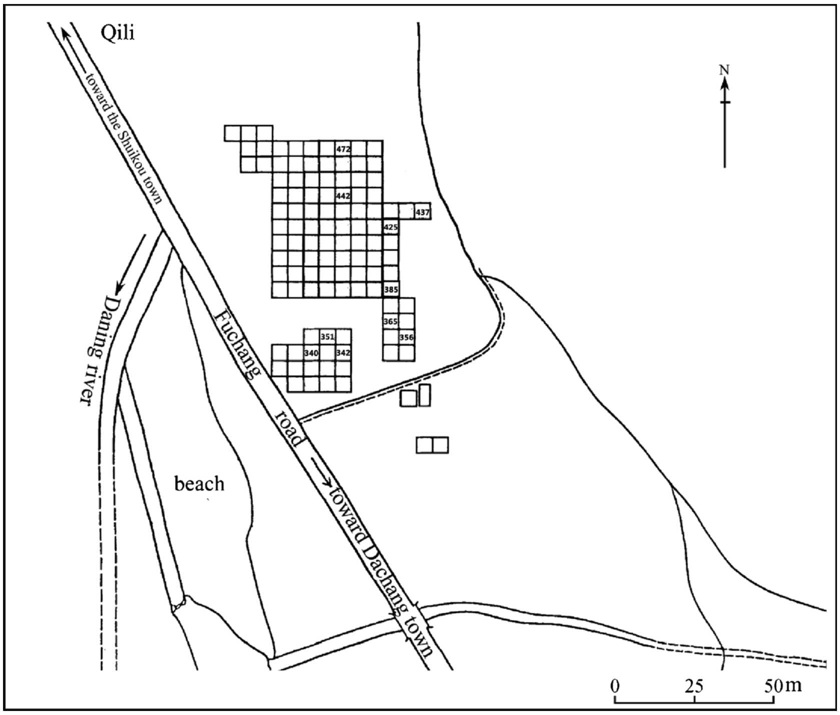

Fig. 4 The grid distribution map of samples from Shuangyantang site

Table 2 The results of two runs for the SRM981 determination and the average analytical error

Number	^{206}Pb/^{204}Pb	^{207}Pb/^{204}Pb	^{208}Pb/^{204}Pb	^{207}Pb/^{206}Pb	^{208}Pb/^{206}Pb
1	16.934	15.486	36.682	0.915	2.166
2	16.933	15.486	36.672	0.915	2.166
Analytical error (%)	0.002	0.002	0.004	0.004	0.004

these bronzes was common lead. The Pb isotope results were plotted in ^{208}Pb/^{206}Pb versus ^{207}Pb/^{206}Pb diagrams (Fig. 5), which were relatively concentrated.

Previous research pointed out that it was difficult to determine whether the lead isotope of the low-lead bronzes with 2~3 % lead was indication of copper or lead ore. Low-content lead could be intentionally added or from lead pollution in the process of casting or waste recycled (Nickel et al. 2012; Young and Casadio 2010; Ying et al. 2004). However, nine samples out of 12 from Shuangyantang site were low-lead bronzes, and lead isotope values of these samples were relatively concentrated and the probability that all the samples were polluted to the same degree was small, and Professor Gale NH et al. once stated that “It seems safe to say that copper alloy artifacts containing lead in the range from 50 ppm to 4 % are suitable for the provenancing of their copper by lead isotope analysis,” so those lead isotope ratios should be indication of copper ore source.

The lead isotope values of the bronzes unearthed at Shuangyantang site showed that the lead in the copper ore, slag, and bronze wares was common lead. Lines of research on the lead isotope of the bronzes during Shang-Zhou period showed that bronzes containing HRL were firstly found in Yanshi city, Zhengzhou city of the Shang Dynasty, and the amount of it increased to half of all bronze wares in Erligang period (~1600 BC–1300 BC) and 70 % in phases I and II of Yin Ruins, then decreased significantly in phase III of Yin Ruins, and disappeared in phase IV of Yin Ruins (~1300 BC–1100 BC) (Zhengyao 2008). The fact that the lead in the bronzes from Shuangyantang site was common lead confirmed that the lead in the bronzes of the Zhou Dynasty was common lead instead of HRL.

