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Provenance identification of the high-fired glazed wares excavated from the Late Jin Dynasty (Dong Xia State) sites in Russia's Primorye Region

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ABSTRACT

In this work, twenty-three pieces of high-fired glazed samples from the archaeological excavations of the Dong Xia sites in Russia's Primorye region were studied. In addition to visual observation, energy dispersive X-ray fluorescence (EDXRF) and field emission scanning electron microscopy (FESEM) techniques were jointly applied to the analysis of the chemical composition and microstructure of the samples. The major emphasis was placed on the provenance identification of the white, opalescent glazed, and celadon samples. Seven excavated fine whiteware samples were confirmed as the products from the Ding Kiln in the Jin Dynasty, and nine samples as the Jun-series wares. The most important conclusion out of the study is that two celadon samples with opaque glaze were proved to be the Ru Kiln wares. This finding implies that the Ru ware is identified for the first time in the Dong Xia sites of Russia's Primorye region. This work is a successful case of interdisciplinary cooperation in the medieval archaeometry of the Far East.

1. Introduction

This work is devoted to the high-fired glazed wares excavated from the Late Jin Dynasty (Dong Xia State) sites in Russia's Primorye Region. The Jin Dynasty lasted for 120 years (1115–1234 CE) and was first founded by Jurchen people. During its peak evolution, the Jin Empire covered an extensive area geographically, including the north of Huaihe River, the northeast of Qinling Mountains in China, and Russia's Far East of today. The economy of the Jin Dynasty was inherited mainly from the Northern Song Dynasty (960–1127 CE), and the ceramics and iron-smelting industries were prosperous. Russia's Primorye region of today was once under the jurisdiction of "Xu Pin Lu", an administrative division of the Jin Dynasty. From 1215 to 1233, this region was controlled by the Dong Xia State, a short-lived kingdom established in Northeast China by Jurchen warlord *Puxian Wannu* in 1215 during the Mongol conquest of the Jin Dynasty. It was eventually conquered by the Mongolians and was later put under the Liaoyang province by the Yuan Dynasty (1271–1368 CE). The locations of the capital of "Xu Pin Lu" of the Jin Dynasty and the capital Kai Yuan City of the Dong Xia State were tracked down to today's Krasny Yar of Ussuriisk in the downstream of the Suifen River. Over 30 mountain-walled towns, such as Krasny Yar, Shaiga, and Anan'evka etc., make up the main body of the relic sites of Russia's Primorye region (Artemieva and Usuki, 2010; Khorev, 2012). Most of the mountain-walled towns discovered in Russia's Primorye region were dated to the period of the Dong Xia. The high-fired glazed wares excavated from these sites shed new light on understanding people's lives in "Xu Pin Lu" and the Dong Xia State, and their commercial and cultural exchanges with the inland regions of China (Peng, 2016).

The archaeology of Russia's Far East, which is closely associated with the cultural relics studies of the XII-XIII century AD, had experienced a qualitative breakthrough in 1963 when the Russian scholar E.V. Shavkunov for the first time excavated the unique Shaiga site in the Primorye region. This relic site became the benchmark for the Medieval Archaeology in Russia's Far East. Many of these sites were discarded after usage for about 20 years. Fortress sites and fortification system used for defending the invasion of the Mongolians were discovered. Jurchen people created their unique traditions and culture, which were passed down even after the downfall of its political regime (Jilin Provincial Institute of Cultural Relics and Archaeology, Institute of History, Archaeology and Ethnography of the Peoples of the Far East, 2013).

At the Dong Xia sites in Russia's Primorye region of today, high-fired glazed wares were the common excavated relics. E.I. Gelman, a Russian

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scholar, provided valuable details regarding their type, number, style, chronological sequence, and the possible kilns that produced the excavated high-fired glazed wares (Gelman, 2005, 1999). According to the observation of Chinese archaeologist Peng Shanguo (Peng, 2013), the excavated greenish-whitewares came from the Hutian kiln in Jingdezhen of the Southern Song Dynasty (1127–1279 CE), and the wares with a glaze similar to that of the Jun wares were considered to have been produced by kilns in Henan, China. The coarse whiteware with engobe, the black-and-whiteware (underglaze ware with black patterns painted on the white engobe), and the black-glazed ware were attributed to the Jiang Guan Tun Kiln in Liaoyang, Liaoning Province of China. The excavated high-fired glazed wares included a large share of fine whiteware produced by the Ding Kiln in Quyang, Hebei Province of China (Peng, 2007).

Previous conjectures about the kilns that produced the wares excavated from the Dong Xia sites in Russia's Primorye region were mostly based on the visual comparative observation with no in-depth scientific investigations. However, the cutting-edge development trend of archaeology is the scientific and technical archaeology, which, in particular, allows one to determine the provenance using the state-of-the-art technologies.

In this work, twenty-three pieces of samples were studied, based on the archaeological excavations of the Dong Xia sites in Russia's Primorye region in 1972–2015 by the Institute of History, Archaeology and Ethnography of the Peoples of the Far East, Russian Academy of Sciences. In addition to visual observation, the chemical composition and microstructure of the samples were analyzed to determine the provenance of the excavated white, opalescent glazed and celadon shards. The research has provided a deeper insight into the provenance identification of the ceramic wares from the Dongxia Sites. Moreover, it has provided important evidence to understand the intrinsic features of the social and trade situations in the Dong Xia State. This work is also a case of interdisciplinary cooperation in medieval archaeometry of Russia's Far East.

2. Description and classification of the excavated ceramic samples

All twenty-three samples were excavated from the Dong Xia sites. The excavation site, year of excavation, color and texture of the samples are listed in Table 1. The pictures of the samples are shown in Figs. 1–4. The map showing the locations of the excavation sites is depicted in Fig. 5.

3. Experimental

3.1. Methods of study

The microstructure of the cross-sections was studied using an FEI Magellan 400 field emission extreme high-resolution SEM scanning electron microscope (FEI Co., US). The cross-sections were ground and polished, then ultrasonically cleaned by water and ethanol, 3–5 times for 15 min. After that, all samples were dried in drying oven for 12 h. The polished cross-sections were lightly etched with 1% HF for 60s.

The chemical composition of the bodies and glazes was examined by energy-dispersive X-ray fluorescence technique via an EDAX Eagle III XXL spectrometer (produced by EDAX unit of AMETEK, Inc., US). EDAX EAGLE III was equipped with the 40 W (40 kV,1000 μA) X-ray tube. The diameter of X-ray focus was 300 μm, and Si(Li) detector was used to determine the characteristic X-ray. Surface scan mode was used for testing. The content of each oxide was the mean value of four data points of the sample. The counting time was 632 s. High voltage and low current (40 kV, 160 μA) was used to analyze the content of MnO, Fe_2O_3, CuO, ZnO, As_2O_3, PbO_2, Rb_2O, SrO, Y_2O_3, and ZrO_2. Low voltage and high current (12 kV,600 μA) was used to analyze the content of Na_2O, MgO, Al_2O_3, SiO_2, P_2O_5, K_2O, CaO, and TiO_2.

The calibration process involved several steps. First, a series of 13 standard samples were formulated using the standard geological minerals. With the test results of the 13 standard samples, the calibration curves were established for each element to represent the relationships between the intensity of the element characteristic peak and the element concentration. With the calibration curves available as references, the EDXRF data were calibrated accordingly. The oxides with more than 0.1 wt% content were listed in Table 2.

2D correspondence analysis, which is one of the multivariate statistical analysis methods, was used to process the data, following the method of Luo H. (Luo, 1997). 2D correspondence analysis is developed on the basis of factor analysis, which combines the characteristics of r-mode and q-mode analysis. 2D correspondence analysis can cluster samples, and can project variables and samples on one factor plane. Through 2D correspondence analysis, we can not only see the classification relations of different samples, but also see the main differences of chemical composition of different samples.

3.2. Methods for determining the provenance of the excavated ceramic samples

Firstly, the chemical compositions of the bodies and glazes by EDXRF were compared to those of the reference wares by EDXRF. Samples from the Ding kiln, Jun kiln, Ru kiln, Southern Song Guan kiln and Longquan kiln were used as reference wares for comparison. The chemical compositions of the reference wares by EDXRF were from the Ancient Ceramics Database of Shanghai Institute of Ceramics, Chinese Academy of Sciences. The information of the reference samples is shown in Table 2. The chemical composition data of the reference wares are presented in supplementary online information.

If the chemical compositions of a sample were consistent with the reference data, a preliminary conclusion was made concerning a possible kiln of producing the ceramic ware. Moreover, the microstructure analysis was further carried out and compared with the microstructure of the sample from the target kiln. If both the chemical composition and the microstructure were consistent with those of the wares from the target kiln, the exact provenance of a ware shard was identified.

4. Results and discussion

4.1. Chemical compositions of the excavated ceramic samples

The chemical compositions of the excavated samples are given in Table 3.

4.2. Provenance of the excavated ceramic samples

4.2.1. Provenance of the excavated whiteware

The whitewares excavated from the Dong Xia site can be subdivided into coarse and fine whiteware species. This categorization of “coarse” and “fine” wares is based on whether or not a layer of engobe is applied by visual observation. Engobe can cover up the color and defects of the coarse body. However, fine whiteware has no engobe and the body is whiter and more compact than that of coarse whiteware. The appearance of the excavated fine whiteware is very similar to that of the whiteware produced by the Ding kiln from the late Northern Song Dynasty to Jin Dynasty. The fine whiteware was produced in large quantities by the Ding Kiln in the Northern Song and Jin Dynasties. Using the Ancient Ceramic Database of Shanghai Institute of Ceramics, Chinese Academy of Sciences, the chemical composition of the fine whiteware of the late Song Dynasty to Jin Dynasty was retrieved and compared to that of the excavated samples using the 2D correspondence analysis. The wares in the above database were excavated from the Ding Kiln in 2009 by a joint team of Peking University and Hebei Institute of Cultural Relics, which ensured a high reliability of the respective archaeological stratigraphy data.

Table 1
Description of the ceramic samples excavated from the Dong Xia sites.

