Eurasian Journal of Physics and Functional Materials

2022, 6(4), 266-274

Neutron-tomographic study of the structural features of a bronze mirror found in the Akterek burial complex

N. Torezhanova[*,1], O. Myakisheva[1], B. Mukhametuly[2,3], M. Kenessarin[2,3], R. Baitugulov[2,4], A.K. Bekbayev[2,3], K.M. Nazarov[2,3]

[1]Central State Museum of the Republic of Kazakhstan, Almaty, Kazakhstan

[2]Laboratory of Neutron Physics, The Institute of Nuclear Physics of Ministry of Energy, Almaty, Kazakhstan

[3]Frank Laboratory of Neutron Physics, Joint Institute for Nuclear Research, Dubna, Russia

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

E-mail: nabi2nabi@mail.ru

DOI: **10.32523/ejpfm.2022060402**
Received: 18.12.2022 - after revision

This work presents a study on the structure of a bronze mirror found in the Akterek burial complex by using neutron radiography and tomography. The manufacturing features and the physical state of this mirror was analyzed by three-dimensional model. It turned out that the rim on the edge was made of a different alloy, and the border between the rim and the base of the mirror is also visually visible. The morphological features and spatial distribution of pores and voids formed during secondary casting with the application of various patterns and ornaments have been studied in detail. The neutron tomography data made it possible to study the remains of a thin layer of corrosion on the surface of the mirror.

Keywords: neutron; radiography; tomography; bronze mirror; Akterek complex

Introduction

In the activities of historians in the field of preservation of objects of archeology and history, cultural heritage, an important area is research related to determining the degree of preservation and degradation of objects: they are mainly related to the study of the corrosion layer and phase composition, the identification of surface and volumetric internal defects, structural features of the structure of the archaeological site. The results of such research can provide valuable information about the true nature of archaeological objects and provide useful and interesting facts about the likely history of such objects, regardless of their stylistic and typological design [1]. Therefore, recently, in the studies of the characteristics of archaeological objects, an interdisciplinary approach has been widely used, which involves the use and combination of physical, chemical and biological methods [2].

Recently, cooperation has been started and a memorandum of cooperation has been signed between the Institute of Nuclear Physics of the Ministry of Energy of the Republic of Kazakhstan and the Central State Museum of the Republic of Kazakhstan. Joint and complementary archaeological and physical research allows qualitatively and quantitatively to study unique and valuable objects found from various archaeological sites on the territory of the Republic of Kazakhstan.

One of the first objects of research was a bronze mirror found with the specialists of the Museum during excavations at the Akterek barrow, Almaty region [3–5]. To get an idea about the studied object, namely the distribution of materials, the current state, origin and possible defects in the volume of the sample, it is enough to involve imaging methods of non-destructive testing. One of the promising methods of non-destructive testing is neutron imaging (radiography and tomography). Neutron radiography is a technique for obtaining an image or shadow projections of a sample using a neutron beam [6]. A practical development of this method is neutron tomography, in which a volumetric reconstruction of a three-dimensional model of the sample is performed from a set of individual radiographic projections [7]. The advantage of neutron radiography and tomography methods in the study of archaeological metal objects, in this case a bronze mirror, is the nature of the interaction of a neutron with a matter, which determines the deep penetration of neutrons into the thickness of massive objects, sensitivity to the distribution of hydrogen-containing compounds, sensitivity to differences in material density and chemical composition [8, 9].

In order to demonstrate the possibilities of the neutron tomography method, this paper presents the results of studies of a bronze mirror included in the collection of the Central State Museum of the Republic of Kazakhstan. By analyzing the three-dimensional structural data of neutron tomography, information was obtained on the spatial distribution of various phases, the presence of internal defects, inhomogeneities and the spatial distribution of pores inside the mirror.

Material and descriptions

The archaeological expedition of the Central State Museum of the Republic of Kazakhstan in the burial complex of the Akterek burial ground, mound No. 7 (Republic of Kazakhstan, Almaty region) found a disc-shaped mirror with a flat rectangular handle, there is a high rim along the edge, ornamented with two dragon fish with wings [10]. One wing flies over the water, the other in the clouds. Made of bronze, casting technique in deep relief. The art interpretation and expressiveness of individual elements quite realistically convey the signs of a carp. Presumably belongs to the Late Han dynasty. Diameter 92.(1) mm and weight 173.(5) grams The thickness of the mirror handle has a size of 15.0(8) mm. The date of the complex burial of the XIII-XIV centuries. The real photo of the front and back views are shown in Figure 1.

Figure 1. Real photo of the front (a) and back (b) sides of the bronze mirror.

Experimental instrument

This study was carried out on the 1st horizontal channel of the WWR-K research reactor using the TITAN experimental facility [11, 12]. This facility is intended for obtaining neutron radiographic images with thermal neutrons and computed tomography of samples and is similar in principle to x-ray imaging. Thermal neutrons are obtained as a result of the process of thermalization of fast neutrons, obtained from a fission reaction, in a water moderator with a temperature of about 300 K.

