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Phase composition and internal structure of Bronze Age ceramics from the Eleke Sazy-2 settlement studied by X-ray diffraction and microtomography

A.Zh. Zhomartova[1,2,*], S.E. Kichanov[2], D.S. Shaimukhanova[3], R.S. Zhumatayev[3], K.M. Nazarov[1,2], M.R. Kenessarin[1,2], B. Mukhametuly[1,2,3]

[1]Institute of Nuclear Physics, Almaty, Kazakhstan

[2]Joint Institute for Nuclear Research, Dubna, Russian Federation

[3]Al-Farabi Kazakh National University, Almaty, Kazakhstan

E-mail: zhomartova@jinr.ru

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This paper presents the results of a non-destructive study of ceramic fragments from the Bronze Age settlement of Eleke Sazy-2 located in the foothills of Tarbagatay, Eastern Kazakhstan. The aim of the work was to characterize the raw materials used for pottery production and to obtain information on vessel manufacturing technology. X-ray powder diffraction (XRD) and X-ray microtomography were applied to determine the phase composition and internal structure of the ceramic fragments. The crystalline fraction of the ceramics is dominated by quartz (SiO_2), mica ($KAl_2[AlSi_3O_{10}](OH)_2$), and feldspar ($NaAlSi_3O_8$). The close similarity of phase composition among most of the samples suggests the use of related local raw materials and comparable technological approaches. Microtomographic data revealed the spatial distribution of dense mineral inclusions and pores formed during firing. In several samples, coarse angular grains with relatively high X-ray attenuation were observed, whereas other samples contained mainly fine rounded inclusions. The volumetric fractions and equivalent diameters of these grains were estimated from reconstructed three-dimensional models. The combined XRD and X-ray microtomography results indicate that at least two raw material preparation strategies may have been used, possibly associated with different mineral sources, including riverine and mountain environments. The study provides new archaeometric data on pottery production at the only currently investigated Bronze Age settlement in the foothills of Tarbagatay.

Keywords: archaeological ceramics; Eleke Sazy-2; X-ray powder diffraction; X-ray microtomography; equivalent diameter; archaeometry

Introduction

Archaeological ceramics are an important source of information on ancient societies, their material culture, and production technologies. The study of ceramic artifacts makes it possible to reconstruct technological processes, identify raw-material sources, and trace economic and cultural connections among ancient communities [1, 2].

The Eleke Sazy valley is located on the northern slope of the Tarbagatay Ridge in Eastern Kazakhstan and forms an enclosed basin surrounded by low mountains and crossed by small rivers that are tributaries of the Kargyba River. A number of archaeological monuments from different periods are concentrated in this landscape, and their investigation has yielded valuable material for the study of ancient technologies and cultural traditions [3, 4]. The broader Shilikty-Tarbagatay region also occupied an important position in long-term cultural exchange processes [5].

The Eleke Sazy-2 settlement lies in the northeastern part of the valley near the confluence of the Bolshaya Kargyba, Malaya Kargyba, and Koldenen rivers. The monument was discovered by A.E. Rogozhinsky and I.V. Merz, who identify the site as the first Andronovo settlement in the Tarbagatai Mountains, dating it to the 18th–16th centuries BC, and draw attention to the fact that the monument is the highest in Eastern Kazakhstan [6]. Excavations conducted in 2022 by the Shilikty-Tarbagatay expedition covered 495 m^2 divided into thirty-nine 3×3 m squares and yielded 1015 ceramic fragments, of which about 90% belong to thin-walled vessels and 10% to thick-walled vessels [7].

In the present work, ceramic fragments from Eleke Sazy-2 were examined using X-ray powder diffraction (XRD) and X-ray microtomography. XRD provides information on the crystalline phase composition of ceramics and can be used to assess the mineral nature of raw materials and the possible effects of firing [8]. X-ray microtomography complements diffraction analysis by visualizing the three-dimensional internal structure of the ceramic body, including pores, dense inclusions, and cracks, and enables their quantitative characterization [9]. The aim of the present work is to characterize the phase composition and internal structure of ceramic fragments from the Eleke Sazy-2 settlement using X-ray powder diffraction and X-ray microtomography and to evaluate possible differences in raw material preparation and production technology.

Materials and methods

Ceramic samples

Several representative ceramic fragments (Figure 1) were selected for the study to characterize the pottery assemblage of the Bronze Age population of Eastern Kazakhstan.