Sample no.	Archaeological site	Year of excavation	Description
Coarse whiteware: thick and rough body, transparent glaze, a layer of engobe applied on the body and beneath the glaze			
1	Shaiga	1973	Coarse whiteware shard
2	Shaiga	1976	Coarse whiteware shard of a plate
3	Shaiga	1976	Coarse whiteware shard of a plate
Fine whiteware: thin, white and well-sintered body, transparent glaze			
4	Shaiga	1983	Fine whiteware shard of a bowl
5	Shaiga	1982	Fine whiteware shard of a bowl
6	Shaiga	1991	Fine whiteware shard of a plate
7	Anan'evka	1980	Fine whiteware shard of a bowl
8	KrasnyYar	2015	Fine whiteware shard of a plate
9	KrasnyYar	2015	Fine whiteware shard of a plate
10	South-Ussuriisk	2013	Fine whiteware shard of a plate
Opalescent glazed ware with copper-red decoration: gray body, a visible white interaction layer at the interface between the body and glaze			
11	Anan'evka	2002	Opalescent lavender-and-blue glazed shard, a green spot in the middle of the lavender area
12	South-Ussuriisk	2013	Opalescent lavender-and-blue glazed shard
13	South-Ussuriisk	2014	Opalescent lavender-and-blue glazed shard
Opalescent glazed ware: gray body,			
14	Shaiga	1974	Opalescent blue glazed shard of abowl
15	Ekaterinovka	1999	Opalescent blue glazed shard of abowl
16	Shaiga	1976	Opalescent blue-green glazed shard
Opalescent glazed ware: gray body, a visible white interaction layer at the interface between the body and glaze			
17	Shaiga	1974	Opalescent blue-green glazed shard of a bowl
18	KrasnyYar	2015	Opalescent blue glazed shard of a plate
Translucent glazed ware: thick gray body, a visible interaction layer between the body and glaze			
19	Ekaterinovka	1999	Translucent green glazed shard of a bowl
Opaque glazed celadon: thin gray body, thin glaze			
20	Shaiga	1972	Opaque, pale blue-green glazed shard
21	Shaiga	1972	Opaque, pale blue-green glazed shard of a bowl
ware with upperglaze black: white body, transparent glaze with upper-glaze decoration of black streaks			
22	Shaiga	2005	Shard of a bowl, blackened by smoke
Pottery with underglaze green: white body, transparent light green glaze with underglaze decoration of dark green pattern			
23	Shaiga	1990	Shard of a bowl

4.2.1.1. The difference between the coarse and fine whiteware. Firstly, the 2D correspondence analysis was applied to determine the chemical composition of the bodies and glazes of the coarse whiteware (sample 1–3 from the Shaiga site) and the fine whiteware (sample 4–10 from four sites, including Shaiga). The chemical compositions of both bodies and glazes differed significantly between the coarse and fine whiteware, which permitted a reliable differentiation of the above two ware types. Apparently, the two types of whiteware were different in both ingredients and formulae.

The significant differences between bodies of the coarse and fine

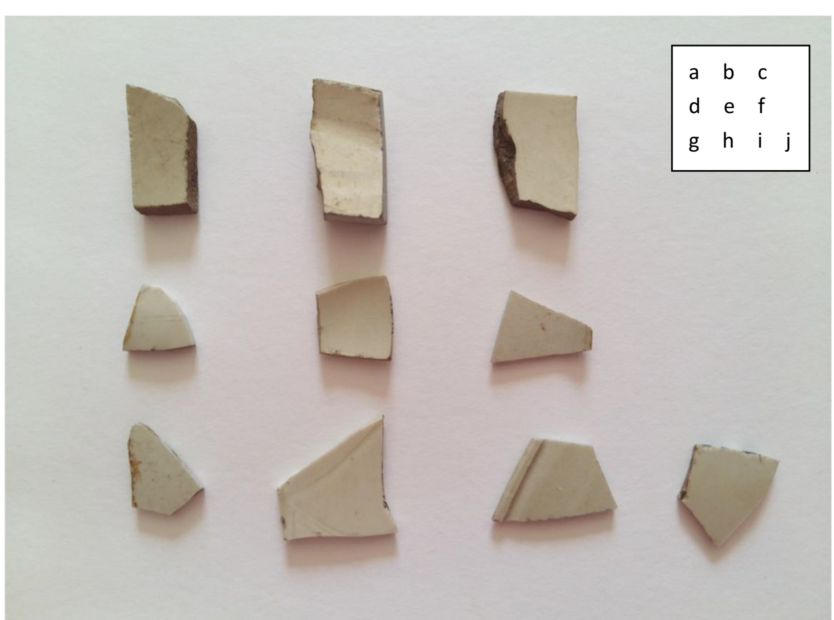


Fig. 1. Whiteware shards excavated from the Dong Xia sites in Russia's Primorye region. (a)–(j): Sample 1–10.

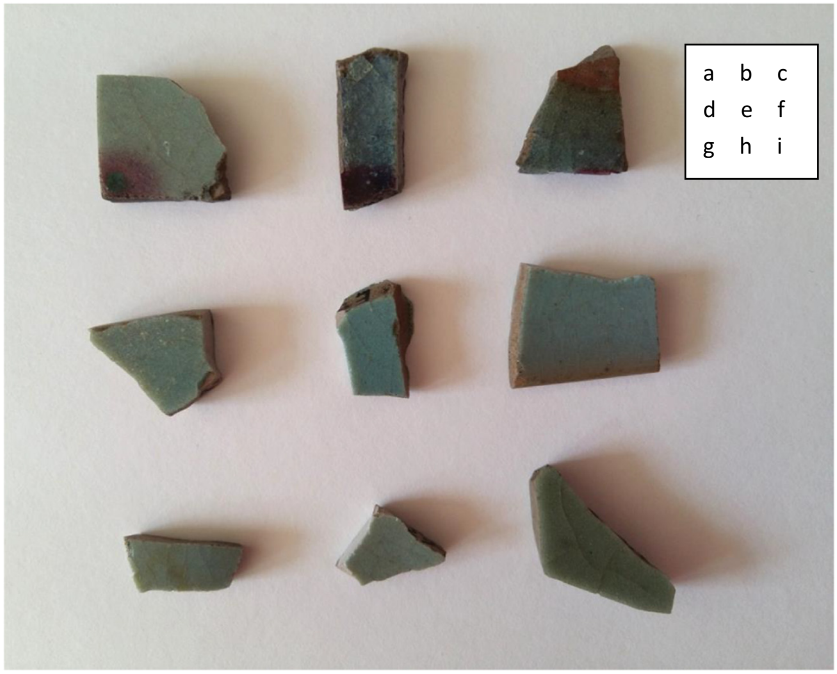


Fig. 2. Opalescent and translucent glazed shards excavated from the Dong Xia sites in Russia's Primorye region. (a)–(h): Sample 11–18, (i): Sample 19.

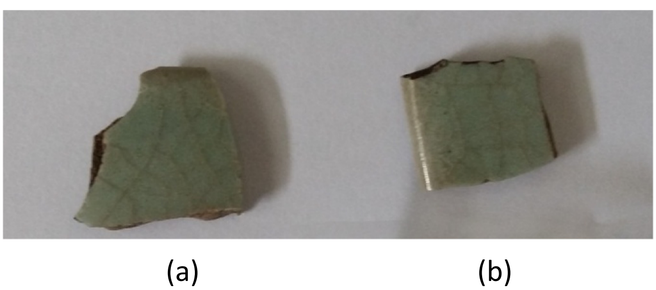


Fig. 3. Opaque glazed celadon shards excavated from the Dong Xia sites in Russia's Primorye region. (a)–(b): Sample 20–21.

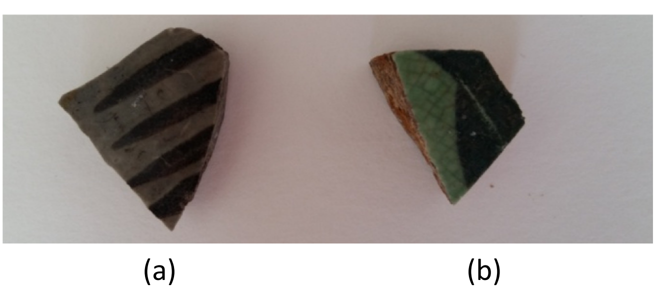


Fig. 4. Painted ceramic shards excavated from the Dong Xia sites in Russia's Primorye region. (a)–(b): Sample 22–23.

whitewares can be seen in Fig. 6. The contents of Fe_2O_3 and TiO_2 were lower in the bodies of the fine whiteware, while the contents of CaO were higher. The average content of Al_2O_3 in the bodies of the seven samples of the fine whiteware was 27.83 wt%, which was higher than that in the bodies of the three samples of coarse whiteware (25.79 wt %).

As shown in Fig. 7, the contents of K_2O and CaO in the glaze of the fine whiteware were much lower than those in the coarse one, while the contents of Al_2O_3 and MgO exhibited the opposite trend. The total content of flux agent in the glaze of the fine whiteware was lower than that in the coarse one.

Fig. 8 shows the FESEM images of coarse whiteware sample 2 and fine whiteware samples 4 and 8. The microstructure of coarse and fine whiteware varies greatly. There are more holes in the body of sample 2 (Fig. 8a) than in the bodies of samples 4 and 8 (Fig. 8b, c). Under the 40 μm-thick transparent glaze of sample 2, there is a 100 μm-thick layer of engobe which contains quartz particles and a small amount of needle-like mullite crystals. The texture of the engobe is obviously more delicate and compact than that of the body.

4.2.1.2. Comparison of the excavated samples of the fine whiteware from the Dong Xia sites and the fine whiteware from the Ding Kiln. The chemical compositions of the whiteware samples of the late Northern Song Dynasty to Jin Dynasty from the Ding Kiln were used as the reference data. These samples were excavated from the Jianci Ridge site and the Bei Town site.

It can be seen from Fig. 9 that the chemical compositions of the bodies of the excavated samples of the fine whiteware were similar to those of the Jin Dynasty from the Ding Kiln. For the Ding wares, the contents of CaO in the bodies of the fine whiteware of the late Northern Song Dynasty and early Jin Dynasty were higher and ranged from 0.5 to 5 wt%. In contrast, the content of CaO in the bodies of the Ding ware samples of the Jin Dynasty was about 1.5 wt%. This feature was basically consistent with the CaO contents in the bodies of the seven fine whiteware samples from the Dong Xia sites.

As shown in Fig. 10, the chemical compositions of the Ding ware glaze varied slightly in the fine whiteware of the late Northern Song Dynasty to Jin Dynasty. The Ding ware sample points were mixed, though the content of CaO in the glaze fluctuated considerably from 3.5 to 16 wt%. The sample points of the seven fine whiteware samples (sample 4–10) were mixed with those of the Ding wares. Thus, it was possible to determine the close correlation of the samples excavated from the Dong Xia sites with those of the late Northern Song Dynasty and Jin dynasties from the Ding kiln.

Based on the chemical compositions of both the bodies and glazes of the seven samples of the fine whiteware (sample 4–10), they were the most similar to the fine white Ding wares of the Jin Dynasty.

