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A fluorite bead from Bronze Age Tianshanbeilu cemetery, Xinjiang, Northwest China

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Abstract
The Tianshanbeilu cemetery is the largest Bronze Age cemetery in eastern Xinjiang, China, and plays important roles in connecting the Eurasian interior to the Hexi Corridor, and further to the Central Plains region. Utilizing micro-XRF and Raman spectroscopy techniques, we identified a fluorite bead at this cemetery. This barrel bead is the earliest record of such a fluorite bead in China, dating back to approximately 1385–1256 BCE. Comparing the unearthed records of fluorite beads in eastern China spanning from the Neolithic Age to the Western Zhou dynasty, we notice that in the early Western Zhou period, fluorite beads found in élite burials are only in barrel or biconical shapes, both of which first appeared in the eastern region of Xinjiang, such as the Tianshanbeilu cemetery in Hami and the Yanghai cemetery in the Turpan Basin. We proposed that the barrel fluorite bead drilled by metal tubular drill first appearing in the Tianshanbeilu cemetery might have spread eastward to the Central Plains region and finally constituted a component of the ritual revolutions during the Western Zhou dynasty.

Keywords Fluorite bead, Bronze Age, Tianshanbeilu cemetery, Xinjiang

Introduction

Situated in Hami oasis on the eastern end of the Tianshan Mountains, Tianshanbeilu cemetery consists of more than 700 graves with two kinds of burial chambers, shaft pits and mud-brick shaft pits [1]. Most recently the cemetery has been dated to 2011–1029 cal BC by extensive radiocarbon dates [2], marking it the earliest and longest-used known cemetery in eastern Xinjiang.

Due to its long chronology and key location, the Tianshanbeilu cemetery is characterized by a two-way traffic of cultural intermediary (Fig. 1). The site is well known for its elaborate painted ceramics clearly derived from eastern traditions such as the Hexi Corridor, contrasting to simple grey wares from the pastoralist cemeteries [3]. Both typological and technological comparisons of copper and bronze artefacts reveal that the Tianshanbeilu cemetery closely linked the Hexi Corridor with the Eurasian steppes.

Multiple lines of bioarchaeological and genetic evidence indicate Tianshanbeilu was a cultural intermediary between eastern and western populations. Isotopic paleodiet studies at Tianshanbeilu show that the population generally consumed both wheat (C_3) and millet (C_4) and at least one of the earliest dated individuals had an exclusive diet of millet (1940–1765 cal BC [4]). In the early 2nd millennium BC, eastwards from Hami there is no evidence for the herding cultures, while westwards there is no evidence for the painted pottery used

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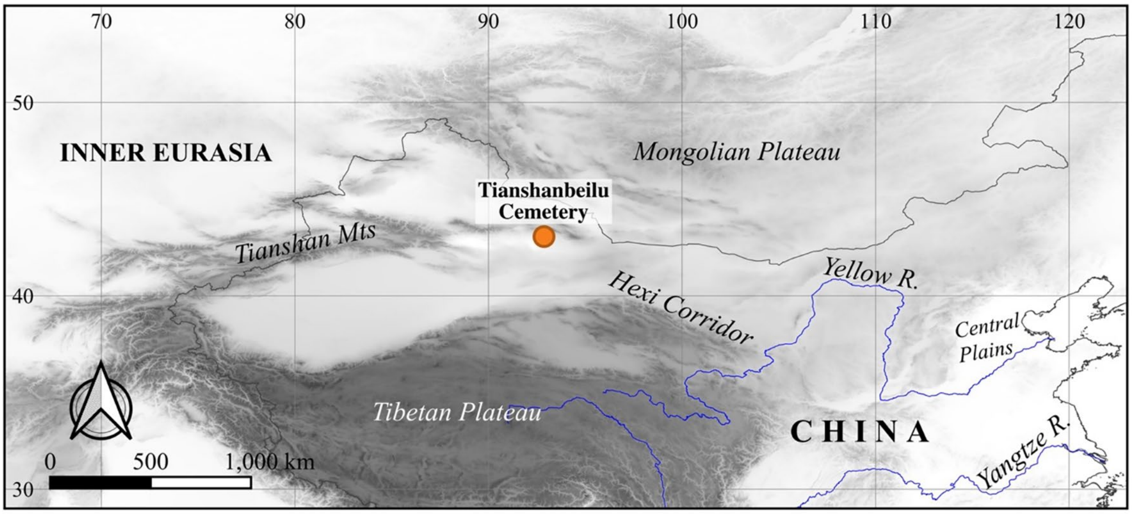

