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Copper metallurgy in prehistoric upper Ili Valley, Xinjiang, China

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ABSTRACT

The upper Ili Valley in northwest Xinjiang is a crucial place for the study of early interactions between the Eurasian Steppe and northern China. This paper presents scientific analytical results and examines the use and production of copper alloys in the region with regard to the transregional exchange of materials and technology. The substantial proportion of unalloyed copper and clear drop in tin concentrations in the bronze samples from the Iron Age indicates the decrease of tin usage compared with the Bronze Age. The shift between the Bronze and Iron Ages can also be seen from the lead isotope results. The changes of the material (alloys) and metal source(s) between the Bronze and Iron Ages in the upper Ili Valley imply movements of objects, raw materials and related technical practises.

KEYWORDS

Early metallurgy; Copper/bronze; Lead isotope; Technological diffusion; Upper Ili Valley

ELECTRONIC SUPPLEMENTARY MATERIAL

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1. Introduction

The early use and production of metal artefacts have been widely quoted as one of key pieces of evidence indicating China's involvement in a prehistoric transcontinental interaction network across Eurasia (e.g., Fitzgerald-Huber 1995; Sherratt 2006; Jaang 2015). In the vast territory spanning from the Urals to the Yellow River, Xinjiang is of particular interest given its intermediate position in eastern Eurasia. The mountainous routes of the Tianshan range were bridges for east-west interaction millennia before the Silk Roads were historically documented (e.g., Mei 2000; Linduff 2004; Millward 2007; Frachetti et al. 2017). The Ili River Valley in northwest Xinjiang was one such major artery for early interactions.

The Ili River originates in the middle of the Tianshan mountain range and flows westward into the Lake Balkhash. Its mainstream and principle tributaries, namely the Kax, Künes and Tekes rivers irrigate the rugged and mountainous upper Ili Valley (UIV) region, which forms the major part of present-day Ili Kazakh Autonomous Prefecture in northwest Xinjiang, China (Fig. 1). The contiguous mountain systems of the Tianshan range separate the UIV region from the Dzungarian and Tarim basins in the north and south respectively, and also obstruct dry/hot currents of air from the deserts and dry/cold ones from Siberia. This topographic setting forms a humid and temperate climate in the UIV that has nurtured the development of human societies for thousands of years (Xu et al. 2011; Wang et al. 2015).

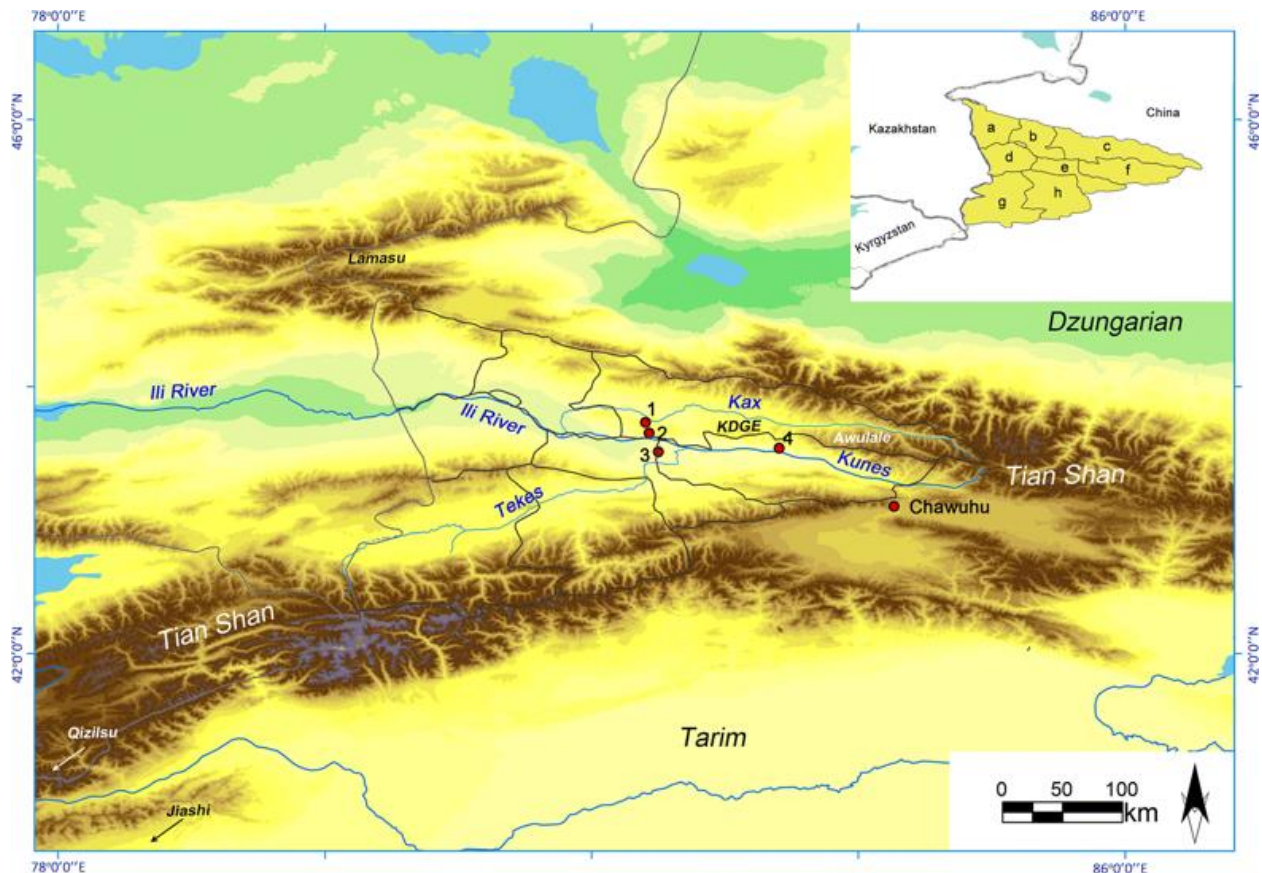


Fig. 1

Map of the upper Ili Valley region showing the position of counties and sites mentioned in the text. Counties: **a** Huocheng. **b** Yining. **c** Nilake. **d** Chaqal. **e** Gongliu. **f** Xinyuan. **g** Zhaosu. **h** Tekes. Sites: **1** Nulasai; **2** Yuantoushan; **3** Aga'ersen; **4** Yutang.