All seven samples of the fine whiteware excavated from the Dong Xia sites were of high quality. The body was white, and the glaze was transparent and had a white color tainted by yellow, which were typical

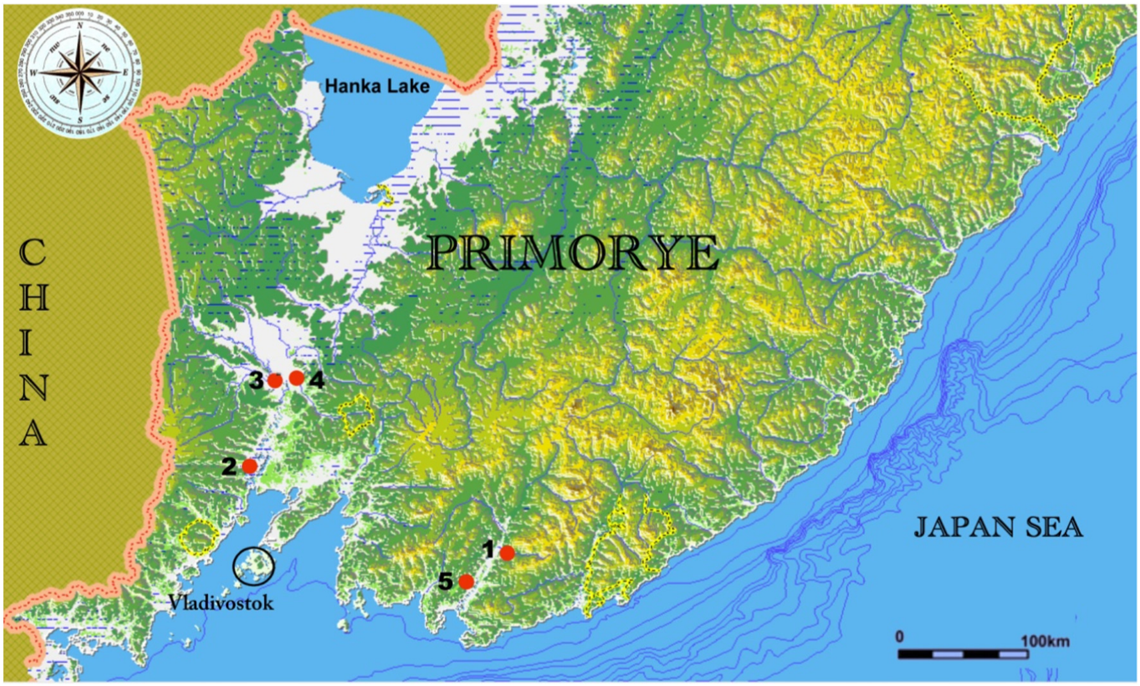


Fig. 5. A map showing the locations of the Dong Xia sites in Russia's Primorye region.
Shaiga (1), Anan'evka (2), Krasny Yar (3), South-Ussuriisk (4), Ekaterinovka (5).

Table 2
Information of the reference samples.

Kiln Site	Time	Number	Excavator
Ding Kiln	Late Northern Song-Early Jin	14	Beijing University, Hebei Institute of Cultural Relics
	Late Jin	29	
Jun Kiln	Jin	47	Henan Provincial Institute of Cultural Relics and Archaeology
Longquan Kiln	Southern Song	5 (Longquan dark-body Guan-type wares)	Zhejiang Provincial Institute of Cultural Relics and Archaeology.
Southern Song Guan kiln	Southern Song	9	Hangzhou Institute of Archaeology
Ru Kiln	Late Northern Song	18	Henan Provincial Institute of Cultural Relics and Archaeology
Zhanggongxiang Kiln	Late Northern Song-Jin	7	Henan Provincial Institute of Cultural Relics and Archaeology

features of wares from the Ding Kiln in the Jin Dynasty. As shown in the FESEM images of the cross-sections of the sample 4 and sample 8 (Fig. 8b, c), the glazes were uniform and contained very few air bubbles, and there was no body-glaze reaction layer. These findings complied with the microstructure features of the fine white Ding wares of the Jin Dynasty. According to our studies, most fine whiteware from the Ding Kiln (including the Five Dynasties, Song Dynasty, and Jin Dynasty) had no body-glaze reaction layer. The low contents of CaO and Al_2O_3 at the body-glaze interface led to the composition of glaze outside the primary crystallization region of anorthite and the failure to form the anorthite body-glaze reaction layer. Rapid cooling in the kiln may be another reason for the absence of precipitation of crystals at the body-glaze interface due to limitations in the dynamic conditions.

Based on the visual observation, the comparison of chemical compositions of bodies and glaze, and the microstructure analysis, we concluded that the seven samples of the fine whiteware (sample 4–10) were the products by the Ding Kiln in the Jin Dynasty.

4.2.1.3. Brief introduction and discussion of the Ding kiln whiteware. The Ding Kiln, one of the five famous kilns in the Song Dynasty, is located in Quyang County, Baoding City, Hebei Province of China. This primary historical and cultural site is protected at the national level since 1986. Ding Kiln was founded in the Tang Dynasty (618–907 CE) and closed in the Yuan Dynasty (1271–1368 CE). Under the influence of the Xing kiln in the middle to late Tang Dynasty, the Ding Kiln also began to make whiteware. By the Song Dynasty, the Ding Kiln became the top kiln that produced whiteware in China. The Ding Kiln had a large production scale, and were sold to many different places. Moreover, tributes of whiteware produced by the Ding Kiln were sent to the court for the longest time among all kilns in the Northern Song Dynasty and Jin Dynasty. The Ding Kiln had an important status in China's history of ceramics and produced a lasting effect on the ceramics industry in later generations. The Ding Kiln had an extremely high archaeological value. The whiteware artifacts produced by the Ding Kiln were outstanding representatives of whiteware in the North China, which embodied some of the breakthroughs in the ceramics-making technology. Fine whitewares made with high-quality kaolin available near the kiln site were well known for their high quality, delicacy, and beauty. The high-temperature firing was guaranteed by the use of the semi-down-draft dome kiln, as well as abundant wood or coal resources near the Ding Kiln site. In the late Northern Song Dynasty, the upside-down firing technique and stacking-ring combined sagger were invented by the Ding Kiln. These new techniques not only optimized the utilization of space inside the kiln but also minimized the deformation of wares. These technical innovations improved both the quality and output of ceramic wares and promoted the development of China's ceramics-making industry. The well-known Jingdezhen ceramics-making industry was greatly influenced by these techniques. For the fine whiteware produced by the Ding kiln, decorative techniques include engraving, incision, impression, etc. These techniques made it possible

Table 3
The chemical compositions of the excavated samples (wt%).

No.	Na_2O	MgO	Al_2O_3	SiO_2	K_2O	CaO	TiO_2	Fe_2O_3	CuO
Coarse whiteware from the Shaiga site (sample 1,2,3)									
1-b	0.43	1.47	26.96	64.43	1.74	0.49	0.79	2.69	
1-g	0.55	1.32	16.77	71.28	4.49	3.07	0.12	1.40	
2-b	0.61	1.10	24.95	67.24	1.82	0.47	0.93	1.87	
2-g	0.58	2.26	14.33	66.94	6.82	6.59	0.31	1.18	
2-e	0.24	0.67	38.35	55.82	2.00	0.41	0.51	1.00	
3-b	0.27	2.50	25.47	65.39	1.96	0.56	0.93	1.91	
3-g	0.30	2.73	15.27	68.24	4.77	6.20	0.35	1.15	
Fine whiteware from the Shaiga site (sample 4,5,6)									
4-b	0.31	2.06	29.34	62.63	1.44	1.48	0.48	1.26	
4-g	0.35	2.64	16.72	72.69	1.99	3.24	0.15	1.21	
5-b	0.07	2.07	26.34	65.88	1.43	1.40	0.60	1.22	
5-g	0.68	3.59	18.59	68.42	2.01	4.31	0.11	1.28	
6-b	0.07	2.65	26.81	64.37	1.88	1.32	0.57	1.33	
6-g	1.29	2.92	16.22	71.59	2.37	3.27	0.10	1.23	
Fine whiteware from other sites (sample 7,8,9,10)									
7-b	0.25	1.59	30.21	62.61	1.18	1.16	0.63	1.38	
7-g	1.01	2.22	16.13	71.74	3.71	2.65	0.23	1.31	
8-b	0.16	2.51	29.84	61.96	1.60	1.24	0.56	1.14	
8-g	0.32	2.72	16.72	70.42	2.88	4.65	0.16	1.12	
9-b	1.11	2.45	23.37	67.15	1.81	1.24	0.52	1.37	
9-g	1.53	3.27	16.91	69.49	2.24	4.21	0.13	1.24	
10-b	0.32	1.49	28.88	63.82	1.24	1.09	0.74	1.41	
10-g	0.07	2.50	16.47	73.57	1.99	2.93	0.19	1.29	
Opalescent glazed ware with copper-red decoration (sample 11,12 13)									
11-b	0.67	1.05	27.85	64.34	1.75	0.67	0.75	1.92	
11-g (blue glaze)	0.98	1.27	10.32	72.20	3.41	9.00	0.11	1.71	
11-g (green spot)	1.40	1.05	7.56	53.19	2.15	11.58	0.10	1.43	20.53
11-g(lavender glaze)	0.44	1.06	9.97	72.86	3.29	9.02	0.12	1.80	0.45
12-b	0.59	1.14	25.65	65.92	1.78	0.72	0.90	2.30	
12-g (blue glaze)	1.32	0.73	10.66	74.81	3.90	6.08	0.09	1.42	
12-g(lavender glaze)	1.40	1.08	10.68	72.40	3.82	7.14	0.10	1.46	0.92
13-b	0.35	1.37	25.53	65.19	2.25	1.03	0.83	2.46	
13-g(blue glaze)	1.15	0.76	10.71	75.00	3.76	6.08	0.08	1.47	
13-g(lavender glaze)	1.11	1.07	9.73	75.10	3.52	6.25	0.09	1.49	0.64
Opalescent glazed ware (sample 14,15,16)									
14-b	0.32	1.38	23.71	67.41	2.11	1.12	0.56	2.39	
14-g	0.94	1.43	9.86	70.62	2.61	11.62	0.13	1.80	
15-b	0.26	1.54	27.71	64.03	2.04	0.59	0.79	2.03	
15-g	0.46	1.54	10.33	71.88	3.13	9.61	0.15	1.90	
16-b	0.23	1.24	26.44	65.85	1.92	0.46	0.79	2.06	
16-g	0.33	1.42	10.36	71.97	3.19	9.78	0.14	1.81	
Oalescent glazed ware (sample 17,18)									
17-b	0.31	1.89	27.28	64.24	1.89	0.56	0.82	1.99	
17-g	0.73	1.52	10.02	71.15	3.39	9.88	0.14	2.16	
18-b	0.26	1.30	28.37	63.47	1.92	1.04	0.69	1.95	
18-g	1.28	2.16	10.26	70.94	2.93	9.46	0.12	1.85	
Translucent glazed ware (sample 19)									
19-b	0.21	1.28	26.95	64.74	1.89	0.68	0.73	2.52	
19-g	1.12	0.59	11.16	73.52	3.43	7.56	0.11	1.52	
Opaque glazed celadon (sample 20,21)									
20-b	0.26	1.46	29.27	62.71	1.72	0.39	0.86	2.32	
20-g	0.72	1.28	13.96	67.40	4.65	9.25	0.13	1.61	
21-b	0.07	1.24	28.57	63.89	1.67	0.42	0.86	2.28	
21-g	1.01	1.52	14.16	66.33	4.75	9.49	0.14	1.61	
Ware with upperglaze black (sample 22)									
22-b	0.43	0.65	23.40	68.72	1.15	1.93	0.72	1.98	
22-g(upperglaze black)	1.45	2.10	14.27	70.43	4.37	3.26	0.26	2.85	
Pottery with underglaze green (Sample 23)									
23-b	0.38	0.87	31.58	60.68	1.56	0.49	1.15	2.29	

No.	Al_2O_3	SiO_2	P_2O_5	PbO_2	K_2O	CaO	Fe_2O_3	Ni_2O_3	CuO
23-g (pale green transparent glaze)	3.62	32.98	0.80	54.77	0.33	0.80	0.57	0.39	5.74
23-g(glaze + underglaze dark green)	4.68	37.12	0.83	50.01	0.46	0.87	0.53	0.38	5.12

b-body, g-glaze, e-engobe.