Fig. 1 Location of the Tianshanbeilu cemetery, China

by farmers consuming millet [5]. These scenarios further confirmed by ancient human mitochondrial genomes. Wang et al. [6] found that Bronze Age residents in Tianshanbeilu were a genetically mixed population, both to ancient Gansu-Qinghai populations and to the West Eurasian–related populations. The presence of some West Eurasian–related haplogroups in Bronze Age eastern Xinjiang is further consistent with the presence of burial forms and ornaments at the Tianshanbeilu site.

Tianshanbeilu people decorated their bodies with various kinds of ornaments, among which were of various forms and diverse materials [2]. The most common earrings, necklaces and bracelets were made of copper, turquoise or carnelian beads. A few faience beads could be the result of exchange with the steppe population in the north [7]. Cowrie shell, *Monetaria moneta*, may have been stitched on fur belts as headbands. Pendants made of copper, carnelian, or bones were commonly used to decorate clothes. All these personal ornaments indicate broad exchange networks surrounding the Tianshanbeilu site.

Among these personal ornaments excavated from Tianshanbeilu, a translucent bead found in the grave of M099 was originally marked as "quartz水晶" by the excavator and mostly ignored for a long time. Considering that various minerals might be misidentified as quartz, we here conduct a series of scientific analysis on this specimen.

Materials and methods

The grave M099 is a rectangular shaft pit grave. A single set of a juvenile skeletal remains in poor preservation was found at the bottom of the burial chamber, with only vertebrae, parts of limb bones, and fragments of the skull (Fig. 2A). The body was in flexed position on the left side, with skull towards the northeast, facing south. A total of five artifacts were unearthed. One is a double-handled pottery jar (Fig. 2B), found on the chest of the human remains. Two are copper ornaments, separately discovered on the chest and near the shin bones. One carnelian bead and one purportedly "quartz" bead were found around the neck of the human remains. Registered as M099:5, the long barrel bead is light green and highly transparent with a perforation through the center. It is

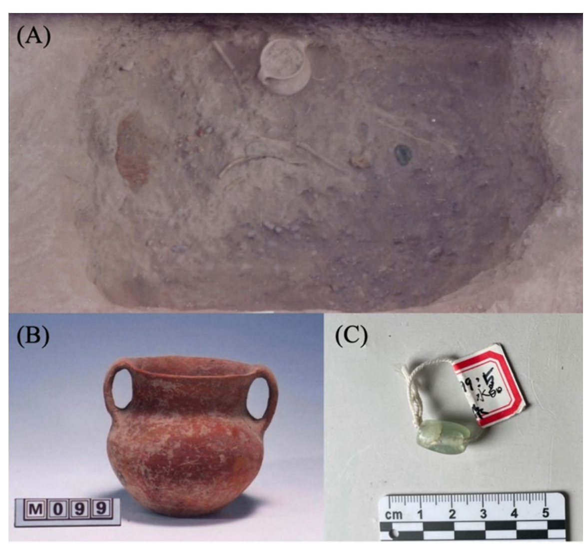

Fig. 2 **A** The grave M099 at the Tianshanbeilu cemetery; **B** the pottery jar from the grave; **C** the purportedly "quartz" bead found in the grave

1.5 cm long, with a diameter ranging from 0.7 to 1 cm, a hole diameter of 0.3 cm, and weighs 2.65 g (Fig. 2C).