Since the 1970s, copper and bronze artefacts dated to the prehistoric period have been reported from the UIV region, including the Aga'ersen hoard in Gongliu, the Yutang (鱼塘, fishpond) cemetery site of No. 1 company, 71 regiment in Xinyuan and many stray finds (e.g., Wang 1987; Wang and Cheng 1989; Li and Dang 1995). The significance of these finds was immediately realised through comparison with similar artefacts found in Central Asia. The shaft-hole axes, sickles and related items from the Aga'ersen group were associated with the Andronovo complex in Kyrgyzstan and Semirechye in eastern Kazakhstan, and dated to the later part of the second millennium BCE (Wang 1985a, b; Peng 1998); while the cauldrons, square plates and altars were ascribed to the Saka or other pastoralist groups in neighbouring regions (Wang 1985a, b; Chen and Hiebert 1995; Gong 1997). Furthermore, the discovery of mining and smelting remains of the first millennium BCE at Nulasai and Yuantoushan in Nilake county and relevant research work have provided solid evidence for local metallurgical production in late prehistoric Xinjiang (Wang 1985a, b; Wang 1986; Mei and Li 1998).

Recently, further archaeological excavations and research into prehistoric Xinjiang have resulted in remarkable progress toward the establishing of developmental sequence of prehistory cultures (see the reviews by Song 2009; Liu and Li 2012; Marchella et al. 2017). Several monographs have systematically examined the available archaeological evidence within a comprehensive spatial-temporal framework (Han 2007; Shao 2007; Guo 2012). In the UIV region, recent excavations have revealed a number of burial and settlement sites and provided provenanced comparand for many chance finds of metal objects previously known in the region (e.g., Liu and Guan 2002; Ruan 2011; Ruan 2012). Despite the long-running controversy over the periodization of Xinjiang's Bronze Age and Iron Age (Wang 1985a, b; Chen 1987; Lu 1995), it is now generally accepted that the archaeological cultures in the prehistoric UIV region can be broadly divided into two separate phases, namely the Bronze Age and the Iron Age, with the transition taking place in the early first millennium BCE (Mei 2000; Chen 2000; Han 2007). It has also been realised that since the Bronze Age, the valley had been closely connected with the Inner Asia highlands and the steppes further north and west (Frachetti 2012; Mei and Colin 1999).

Since the 1990s, several hundred early copper and bronze objects found in Xinjiang have been investigated and scientifically examined, demonstrating both their typological and technological similarities to those from neighbouring areas, such as the Gansu-Qinghai regions in the east and the steppes in the north and west (e.g., Chen and Hiebert 1995; Mei et al. 1998; Mei and Colin 1999; Mei 2000; Qian 2006; Ling 2008; Mei et al. 2013). It has been argued that Xinjiang may have played an intermediate role in introducing metallurgy from the steppes into northwest China, and further east, eventually reaching the agricultural societies of the middle-lower reaches of the Yellow River (Mei 2000; Li 2005; Hwang 2014). Taking into consideration, Xinjiang's vast territory and diverse archaeological cultures, however, further archaeometallurgical, especially lead isotopic, research is still needed to enhance our understanding of the early use and production of metals in Xinjiang and their relationship with neighbouring regions. This paper, based on the scientific analysis of chemical composition,

metallography and lead isotopes, examines the use and production of copper alloys in the prehistoric UIV region and discusses related issues, such as the transregional exchange of materials and technology.

2. Materials and methods

The copper/bronze artefacts in this research come from the collection of the Ili Kazakh Autonomous Prefecture Museum. The whole assemblage of samples is composed of 33 copper/bronze objects covering different typological categories, such as vessels, implements, weaponry and altars/lamps. Two copper ingots were also sampled for this research. It should be pointed out that most of the objects sampled here are chance findings from the UIV regions, especially the counties of Gongliu (Toqquz Tara), Xinyuan (Künes) and Tekes (see Fig. 1); therefore, our understanding of their archaeological background is rather limited. In the light of recent archaeological discoveries and comprehensive typological studies on early metal objects in Xinjiang and neighbouring areas (Ruan 2011, 2013), we are, however, able to differentiate these objects into two different complexes. The shaft-hole and socketed axes, sickles, gouge and single-blade knives (Fig. 2: 1–4) are analogous to the ones from the Aga'ersen hoard. Therefore, they can be ascribed to the Bronze Age Andronovo horizon, which is dated, in the region of Semirechye, from the middle second to early first millennium BCE (Shao 2007; Yang et al. 2016). The large cauldrons, altars/lamps, ingots and statues (Fig. 2: 5–9) can be assigned to the Iron Age Suodunbulake or Ili Valley archaeological cultures of pastoralist societies such as the Saka people in Inner Asia (Grigor'ev and Ismagil 1997; Guo 1999, 2007, 2012). Supplementary Table S1 provides the collection records, origin and the periodization/dating for all the objects studied.



Fig. 2

Examples of prehistoric copper/bronze objects discovered in the Ili region. **1** Shaft-hole axe (XY0179); **2** Socketed-axe (XY0489); **3** Sickle (XY0019); **4** Knife (XY0493); **5** Altar/lamp (XY0196); **6** Human statue (XY0173); **7** Ingot (XY0024); **8** Tripod cauldron (XY0477); **9** Circle-footed cauldron (XY0059); (1–4 Bronze Age, 5–9 Iron Age; scales differ, see Supplementary Table S1 for sizes of objects)

In this research, small metal pieces of 24 objects were sawn off from casting seams or broken points for chemical, metallographic and lead isotopic analysis. Patina samples were taken from the surface of a further 9 intact objects for lead isotopic analysis. The metal pieces were mounted in epoxy resin, then ground with sand paper and polished with diamond paste down to 0.5 mm. Metallographic and chemical analyses of the metal samples were conducted with a Leica DM4000M optical microscope and a Zeiss EVO18 scanning electron microscope equipped with a Bruker X-Flash 5010 dispersive spectrum analyser (SEM-EDS) at the University of Science and Technology Beijing (USTB). The analytical conditions of SEM-EDS were set at an acceleration voltage of 20 kV, a working distance of ~ 15 mm and an acquisition time of 60 s.

Lead isotopes were analysed using a VG Elemental multicollector inductively coupled plasma mass spectrometer (MC-ICP-MS) at Peking University's School of Earth and Space Sciences. Standard material SRM981 was repeated during the analysis, indicating that the overall analytical 2σ error for all lead isotope ratios was less than 0.04%. Detailed description of methodology and sample preparation procedure for lead isotopic analysis can be found in Cui et al. (2010). Though there is concern that possible contamination from the burial environment may alter the lead isotope composition of the patina in metals with a low-lead content (Gale and Stos-Gale 2000), the results of corrosion from 9 intact objects are retained here as additional data for further comparison and discussion.

3. Results

Chemical and lead isotopic ratios of the analysed samples are listed in Table 1. Although 1 wt% has been proposed as a potentially more meaningful cutoff value for the classification of archaeological copper alloys (Eaton and Mckerrell 1976; Northover 1989; Bray et al. 2015; Pollard et al. 2015), this paper follows the conventional threshold of 2 wt%, which has been widely adopted for Chinese materials to avoid confusion in comparing our results with published data (Wen 1980). According to this criterion, the 24 objects sampled with metal pieces can be divided into two groups, namely unalloyed copper (Cu, 18) and tin bronze (Cu-Sn, 6). This distribution of material types (having both Cu and Cu-Sn) is consistent with previous studies (Ling et al. 2008). It is worth mentioning that the relatively low levels of lead and arsenic in UIV's objects set them apart from both central China and the Gansu-Qinghai area in terms of typical metal composition. Sulphur, iron and antimony are present in some samples as minor/impurity elements, with the former two being likely correlated with each other.