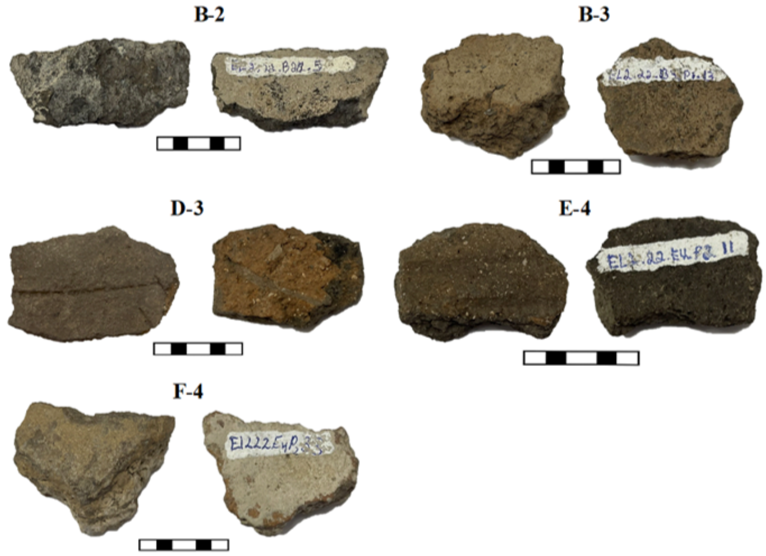

Figure 1. Photograph of the examined samples showing the outer and inner surfaces. Scale bar: 2 cm.

X-ray powder diffraction

X-ray powder diffraction measurements were carried out using a SAXS/WAXS XEUSS 3.0 instrument (XENOCS) equipped with a GeniX 3D source (Mo - $K\alpha$ radiation, $\lambda = 0.71078$ Å) and an Eiger2 R 1M two-dimensional detector (Dectris). The analysis requires only a few milligrams of powdered material and does not cause significant damage to the specimen; therefore, for valuable archaeological materials it can be regarded as a practically non-destructive method. The diffraction data were analyzed and refined with the FullProf software package using the Rietveld method, which allows the mass fractions of the identified phases to be estimated [10].

X-ray microtomography

X-ray microtomography was employed to reconstruct the internal three-dimensional structure of the ceramic fragments without destructive sampling. The method is based on recording a series of two-dimensional projections during sequential rotation of the specimen followed by tomographic reconstruction. The measurements were performed on an IMAX microtomography (PRODIS.COMPACT) with a Spellman X-ray source [11]. The tube voltage was 70 kV and the tube current was 50 μA. A copper filter was used to suppress the low-energy part of the spectrum. Up to 900 projections were collected with an angular step of 0.4°. Image correction, reconstruction, and visualization of the three-dimensional data were performed using ProDIS, ImageJ including the BoneJ module, and VGStudio MAX [12]. The resulting volume data were used to analyze inclusions, pores, and cracks.

Results and Discussion

X-ray powder diffraction

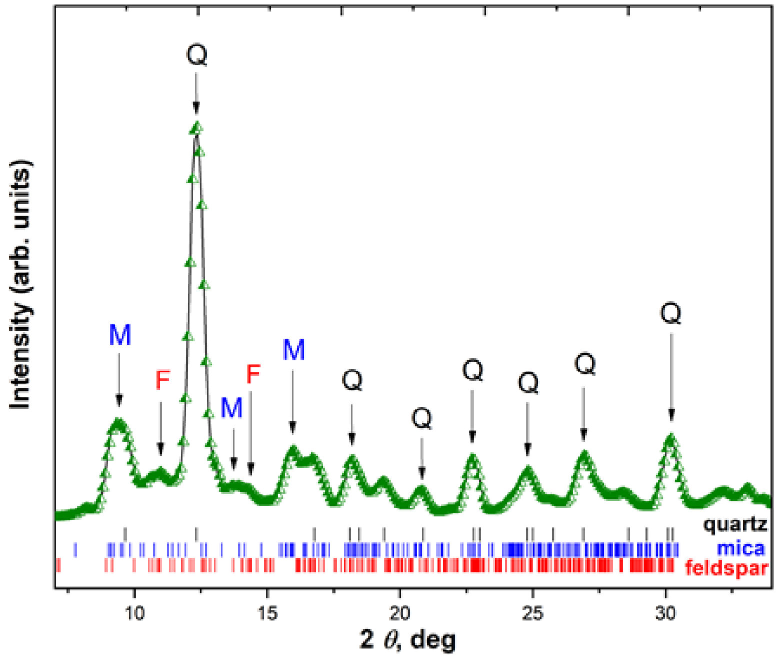

Figure 2. Diffraction pattern of ceramic fragment B-3 from Eleke Sazy-2. Experimental points, calculated profile, and Bragg positions of quartz, feldspar, and mica are shown.

