

## SCANNING ELECTRON MICROSCOPY STUDY OF THE MICROSTRUCTURE OF ANCIENT ISSYK-KUL CERAMIC

G. T. Oruzbaeva<sup>1</sup>

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The microstructure of ancient Issyk-Kul ceramic was studied using a scanning electron microscope, making it possible not only to see the relative arrangement of the mineral particles, pores, and microcracks but also to perform a quantitative analysis of the main microstructural metrics. The ceramic samples from the bottom of Lake Issyk-Kul (Kyrgyzstan), where the ruins of many ancient settlements are located, were studied.

**Key words:** Issyk-Kul valley, microstructure of ancient ceramic, scanning electron microscope.

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The historical and archaeological monuments of Lake Issyk-Kul, a high-mountain jewel of Kyrgyzstan, draw the attention of many scientists [1–3], but the use of modern methods of scientific research there is just beginning. At the same time a comprehensive professional study of archaeological artifacts can supplement information on the history and culture of civilizations and aid in the reconstruction of the historical and cultural legacy of Kyrgyzstan.

In the study of ancient Issyk-Kul ceramic it is important to study not only its chemical and mineralogical composition [4, 5] but also the microstructure, which determines many of its properties. It incorporates information on the size and shape of the clay particles, the strength of the clay rock and the conditions under which it is formed, all on account of the specific combination of various morphometric, geometric, and energetics metrics. In archaeological work the analysis of the microstructure of the Issyk-Kul ceramic is quite meager.

The microstructure of ceramic is studied using an optical microscope, transmission electron microscope (TEM) [6], and scanning electron microscope (SEM) [7] as well as other instruments. Each one possesses its own optimal resolving power and its own advantages and limitations.

In the optical as well as transmission electron microscopes the image of the object is formed simultaneously at all of its points, which results in a distortion of the real microstructure.

The principle of image formation in a scanning electron microscope is different. In a high-resolution electron micro-

scope the image of an object is formed successively in time, from one point of the object to another, by moving along the surface of the object a sharply focused beam of electrons — an electronic probe, as a result of which the obtained images are most informative and clear. The preparation of objects for SEM is significantly simpler than for TEM.

The microstructure was investigated using samples from the Tosor River basin (sample No. 1), village of Zharkynbaevo (sample No. 2), and village of Kan-Dobo (sample No. 3). These samples were kindly provided by Professor K. Sh. Tabaldyev of Kyrgyz-Turets University ‘Manas.’

The studies were performed using the VS-300 SEM in the solid-state physics laboratory at the B. El'tsyn Kyrgyz-Russian Slavic University. SEM photographs of samples are displayed in Fig. 1.

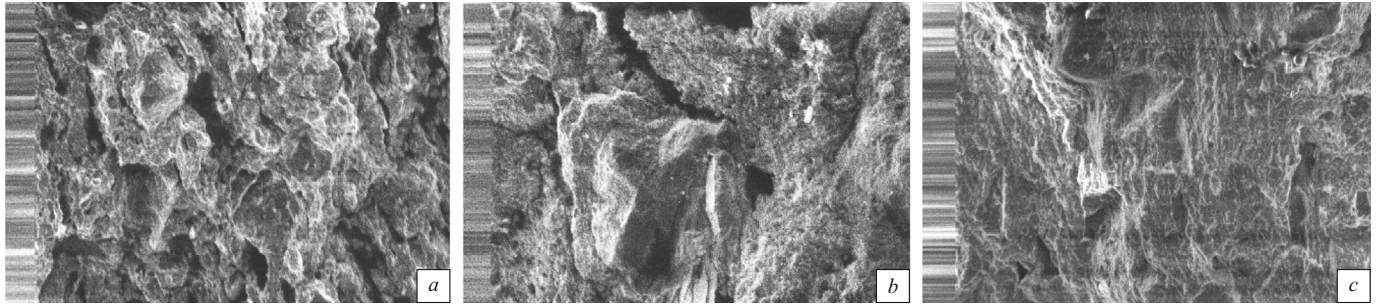
The microstructure of samples according to the SEM images was studied with the aid of STIMAN software [8] in the laboratory of soil science and technical soil reclamation in the Faculty of Geology at Lomonosov Moscow State University.

The SEM images obtained with magnifications  $\times 48$ ,  $\times 102$ ,  $\times 200$ , and  $\times 400$  were studied for each experimental sample. The quantitative metrics of the microstructure were obtained as a result: the total porosity, area and perimeter of the pores, the average values of the area, perimeter, and diameter of the pores, and the filtration permeability and specific surface area. The results of the microstructure analysis are presented in Table 1.