The sample was subjected to non-invasive analysis to identify its minerals. The elemental composition of the specimen M099:5 was obtained using the Bruker M4 TORNADO^{PLUS} micro XRF spectrometer (μXRF). This instrument can detect super light elements with its dual large area detectors and light element X-ray tube, allowing for identifying minerals easily by clearly highlighting the fluorine, oxygen, and carbon. The analysis was performed at the Institute of Geology and Geophysics, Chinese Academy of Science (IGGACS). A polycapillary optic focused a beam spot size 20 μm diameter, and a 30 mm^2 dual silicon drift detector were used with a dwell time of 60 s per point. Multi-point mode was used to measure 10 points from the bead. Quantitative XRF analysis for all points were undertaken with the characterization software provided by Bruker Micro Analytics. The 16 elements quantified were summed, normalized to 100%, and the elemental data expressed as percentages. The bundled calibration of the instrument was used to calculate elemental concentrations. Means and standard deviations of the concentrations of fluorine (F), sodium (Na), magnesium (Mg), aluminum (Al), silicon (Si), phosphorus (P), sulfur (S), chlorine (Cl), potassium (K), calcium (Ca), titanium (Ti), chromium (Cr), iron (Fe), nickel (Ni), copper (Cu), and gallium (Ga) are reported.

Raman spectroscopy was performed at room temperature using a Witec alpha300R confocal Raman microscope at IGGCAS. The laser beam was focused on the sample surface using a 50× Zeiss microscope objetive (NA = 0.75). The Raman data has been calibrated with a silicon peak of 520.7 cm^{-1}. The spectra were collected with a 488 nm laser with a spectral acquisition time of 1s and 20 accumulations for each measurement. The investigated spectra range from 100 to 4500 cm^{-1} using 300 grooves/mm optical grating. Ten measurements were operated for the specimen.

Results

The μXRF measurements performed provide an overall distribution of at least 10 recognizable elements in the bead M099:5 (Fig. 3A). Sorting of the elements by their main phase shows that the bead is rich in Ca and F, corresponding mineralogically to fluorite, CaF_2. Semi-quantitative compositional analysis to the μXRF spectrum yields average content of fluorine 15.8 ± 4.4% and calcium of 81.1 ± 3.8% (n = 10; Table 1).

The bead also exhibits the fluorite diagnostic T_{2g} Raman band at 323 cm^{-1}, which is formed by phonon vibration of Raman activity. Therefore, this bead is most likely fluorite (Fig. 3B). Beside the diagnostic feature, a set of bands appears in the frequency-shift region between 1000 and 2500 cm^{-1}. These bands result from fluorescence of REE^{3+} ions in fluorite-structure hosts [8], supportive the identification of the analyzed specimen as fluorite.

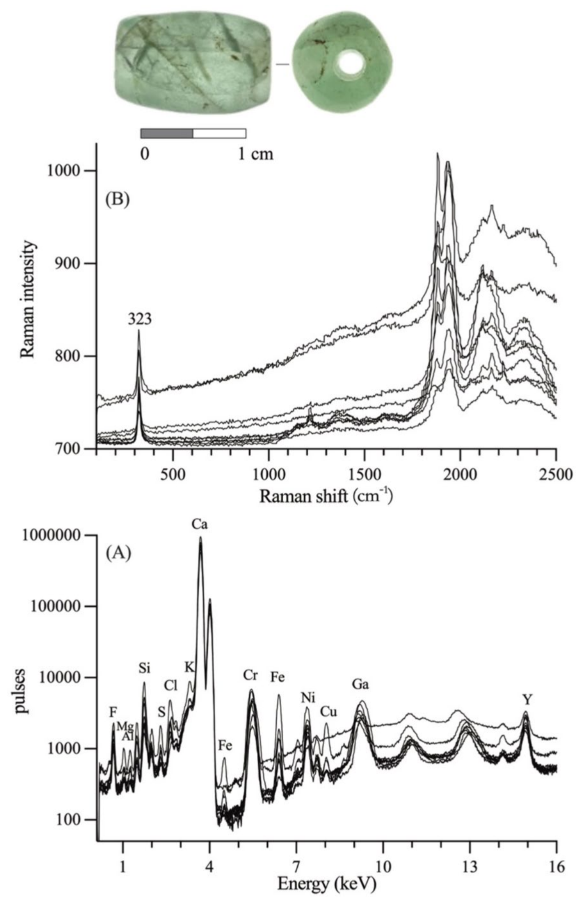

Fig. 3 **A** XRF spectrum of the bead of M099:5, showing the detectable elements of F, Mg, Al, Si, S, Cl, Ca, Mn, Cr, Fe, and Ga; **B** The Raman spectrum of the bead. The inset above is the specimen