The phase composition of the ceramic fragments was studied by XRD. Figure 2 shows a representative diffraction pattern for sample B-3. The most intense reflections correspond to quartz with a trigonal crystal structure $P3_121$ [13]. Calculated parameters of the unit cell of the quartz phase: $a = 4.908(5)$ Å and $c = 5.414(4)$ Å [14]. Quartz is the dominant phase in all samples except D-3, indicating its major contribution to the ceramic body. The second phase of ceramic fragments is mica with a monoclinic crystal structure $C2/c$. Calculated average unit cell of the mica phase: $a = 5.187(6)$ Å, $b = 9.307(5)$ Å, $c = 20.181(1)$ Å, $\beta = 95.5(2)^\circ$. Figure 2 also shows additional low-intensity peaks that correspond to the feldspar phase. The feldspar crystal structure is described by the triclinic syngony of the space group $C\bar{1}$ with the lattice parameters: $a = 8.23(6)$ Å, $b = 12.78(5)$ Å, $c = 7.08(1)$ Å, $\alpha = 91.17(3)^\circ$, $\beta = 115.54(2)^\circ$, $\gamma = 90.14(1)^\circ$. The content of the observed phases in the ceramic fragments, such as quartz, mica, and feldspar, was calculated using the Rietveld method, and the proportions of these phases were obtained (Figure 3).

The occurrence of mica may reflect the addition of crushed rock rich in mica-bearing minerals, for example granito-gneiss material, as temper, or its natural presence in the clay used for vessel production. At the same time, the presence of quartz-feldspar mineral associations may indicate the use of river sand. This is also evidenced by microcracking on the surface of some fragments, which are often formed as a result of the addition of river sand [15]. Therefore, the variations in mica and feldspar content may reflect differences in raw-material source, manufacturing technology, or vessel function.

Comparison with earlier studies from the same region suggests long-term use of local raw materials for ceramic production from the Bronze Age through later periods [16, 17].

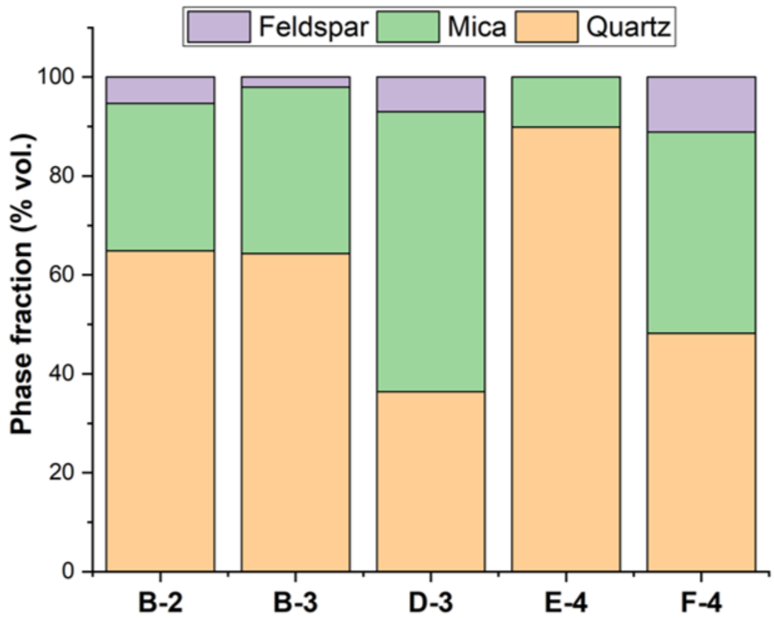

Figure 3. Percentage content of quartz, mica, and feldspar in ceramic samples from Eleke Sazy-2.

X-ray microtomography

The spatial distribution of mineral inclusions and pores was investigated by X-ray microtomography. Figure 4 presents reconstructed three-dimensional models and representative longitudinal slices of the analyzed ceramic fragments.