Histograms of the distribution of the structural elements over the equivalent diameters, areas, and form factor were constructed from the results of the analysis, and the dependence of the form factor of the pores on their area was ob-

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<sup>1</sup> I. Razzakov Kyrgyz State Technical University (KGTU), Bishkek, Kyrgyzstan (e-mail: g\_oruzbaeva@hotmail.com).



**Fig. 1.** SEM photographs of samples of a ceramic body: *a*) Tosor River basin, 2nd millennium BC (No. 1), magnification  $\times 300$ ; *b*) village of Zharkynbaevo, 2nd – 3rd millennium BC (No. 2), magnification  $\times 300$ ; *c*) village of Kan-Dobo, 10 – 12th centuries (No. 3), magnification  $\times 220$ .

tained, separate categories of pores were identified in the histograms, and their contribution in the total porosity of the sample was determined. We shall examine the results of a quantitative analysis of the microstructure for the example of sample No. 1 — Tosor River basin (2nd millennium BC), which are displayed in Fig. 2.

According to the data obtained from an analysis of the microstructure of No. 1 using the SEM images the porosity is equal to 12.95% and the specific surface area  $0.043 \mu\text{m}^{-1}$ .

Three categories of pores are present in the pore space (Fig. 2*a*). In Fig. 2  $N_i/(Nl)$  is the probability density;  $N_i$  is the number of pores in the studied interval;  $N$  is the total number of analyzed pores;  $l$  is the length of the interval. These are small ( $D_1$ ) and large ( $D_2$ ) intergrain micropores as well as macropores ( $D_3$ ). The category- $D_1$ , small, intergrain micropores are most numerous in the microstructure. They possess an anisometric strongly elongated shape, and the average equivalent diameter is equal to  $5.81 \mu\text{m}$ . In spite of their large number in the pore space the contribution in the total porosity is very small because of the small size and is equal to about 14%.

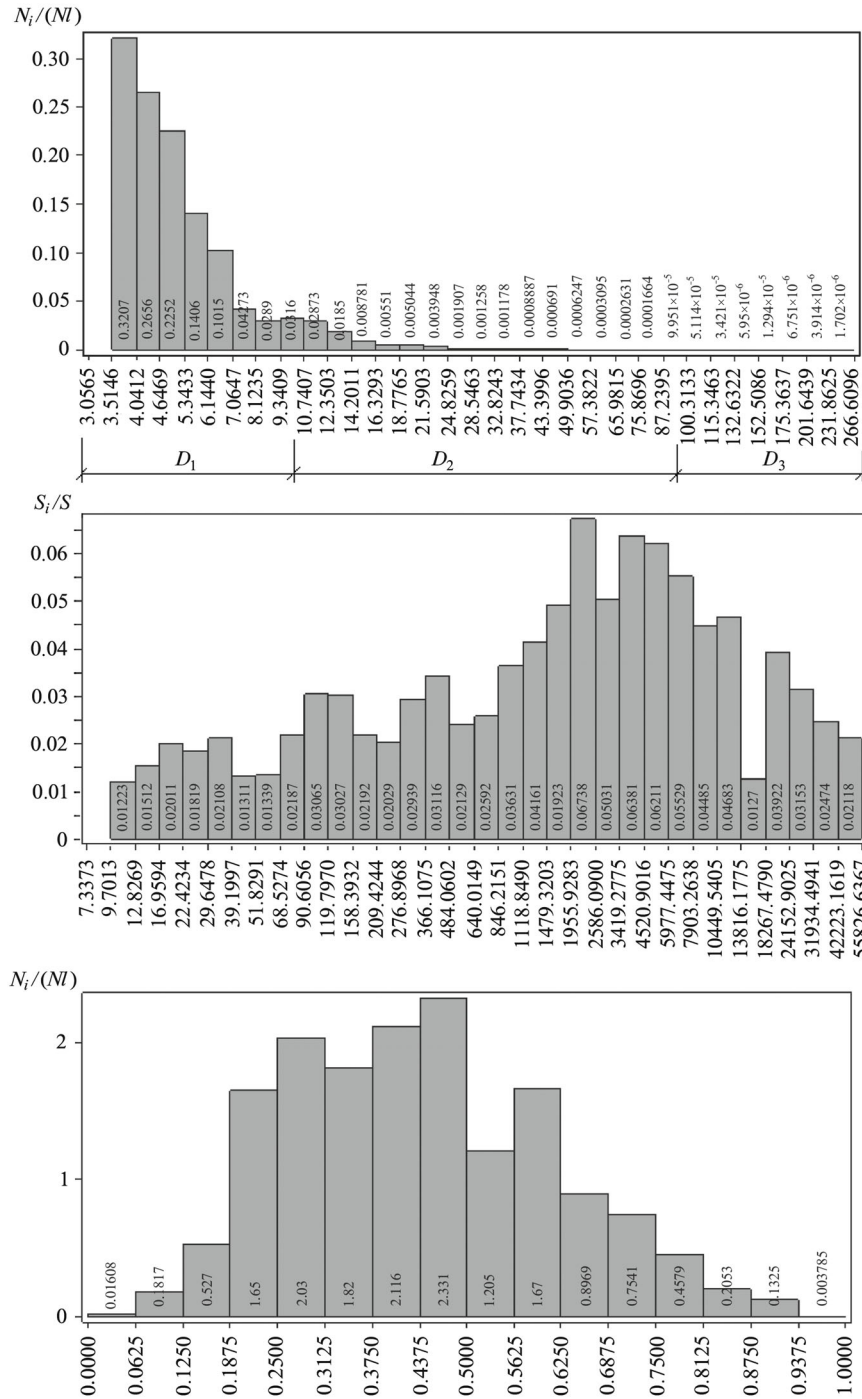
The large  $D_2$  intergrain micropores with anisometric shape and average equivalent diameter  $37.35 \mu\text{m}$  are present in smaller amounts, but they form the bulk of the pore space. The total contribution reaches 59% of the total porosity. The  $D_3$  macropores make the smallest contribution in the pore space. They have an isometric shape and average equivalent diameter  $172.03 \mu\text{m}$ , and they comprise up to 27% of the total porosity.