Table 1

Chemical and lead isotopic compositions of copper/bronze objects from the upper Ili Valley regions.

No.	Objects	Period	Origin	Chemical composition (SEM-EDS, wt%)										Lead isotope ratios		
				Cu	Sn	As	Pb	Sb	Cl	O	S	Fe	Ag	²⁰⁶ Pb/ ²⁰⁴ Pb	²⁰⁷ Pb/ ²⁰⁴ Pb	²⁰⁸ Pb/ ²⁰⁴ Pb
D0013	Knife	Bronze Age	Tangjiale memorial in Xiinyuan	83.6	15.4	-	-	-	0.8	0.1	0.1	-	-	18.126	15.562	37.975
XY0018	Socketed axe	Bronze Age	Gongliu County					/						18.071	15.573	38.038
XY0019	Sickle	Bronze Age	Gongliu County	97.8	-	1.0	-	-	0.4	-	0.7	0.1	-	18.098	15.554	37.991
XY0033	Spear head	Bronze Age	Nileke					/						18.007	15.567	38.013
XY0072	Sickle	Bronze Age	Taleda, Xinyuan County	99.8	0.1	-	-	-	-	-	0.1	-	-	18.613	16.075	39.381
XY0179	Shaft-hole axe	Bronze Age	Gongliu County	79.9	7.7	-	-	-	2.1	9.5	0.6	0.3	-	17.982	15.556	37.901
XY0488	Knife	Bronze Age	Qiaolake Tiereke, Tekesi County	91.0	8.3	-	-	-	0.1	0.3	0.2	0.1	-	18.255	15.582	38.084
XY0489	Socketed-axe	Bronze Age	Tekesi County	91.3	8.5	-	-	-	-	-	0.1	0.1	-	17.888	15.593	37.816
XY0493	Knife	Bronze Age	Qiaolake Tiereke, Tekesi County	91.6	6.9	-	-	-	1.0	0.2	0.2	0.1	-	18.377	15.800	38.679
XY0020	Chisel	Iron Age	Gongliu County	91.3	2.0	-	-	0.1	1.0	5.4	0.2	-	-	18.174	15.580	38.101
XY0024	Ingot	Iron Age	Wusun mountain, Zhaosu county	99.6	-	-	0.4	-	-	-	-	-	-	18.234	15.576	38.080
XY0027	Ingot	Iron Age	Central town of Gongliu County	98.1	-	-	-	-	-	-	1.9	-	-	18.105	15.559	38.002
XY0031	Sword	Iron Age	Boarder post at Hasang, Zhaosu County					/						18.147	15.568	38.026
XY0048	Sword	Iron Age	Xiaolake Tiereke, Tekesi County					/						18.260	15.582	38.106
XY0058	Altar/lamp	Iron Age	71 Regiment in Xinyuan County					/						18.263	15.620	38.180
XY0059	Cauldron	Iron Age	71 Regiment in Xinyuan County	97.1	0.7	-	-	-	1.0	-	1.1	0.1	-	18.095	15.550	37.981
XY0061	Cauldron	Iron Age	Tasi Bier, Gongliu County	98.4	1.5	-	0.2	-	-	-	-	-	-	18.108	15.554	37.993
XY0143	Altar/lamp	Iron Age	Village 4, Xiata, Zhaosu County					/						18.478	15.646	38.374
XY0148	Cauldron	Iron Age	Subutai, Nileke County	99.7	0.2	-	-	-	-	-	-	0.1	-	18.345	15.683	38.562
XY0150	Cauldron	Iron Age	Tekesi County	98.3	0.5	0.4	0.3	0.2	-	0.2	-	-	-	18.126	15.583	37.972
XY0151	Cauldron	Iron Age	Xinyuan County	98.9	1.0	-	-	0.1	-	-	-	-	-	18.101	15.553	37.989
XY0152	Cauldron	Iron Age	Unknown	65.3	0.2	-	-	0.1	18.7	15.4	0.1	0.2	-	17.677	15.562	37.978
XY0153	Cauldron	Iron Age	Mohe, Gongliu County					/						18.083	15.549	37.974
XY0156	Square plate	Iron Age	Mohe, Gongliu County	99.7	-	-	-	-	0.1	-	-	0.1	0.1	18.362	15.643	38.418
XY0173	Human statue	Iron Age	Gongliu County	97.1	2.7	-	-	-	-	-	-	0.1	0.1	18.483	15.726	38.555

No.	Objects	Period	Origin	Chemical composition (SEM-EDS, wt%)										Lead isotope ratios		
				Cu	Sn	As	Pb	Sb	Cl	O	S	Fe	Ag	²⁰⁶ Pb/ ²⁰⁴ Pb	²⁰⁷ Pb/ ²⁰⁴ Pb	²⁰⁸ Pb/ ²⁰⁴ Pb
XY0185	House bite	Iron Age	Dunmaili, Gongliu County	88.1	0.5	1.7	0.4	0.3	1.6	7.3	0.1	-	-	18.175	15.599	38.113
XY0187	Chisel	Iron Age	Central town of Gongliu County	97.1	1.7	-	-	-	0.2	0.2	0.7	0.1	-	18.103	15.558	37.976
XY0195	Altar/lamp	Iron Age	Nalati, Xinyuan County					/						18.309	15.635	38.150
XY0476	Cauldron	Iron Age	Unknown	96.4	2.2	0.6	0.4	-	0.1	-	-	-	0.3	18.092	15.546	37.969
XY0477	Cauldron	Iron Age	Zhaosu County	99.2	0.3	-	-	0.1	0.2	-	-	-	0.2	18.542	15.662	38.747
XY0478	Cauldron	Iron Age	Kushitai, Tekesi County	99.7	-	-	-	-	-	-	-	-	0.3	18.529	15.654	38.673
XY0479	Cauldron	Iron Age	Tekesi County	99.5	0.2	-	-	-	0.1	-	-	-	0.2	18.528	15.742	38.783
XY0657	Mirror	Iron Age	Qiongkeke site, Nileke County					/						18.093	15.579	38.082

Several micro-areas were analysed in each sample, and the mean value was used as bulk composition in order to balance out the error caused by potential heterogeneity within samples. The results are normalised with oxygen and chlorine contents to indicate the corrosion condition of the samples. “-” means the value is below the detection limitation, “/” means the data are not available

The lead isotopic ratios (LIR) of the 33 samples (24 metal and 9 patina) were measured here ranging from 17.677 to 18.613 for $^{206}\text{Pb}/^{204}\text{Pb}$, from 15.546 to 16.075 for $^{207}\text{Pb}/^{204}\text{Pb}$ and from 37.816 to 39.381 for $^{208}\text{Pb}/^{204}\text{Pb}$. Given that the low-lead concentration of all analysed samples (always less than 0.5 wt% and mostly below the detection limit), and the fact that the contribution of lead from tin is generally negligible (Gale and Stos-Gale 2000; Molofsky et al. 2014), it is reasonable to consider the lead as an impurity of the copper rather than as a separate material that was deliberately added in. Therefore, the lead isotopic ratios of the objects very likely reflect the signature of their copper source(s). The non-clustered distribution of the patina sample's LIR data suggests lead contamination from the burial environment was not significant, and the data can be used to represent the metals on which the patina was taken.