The production processes of the bronze are mining, smelting, and casting. Copper ore, casting slag, bronze fragments, and bronze arrowheads unearthed at Shuangyantang site, respectively, reflected the phase of mining, casting, and alloying. It can be found by comparing the lead isotope ratios of different types bronzes that ^{207}Pb/^{206}Pb ratios of these bronze samples were 0.8443~0.8768 with a difference of 0.0325 and ^{208}Pb/^{206}Pb ratios of these bronze samples were 2.0939~2.1386 with a difference of 0.0447 (Fig. 6). It could be observed in Fig. 6 that the lead isotope ratios of the bronzes from Shuangyantang site were very close. Geochemical case studies carried out in many parts of the world have provided us convincing evidence that by looking at bronzes’ geochemical compositions (lead isotopic distribution, for instance), we can often get a clue of where their raw materials (copper ores) might come from; following the same way of thinking, slags which are quite often discovered at archaeological sites and very likely survived from the casting activities will contain the same kind of information, which hopefully help us recognize the functional use of sites. As slags are obviously not something that were worthwhile for long-distance trade or exchange in ancient times considering the huge amount of cost

Table 3 Cu, Sn, and Pb content and Pb isotopic composition

Number	Type	XRF (wt%)			Pb isotopes				
		Cu	Sn	Pb	^{206}Pb/^{204}Pb	^{207}Pb/^{204}Pb	^{208}Pb/^{204}Pb	^{207}Pb/^{206}Pb	^{208}Pb/^{206}Pb
SX01	Casting slag	9.29	0.88	0.24	17.752	15.255	37.559	0.8593	2.1157
SX02	Bronze fragment	73.27	24.84	1.80	17.997	15.433	38.039	0.8575	2.1136
SX03	Bronze fragment	91.35	6.87	1.75	18.270	15.612	38.522	0.8546	2.1085
SX04	Bronze fragment	88.56	10.31	1.10	18.626	15.726	39.001	0.8443	2.0939
SX05	Arrowhead	65.14	28.35	6.4	18.275	15.625	38.805	0.8550	2.1234
SX06	Bronze fragment	72.60	25.63	1.70	18.002	15.547	38.171	0.8637	2.1204
SX07	Bronze fragment	75.76	22.78	1.42	17.949	15.570	38.260	0.8675	2.1316
SX08	Arrowhead	70.49	29.01	0.45	17.823	15.626	38.115	0.8768	2.1386
SX09	Arrowhead	98.53	0.68	0.75	17.982	15.635	38.076	0.8695	2.1175
SX10	Bronze fragment	74.99	20.99	3.82	18.136	15.610	38.494	0.8607	2.1226
SX11	Bronze fragment	63.03	21.53	15.25	18.632	15.733	39.018	0.8444	2.0942
SX12	Copper ore	83.75	0.04	0.20	18.313	15.727	38.715	0.8588	2.1141

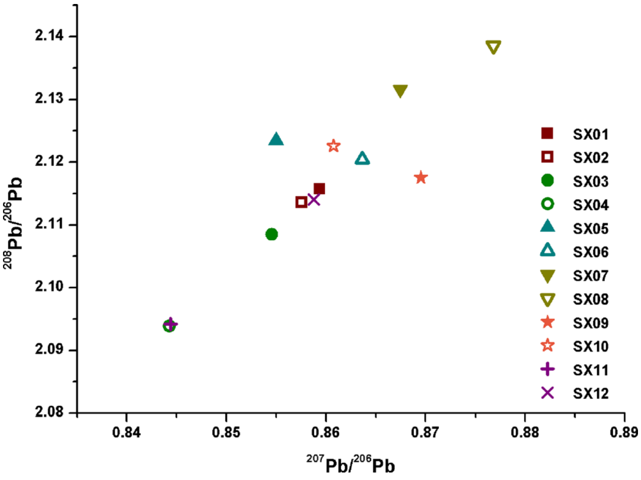

Fig. 5 Pb isotope values of samples from Shuangyantang site

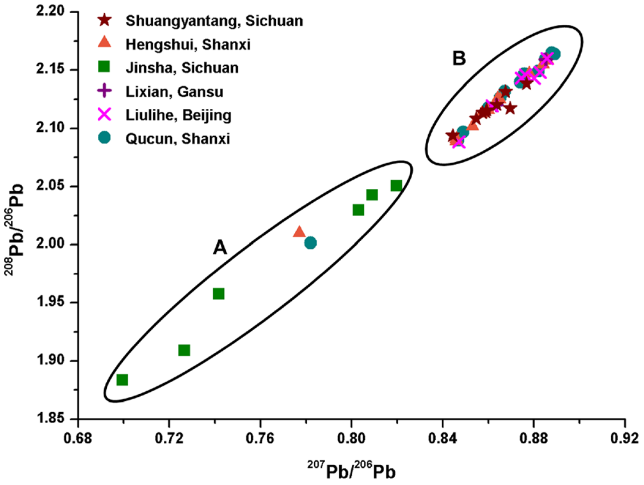

Fig. 7 Pb isotope values of low-lead bronzes from Shuangyantang and other sites in China during Zhou period

and labor, we can be quite confident to say that bronze casting activities were very likely to be carried out at the Shuangyantang site.