Different categories of pores come to light in the histogram of the distribution over the total areas. The pore categories manifest as several local peaks in the histogram. This indicates the existence of several combinations of structural elements, which can be interpreted as individual groups of pores with definite size.

To determine the form factor an ellipse is inscribed within the contours of the studied structural elements, and the form factor  $K_f$  is calculated as the ratio of the axes of this ellipse. The algorithm also makes it possible to evaluate the shape of curved pores using the same principle. This factor varies from 1.00 in structural elements of isometric shape to 0.01 in the structural elements with a strongly elongated shape (Fig. 2*c*).

**TABLE 1.** Final Results of the Analysis of SEM Images

Parameter	Sample No.		
	1	2	3
Dating, century	2nd millennium BC	II – III centuries	X – XII centuries
Number of pores	16,910	26,861	35,450
Porosity, %	12.9533	11.8197	6.9615
Total area of pores, $\mu\text{m}^2$	2,635,737.6365	2,447,053.4937	1 557 879.6084
Total perimeter of pores, $\mu\text{m}$	872,498.5576	1,000,764.1449	1 129 172.4445
Average diameter, $\mu\text{m}$ /dispersion	8.6978/122.8055	6.9911/67.1172	5.8208/22.0721
Average area, $\mu\text{m}^2$ /dispersion	155.8686/968 691.3403	91.1006/577 851.8407	43.9458/102 370.9673
Average perimeter, $\mu\text{m}$ /dispersion	51.5966/8599.8480	37.2571/3946.4572	31.8525/1195.0788
Specific surface area, $1/\mu\text{m}$	0.0433	0.0487	0.0514
$D_{\min}$ , $\mu\text{m}$	3.5146	3.4288	3.5146
$D_{\max}$ , $\mu\text{m}$	266.6096	187.4236	258.0634



**Fig. 2.** Results of a quantitative analysis of the microstructure of sample No. 1: a) histogram of the distribution of the pores over the equivalent diameter  $D$ ; b) histogram of the distribution over the total areas  $S$ ; c) histogram of a distribution over the form factor  $K_f$ .

In summary, by analyzing SEM images in terms of the small sample sizes the metrics of the microstructure were determined quickly and reliably, the type of microstructure was determined, the special features of the microstructure were brought to light, first and foremost, the special features of the pore space which are the basis of the specific strength and

deformational properties of ceramics. The results of this analysis can serve as a basis for bringing to light the properties of ceramic raw material and the technology of preparing the molding body and for determining the firing temperature and some techniques used in processing as well as work on the restoration of ancient Issyk-Kul ceramic.

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