In all samples, inclusions and pores of different sizes were observed (Figure 4). Based on X-ray attenuation contrast, three material components can be distinguished in the ceramic body. It is assumed that the yellow areas in the reconstructed images correspond to chamotte (Figure 4). However, determining the accuracy of these conclusions remains difficult, since chamotte contrasts poorly with the original raw material of the vessel [9] and can have attenuation values close to those of the ceramic matrix. Large inclusions with high X-ray absorption coefficients were found in samples B-2, D-3, and F-4. The characteristic equivalent diameters (Figure 5) of these grains are mainly in the range of 0.3–1.4 mm, while the largest grain reaches approximately 3.5 mm and corresponds to approximately 0.6% of the total fragment volume. In sample B-2, the total volume of inclusions is 6.17% of the fragment volume, while in sample B-3, the inclusions are significantly smaller, their volume is 1.2% of the total sample volume. Based on this, the presence of large and medium-grained fractions and their smoothed-angular outlines in the samples, as well as their uneven distribution in fragments B-2, D-3, and F-4, may be attributed them to artificial additives in the form of crushed grit [15].

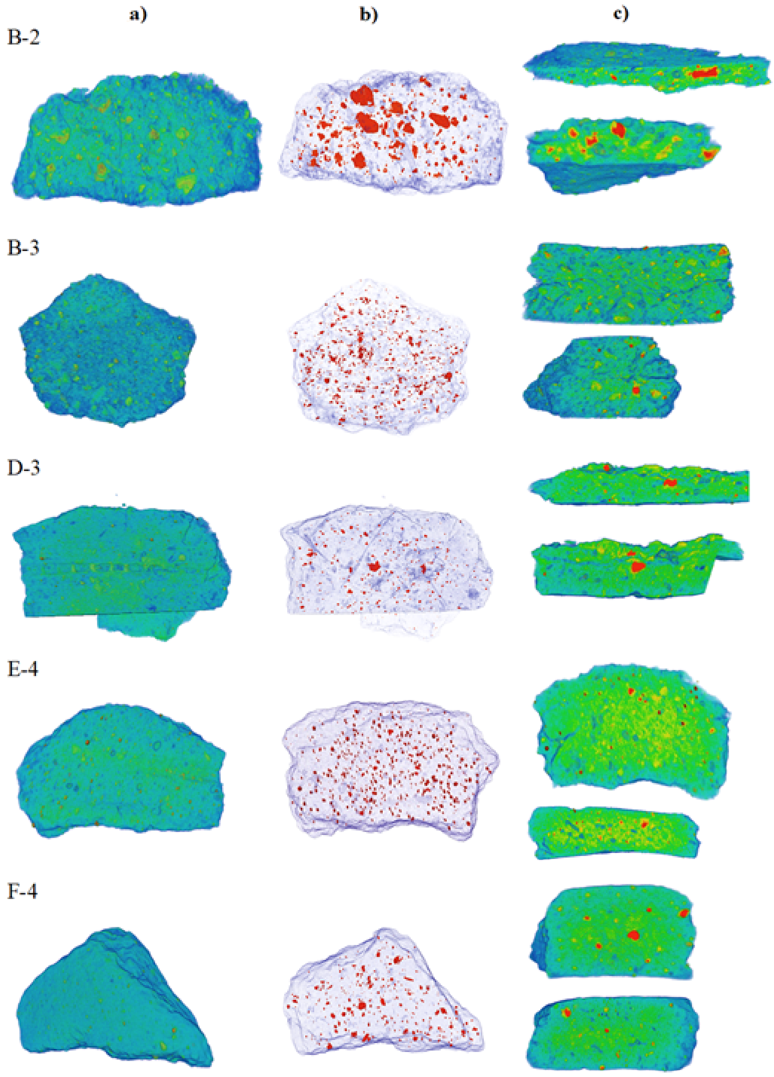

Figure 4. (a,b) Three-dimensional tomographic reconstruction and (c) representative longitudinal slices of a ceramic fragment.