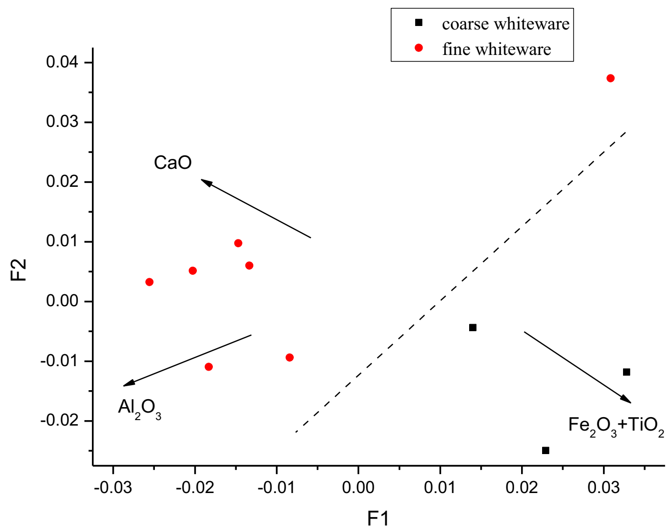


Fig. 6. The 2D correspondence analysis of the bodies of the coarse and fine whiteware samples excavated from the Dong Xia sites.

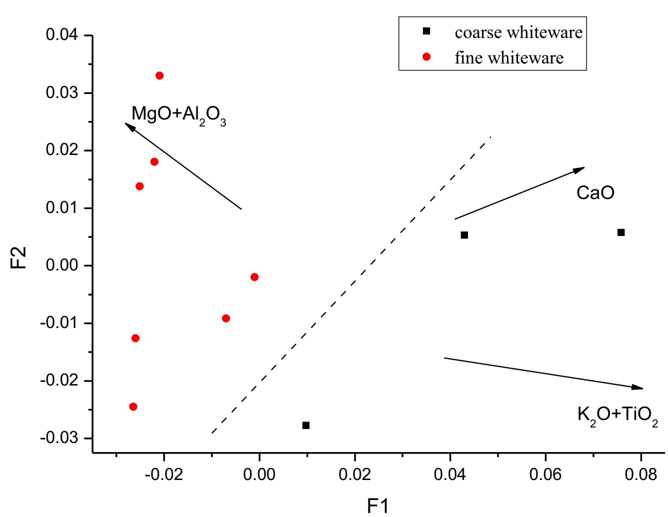


Fig. 7. The 2D correspondence analysis of the glazes of the coarse and fine whiteware samples excavated from the Dong Xia sites.

to produce exquisite, elegant, and unique decorative patterns on the ware. 69 Ding whiteware sherds from the 2009 excavation were analyzed using laser ablation techniques (ICP-AES). Ding wares of different periods can be distinguished by their chemical compositions, since the Ding bodies and glazes show some minor characteristics of their age (Cui et al., 2012).

Glaze color of the Ding ware began to change dramatically from the middle Northern Song Dynasty. That is, the white color tainted by green in the early Song Dynasty switched to the white color tainted by yellow. This transition was related to the application of coal fuel instead of wood and was completed in the late Northern Song Dynasty. Wares with "尚食局" (Shang Shi Ju) (agency for supplying food for the court) and "尚药局" (Shang Yao Ju) (agency for supplying medicines for the court) marks were excavated from the strata corresponding to the late Northern Song Dynasty (Beijing University, Hebei Provincial Institute of Cultural Relics, 2014). The Jin Dynasty corresponded to the historical period when the Ding Kiln was the most prosperous and developed, wherein the production scale and the number of wares produced reached a peak. Nearly all wares were produced via the upside-down firing process. Wares with a dragon design on the inner wall and "东宫" (East Palace) mark on the bottom were excavated. A few Bowls with "尚食局" (Shang Shi Ju) mark and a Capricorn pattern (a creature with the head of a dragon and the body of a fish, which originated from Indian Buddhism) engraved on the inner wall were also excavated (Beijing University, Hebei Provincial Institute of Cultural Relics, 2014). These wares have provided important clues for determining the year of production and the features of the wares sent as tribute.

During the Jin Dynasty, whitewares produced by the Ding Kiln were widely discovered in the middle and east of Northeast China, as well as in today's Primorye region (Peng, 2013). This was not only the result of trade between the remotely located "Xu Pin Lu " and the inland region of China but also a reflection of the prosperity of the Ding Kiln in the Jin Dynasty.

An important discovery was a bowl with the "尚食局" (Shang Shi Ju) mark in cursive script excavated from the Anan'evka site. Its inner wall is engraved with Capricorn, flowers, and plants. Its inner bottom is engraved with lotus flowers, while the "尚食局" (Shang Shi Ju) mark is vertically engraved by the cursive script in the middle of the outer bottom (Fig. 11). This particular bowl with the "尚食局" (Shang Shi Ju) mark was a tributed ware to the court from the Ding Kiln, which resembled a plate with "尚食局" (Shang Shi Ju) mark engraved with the Capricorn pattern that was produced in the Jin Dynasty and excavated from the Ding Kiln site (Beijing Art Museum, 2012). Therefore, there is reason to reckon that this bowl is a Ding ware produced in the Jin Dynasty. The bowl might have been taken by *Puxian Wannu's* troops to the Anan'evka walled town of the Dong Xia State. As the bowl is considered very cherished, no destructive compositional and structural analyses were performed.

Three samples (No.1–3) of the coarse whitewares excavated from the Dong Xia site were not products of the Ding Kiln. It is likely that they were produced by local kilns in the Northeast China, such as the Gang Wa Kiln and Jiang Guan Tun Kiln. However, the exact provenance is hard to determine due to the lack of reference samples or data.

4.2.2. Provenance of the excavated celadon and opalescent glazed ware

4.2.2.1. Classification of samples 11–21 based on chemical composition. Figs. 12 and 13 depicted the results of the 2D correspondence analysis of the bodies and glazes of the eleven samples (No. 11–21). The chemical compositions of the bodies and glazes (especially glazes) of sample 20 and 21 were much different from those of other samples. The contents of Al_2O_3 and K_2O in the glaze of sample 20 and 21 were higher than those in other samples, while the content of Al_2O_3 in the bodies was slightly higher than in other samples. Thus, sample 20 and 21 differed from the remaining nine samples. The chemical compositions of the glaze 11–19 were similar, with the Al_2O_3 content of about 10 wt%. However, the sample points were distributed scatteredly due to the differences in the contents of Na and Mg. Although sample 19 had a translucent green glaze, which looked different from the typical Jun glaze, its chemical composition was close to that of the Jun glaze.

4.2.2.2. Comparison of the excavated samples 11–19 and the Jun Kiln ware. The chemical compositions of the glazes were compared between the sample 11–19 and the Jun wares (Fig. 14). The reference Jun wares were from the excavation of the Juntai site in Yuzhou, Henan. The results showed that the chemical compositions of the glazes in the nine celadon samples were similar to those of the Jun glazes. Several sample points were separated from the cluster of the remaining sample points on the graph because of the fluctuation of the contents of Na and Mg in the glaze. The average content of Al_2O_3 in the Jun glaze was about 10 wt%, and the average content of SiO_2 in the Jun glaze was 72 wt%.

Eight samples with opalescent glazes (sample 11–18) had a similar appearance to the Jun glaze, in terms of the blue or blue-green color and opalescence, related to the characteristic phase separation structure of the Jun glaze. Besides, the glazes of sample 11–13 exhibited a lavender color due to the presence of the copper element (Table 2), attributed to the coloring effect by Cu and Cu_2O (Kingery and Vandiver,

