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Nondestructive analysis of faience beads from the Western Zhou Dynasty, excavated from Peng State cemetery, Shanxi Province, China

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The faience in China suddenly appeared in the Western Zhou Dynasty (1046–771 BC), and its production is considered to be influenced by the West. In this paper, the microstructure and chemical compositions obtained by synchrotron radiation micro-computed tomography (SR-μCT) and μ-probe energy dispersion X-ray fluorescence spectrometry (EDXRF) were combined to disclose the manufacturing information of faience beads excavated from Peng State cemetery in Hengshui, Shanxi Province, China, dated to the Western Zhou Dynasty (1046–771 BC). Based on inner structural features obtained by SR-μCT, it was found that these faience beads could be divided into two types: glazed faience and glassy faience. According to the structural information revealed by the CT slices, it is inferred that these beads were first formed on an organic cylinder and then glazed using the direct application method. The possible sources of copper colorant are copper ores. In addition, the glaze chemical compositions are distinct from Na_2O–CaO–SiO_2 glaze or glass in the West, and thus, Western Zhou faience should have an indigenous origin in China. Furthermore, the manufacturing features are consistent with the techniques of proto-porcelain during the same time period, but the glaze recipe is distinct from that of proto-porcelain and early glass in China. Consequently, it is proposed that faience in the Western Zhou Dynasty was not the precursor of early glass in China.

1 Introduction

Faience, namely glazed quartz, was produced in many ancient civilizations, such as ancient Egypt, Mesopotamia, Indus Valley and China.[1–4] The Chinese faience, also called "liaoqi" or "artificial quartz beads" by Chinese archaeologists,[5] suddenly appeared and became popular in the Western Zhou Dynasty (1046–771 BC), at least 2000 years later than in the West. The Chinese faience production continued throughout the Spring and Autumn period (770–476 BC) and the Warring State (475–221 BC) until the Han Dynasty (202BC–AD220).[4] Most faience artifacts in the Western Zhou Dynasty are perforated small beads, which were found in Northern China and used for forming fabulous necklaces with jade as a symbol of status and wealth.[6] Some scholars think the manufacturing technology of Chinese faience may have been influenced by West Asia and Egypt in terms of the chemical composition results[7,8] and the cultural connections between China and the West 3000 years ago.[9–12] Given that chemical compositions of faience glaze are similar to those of early glass and that faience predates glass in ancient Egypt, the faience in the West is thought to be the precursor of ancient glass in the West.[13,14] Similarly, the faience in China is also considered to be the precursor of glass or even proto-glass.[15,16] Obviously, studies on early Chinese faience will contribute much to further our understanding of the origin of Chinese glass and the cultural communication between the West and the East.

Previous studies on Chinese faience in the Western Zhou Dynasty have mainly focused on the chemical composition of the glaze, which contributed to the investigation of the raw materials used in faience production.[5,17,18] In addition, X-ray diffraction analysis (XRD) was undertaken to distinguish Chinese faience from other glazed products. The XRD results indicated that faience mainly shows the diffraction peak of quartz, but other glazed products exhibit diffraction peaks of some other mineral materials.[6,15,17] As some clay remained in the perforation, it was proposed that Chinese faience beads were formed by a modeling or core forming technology.[18] However, it is difficult to judge whether the remaining clay was introduced in the burial environment or not. In fact, scientific evidence for the shaping and glazing methods employed in Western Zhou faience production is still lacking.

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Scanning electron microscopy (SEM) combined with attached analytical techniques is usually used to examine polished cross-sections of both ancient faience samples and laboratory replicates. Based on the analytical and microstructure data, raw materials and methods of fabrication employed in ancient Western faience production have been deduced.[19–22] The natron, plant ash and mixed flux were used as fluxes.[23–25] There are three glazing technologies for faience, namely direct application, efflorescence, and cementation methods, but it is very difficult to confirm by SEM which methods were used in glazing.[1,14,26,27] Recently, optical coherence tomography (OCT) was used to analyze the microstructural features of faience and provide some useful information to determine the glazing methods applied in ancient Egyptian faience.[28] In addition, synchrotron radiation micro-computed tomography (SR-μCT) has been applied to distinguish the material types and research the detailed manufacturing procedure of faience eye beads in China,[29] as well as to preliminarily analyze the fabrication technology of one Western Zhou faience bead.[30] Compared to SEM, SR-μCT could provide inner structural information of faience beads from different angles nondestructively.