Discussion

Distribution and early utilization of fluorites in China

Since the fluorite deposits in China enjoy high grades and are easy to exploit, China had the second-largest reserves around the globe, which was 46 million tons of reserve base [9, 10]. The identified fluorite resources are mainly distributed in northern and southeastern China (Fig. 4; [10]).

At least partly due to its available resources and ease of processing (with a Mohs hardness of 4), fluorite was one of the earliest gemstones utilized during the prehistoric period of China (Fig. 5). In the lower reaches of the Yangtze River, an arc-shaped fluorite pendant, *huang*璜, has been discovered at the contemporaneous Kuahuqiao site (6000–5500BCE, Fig. 5A, site 1 in Fig. 4), and

Table 1 Compositional analysis to the bead of M099:5 based on µXRF spectrum

Point#	F	Mg	Al	Si	*P*	S	Cl	K	Ca	Ti	Cr	Fe	Ni	Cu	Ga
M099:5-001	27.1	0.1	0.1	0.4	0.0	0.0		0.1	71.3		0.4	0.0			
M099:5-002	19.0		0.1	0.3	0.0	0.0			80.1		0.4	0.0		0.0	0.2
M099:5-003	16.8	0.1	0.1	0.4	0.0	0.0		0.1	82.0			0.1		0.0	0.1
M099:5-004	13.4	0.1	0.2	0.6	0.0	0.0		0.1	84.8	0.0		0.1		0.0	0.1
M099:5-005	14.9	0.5	0.7	1.5	0.1	0.1	0.1	0.3	79.2	0.1	0.6	0.3		0.1	0.2
M099:5-006	13.6	0.2	0.3	0.8	0.0	0.0	0.1	0.2	83.3	0.0	0.6	0.0		0.0	0.2
M099:5-007	13.6	0.2	0.4	1.0	0.0	0.0	0.1	0.1	82.9	0.0	0.6	0.1		0.0	0.3
M099:5-008	14.7	0.3	0.3	0.8	0.1	0.0	0.0	0.1	82.7	0.0	0.4	0.1	0.0	0.0	0.2
M099:5-009	12.3	0.5	1.0	2.4	0.1	0.0	0.1	0.3	81.0	0.1	0.6	0.3		0.0	0.2
M099:5-010	13.0	0.3	0.6	1.3	0.1	0.0	0.1	0.2	83.3	0.0		0.1		0.0	0.1
Mean	15.8	0.2	0.4	1.0	0.1	0.0	0.1	0.2	81.1	0.0	0.5	0.1	0.0	0.0	0.2
Std.	4.4	0.1	0.3	0.6	0.0	0.0	0.0	0.1	3.8	0.0	0.1	0.1	0.0	0.0	0.0