Metallographic observation shows that most of the cauldron samples have a typical as-cast microstructure of α -copper grains with non-metallic inclusions (e.g., Cu_2O and Cu_2S) as globules and sometimes enrichment of soluble impurities by coring segregation at grain boundaries (Fig. 3a). The disappearance of casting segregation and relatively large grain size of one cauldron sample (XY0059), together with the residual ash on the surface, suggests extensive firing during its usage (Fig. 3b). On the other hand, samples from tools, such as knives and sickles, are often found to have a metallographic structure of hot or/and cold working. The matrix of equiaxial/twin grains and elongated non-metallic inclusions indicates the experience of hot-working and/or cold-working then annealing (Fig. 3c), while the presence of grain deformation and slip bands suggests the application of further cold-working (Fig. 3d).

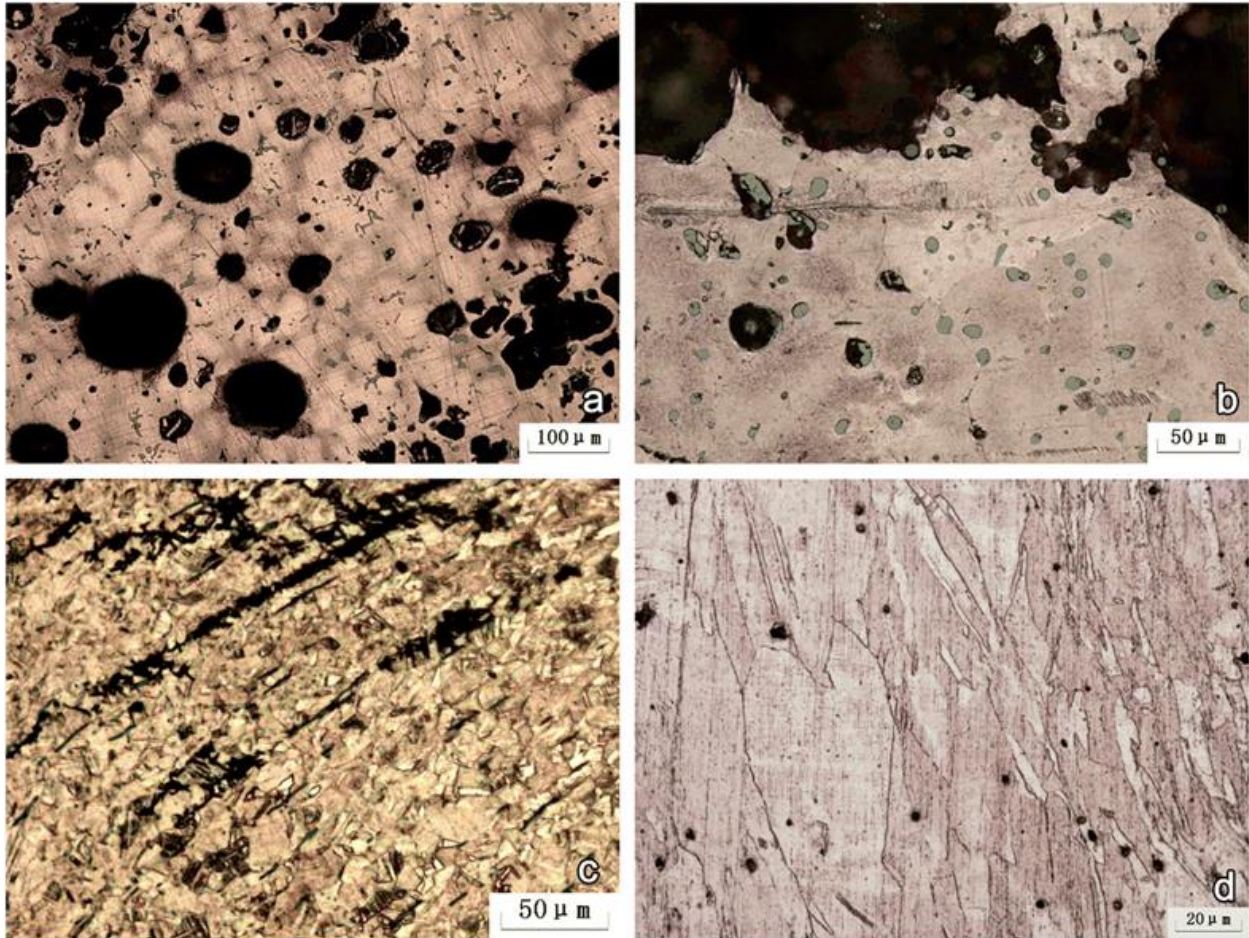


Fig. 3

Photomicrographs of typical metallographic structures identified among copper/bronze objects from the Ili region. **a–b** As-cast microstructure of unalloyed copper, noting the abundant porosity (**a** cauldron XY0476) and occurrence of grey-blue non-metallic inclusions (**b** cauldron XY0059). **c–d** Hot/cold worked microstructure of recrystallised equiaxed and twin grains, the aligned and elongated inclusions (**c** knife XY0488) and grains (**d** sickle XY0072) are also visible

4. Discussion

4.1 Shift between the Bronze and Iron Ages

In his pioneering work, Mei (2000) systemically examined metal objects of prehistoric Xinjiang, emphasising their importance for revealing cultural and technological exchanges across the Eurasian continent. Since then, ongoing archaeological and archaeometallurgical studies have furthered our understanding of the topic considerably (e.g., Qian 2006; Shao 2007; Ling et al. 2008; Guo 2012; Mei et al. 2013; Ruan 2013; Jia et al. 2017). Several important conclusions can now be drawn about Xinjiang's early metallurgy. Firstly, the emergence of the Andronovo complex in northwestern Xinjiang can be traced back to the first part of the second millennium BCE. Metal objects of Andronovo type have been identified in the Ili Valley and neighbouring regions in the western Tianshan, and the relative abundance of tin bronze among the examined objects suggests a technological affinity with bronze cultures of the Eurasian Steppe (Mei and Colin 1999). Secondly, UIV's connection with the steppes, especially with the Semirechye region in eastern Kazakhstan persisted during the Iron Age. Several identical types of objects, such as tripod cauldron, altar/lamp and large square plate, demonstrate the resemblance of metalwork in these regions (Grigor'ev and Ismagil 1997; Guo 2007). Thirdly, it is gradually being recognised that the early copper/bronze metallurgy in northwest Xinjiang, Dzungarian, Tarim and Hami is more diverse in terms of technological practises than previously assumed, for instance, in types of artefacts and materials—and our data add to this diversifying picture. The relationship among the metallurgical traditions of these distinct regions in Xinjiang, and their possible connections to the east with the Hexi Corridor, remains obscure.