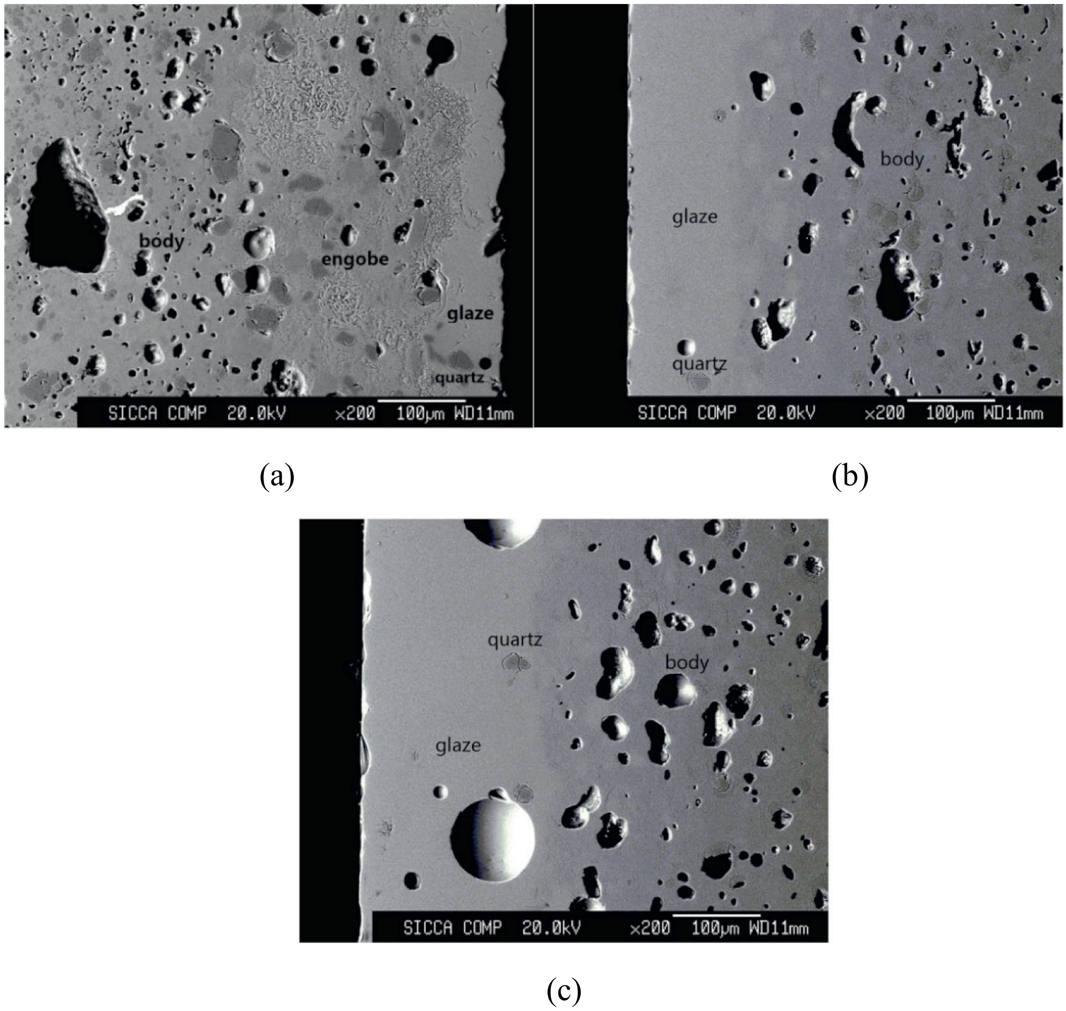


Fig. 8. FESEM images of the cross-sections of the excavated whitewares (a) coarse whiteware sample 2 (b) fine whiteware sample 4, (c) fine whiteware sample 8.

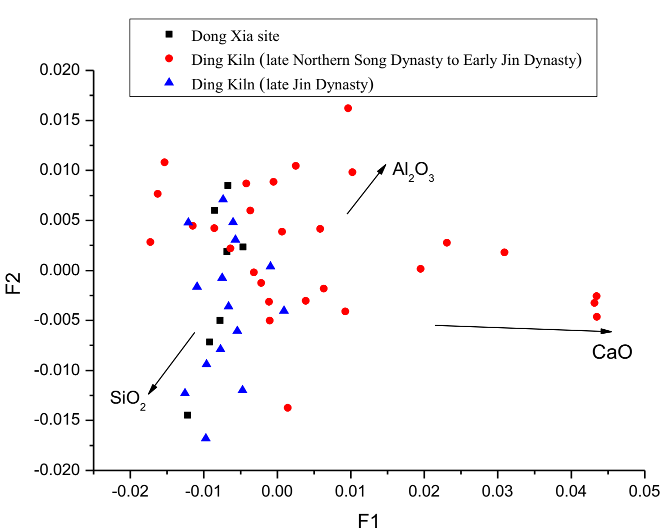


Fig. 9. The 2D correspondence analysis of the bodies of the fine whiteware samples excavated from the Dong Xia site and those of the late Northern Song Dynasty to Jin Dynasty from the Ding Kiln.

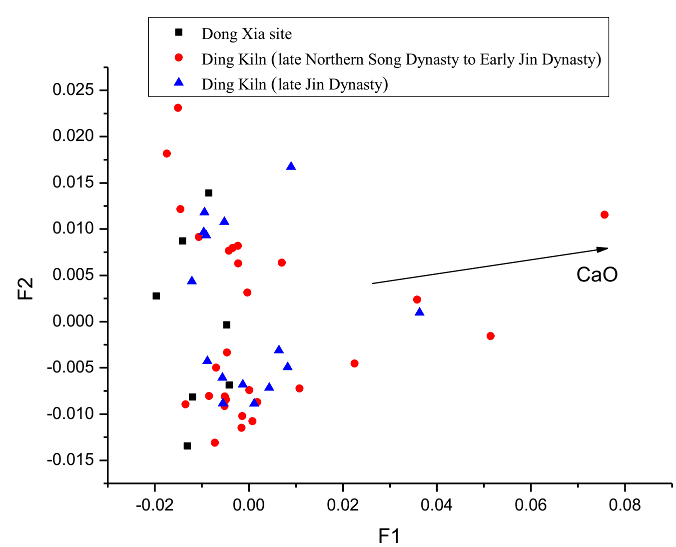


Fig. 10. The 2D correspondence analysis of the glazes of the fine whiteware samples excavated from the Dong Xia site and those of the late Northern Song Dynasty to Jin Dynasty from the Ding Kiln.

(a) (b)

Fig. 11. A whiteware bowl with "尚食局" (Shang Shi Ju) mark excavated from the Anan'evka site. (a) the front of the bowl, (b) the back of the bowl.

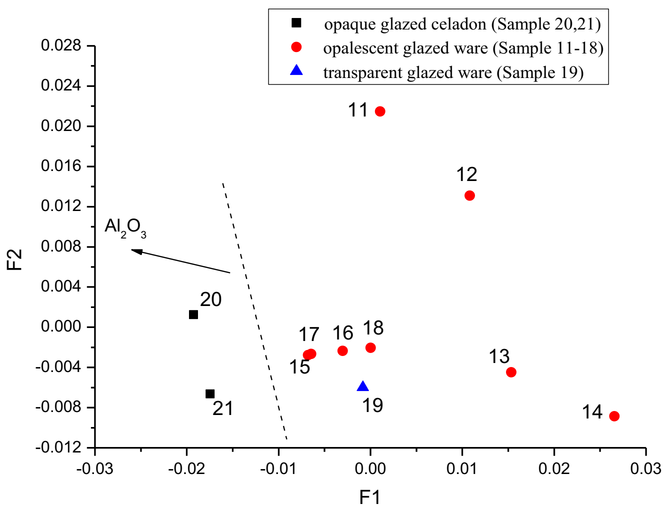


Fig. 12. The 2D correspondence analysis of the bodies of the ware samples excavated from the Dong Xia sites.

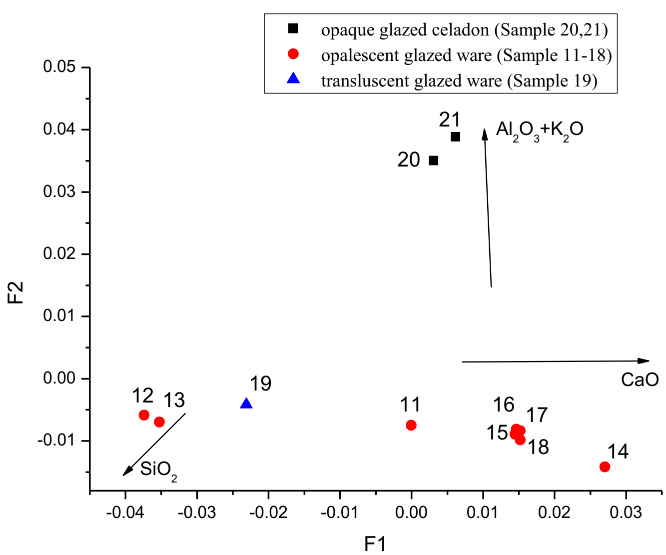


Fig. 13. The 2D correspondence analysis of the glazes of the samples excavated from the Dong Xia sites.

1983). In our opinion, these samples were not necessarily produced by the Jun Kiln in Yuzhou, Henan province, they were considered to belong to the products from the Jun kiln series. According to archaeological excavations, in the region of Henan and Hebei provinces, a few

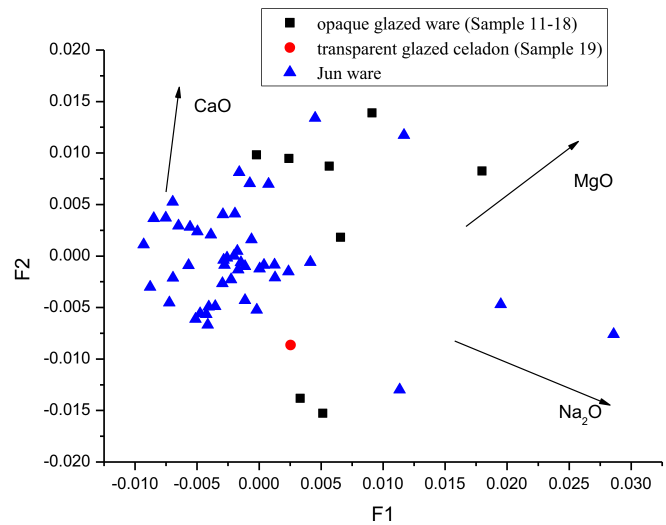


Fig. 14. The 2D correspondence analysis of the glazes of sample 11–19 excavated from the Dong Xia site and those from the Jun kiln site in Yuzhou of Henan province.

folk kilns produced Jun-type ware, so we call it Jun-series ware. By visual observation, the Jun-series wares from the Krasny Yar site may include the products of the late Northern Song Dynasty, the Jin Dynasty, and the Yuan Dynasty. Which implied that the Krasny Yar walled town continued to be inhabited to the Yuan Dynasty after the downfall of the Dong Xia State.

4.2.2.3. Brief introduction and discussion of the Jun kiln opalescent glazed ware. It is conventionally adopted that the Jun Kiln was founded in the Northern Song Dynasty and closed in the Yuan Dynasty. However, disputes are still going on as to the start time of the Jun Kiln. Some scholars precluded that the Jun wares were produced from the Jin Dynasty, as indicated by the thermoluminescence dating results (Miao et al., 2016). Jun-series wares were produced, used, and widely circulated in the Jin Dynasty. The blue and blue-green color was mainly attributed to the selective scattering produced by the phase-separated structures in the glaze to the visible light. Such periodic phase-separated structures of the submicron scale are amorphous photonic structures (Li, 2015).