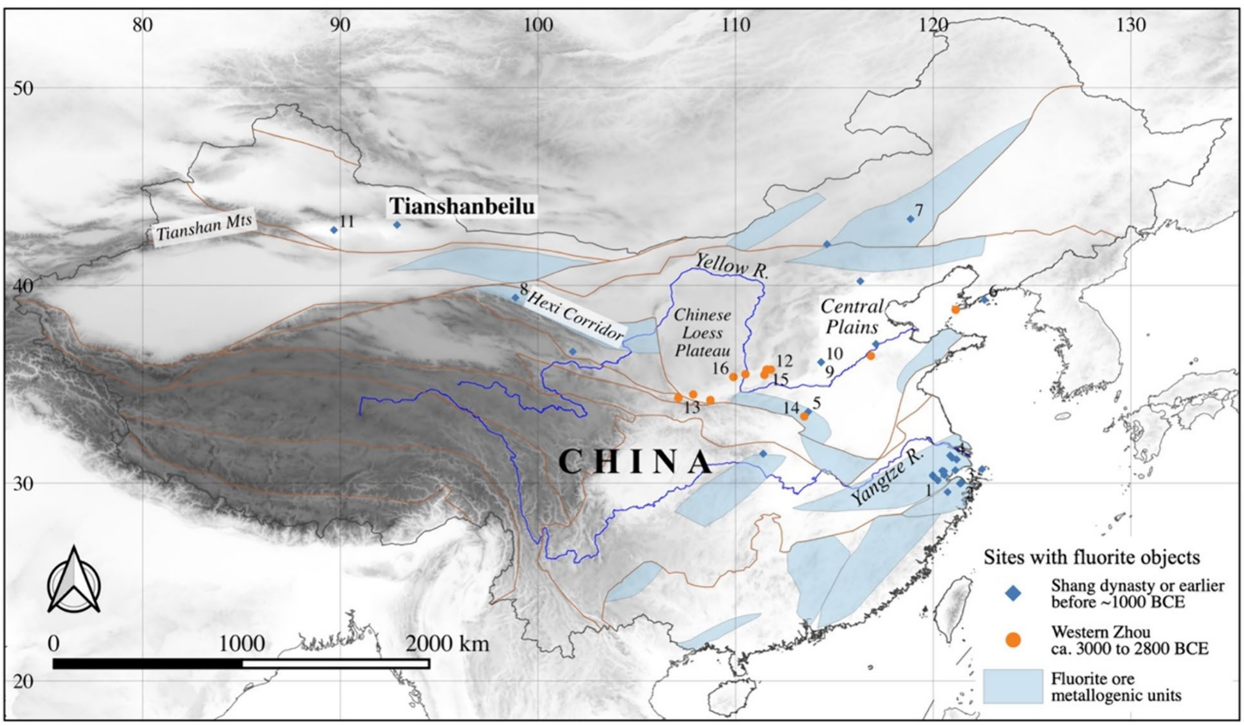

Fig. 4 Distribution of fluorite artifact sites in ancient China from prehistoric through Western Zhou dynasty. The fluorite ore metallogenic units in China are adopted from references [10]. Sites presented in the map: 1 Kuahuqiao [11]; 2 Tianluoshan [12]; 3 Fujiashan [13]; 4 Zhaolingshan [14]; 5 Jiahu [15]; 6 Xiaozhushan [16]; 7 Narisitai [16]; 8 Ganguya [17]; 9 Yinxu [18]; 10 Fuhao [19]; 11 Yanghai [20]; 12 Dahekou [21]; 13 Rujiazhuang [22]; 14 Yingguo [23]; 15 Beizhao [21]; 16 Liujiawa [24]

penannular fluorite pendant, *jue*玦, has been unearthed from the Hemudu culture Tianluoshan (Fig. 5B, s.2 in Fig. 4) and Fujiashan sites (Fig. 5C, s.3 in Figs. 4 and 5000–4000 BCE). Slightly later, Liangzhu culture site at Zhaolingshan (Fig. 5D, s.4 in Figs. 4 and 3000–2300 BCE) revealed pieces of fluorite inlay. On the Central Plains, 28 fluorite ornaments have been unearthed from 9000-year-old Jiahu site (Fig. 5E, s.5 in Fig. 4), among which the cross-section of the fluorite beads is circular. On the eastern margin of the Mongolia Plateau, a fluorite fish-shaped ornament had been found at the Hongshan culture Narisitai site (Fig. 5F, s.7 in Fig. 4). By the late 2nd millennium BCE, spherical fluorite beads were found at Xiaozhushan site (Fig. 5G, s.6 in Fig. 4) at the