Peng State cemetery, located in Hengshui, Jiang County, Shanxi Province, China, is a famous archaeological site of the Western Zhou Dynasty. This site was excavated from 2004 to 2007 and won the honor as one of the top 10 archaeological discoveries of 2005 in China. Many faience beads and tubes were discovered at this site, and most of them were found near the neck or stomach of the deceased, indicating that the faience beads were part of a necklace.[31]

In this study, SR-μCT was applied to determine the manufacturing procedure used to fabricate the faience beads from Peng State cemetery. Energy dispersive X-ray fluorescence (EDXRF) was used to analyze chemical compositions and determine the glaze recipe. In the following sections, the chemical compositions of early faience, glass and proto-porcelain, which have been previously published, will be brought together to discuss their relationship and explore the development of Chinese faience.

2 Materials and experiment

2.1 Samples

Sixteen faience beads were selected from five tombs of Peng State cemetery. These beads were first observed under an optical stereomicroscope, and the exterior blue-green glassy surface was interspersed with many semi-opaque quartz particles. All the beads were scanned by using SR-μCT, and EDXRF was used to examine the glaze of six typical beads, which are described in Table 1 and Fig. 1. The diameter of these faience objects ranges from 4 mm to 12 mm; the length varies from 5 mm to 13 mm. Before any experiment, the beads were cleaned in an ultrasonic tank using deionized water to remove attached soils.

Table 1 Description of some ancient Peng State faience beads

Sample	Description
M2161-1	Like a jujube pit; blue outside with a quartz body; length, 8.16 mm; aperture, 2.7 mm; diameter, 5.57 mm
M2036-1	Tube; blue outside with some remarkable setting marks; some cinnabar adhering to the inner layer; length, 12.75 mm; aperture, 2.6 mm; diameter, 4.65 mm
M2047-1	Like an egg; blue and green outside; aperture, 3.43 mm; diameter, 6.12 mm
M2055-2	Fragment of a tube with a blue surface; white quartz body; length, 6.59 mm; diameter, 4.71 mm
M2002-1	Like an abacus bead; blue and green outside; length, 11.95 mm; aperture, 2.64 mm; maximum diameter, 11.63 mm
M2047-2	Like an egg; aperture, 3.01 mm; diameter, 5.78 mm. It naturally broke into two parts after examination due to a crack on the body

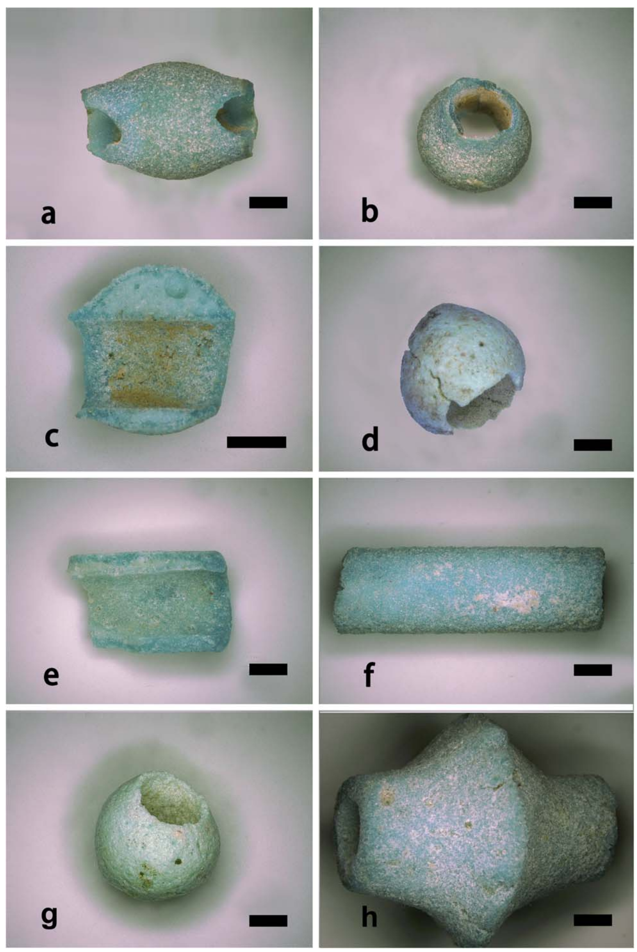

Fig. 1 Photographs of some typical faience objects. (a and b: M2161-1; c and d: M2047-2; e: M2055-2; f: 2036-1; g: 2047-1; h: 2002-1. The scale bar is 2 mm.)