The analytical results reported in the present paper show that in the UIV, tin bronze is the major material that was used for tools/weapons during the Bronze Age, while the material for casting the Iron Age cauldrons is mainly unalloyed copper. These results are consistent with the published data from this region (Ling et al. 2008; Mei et al. 2003; Mei et al. 2005). Taking chronology into account, however, an interesting trend emerges, that is the proportion of tin bronze decreased markedly from the Bronze Age to the Iron Age. Although the currently available data are relatively limited, this shift is even more notable when we compare only the tin concentration of tools/weapons that is available for both periods. While the tin concentration of most of the Bronze Age samples exceeds 6%, with an average of 6.7%, the Iron Age samples have much lower tin, all less than 3% and around 1.4% on average (Table 1). This trend implies a potential shortage of tin supply in Iron Age UIV, which seems to agree with the situation in Cis-Baikal and Tuva noticed by Hsu et al. (2016).

The shift between the Bronze Age and the Iron Age can also be seen from the lead isotope results. In Fig. 4, lead isotopic data are presented in scatter plots split by chronological periods. They show that most of the Iron Age samples are clustered together with $^{206}\text{Pb}/^{204}\text{Pb}$ ratios of 18.1 ~ 18.4 and $^{207}\text{Pb}/^{206}\text{Pb}$ ratios of 0.85 ~ 0.86. Despite the overlaps in both diagrams, 6 out of 9 Bronze Age samples can be separated from the Iron Age ones with lower $^{208}\text{Pb}/^{206}\text{Pb}$ or higher $^{206}\text{Pb}/^{204}\text{Pb}$ ratios, implying that the copper resource(s) for the Bronze Age were likely different from those used in Iron Age UIV.

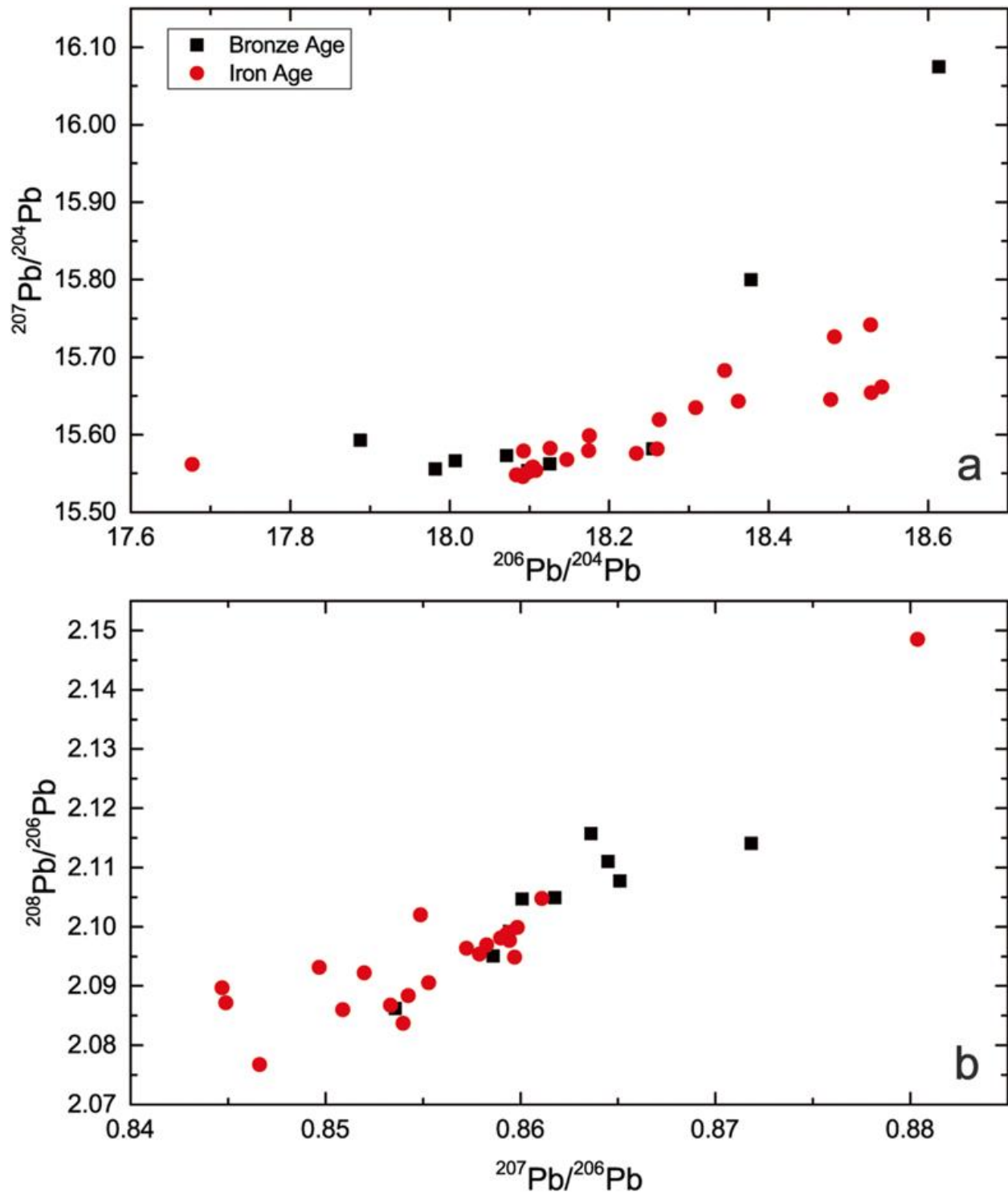


Fig. 4

Plots of lead isotope ratios of copper/bronze objects from the Ili region

4.2 Localised metallurgy in the upper Ili Valley

The differences in chemical and lead isotopic composition indicate some changes in the technological practises and supply of raw materials for the copper/bronze objects found in the UIV between the Bronze and Iron Ages. One plausible explanation for these changes could be the localisation of metallurgy in the UIV during this transition. While copper deposits along the Awulale and other mineralisation belts in the middle and northern part of the valley may have been potential sources available to ancient inhabitants (e.g., Chen 2003; Ma et al. 2017), the identification of ancient mining/smelting sites at Nulasai and Yuantoushan in Nileke County provides concrete evidence for local metallurgical production in the region (Wang 1984; Wang 1985a, b).