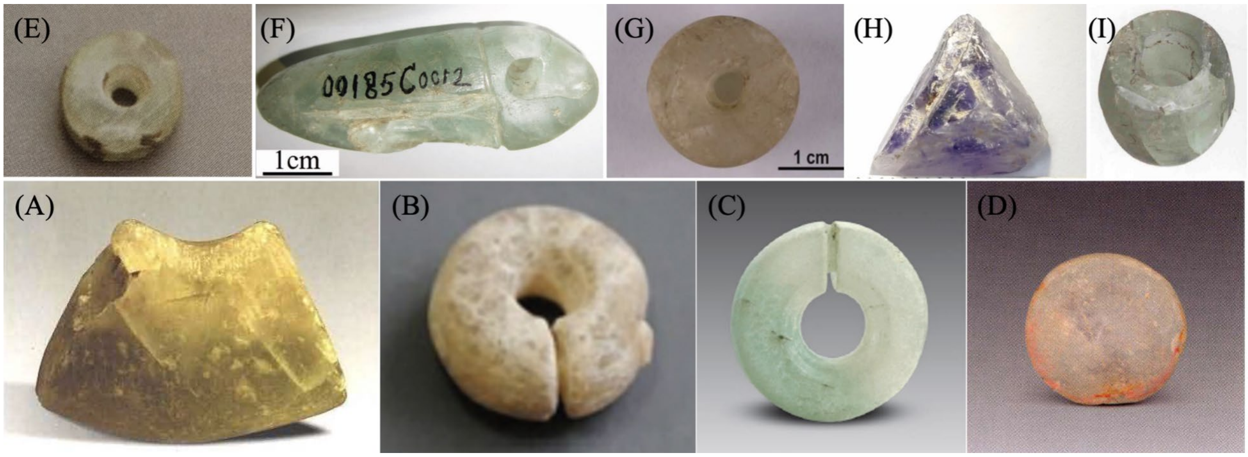

Fig. 5 Selected early fluorites in China. South China. **A** Kuahuqiao [11], T0152Hu IV:1; **B** Tianluoshan [12], T203 [7]:4; **C** Fujiashan [13], T121 [8]:14; **D** Zhaolingshan [14], M18:32; North China: **E** Jiahu [15], M318:1–3; **F** Narisitai [16], 00185C0012; **G** Xiaozhushan [16], 2009LCX T1311 [9]:5; **H** Ganguya [17], M60:8–13; **I** Fu Hao tomb [19], 1976AXTM5:1250

coastal area and a piece of fluorite ore was excavated at the Ganguya site (Fig. 5H, s.8 in Fig. 4) in the inland Hexi Corridor. The most notable is that a barrel fluorite pendant was unearthed at the tomb of Fu Hao (Fig. 5I, s.9 in Fig. 4). Besides its form of barrel, its size also is usually large, measuring 4.23*4.85 cm.

Comparing with the fluorite distribution map of China (Fig. 4), sites containing fluorite are mainly located in the eastern part of China, and most sites prior to the Western Zhou are situated not far from the fluorite ore metallogenic units [10]. For example, the pre-Zhou dynasty sites utilizing fluorite are mainly concentrated in the region along the middle and lower reaches of the Yangtze River (Fig. 4), where the fluorite ores widely distributed [10]. Some researchers argued that ancient people first used low-hardness materials including fluorite (Mohs hardness, 4) and steatite (Mohs hardness, 1), followed by high-hardness materials like agate and nephrite [21, 25]. Before the rise of nephrite jade at the end of Majiabang culture (ca. 4000 BCE), fluorite once dominated the materials for personal ornaments in the Lower Yangtze River Basin [21]. During this period, the Yin Ruins site (s.9 in Fig. 4), as well as Fu Hao's tomb (s.10 in Fig. 4), stands out as a notable exception, being far from any mining areas, and the form of fluorite unearthed there is significant different from those found at other sites (Fig. 4).

The fluorite bead presented here is circular in cross section and is classified as long bi-truncated barrel shape based on the length to width ratio [26]. Judged from the associated pottery and stratigraphy, the grave bearing the fluorite bead, M099, is assigned to the Tianshanbeilu Phase III (personal communications with Dr. TONG Jianyi), corresponding to 1385–1256 cal BC [2]. To the best of our knowledge, the bead excavated from the Tianshanbeilu cemetery is the earliest among its type in China.