2.2 SR-μCT scanning

The faience beads were scanned by SR-μCT at the Shanghai Synchrotron Radiation Facility (SSRF), Shanghai, China. During the experiment, the scanned beads were placed on an open sample platform. The parallel SR X-ray with the height of 4 mm and width of 2 cm was directed at the object with a source

Table 2 The chemical compositions of the faience glaze and glass (wt%)

Sample	SiO_2	K_2O	Na_2O	CaO	CuO	Fe_2O_3	Al_2O_3	P_2O_5	MgO
M2161-1	82.97	1.37	1.88	4.48	2.63	0.62	1.85	0.31	3.06
M2036-1	94.16	0.43	1.29	0.09	1.84	0.14	1.26	0.21	0.41
M2047-1	88.08	1.07	1.55	0.79	1.47	0.18	5.46	0.29	0.84
M2055-2	90.05	2.02	1.17	0.28	1.61	0.17	2.93	0.35	1.18
M2002-1	91.23	0.86	1.22	0.28	2.59	0.29	1.76	0.31	0.74
M2047-2	90.87	0.6	2.51	0.33	1.76	0.18	2.36	0.29	0.9
G6-Fengxi[5]	94.0	0.3	0.3	0.4	0.8	0.4	0.7	—	0.2
G1-Luoyang[5]	>90	3.4	1.2	0.4	1.6	0.2	0.3	—	0.3
HNWII 67-Ying State[17]	92.98	0.51	1.38	0.30	2.06	0.31	1.26	0.47	0.68
Jiangling[4]	71.63	10.92		2.95		1.99	6.53		

energy setting of 30 keV. The CCD detector had a space resolution of 9 μm. The scan time was about 10 minutes. Scan data were imaged and analyzed using Mimics 12 (Materialise, Belgium). Micro-CT produces the slice of an object in terms of the differences in the absorption differences of X-ray, and the variation of brightness on the slice reflects the variation in density and chemical composition.[32]

2.3 EDXRF analysis

Chemical composition analysis was performed on an Eagle III μ-Probe EDXRF spectrometer (EDAX, USA) with a Mo tube and a 125 μm Be window, which had been calibrated using appropriate primary standards. The detector was a liquid-nitrogen-cooled Si (Li) crystal with a resolution of about 160.3 eV at Mn K. The operation conditions of the X-ray tube included an accelerating voltage of 50 kV and current of 800 μA. The X-ray beam spot was set at 0.3 mm. The incidence angle of the spectrometer was 65°, and the emergence angle was 60°. VISION32 software with all the basic EDX functions was used to analyze the obtained spectra. The concentrations of the analyzed major and some minor elements were beyond the detection limit of 20 ppm. The relative errors were 1–3% for elements present at the level of 1 wt% or more, and up to 10% for elements present at the level of 0.1 wt% or less. All the samples were tested in a vacuum atmosphere. The results of six representative faience beads are listed in Table 2 with some previously published data of faience glaze in the Western Zhou Dynasty and glass of the Warring State period.