Yuantoushan was reported as a mining site with an absence of technological remains relating to smelting activities, and dated to cal 1191–236 BCE (BK77001, 95.4% probability, curve: IntCal 13) by carbon dating of a charcoal sample (Wang 1985a, b; IoA, CASS 1992). We visited the site in summer 2016 and identified one stone hammer despite of a serious damage resulting from modern mining in the area (Fig. 5). At the Nulasai site, more than a dozen ancient mining shafts and smelting slag heaps, from which five “matte ingots” were unearthed, have been recorded (Wang 1984, 1985a, b). A pit timber from the mining shaft at Nulasai was radiocarbon dated to cal 752–206 BCE (WB82–51, 95.4% probability, curve: IntCal 13) (IoA, CASS 1992). Mei and Li (1998) suggested a relatively sophisticated technical flowchart for Nulasai’s smelting process based on examination of the ore and slag samples: plumbiferous copper sulphide ores were roasted first, the subsequent smelting producing lower-grade matte; the matte was then roasted and arsenious ores were added to for the second smelting, which resulted in arsenic-containing higher-grade matte; the resulting matte was further roasted and smelted, yielding a product of Cu-As-Pb alloys.



Fig. 5

Modern mining trench and ancient stone hammer found at the site of Yuantoushan (photo taken in 2016)

To assess the relationship between the metal objects and local metallurgical practises in the UIV, published lead isotope data for copper mines in the relevant regions together with slag from the Nulasai site and ingots discovered in various places within the UIV were collected for comparison with the data generated by this research. The results show that the lead isotope ratios of two copper deposit belts in the Ili region, Awulale (including Yuantoushan and Nulasai mines) and Kedenggaoer, cluster together in a relatively narrow area and are distinguished from the others, although the occurrence of outlying data is also notable (Fig. 6a). Most of the data for the ancient slag from Nulasai and ingots fall in the distribution region of the Ili mines; in particular, the data for the slag samples and one ingot (No. 121) are almost identical, strongly indicating a common origin and defining the data distribution of local metallurgical production at Nulasai (Fig. 6b). There is one Bronze Age sickle (XY0019) that overlaps with the Nulasai slag together with two others (XY0018 and D0013, see Table 1 and Supplementary Table S1) close to it, but the rest are notably different (Fig. 6c). On the other hand, half of the Iron Age samples cluster together in the distribution region of the Awulale deposit belt, and the data of six of these samples cannot be differentiated from the Nulasai slag, and so are very likely to originate from the Ili ore and local metallurgical production. The other half of the Iron Age samples, having a relatively broad range of lead isotopic ratios, tend to be varied and extraneous (Fig. 6d).

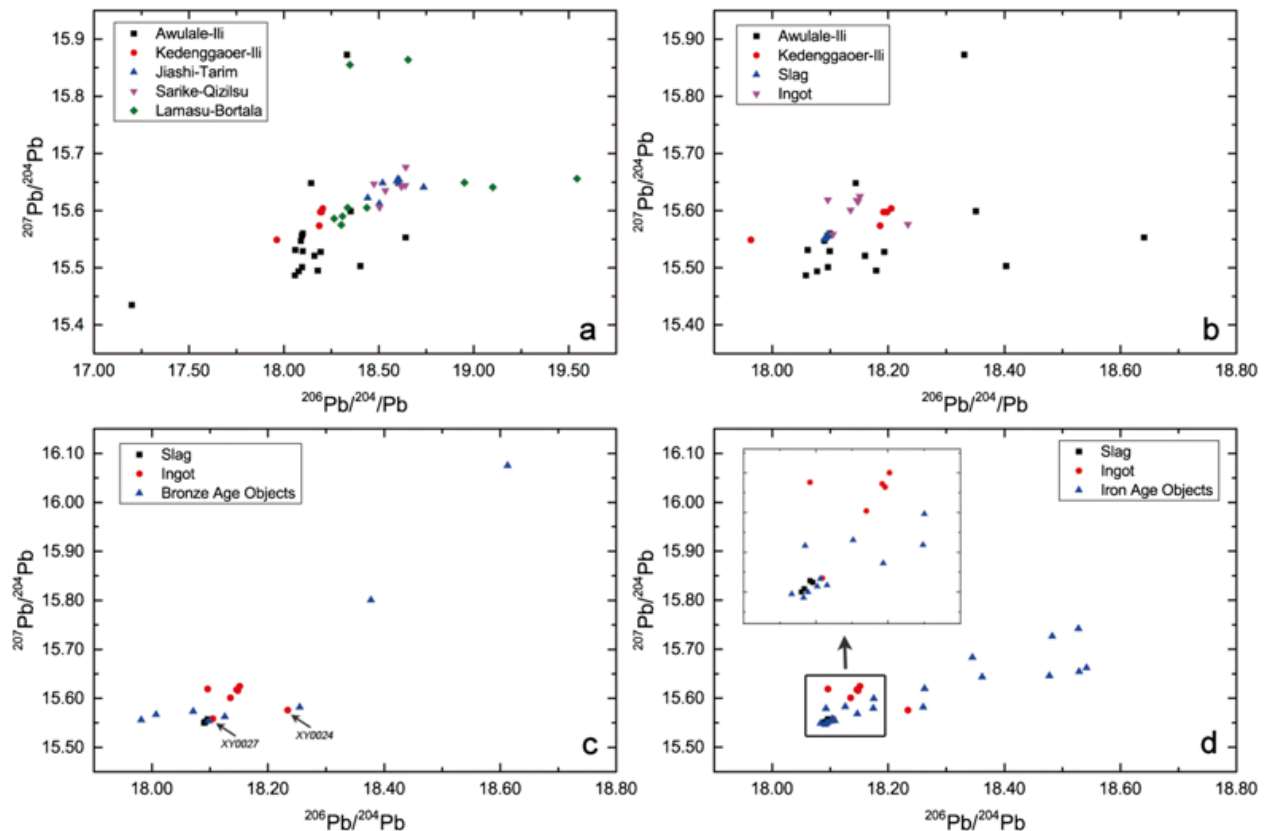


Fig. 6

Plots of lead isotope ratios of mines in Ili and related region (a), technological remains and ingots discovered in Ili (b) and their relations with analysed objects (c, d). (Slag and ingots samples are all from the Nulasai site except two, XY0024 and XY0027 from this research)

The aforementioned observations are significant for an understanding of the flow of material/objects and metallurgical development in the UIV and its neighbouring regions. Current data suggest that most of the objects dating to the Bronze Age found in the UIV region were probably imported from other places as indicated by their lead isotope composition, which is distinct from the mines and technical remains in the region. The only sample (sickle XY0019) that has similar isotopic data of the local mines and slags may in fact be ascribed to a later period, given that it is comprised of unalloyed copper with relatively high-arsenic concentration. Local copper smelting in the UIV emerged probably from the early first millennium BCE and accounted for a substantial proportion of the Iron Age objects discovered in the region. Several copper/bronze objects from the Chawuhu cemetery site in Hejing (ca 1000–500 BCE) have been revealed to have similar lead isotope ratios (Mei et al. 2002b), implying the potential exportation of metals from the UIV to neighbouring regions. In the meantime, a number of objects could still have been imported, though their origins are probably varied and distinct from the one(s) in the Bronze Age. The decrease of tin content in the Iron Age objects is suggestive of a tin shortage similar to that of several regions in the eastern Eurasian Steppe (Hsu et al. 2016), suggesting also inconsistent technological practises and material supplies between the two periods.