Comparison of lead isotope values of the low-lead bronzes from Shuangyantang site with those from other sites during the Western Zhou period

Although there was precedent that the lead content of the low-lead bronze could be 4 % (Gale and Stos-Gale 2000; Tiemei 2008), we would like to choose the bronzes with lead less than 2 % to trace copper ore source in order to ensure that the data of lead isotope ratios could be indications of copper ore. Table 3 shows that there were nine samples with lead less than 2 %.

The lead isotope values of the low-lead bronzes from Shuangyantang site and other western Zhou sites in China (Zhengyao 2008; Puheng et al. 2012; Lajiang 2010; Jianfeng and Xiaohong 2008) were summarized in ^{208}Pb/^{206}Pb versus ^{207}Pb/^{206}Pb diagrams (Fig. 7). The ^{207}Pb/^{206}Pb value reflected the model age of the ore deposit, and the ^{208}Pb/^{206}Pb value and the U/Th ratio of the fluid medium were responsible for the ore formation. It can be observed that there was a linear relationship between ^{208}Pb/^{206}Pb and ^{207}Pb/^{206}Pb of low-lead bronzes from Western Zhou sites. The values can be obviously divided into two areas: with anomalous lead (HRL) (A) and with normal lead (B). The lead isotope values of the bronzes from Jinsha site were concentrated mainly in area A, and those from other sites were concentrated mainly in area B. This result illustrated that the ore used in the Central Plain which mainly contained common lead was different from that in southwest area during the Western Zhou period.

Area B in Fig. 7 was enlarged in Fig. 8. It could be observed that there was some overlap between the lead isotope ratios of the bronzes from Shuangyantang site and those from tombs in Peng State (Hengshui, Shanxi) and Jin State (Qucun, Shanxi). This indicated that copper ores used in these sites were possibly identical and there may be ore exchange among these sites.