Sample E-4 differs from the others by a high content of relatively homogeneous rounded inclusions with sizes of about 0.2–0.4 mm and by the absence of feldspar according to the XRD data. This sample was likely produced from a lean clay. According to Bobrinsky, inclusions of this type are difficult to classify as artificial additives because they may reduce the plasticity of the raw material [18]. All these differences allow us to conclude that this sample was probably made using different raw materials. At the same time, sample B-3 is structurally the closest specimen to E-4 because it is also dominated by fine inclusions in the range 0.2–0.6 mm. Despite the presence of a small number of larger grains up to 1.1 mm, these may still be regarded as natural impurities [15].

Pores of different shapes were also identified in all studied samples. In B-2, elongated voids with blade-like outlines are clearly visible; the largest of them is approximately 1 cm long. Such pore morphology is characteristic of cavities formed after the burnout of chopped straw or other elongated organic temper [19, 20].

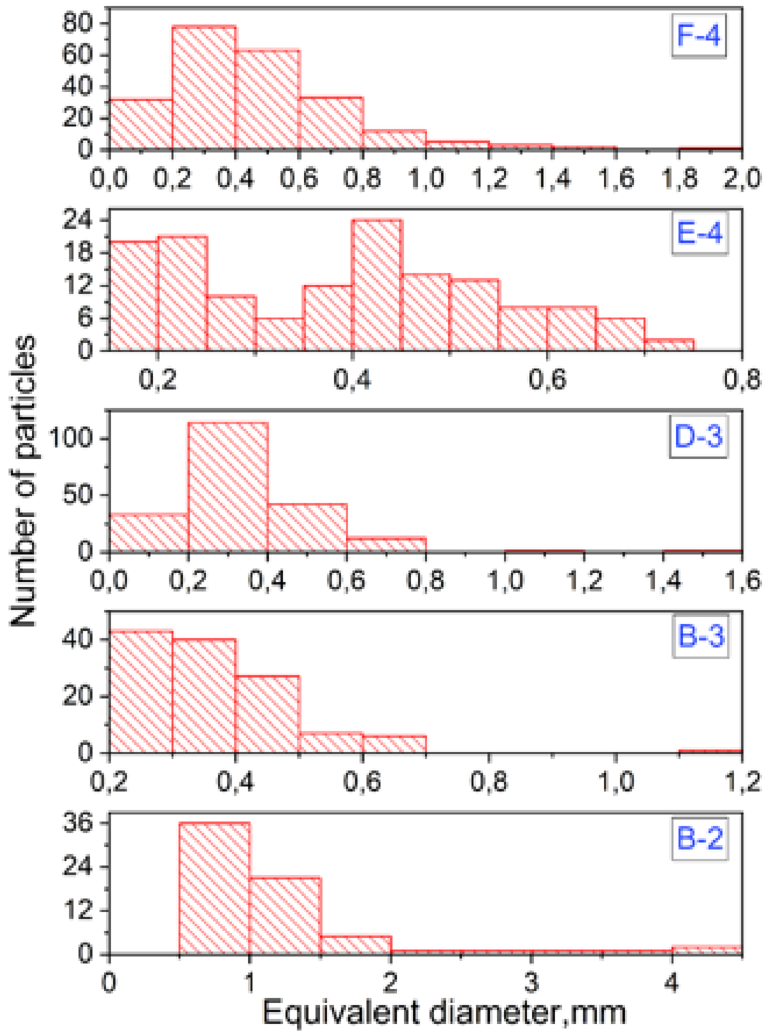

Figure 5. Distribution of equivalent diameters of inclusions observed in a ceramic fragment from the Eleke Sazy-2 complex.

Conclusion

The combined non-destructive investigation of ceramics from the Eleke Sazy-2 settlement has provided new information on the production technology of Bronze Age pottery in the Tarbagatay foothills. First, the similarity in phase composition among most fragments indicates the use of closely related primary raw materials. Second, the internal distribution of inclusions and pores revealed both common structural traits and substantial differences among individual samples. Taken together, the X-ray diffraction and X-ray microtomography results suggest the use of at least two raw-material sources, probably riverine and mountain-derived.

The analogy with samples from the Eleke Sazy burial ground, attributed to the Early Iron Age and the medieval period, may reflect the long-term continuity of cultural traditions in the region, implying intergenerational transmission of basic pottery-making knowledge with gradual adaptation but without fundamental changes in technological practice. Since Eleke Sazy-2 is currently the only investigated Bronze Age settlement in the foothills of Tarbagatay, its ceramics provide an important reference assemblage for future comparative archaeometric studies in Eastern Kazakhstan and neighboring regions.

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