Sample 19 had a translucent glaze in green color, and its chemical composition agreed with that of the reference Jun wares (Fig. 14). The visual appearance of sample 19 looked different from Sample 11–18, a blue opalescence, which is typical of the phase-separated glaze of the Jun ware, was not observed. This is because the firing temperature of sample 19 was relatively high and exceeded the highest immiscibility temperature of the glaze, and the cooling rate might not be slow

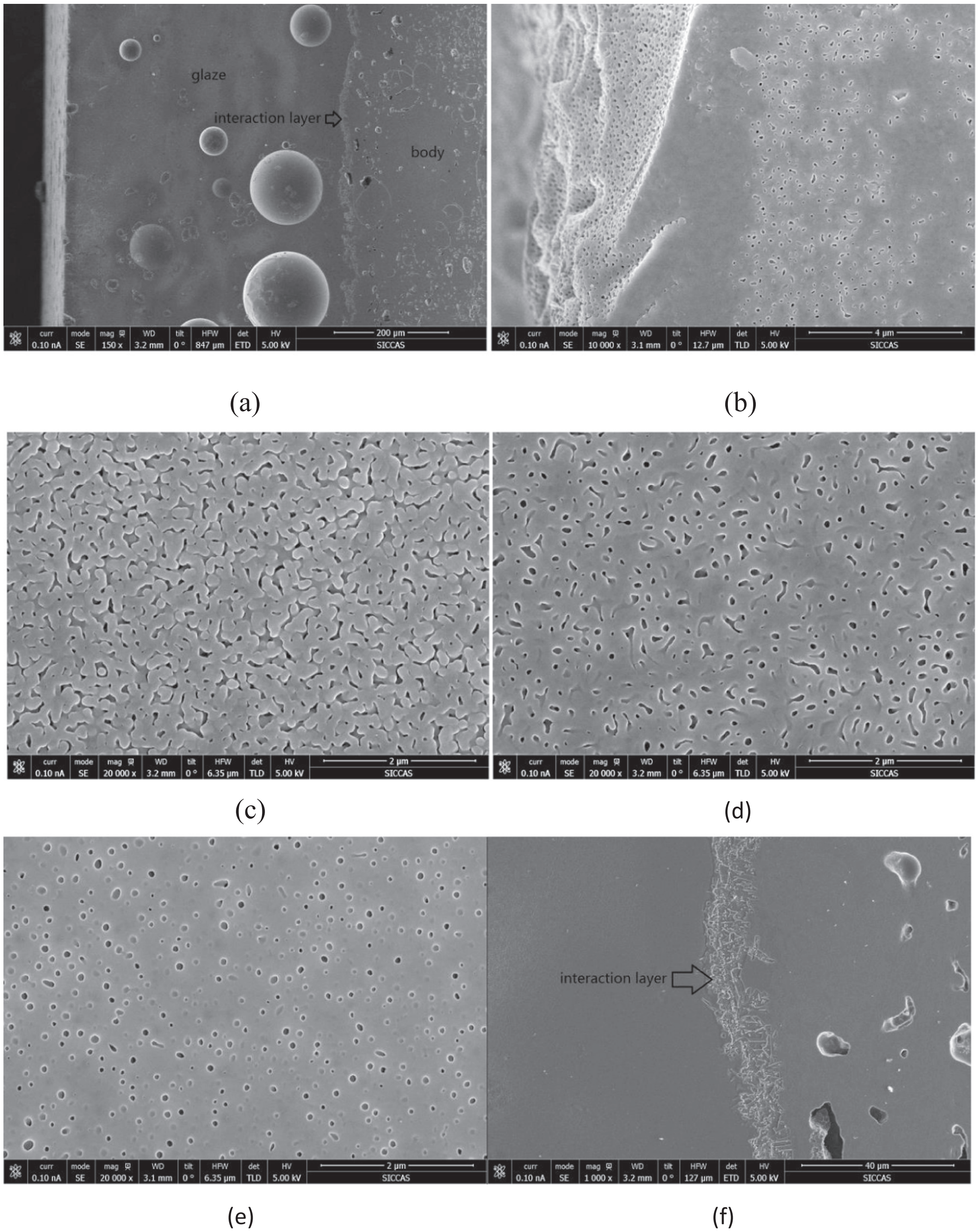


Fig. 15. FESEM images of the excavated sample 14
(a) cross section of the glaze layer, showing bubbles, rough texture of nano-sized phase separation, and an interaction layer with dense anorthite crystallization at the glaze-body interface
(b) closeup of (a), showing discrete droplet and worm-like phase separation near the exterior surface of the glaze
(c) semi-interconnected phase separation
(d) discrete worm-like phase separation
(e) discrete droplet phase separation
(f) well-developed interaction layer of anorthite crystals at the interface between the body and the glaze.

enough. The green color of sample 19 mainly came from the coloring effect of Fe^{2+}/Fe^{3+} ions. Thus, both the composition and microstructure influenced the glaze color and texture.

The microstructure was analyzed across the thickness of the glaze, from the exterior to interior surface. In terms of phase separation behavior, a great deal of variability was to be expected in a single glaze. The entire glaze layer presented a phase separation structure (Figs. 15–17), the composition of the different positions in the glaze was fluctuating, local variations were caused by coarse grinding and incomplete mixing of the raw materials. Actually small variations in the

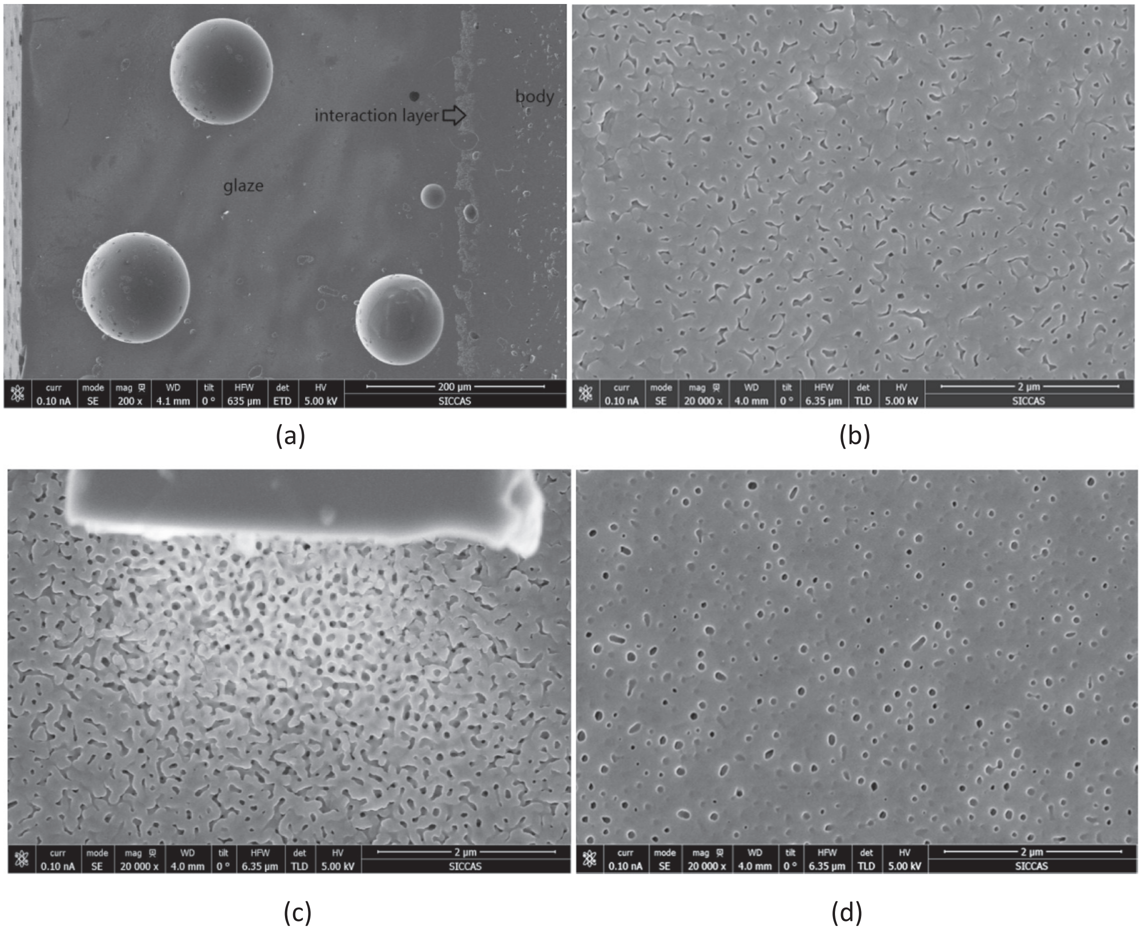


Fig. 16. FESEM images of the excavated sample 17.
(a) cross section of the glaze layer, showing bubbles, rough texture of nano-sized phase separation, and an interaction layer with dense anorthite crystallization at the glaze-body interface
(b) discrete worm-like phase separation
(c) local interconnected phase separation structure formed due to a local rise of calcia-alumina ratio in the glaze caused by a local crystallization of anorthite
(d) discrete droplet phase separation.

calcia-alumina ratio markedly influenced liquid-liquid phase separation behavior (Kingery and Vandiver, 1983), so the scale and shape of phase separation were changeable too.

The chemical composition of Chinese high-fired glaze can be roughly represented with the SiO_2–Al_2O_3–CaO ternary. The Jun glaze composition is located in the low calcia region within the liquid-liquid immiscibility boundary above 950 °C, the discrete phase in phase-separation should be rich in CaO (Fe_2O_3, MgO), the Ca-rich phase was etched out by HF to observe the microstructure clearly. The discrete phase can be in the shape of droplet, ellipsoid or worm (Figs. 15b, d, 16b, d, 17c). Phase separation in glazes is primarily determined by the strong immiscibility tendency between SiO_2 and CaO (Mazurin and Porai-Koshits, 1984). The SEM images showed that in the glaze layer, there were bubbles, rough texture of nano-sized liquid-liquid phase separation, and an interaction layer with dense anorthite crystallization at the glaze-body interface (Figs. 15a, b, 16a, 17a). In some regions of the glazes, semi-interconnected phase separation structure can be observed because the local calcia content is relatively high (Figs. 15c, 17b), but not high enough to form an interconnected structure. An interconnected phase-separation structure was observed due to a local rise of the calcia-alumina ratio in the glaze caused by a local crystallization of anorthite (Fig. 16c). The formation process of phase separation is complicated, influenced by the variations in the chemical composition, the viscosity of melt, and the firing conditions such as temperature and atmosphere. Going into the interior of the glaze and approaching the interaction layer at the glaze-body interface, the calcia-alumina ratio in the glaze is further reduced due to the diffusion of aluminum ions to the glaze, and the Ca-rich phase tended to become discrete droplet (Figs. 15e, 16d). Cylindrical anorthite crystals grew between the glaze and the body, forming a white interaction layer which can reflect light (Fig. 15f).

4.2.2.4. Comparison of the opaque glazed celadon sample 20–21 and the opaque glazed celadons from the Southern Song Guan Kiln, the Longquan Kiln of the Southern Song Dynasty and the Ru Kiln. The excavated sample 20 and sample 21 had a light blue-green opaque glaze and thin gray body. The chemical compositions of these two samples were compared to those of the opaque-glazed celadons from the Southern Song Guan kiln, the Longquan Kiln of the Southern Song Dynasty (the dark-body Guan-type opaque celadon), and the Ru Kiln.