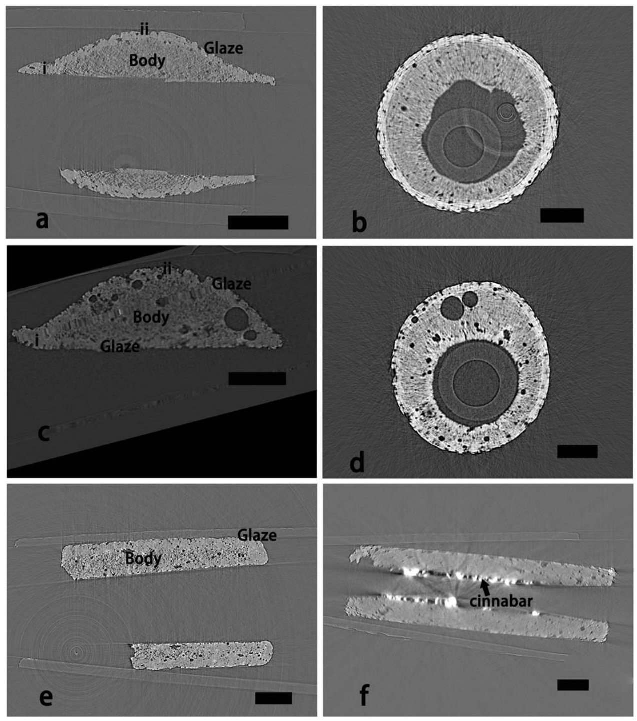

Fig. 2 The CT slices of two different faience types (a and b are the longitudinal section and cross section of sample M2161-1; c and d are the longitudinal section and cross section of sample M2047-2; e is one longitudinal section of sample M2055-2; f is one longitudinal section of sample M2036-1. The scale bar is 2 mm).

3 Results and discussion

3.1 Identification of faience type

As the glaze contained greater amounts of heavy elements such as K, Ca, Fe and Cu (Table 2), it will have a higher X-ray absorption than the quartz body. Thus, the glazed area will be brighter than the quartz body on the CT slices, and the air pores are black. According to structural characteristics reflected by the CT slices (Fig. 2), the faience beads could be divided into two types. Type A: glazed faience. There is an apparent glaze on the bead surface (Fig. 2a–e). Five of six faience objects belong to type A, and the typical CT slices are shown in Fig. 2a and b. Moreover, one glazed faience bead (M2047-2) was glazed on both the outer surface and the inner wall of perforation (Fig. 2c and d). Type B: glassy faience,[20] in which there is no distinct outer layer from the interior. M2036-1 belongs to type B (Fig. 2f).

As shown in Fig. 2, the glassy faience bead (Fig. 2f) is of higher density compared with glazed faience in which air pores and crevices can be clearly observed in the CT slices. The glazed faience artifacts are also distinct in the inner structure. The inner structures of M2161-1 (Fig. 2a and b) and M2055-2 (Fig. 2e) are different from that of M2047-2 (Fig. 2c and d), which contains some big air pores. Micro-CT provides a convenient method to differentiate the glaze, quartz body and air pores. Different types of faience can be easily differentiated in a nondestructive manner, and the differences in the inner structure can also be clearly shown in CT slices. The structural evidence, obtained by examination of CT slices, provides a very effective approach to investigate the overall inner structure of faience.

3.2 The manufacturing process

As shown in Fig. 2a, c, e and f, the longitudinal sections of all faience beads share a similar characteristic, in that the profile lines of the perforation are straight lines. The cross-sections of all faience beads are also similar, since the transverse section of the perforation resembles a circle (Fig. 2b and d). The structural information reflected by CT slices suggested that the shape of the perforation resembles a cylinder. Thus, some kinds of cylindrical inner core should be used to support the perforation during the shaping and firing process. Furthermore, the inner core in the perforation should be composed of organic materials. Otherwise, it would be very difficult to strip, because the clay or metal core would be sintered together with glaze or quartz particles during firing. The shaping procedure of the quartz body could be described as follows: quartz particles were piled on a cylindrical stick and then glazed.

Previous scholars have shown in laboratory replications that three different glazing methods were used in glaze production. For the application glazing method, a slurry is prepared first. The glaze is then applied either by dipping the faience body in the slurry or by pouring the slurry onto the body.[22] For the efflorescence glazing method, the faience was made from a mixture of raw materials including quartz, lime, alkali and colorant. During drying, the soluble salts migrated to the surface, and these salts deposited as an efflorescence layer. Upon firing, when drying is more rapid, more salts were deposited, and thus, the glaze became thicker. However, under the base and the interior wall where drying is slower, the glaze is thinner.[34] For the cementation glazing method, the quartz body was fired at a special glazing power. During firing, glaze constituents like alkalis migrated to and reacted with the quartz surface. After firing, the fused cementation mixture would be wiped off.[29] Although the criteria used to identify the glazing method must be used with caution, the structural evidence, based on the examination of numerous laboratory replications, suggests the variations in glaze thickness would provide some positive information to determine the possible glazing methods used for ancient faience production.[21,22]