It is worth pointing out that the research presented here is still preliminary, and some critical issues related to the UIV's early metallurgy are yet to be settled. For instance, research on the technological remains from Nulasai indicates the production of copper with significant lead and arsenical content (Mei and Li 1998); but confusingly, including the data reported here, very few objects have been identified to have such a compositional feature, and Cu-As-Pb alloys have only been identified among the objects discovered in the Hami region in eastern Xinjiang rather than the Ili region (Mei et al. 2002a; Ling et al. 2008). Where and how were the metallurgical products from Nulasai used? Do the Cu-As-Pb objects in Hami imply an eastward circulation of this especial material? Perhaps it is, more likely that, unalloyed copper may have been produced in addition to the Cu-As-Pb "alloy" given the identical lead isotope ratios of Nulasai's slag and several objects with lead hardly detected. As might be expected, however, with the limitations of available evidence, these questions remain unclear. Further detailed investigations of the archaeological materials with adequate chronological and cultural context are necessary to advance our understanding of prehistoric metallurgy in the upper Ili Valley.

5. Conclusion

Compositional and metallographic analyses have revealed technological features of prehistoric copper/bronze objects discovered in the upper Ili Valley. Tin bronze was the dominant material used during the Bronze Age; but the use of tin seems to have decreased markedly during the Iron Age, as evidenced by the substantial proportion of unalloyed copper and clear drop in tin concentrations in the bronze samples examined. Most of the objects were manufactured by casting; while some cauldron samples show evidence of heating, and some tools/weapons received further hot or/and cold working for finishing. Lead isotope data show that most of the Bronze Age objects were likely imported; conversely, local copper resources appear to have been exploited during the Iron Age, and many of the analysed objects could have been made locally using the local mineral resources. The changes of the material (alloys) and metal source(s) between the Bronze and Iron Ages in the upper Ili Valley imply various movements of objects, raw materials and related technical practises. This research is significant as a further contribution to our understanding of the mechanisms of the adoption of metallurgy in the region.

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7. Supplementary Material

Supplementary data related to this article can be found at:

[12520_2018_679_MOESM1_ESM.docx](#) (8.4 mb)

ESM 1 (DOCX 8560 kb)

[12520_2018_679_MOESM2_ESM.docx](#) (16 kb)

ESM 2 (DOCX 16 kb)

8. References

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Supplementary Table S1. Background and finding information of the objects studied (Photo data source: Ili Museum)


No.	Object	Origin	Remarks	Periodization/dating	Photo
D0013	Knife	Tangjiale Memorial in Xiinyuan	donated chance finding; Length: 25.2 cm	Bronze Age/ca. 14 C BCE	
XY0018	Socketed axe	Gongliu County	donated chance finding; Length: 14 cm	Bronze Age/ca. 14 C BCE	
XY0019	Sickle	Gongliu County	donated chance finding; Length: 23 cm	Bronze Age/ca. 10 C BCE	


No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0033	Spear head	Nileke	donated chance finding; Length: 14.8 cm	Bronze Age/ca. 10 C BCE	
XY0072	Sickle	Talede, Xinyuan County	donated chance finding; Length: 27 cm	Bronze Age/ca. 10 C BCE	
XY0179	Shaft-hole axe	Gongliu County	donated chance finding; Length: 15.5 cm	Bronze Age/ca. 14 C BCE	

No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0488	Knife	Nazituobie, Qiaolake Tiereke, Tekesi County	donated chance finding; Length: 11.1 cm	Bronze Age/ca. 13 C BCE	
XY0489	Socketed-axe	Tekesi County	donated chance finding; Length: 10 cm	Bronze Age/ca. 14 C BCE	
XY0493	Knife	Nazituobie, Qiaolake Tiereke, Tekesi County	donated chance finding; Length: 23.3 cm	Bronze Age/ca. 10C BCE	
XY0020	Chisel	Gongliu County	donated chance finding; Length: 16.7 cm	Iron Age/ ca. 5-3 C BCE	


No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0024	Ingot	Ancient mining site at Wusun Mountain, Zhaosu county	collected in-situ; Diameter: 11 cm; weight: 2090 g	Iron Age	
XY0027	Ingot	Central town of Gongliu County	donated chance finding; Diameter: 24 cm; weight: 13210 g	Iron Age	
XY0031	Sword	Boarder post at Hasang, Zhaosu County	donated chance finding; Length: 25.7 cm	Iron Age/ ca. 8-5 C BCE	

No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0048	Sword	Xiaolake Tiereke, Tekesi County	donated chance finding; Length: 28.5 cm	Iron Age/ ca. 8-5 C BCE	
XY0058	Altar/lamp	71 Regiment in Xinyuan County	Excavated; Broken fragments, width: 12 cm	Iron Age/ ca. 5-3 C BCE	



No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0059	Cauldron	71 Regiment in Xinyuan County	Excavated; Height: 56 cm	Iron Age/ ca. 5-3 C BCE	



No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0061	Cauldron	Tasi Bier, Gongliu County	donated chance finding; Height: 53.5 cm	Iron Age/ ca. 5-3 C BCE	



No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0143	Altar/lamp	Village 4, Xiata, Zhaosu County	collected in-situ hand in by Li Xinlin; Height: 16 cm	Iron Age/ ca. 5-3 C BCE	
XY0148	Cauldron	Subutai, Nileke County	collected in-situ; Height: 44.5 cm	Iron Age/ ca. 5-3 C BCE	


No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0150	Cauldron	Tekesi County	donated chance finding; Height: 59 cm	Iron Age/ ca. 5-3 C BCE	

No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0151	Cauldron	Xinyuan County	donated chance finding; Height: 70 cm	Iron Age/ ca. 5-3 C BCE	
XY0152	Cauldron	Unknown	donated chance finding; Diameter: 51 cm	Iron Age/ ca. 5-3 C BCE	



No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0153	Cauldron	Mohe, Gongliu County	collected in-situ; Height: 40 cm	Iron Age/ ca. 5-3 C BCE	
XY0156	Square plate	Mohe, Gongliu County	collected in-situ; width: 85 × 78.5 cm	Iron Age/ ca. 5-3 C BCE	

No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0173	Human statue	Gongliu County	collected in-situ; Height: 21.5 cm	Iron Age/ ca. 5-3 C BCE	
XY0185	Horse bite	Dunmaili, Gongliu County	collected in-situ; Length: 8.5 cm	Iron Age/ ca. 8-3 C BCE	