Regarding the celadon wares from the Longquan Kiln, the Fe_2O_3 content in the bodies exceeded 4 wt%, and the CaO content in the glazes ranged from 12 to 16 wt%. While these two critical indicators were much lower in sample 20 and 21. Given the large differences in the composition formulae for the bodies and glazes, these two samples were different from the dark-body Guan-type opaque celadon from the Longquan Kiln of the Southern Song Dynasty.

It can be seen from Figs. 18–19 that the chemical compositions of bodies and glazes of sample 20 and 21 greatly differed from the reference celadons of the Southern Song Guan kiln. As shown in Fig. 19,

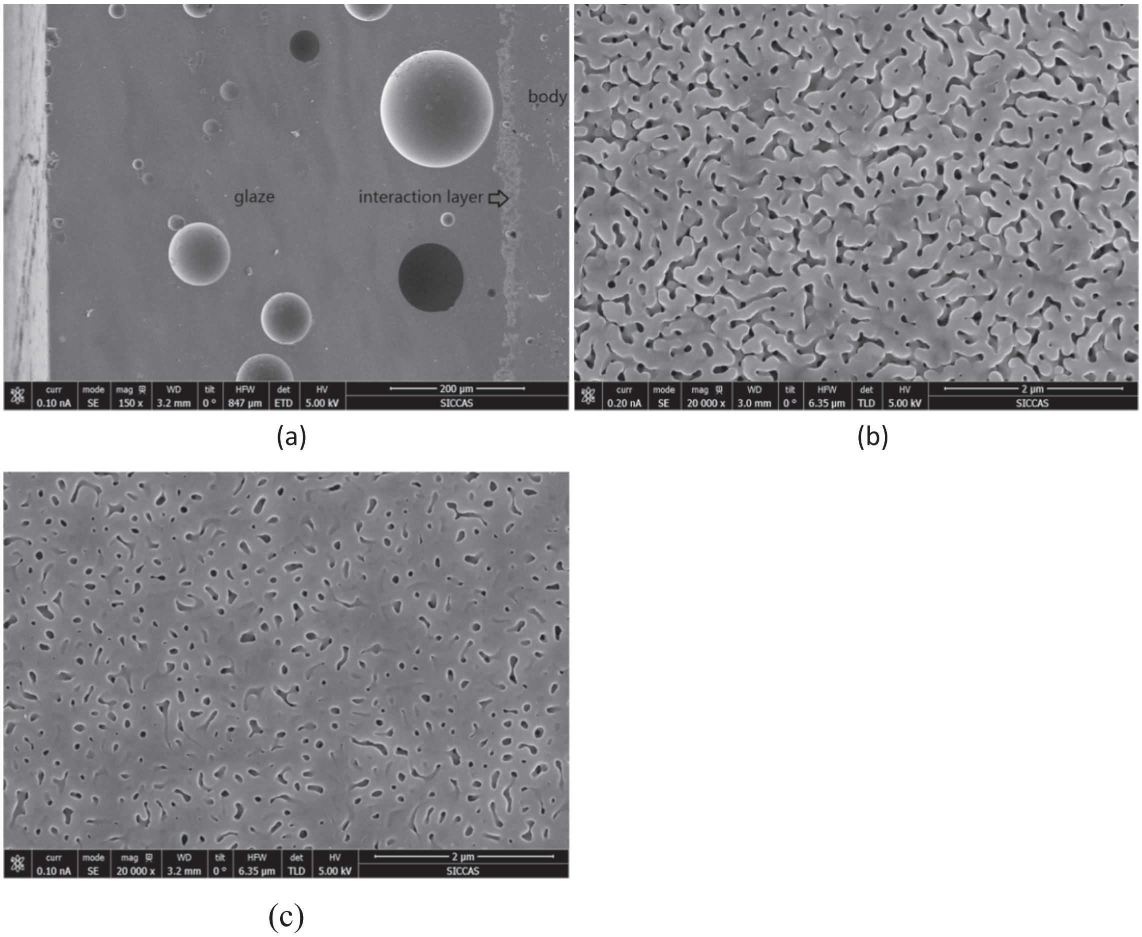


Fig. 17. FESEM images of the excavated sample 18
(a) cross section of the glaze layer, showing bubbles, rough texture of nano-sized phase separation, and an interaction layer with dense anorthite crystallization at the glaze-body interface
(b) semi-interconnected phase separation
(c) discrete worm-like phase separation.

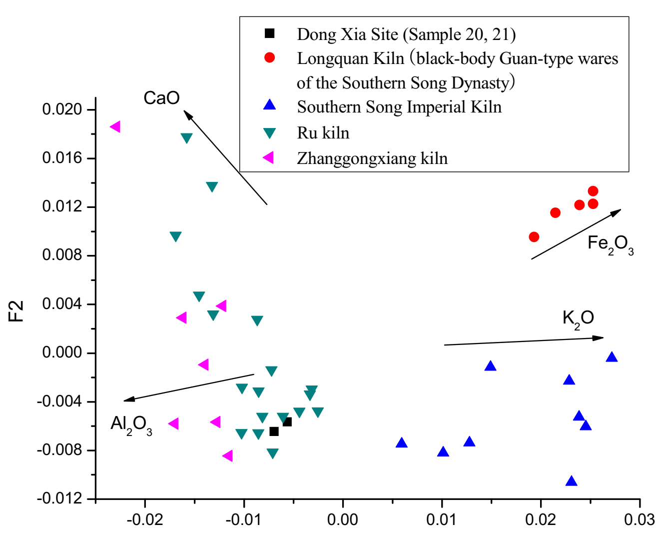


Fig. 18. Comparison of the body chemical compositions for the excavated sample 20–21, the opaque celadon ware from the Southern Song Guan kiln, the Longquan Kiln, the Ru Kiln and the Zhanggongxiang Kiln.

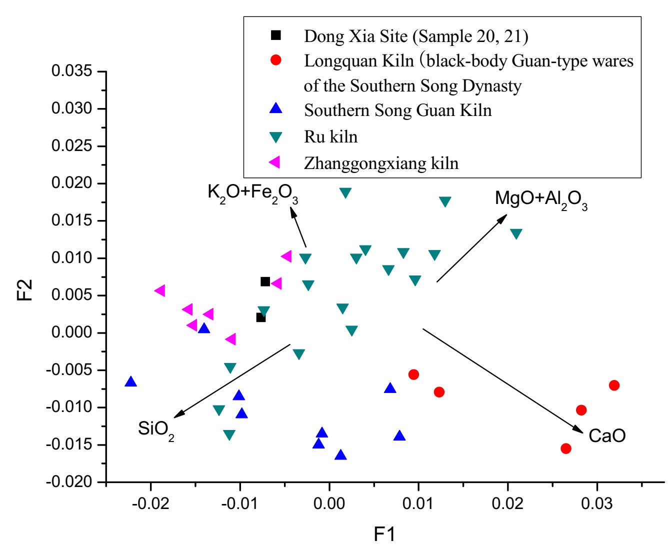


Fig. 19. Comparison of the glaze chemical compositions for the excavated sample 20–21, the opaque celadon ware from the Southern Song Guan kiln, the Longquan Kiln, the Ru Kiln and the Zhanggongxiang Kiln.