The analyzed beads in this study were prepared using a similar glaze recipe (Table 1), and thus, it is deduced that the same manufacturing technique, including the glazing and firing methods, was used. The structural information provided by micro-CT indicated a possible glazing method in faience bead production.[29] As shown in Fig. 2, the glazed faience beads are of variable glaze thickness (Fig. 2a–e), and the glaze at the rim (Fig. 2a position i and Fig. 2c position i) is much thicker than that at other positions (Fig. 2a position ii and Fig. 2c position ii). In addition, the glaze on the outer surface of the special glazed faience bead M2047-2 is of similar thickness with that of the interior layer. These results suggest that glazed faience beads were not be glazed using the efflorescence glazing method, for which the glaze on the outer surface and at rims where the air flows during drying are of the same thickness.[19,22] As the faience was formed on the cylindrical core first, no glaze cover the interior layer or the glaze thickness of the interior layer being the thinnest for drying rate of the interior was much slowly than the outer surface during the efflorescence process. As for the application glazing method, the glaze slurry was applied by painting or dipping. The glaze slurry will flow to the rim during the application process, and thus, the glaze is thickest at the rims after firing.[21] During the dipping process, the slurry would enter the perforation. Consequently, the interior surface would be glazed after firing, as for M2047-2 in Fig. 2c and d. In addition, cementation glazing is characterized by a uniform glaze thickness,[21] and there is no grinding mark in the glaze of those beads under optical stereomicroscopy. Based on the above discussion, it is more likely that glazed faience was glazed using the application method. The possible manufacturing process used to fabricate these faience beads is as follows: the quartz body is formed on a cylindrical stick first, and then the glaze is applied by painting the glaze slurry onto the body or dipping the body in the glaze slurry.

The manufacturing process of faience seemed to be influenced by proto-porcelain production technology. Proto-porcelain production began at least since the Shang Dynasty (1600–1046 BC).[35] However, there are some differences in the application glazing methods between the analyzed faience beads and proto-porcelain. The proto-porcelain is much larger than faience beads. The lip of the porcelain bottom is always not glazed, because that position was in contact with the kiln ground. However, the surfaces of the analyzed faience beads are totally coated with a thin layer of glaze. According to the simulation experiment, when a faience bead was placed directly on the ground before firing, the setting place of the bead would appear grey due to the lack of contact with air during the firing process. Since the faience beads were shaped on a stick, it is recommended that these beads should be fired by hanging in the air and the stick may be used to support the beads.

3.3 The glaze recipe

From the chemical composition data of the faience glaze listed in Table 2, the Peng State faience glaze has a glaze recipe similar to that of other Chinese early faience beads that contain high contents of silicon (more than 80 wt%) and low contents of flux. The silicon should come from quartz. These faience beads are blue-green in color with copper and iron as color-generating elements. The copper content is about 2%, and the iron content is around 0.2%. When the Fe content is lower than 2 wt% in the glaze, it would have no significant influence on the glaze color.[36] Thus, the glaze color variation is deemed to be caused by different local chemical environments of Cu^{2+} ions in the glaze.[33] The faience beads in the Western Zhou Dynasty are the earliest examples of glaze with copper colorant in China. Some Chinese archaeologists thought the source of the copper colorant would be the copper slag.[37] As known, the bronzes in the Shang and Western Zhou Dynasties contained more than 15% Pb and Sn,[38] but very low Pb and Sn contents were detected

Table 3 Typical chemical compositions of Chinese faience, Egyptian faience and Chinese proto-porcelain (wt%)

	Date	SiO_2	Al_2O_3	CaO	MgO	(CaO + MgO)	Na_2O	K_2O	(Na_2O + K_2O)	CuO	Fe_2O_3
Chinese faience[18]	Western Zhou Dynasty	81–94	1–5				1	1		1–9	
Egyptian faience[22]	2000–700 BC	65–75				3–7			15–20		
Chinese proto-porcelain[40]	Western Zhou Dynasty	55–70	8–20	3–15	1–3			3–8			2–10

in these faience glazes. Therefore, bronze scale or copper slag was not used as the source of colorant. In sum, some copper ore, such as malachite or azurite, and the quartz could have been added to the glazing mixture.