Slightly later, the Early Iron Age Yanghai cemetery (Fig. 6A, B and s.11 in Fig. 4), has yielded short barrel beads from its Phase II grave (ca. 10–8 century BCE, corresponding to the Western Zhou dynasty in the Central Plains) and biconical fluorite beads from the Phase III grave (ca. 7–4 century BCE, comparable to the Eastern Zhou dynasty in the Central Plains). Almost at the same time, within the territory of the Zhou Dynasty, including the Central Plains and the southern part of Chinese Loess Plateau, fluorite was widely adopted as a part of stringed beads consisting jade pendant set组玉佩. In general, jade pendant sets are typically unearthed in the most élite burial sites and thus bear ritual significances. Many scholars linked the fundamental changes in type, forms, decorations, functions of jade artefacts, and bronze as well, with the ritual reform occurred around 1000 BCE [27–29]. During this period, the Five-Color system of blue-green, red, white, black, and yellow, had constituted an essential component and was applied as symbolic colors in various ritual scenarios [30, 31]. Regarding to jade pendent set, yellow fluorites may be substitutions of other yellow materials such as yellow agate, as proposed by Wang and colleagues [21].

The arguments can be supported by numerous archaeological materials in the Western Zhou dynasty (Fig. 6C and H). Jade pendant sets with fluorite beads include stringed beads from the middle Western Zhou dynasty Dahekou (s.12 in Figs. 4, [21], Rujiazhuang (s.13 in Fig. 4), and Yingguo (s.14 in Fig. 4) cemeteries, as well as the late Western Zhou cemeteries of Beizhao (s.15 in Fig. 4) and Liujiawa (s.16 in Fig. 4).

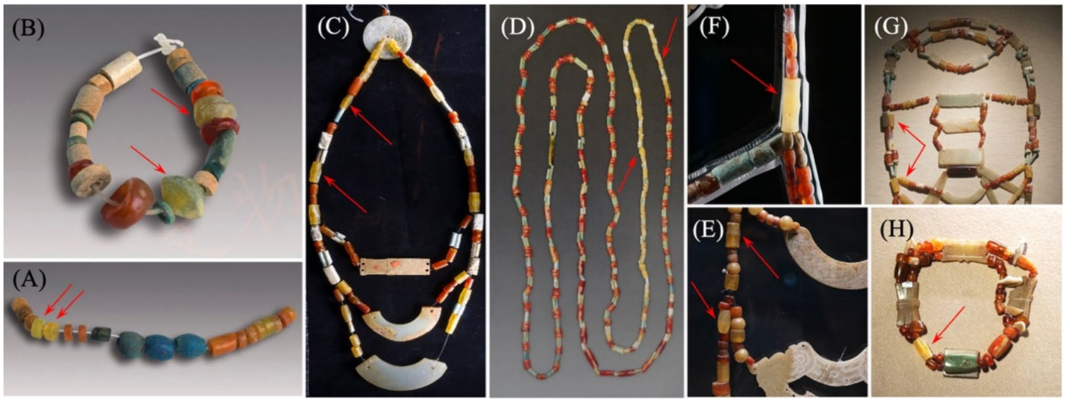

Fig. 6 Selected Western Zhou fluorites. **A** Yanghai [20], IM20:7; **B** Yanghai [20], IIM144:5; **C** Dahekou [21], M2:49; **D** Rujiazhuang [22], BRM2:64; **E** Yingguo [23], M6; **F** Beizhao [21], M31; **G** Beizhao [21], M92; **H** Liujiawa [24], M19

The presence of yellow fluorite beads among the Western Zhou dynasty stringed ornaments suggests the emergence of a distinct tradition of fluorite use during this period. In addition to the shift in utilized fluorite colors, the diverse fluorite forms found in earlier archaeological sites (Fig. 5) are largely absent, with most fluorite appearing in either barrel or biconical shapes (marked by arrows in Fig. 6C and H). Furthermore, whereas pre-Zhou sites containing fluorite artifacts were generally located near geological sources of the mineral, the Western Zhou sites that contained fluorite beads were generally located a considerable distance away from known fluorite deposits (Fig. 4). This distribution implies the rise of long-distance trade and exchange networks facilitating the transport of fluorite across China.