No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0187	Chisel	Central town of Gongliu County	donated chance finding; Length: 13.8 cm	Iron Age/ ca. 5-3 C BCE	
XY0195	Altar/lamp	Nalati, Xinyuan County	collected in-situ; Height: 14.3 cms	Iron Age/ ca. 5-3 C BCE	

No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0476	Cauldron	Unknown	donated chance finding; Height: 38 cm	Iron Age/ ca. 5-3 C BCE	

No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0477	Cauldron	Zhaosu County	donated chance finding; Height:54 cm	Iron Age/ ca. 5-3 C BCE	
XY0478	Cauldron	Kushitai, Tekesi County	donated chance finding; Height: 38 cm	Iron Age/ ca. 5-3 C BCE	

No.	Object	Origin	Remarks	Periodization/dating	Photo
XY0479	Cauldron	Tekesi County	donated chance finding; Diameter: 45 cm	Iron Age/ ca. 5-3 C BCE	
XY0657	Mirror	Qiongkeke site, Nileke County	Excavated; Diameter: 9 cm	Iron Age/ca. 9-5 C BCE	

Supplementary Table S2. Lead isotopic data used for comparison in Figure 6

Sample No.	207Pb/206Pb	208Pb/206Pb	206Pb/204Pb	207Pb/204Pb	Mineral	Origin
NLS12	0.8594	2.0995	18.09	15.547	Chalcocite	Awulale, Ili
NLS15	0.8599	2.1002	18.061	15.531	Chalcocite	Awulale, Ili
NLS32	0.8624	2.1119	18.144	15.648	Chalcocite	Awulale, Ili
109-26	0.8974	2.1815	17.199	15.435	Chalcopyrite	Awulale, Ili
109-27	0.8343	2.0699	18.641	15.553	Bornite	Awulale, Ili
109-31	0.8535	2.0914	18.193	15.528	Bornite	Awulale, Ili
QBLK1	0.858	2.0957	18.099	15.529	Chalcopyrite	Qiongkeke, Awulale, Ili
QBLK2	0.8523	2.0835	18.179	15.495	Chalcopyrite	Qiongkeke, Awulale, Ili
QBLK3	0.8546	2.0941	18.16	15.521	Chalcocite	Qiongkeke, Awulale, Ili
QBLK33	0.8592	2.0825	18.403	15.503	Chalcocite	Qiongkeke, Awulale, Ili
QBLK4	0.8571	2.0921	18.077	15.494	Chalcocite	Qiongkeke, Awulale, Ili
QBLK5	0.8576	2.0931	18.058	15.487	Chalcocite	Qiongkeke, Awulale, Ili
QBLK6	0.8565	2.0912	18.096	15.501	Chalcocite	Qiongkeke, Awulale, Ili
QBLK7	0.85	2.0787	18.351	15.599	Bornite	Qiongkeke, Awulale, Ili
JS-10	0.8348	2.0937	18.736	15.641	Chalcocite	Jiashi, north Tarim
JS-11	0.8416	2.0691	18.595	15.651	Chalcocite	Jiashi, north Tarim
JS-15	0.8471	2.0933	18.441	15.622	Chalcocite	Jiashi, north Tarim
JS-16	0.8416	2.0812	18.601	15.655	Chalcocite	Jiashi, north Tarim
JS-17	0.8437	2.0819	18.503	15.612	Chalcocite	Jiashi, north Tarim
JS-18	0.8412	2.0816	18.607	15.654	Chalcocite	Jiashi, north Tarim
JS-19	0.8409	2.0823	18.607	15.647	Chalcocite	Jiashi, north Tarim
JS-35	0.8449	2.0873	18.52	15.648	Chalcocite	Jiashi, north Tarim
4-54	0.8504	2.0875	18.654	15.864	Chalcopyrite	Lamasu, Bortala
E50	0.8641	2.1008	18.347	15.855	Chalcopyrite	Lamasu, Bortala
LMSY-10	0.8464	2.0849	18.437	15.605	Chalcopyrite	Lamasu, Bortala
LMSY-17	0.8534	2.0907	18.264	15.586	Chalcopyrite	Lamasu, Bortala
LMSY-3	0.8515	2.0875	18.309	15.59	Chalcopyrite	Lamasu, Bortala
LMSY-4	0.8257	2.0422	18.951	15.649	Chalcopyrite	Lamasu, Bortala

LMSY-5	0.8189	2.0239	19.1	15.641	Chalcopyrite	Lamasu, Bortala
LMSY-8	0.8011	1.9804	19.544	15.656	Chalcopyrite	Lamasu, Bortala
LMSY-9	0.8511	2.0819	18.302	15.575	Chalcopyrite	Lamasu, Bortala
K26	0.8564	2.0942	18.186	15.574	Chalcopyrite	Kendenggaer, Ili
K27	0.8571	2.0974	18.205	15.604	Chalcopyrite	Kendenggaer, Ili
K32	0.8571	2.0972	18.198	15.598	Chalcopyrite	Kendenggaer, Ili
K36	0.8574	2.0976	18.192	15.598	Chalcopyrite	Kendenggaer, Ili
K5	0.8656	2.1305	17.963	15.549	Chalcopyrite	Kendenggaer, Ili
2240	0.8603	2.104	18.135	15.601	Ore	Nulasai, Nileke, Ili
2298	0.8597	2.1003	18.095	15.556	Ore	Nulasai, Nileke, Ili
2299	0.8597	2.1006	18.099	15.56	Ore	Nulasai, Nileke, Ili
122-1	0.8606	2.1054	18.151	15.625	Ingot	Nulasai, Nileke, Ili
122-2	0.8605	2.1049	18.148	15.616	Ingot	Nulasai, Nileke, Ili
121	0.8605	2.1049	18.096	15.619	Ingot	Nulasai, Nileke, Ili
60	0.8595	2.1001	18.098	15.556	Slag	Nulasai, Nileke, Ili
65	0.8597	2.1004	18.098	15.556	Slag	Nulasai, Nileke, Ili
66	0.8596	2.1002	18.092	15.552	Slag	Nulasai, Nileke, Ili
67	0.8596	2.1002	18.09	15.55	Slag	Nulasai, Nileke, Ili
68	0.8597	2.1004	18.096	15.557	Slag	Nulasai, Nileke, Ili
102	0.8607	2.1053	18.146	15.618	Ingot	Nulasai, Nileke, Ili
940155	0.861501	2.10294	18.4823	15.293	Slag	Nulasai, Nileke, Ili
940156	0.865893	2.1282	18.3312	15.873	Ore	Nulasai, Nileke, Ili
940157	0.861576	2.10336	18.5443	15.977	Slag	Nulasai, Nileke, Ili
940158	0.862106	2.10368	18.405	15.867	Matte	Nulasai, Nileke, Ili