Some scholars have summarized the chemical compositions of faience in the Western Zhou Dynasty (Table 3). The chemical compositions of the glaze analyzed in this study are not exceptional. The glazes of Egyptian and Near East faience are of the soda-lime-silicate type with soda as the dominant alkali,[22] and the typical glaze compositions are listed in Table 3. Compared with the glaze compositions of Western faience, the Chinese faience only contained 2–3% soda-plus-potash (Na_2O + K_2O), and the potassium was usually the dominant alkali.[18,39] Obviously, the glaze recipe of most of Western Zhou faience in China is distinct from faience in Egypt and the Near East. Therefore, the faience beads in the Western Zhou Dynasty were not imported from the West but had an indigenous origin in China. Also, the Chinese glaze was first coated on the surface of proto-porcelain during the Shang Dynasty (1600–1046 BC), and then it was applied on the faience. The development of Chinese glaze was different from that in the West, where the glaze was first coated on the surface of glazed steatite at the end of the 5th millennium BC.[21] However, as shown in Table 3, the glaze recipe of proto-porcelain is also different from Chinese faience. The porcelain glazes were light yellow and brown in color due to Fe, and it contained high contents of Ca or Fe.[40] The results indicate that the flux and colorant used in the glaze production of proto-porcelain are different from those in the glaze recipe of faience. Therefore, the proto-porcelain glaze does not have too much influence on the faience.

Western scholars have thought of the faience as the precursor of glass, because: (1) the faience predates glass; (2) faience glaze and glass have a similar recipe (Na_2O–CaO–SiO_2); and (3) some faience and glass have been discovered in the same workshop.[2] However, the situation in China is different. Glass production in China started from the late period of the Spring and Autumn and Warring State period (476–221 BC), and there were three types of glass manufactured during this period, including Na_2O–CaO–SiO_2 glass (imported from the West), PbO–BaO–SiO_2 glass (homemade, the most common type), and K_2O–CaO–SiO_2 glass (homemade).[4] Furthermore, as shown in Table 2, the K_2O–CaO–SiO_2 glass (Sample Jiangling in Table 2, Warring State period) has much higher flux contents than the glaze recipe of Western Zhou faience. Thus, although the faience also predates glass in China, the glaze recipe of Western Zhou faience is distinct from that of PbO–BaO–SiO_2 and K_2O–CaO–SiO_2 glass in the later period. Furthermore, the early Chinese glass objects in the Warring State period were produced by mold casting.[18] Therefore, Western Zhou faience has no direct relationship with PbO–BaO–SiO_2 glass. On the other hand, the Warring State craftsmen also produced faience eye beads, and the glaze recipe is similar to K_2O–CaO–SiO_2 glass recipes, implying that the appearance of K_2O–CaO–SiO_2 glass may have some relationship with faience production during the Warring State period (476–221 BC).[29] In the future, more emphasis should be placed on the study of faience and glass in the Spring and Autumn period (770–476 BC) to understand the development of faience in China.

4 Conclusions

The analysis of the faience objects selected from the Peng State cemetery of China suggested that most faience was possibly glazed using the application method, which enriched our understanding of the manufacturing information for Chinese faience. The faience can be divided into two types: glazed faience and glassy faience. The Chinese glassy faience was first reported in this paper. The possible source of copper colorant was copper ore. By the observation of CT slices, the possible manufacturing process of faience involved formation of the quartz body on a cylindrical stick first and then application of the glaze by painting the glaze slurry onto the body or dipping the body in the glaze slurry. This technology may be influenced by Chinese proto-porcelain production technology. However, the faience could possibly be fired by hanging in the air with the support of a stick.

With the beginning of faience production since the early Western Zhou Dynasty, most of Chinese ancient faience was of low flux contents, which was different from West Asia and Egypt faience, which is commonly associated with high sodium and low potassium. The research on the production technology of faience implied that most of the Chinese Western Zhou faience has an indigenous origin in China. The Western Zhou faience showed no direct relationship with the emergence of China's earliest home-made PbO–BaO–SiO_2 glass. With the development of faience production technology since the Western Zhou Dynasty, the faience may have some connection with the appearance of K_2O–CaO–SiO_2 glass during the Warring State period. The glaze was first applied in the proto-porcelain production during the Shang Dynasty, but the chemical composition results suggest that the glaze production technology of proto-porcelain does not have too much impact on the glaze production of faience. Determining the origin of Chinese faience glaze still requires more work.

The structural data obtained by SR-μCT provided valuable information on the glazing methods. The data reflected the overall structural features of ancient faience beads in a non-destructive manner and clearly showed the differences in the inner structure of different objects. The present study has shown that SR-μCT provided a new and effective approach to study the manufacturing technology of ancient faience.

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