Such fluorite beads likely originated from the eastern Tianshan Mountains including the Tianshanbeilu cemetery presented here, and the Yanghai cemetery to the west as well. The fluorite beads spread into Western Zhou territory would benefit from complex cultural contacts between the Zhou people and their neighbors [32]. As exotic cultural elements, fluorite beads in barrel and bi-conical shapes, carnelian beads [33], faience [34], black jet [31], chariots and horse-riding [35], gradually spread eastward from the sites on the Eurasian steppe such as the oasis of Hami where the Tianshanbeilu is situated, and finally fused into Chinese traditional styles.

Furthermore, the complete cleavage of fluorite crystals results in the brittleness of fluorite, making it difficult to work into large-size artefacts or elongated holes with tapered drill. Compared to the fluorite ornaments of the Shang Dynasty or earlier, the emergence of barrel-shaped fluorite beads during the Western Zhou period significantly increased the ratio of hole length to hole diameter, indicating a substantial improvement in fluorite processing technology in the early Western Zhou era.

The specimen M099:5 shows circular remains clearly visible in both ends of the drill holes, indicating that the bead was drilled through two ends by tubular drill (Fig. 7). The Tianshanbeilu cemetery unearthed a large quantity of bronze and copper artifacts, with at least 79 out of 705 tombs containing numerous copper-based tubes about 5 mm in outer diameter. Among these, Tomb M125, which dates to the same period as Tomb M099, contained seven such bronze tubes [36]. We speculate that the fluorite processing in Tianshanbeilu utilized bronze tubular drilling technology, and this technique was later transmitted eastward into the Central Plains region.

Admittedly, the solid connections between the Tianshanbeilu cemetery and Western Zhou ritual material are still insufficient at this stage. The fluorite bead reported here is merely a starting point, the relevant issues warrant more body of evidence.

Conclusion

Through µXRF and Raman spectroscopic analysis, we have identified a fluorite bead among the artifacts from the Bronze Age Tianshanbeilu cemetery in eastern Xinjiang, China. This specimen represents the earliest known long barrel fluorite bead uncovered in China thus far.

Comparative analysis of fluorite beads from Western Zhou élite burials with those from earlier sites reveals different patterns: beads from earlier sites are often proximal to fluorite mines, whereas those from Western Zhou burials show a less direct association with the mine locations. This suggests an intensified long-distance exchange of objects such as fluorite during the Western Zhou period.

Taken the forms and the chronology of the unearthed fluorite into consideration, the Tianshanbeilu fluorite bead predates Zhou dynasty specimens and may have

Fig. 7 **A** Round-folded tin bronze tube from tomb M125; **B** a narrow ring remain in the end of one perforation; **C** a narrow ring remain in the end of another perforation

influenced the distinctive bead styles that subsequently emerged. Contact with Eurasian steppe cultures likely facilitated this growth of extensive trade networks.

Acknowledgements
We also extend our appreciation to director YAN Feng for his kind supports, to TANG Xu for µXRF laboratory assistance, and WANG Lan for his photo courtesy.

Author contributions
Materials, KR, MS, and ZT; Funding acquisition, ZT; Writing—original draft, ZT; Writing—review and editing, MY, CQ, and XL; Investigation, MY, CQ, XL and ZT; Figure preparation, MY, CQ, ZT. All authors read and approved the final manuscript.

Funding
This research is funded by National Natural Science Foundation of China (Grant No. 42072210) and IGGCAS (IGGCAS-201905).

Data availability
All data generated or analyzed during this study are included in this published article.

Declarations

Competing interests
The authors declare no competing interests.

Received: 2 March 2024 Accepted: 16 July 2024
Published online: 26 July 2024

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