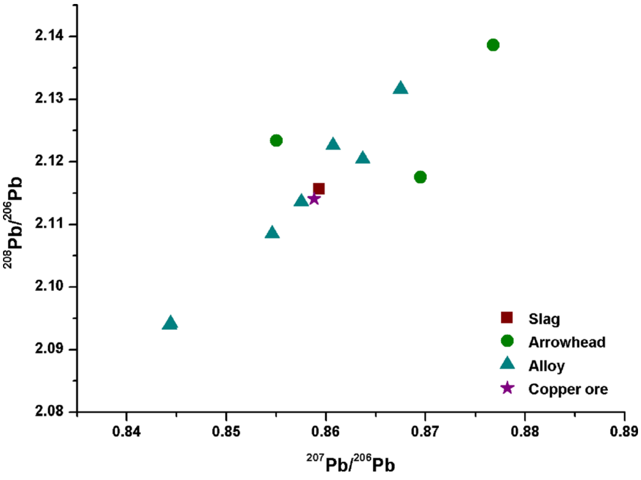

Fig. 6 Pb isotope values for samples of different types

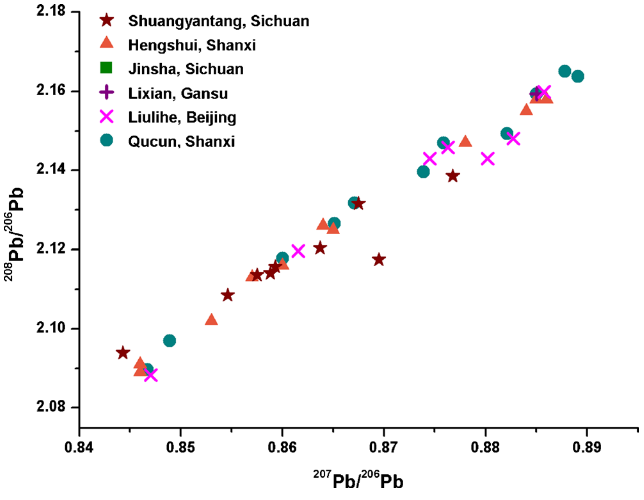

Fig. 8 Enlarged view of range B in Fig. 4

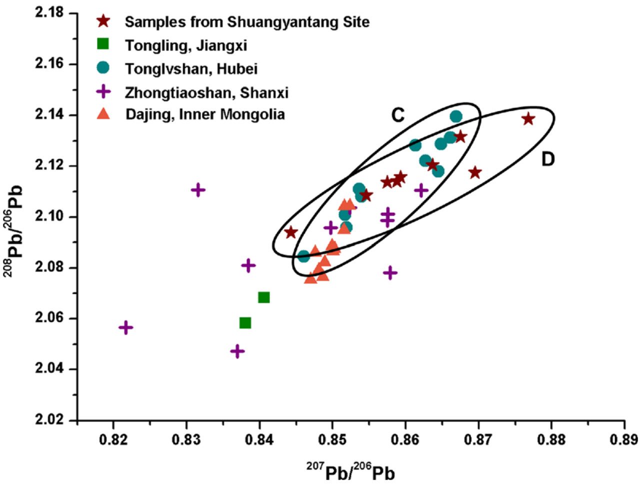

Fig. 9 Lead isotope values of the samples from Shuangyantang site plotted against those from other relevant ore deposits

Comparison of lead isotope values of the bronzes from Shuangyantang site with copper ores

It was recorded in the *Classic of Mountains and Seas* (*Shan Hai Jing*) that there were 14 copper mountains in Shaanxi, Shanxi, and Henan Province and there were 21 regions where copper mine was exploited, mainly distributed in northern Shaanxi, Weibei, northern Shanxi, Yinshan, Yanshan, Dabie mountain, Yiluo, Mufu, and Min mountain. Hitherto, the ancient copper ore sites which have been excavated were Zhongtiao Mountain in Shanxi, Tonglvshan in Hubei, Tongling in Jiangxi, and Dajing in Inner Mongolia (Jianzhong et al. 1994; Jueming and Benshan 1996; Guosheng and Xiaoxia 1997; Guofeng 2007.).

The lead isotope values of the bronzes, copper ore, and slag from Shuangyantang site and copper ores from Shanxi, Hubei, Jiangxi, and Dajing were summarized in ^{208}Pb/^{206}Pb versus ^{207}Pb/^{206}Pb diagrams (Jianzhong et al. 1994; Zhenju et al. 1998; Zicheng et al. 1999; Wen et al. 2004; Huaping 2004; Wenxin et al. 2005; Ben Li 2010; Guotao et al. 2010; Yanbo et al. 2011; Yanbo 2012) (Fig. 9). Area D where lead isotope values of the bronzes from Shuangyantang site concentrated overlapped highly area C where lead isotope values of copper ore from Tonglvshan, Zhongtiaoshan, and Dajing concentrated. This result indicated that the copper ore used in Shuangyantang site may be from these three copper mines. As well known, Dajing copper mine is about 1500 km away from Shuangyantang site, while the other two copper mines are about 450 km away from the site. And, the archaeological excavation indicated that the Tonglvshan copper mine was strip mined by ancient Chinese from Shang dynasty (~1600–1046 BC), while the Zhongtiaoshan copper mine was mined from Warring States (~475–221 BC). Besides, the shape and decoration of the potteries unearthed in Shuangyantang site are similar to those unearthed in western Hubei during Western Zhou period. So, considering on the distance, geography, and cultural factors, the copper ore used in Shuangyantang site may be probably from the Tonglvshan copper mine. Of course, more work, such as trace element analyses, needs to be done in the future in order to confirm the exactly copper ore provenance.

Conclusions

Copper ores, slags, three bronze arrowheads, and seven bronze fragments were investigated by EDXRF and lead isotope analyses in the present research to understand their chemical compositions. By looking at the geochemical patterns and exploring their implications for sourcing copper mines that could have been used for producing the investigated bronzes excavated at the Shuangyantang site, we came to the following conclusions:

1. The investigated bronzes, mostly with low, common lead and narrow-range distributed lead isotopic values, were probably produced by copper ores from a common copper mine. At the time of writing our paper, we cannot address the issue of where exactly were these copper mines located; however, we do suggest that three broader areas could be potential candidates: (1) the Tonglvshan mines in Hubei Province, (2) the Zhongtiaoshan mines in Shanxi Province, or (3) the Dajing copper mines in Inner Mongolia.
2. Both chemically and isotopically, the Western Zhou bronze samples uncovered from the Shuangyantang site are similar to those bronzes produced and used by the Peng and Jin states in the same Western Zhou period which located in Shanxi Province (central China). Whether they really shared a common source of copper mines or whether it is purely a coincidence due to the similar bronze casting technology remained to be explored with more archaeological and geological samples.

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