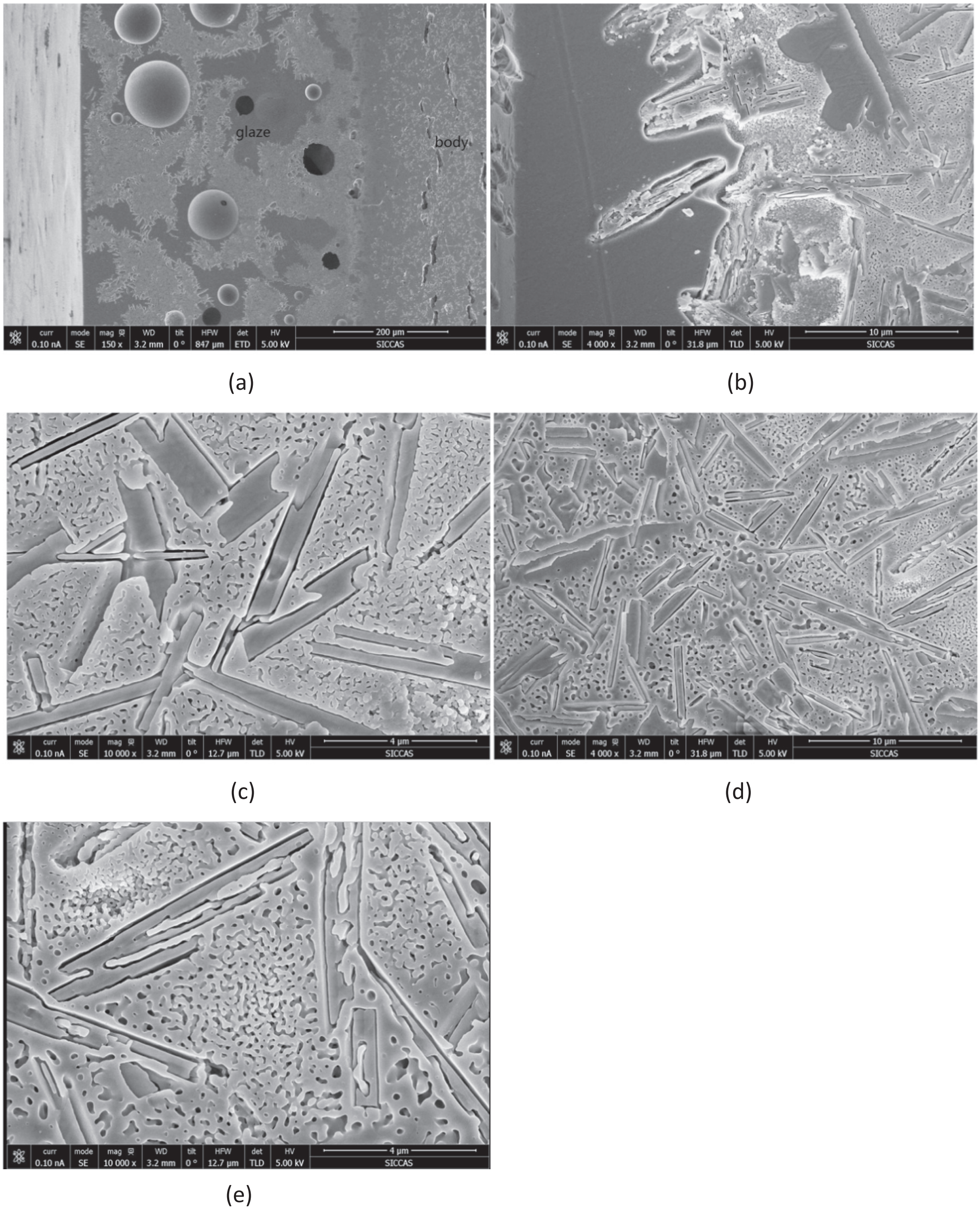
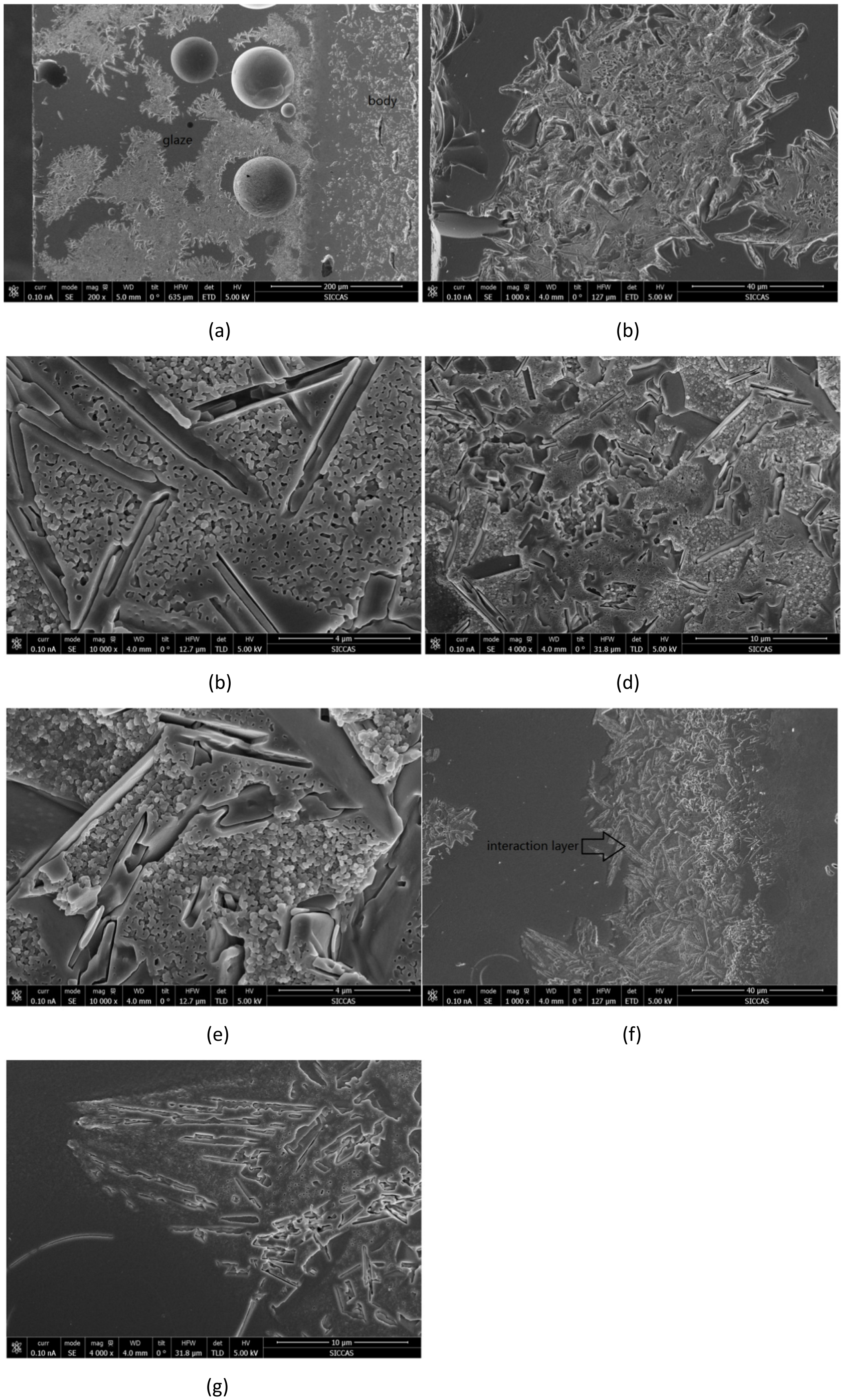


Fig. 20. FESEM images of the excavated sample 20
(a) loose body, an interaction layer with dense anorthite crystallization at the glaze-body interface, and extensive anorthite dispersion within the glaze
(b) rough texture of nano-sized phase separation within the interspaces of anorthite clusters or around the brims of anorthite needles or columns resulting from local chemical variation caused by anorthite crystallization
(c) closeup of (b), showing interconnected phase separation structure
(d) multi-level phase separation structure
(e) closeup of (d), showing both discrete droplet or worm, and interconnected structure in different regions of the glaze.

the glazes of samples 20 and 21 are close to both Ru wares and Zhanggongxiang wares, so it is not possible to make a clear distinction based on the glaze composition. However, as shown in Fig. 18, the bodies of samples 20 and 21 are obviously closer to Ru wares than Zhanggongxiang wares. Therefore, we conclude that samples 20 and 21 belong to the Ru kiln at the Qingliang Temple. Compared with the Ru ware, the body of the Zhanggongxiang ware contains less Fe_2O_3 and the color of body is lighter.

In terms of microstructure, opaque glaze contains light-scatterers within the glaze, such as microcrystals or phase-separation structures or both. The FESEM analysis revealed that both of the glazes of sample 20 and 21 had a unique crystallization-phase separation associated structure (Figs. 20–21), which was consistent with the characteristic microstructure of the Ru glaze (Li et al., 2005).



(caption on next page)

Fig. 21. FESEM images of the excavated sample 21
(a) loose body, an interaction layer with dense anorthite crystallization at the glaze-body interface, and extensive anorthite dispersion within the glaze
(b) rough texture of nano-sized phase separation within the interspaces of anorthite clusters or around the brims of anorthite needles or columns resulting from local chemical variation caused by anorthite crystallization
(c) closeup of (b), showing interconnected phase separation structure
(d) multi-level phase separation structure
(e) closeup of (d), showing both discrete droplet or worm, and interconnected structure in different regions of the glaze
(f) an interaction layer with dense anorthite crystallization at the glaze-body interface
(g) closeup of (f), showing discrete droplet phase separation within the interspaces of anorthite columns.

The chemical composition of the Ru glaze falls into the primary crystalline phase region of anorthite. With the precipitation of anorthite, the local silica/alumina ratio rises dramatically and the glaze composition enters the metatable liquid-liquid immiscibility region above 950 °C, then a crystallization-phase separation associated structure forms (Li, 2015; Li et al., 2005). Within the interspaces of the anorthite clusters or around the brims of the anorthite needles or columns, a rough texture characteristic of the nano-sized liquid-liquid phase separation can always be observed (Figs. 20–21). The phase separation is characterized by variable morphologies, including interconnection, semi-interconnection, isolated worm and droplet. These structures are closely related to the morphology, scale, and packing density of the precipitated anorthite crystals.

According to the chemical composition and microstructural analyses, samples 20 and 21 were identified as the Ru Kiln ware. This is the first identification of the Ru wares from the wares excavated from the Dong Xia sites in Russia's Primorye region.

4.2.2.5. Brief introduction and discussion of the Ru kiln opaque glazed celadon. The Ru Kiln was founded in the late Northern Song Dynasty and located near the present-day Qingliang Temple in Baofeng County, Henan Province of China. The Ru Kiln operated for only about 20 years, and the Ru wares were greatly cherished by the court. There are fewer than 100 pieces of Ru wares preserved to the present day. The Ru ware is featured by a simple style, combined with an implicit and elegant glaze color. Blue-green is considered the best glaze color of the Ru ware, and its glaze opacity is mainly attributed to the crystallization-phase-separated glaze structure (Li et al., 2005). For the Ru glaze, a dual coloring mechanism is in effect, covering the chemical-compositional coloring by the iron ion chromophore and the structural coloring caused by structural inhomogeneities of the nano-sized phase separation structure (Li, 2015).

It was recorded in the "*Qing Bo Za Zhi*" written by Zhou Hui in the Southern Song Dynasty that "*the sales of the Ru wares was allowed only for those wares that were returned or not chosen by the court*." Therefore, in the late Northern Song Dynasty, apart from the Ru wares tributed to the court, some second-class Ru wares were available for sale. It is reasonable to assume that a few Ru wares were carried by Jurchen people to East Liao and then to Russia's Primorye region of today. In 1128, the eighth emperor of the Northern Song Dynasty Huizong (1082–1135 CE), along with the last emperor of the Nothern Song Dynasty Qinzong (1100–1161 CE) and the rest of their family, were taken captive by Jurchens and brought to the Jin capital (present-day's Huining Prefecture, China). Although in 1131 (the first year of Shaoxing period of the Southern Song Dynasty) both Emperors and their families were reported to be sent to Wuguo City (present-day's Yilan of Heilongjiang Province, China), no Ru ware pieces have been yet found there.

In summary, the excavated sample 4–21 were confirmed to be produced by the kilns (Ding King, Jun kiln series, and Ru Kiln) in ancient North China. The kilns were all located within the territory of the Jin Dynasty.

4.2.3. Provenance of the painted ware

The painted sample 22 and sample 23 are preliminarily assumed to be produced by the kilns in Henan and Hebei regions of ancient North China, but the exact provenance was not determined due to the lack of the reference data.

4.2.4. Prospects for future work

According to our visual observation of a large number of celadon shards excavated from the Dong Xia sites in Russia's Primorye region, some of the transparent glazed celadon wares are likely to be produced by the Longquan Kiln in South China and the Yaozhou Kiln in North China. Moreover, the excavated opaque-glazed celadon wares may also include the dark-body celadon ware by the Longquan Kiln in the Southern Song Dynasty, which was an imitation of the product from the Southern Song Guan kiln. The Longquan Kiln had a high production volume and its trading activities are widely spread out in the Song Dynasty. The production output of the Yaozhou Kiln in the ancient North China during the Jin Dynasty was also quite large and its distribution was extensive. In the future, more samples need to be analyzed using scientific methods, and further provenance identification is needed in order to confirm our hypotheses.

5. Conclusions

(1) Chemical composition and microstructural analyses were jointly applied to determining the provenance of the ceramic samples excavated from the Dong Xia sites in Russia's Primorye region.
(2) Seven excavated fine whiteware samples were confirmed as the products by the Ding Kiln in the Jin Dynasty.
(3) Eight opalescent glazed ware samples and one translucent glazed ware sample were confirmed as the Jun-series ware. The eight opalescent glazes are typical of phase separated glazes.
(4) The most important conclusion is that the two celadon samples with pale green opaque glaze were confirmed as the Ru kiln ware. The two opaque glazes are typical crystalline-phase separated glazes. This finding implies that the Ru ware is identified for the first time in the Dong Xia sites of Russia's Primorye region.
(5) This research is a successful case of interdisciplinary cooperation in the medieval archaeometry of the Far East.

Acknowledgment

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