The TITAN facility uses a direct geometry of neutron beam and is located radially with respect to the neutron source, so a sapphire filter is used to reduce unwanted background radiation [13]. The collimation of the thermal neutron beam is formed by an aperture system which consists of several pinholes of different diameters. Depending on the needs of the experiment, it is possible to vary the diameter of the aperture system for different modes: high resolution or high intensity [14].

The neutron image resolution depends on the beam geometry and can be obtained by adjusting the main setup parameters: the incident beam divergence, determined by the ratio of the hole size D and its distance from the sample L, and the sample-detector distance l. For example, in the standard case when

L/D=350 (divergence $0.163°$) and the distance between the sample and the detector (l) is 35 mm, the blurring of the neutron image (d) is approximately ~ 100 μm, described by the relation $d = l * \frac{D}{L}$ [15].

The neutron beam after collimation falls on the research sample, on which it is attenuated due to absorption and scattering process on the sample. The attenuation of the neutron beam mainly depends on the thickness, composition, density of the materials, as well as the energy of the neutron. The neutron beam attenuation process obeys the Beer-Lambert law. The detector system for obtaining a neutron image is equipped with a CCD camera and a scintillation plate. The neutron beam passing through the test sample falls on the scintillation screen and is converted into visible light, then reflected from the mirror and recorded by the camera. As a result of this process, a two-dimensional shadow projection or neutron radiographic image of the sample is obtained. The detected shadow images are then corrected for the camera's dark current and the incident neutron beam is normalized [16].

By using a rotating table to rotate the sample in the range of at least $180°$ and registering shadow projections, it is possible to perform a tomographic reconstruction of the three-dimensional internal structure of the sample. First, each 2D projection image is converted into sinograms containing one row of data for all angles. A single slice of the 3D volume can then be reconstructed from these sinograms. Each rendered layer contains voxels (3D pixels) and is characterized by attenuation coefficients as gray values and can be rendered as a grayscale image. The reconstructed image set is a 3D matrix with attenuation coefficients that can be visualized as the internal structure of the sample using appropriate 3D imaging software.

In the presented work, tomographic experiments were carried out by rotating the sample by $180°$ and with a rotation step of $0.5°$, and the total number of measured angular projections was 360. The size of the field of view was 110x110 mm. The exposure time per one projection was 20 s, and the total time of the tomography experiment was 4 hours. The radiographic angular projections were corrected by subtracting the camera dark current image and normalized to the incident neutron beam using the ImageJ software package [17]. The tomographic reconstruction of the three-dimensional model of the samples was performed using the STP program [18]. The Oimoen algorithm was used to reduce ring artifacts. The size of one voxel in our study was $54 \times 54 \times 54$ μm. Avizo software was used to visualize and analyze the reconstructed 3D data.

Results and Discussion

Neutron radiography results

An example of normalized projections of (at 0 and 180 degrees) obtained from neutron radiographic measurements and subsequently used to reconstruct the internal structure of a bronze mirror is shown in Figures 2 a-b. The rim on the edge contrasts well with the internal elements. Despite this, the ornaments and patterns of the mirror are also distinguishable. In the central part of the bronze mirror, each feather of the wings of the dragon fish and even individual pores are clearly visible. Separate small regions with a high neutron attenuation coefficient are also observed inside the mirror rim. In this case, the weakening of the neutron beam, due to the processes of scattering and absorption, mainly depends on the thickness of the deposited layer or the relief of the patterns.

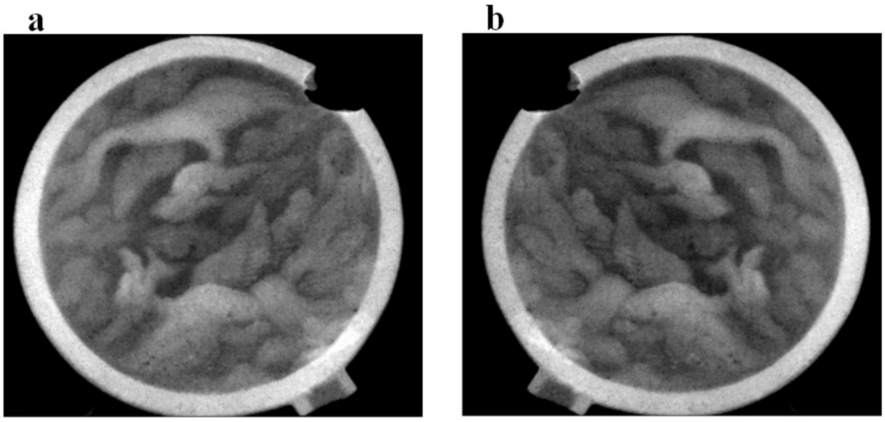

Figure 2. Normalized neutron projections of the front (a) and back (b) side of the bronze mirror. Background is black.

Neutron tomography results

This section of the article presents the results of neutron tomographic analysis. One of the main tasks was to show the internal inhomogeneities, or structural features associated with the manufacture and method of casting, of the bronze mirror. After tomographic reconstruction processes, a set of cross-sectional images was obtained and a three-dimensional model of the test specimen was created. Figure 3 shows front and back views of a 3D model of a bronze mirror, and the color scheme corresponds to neutron beam attenuation coefficients from high values (red) to low values (blue).

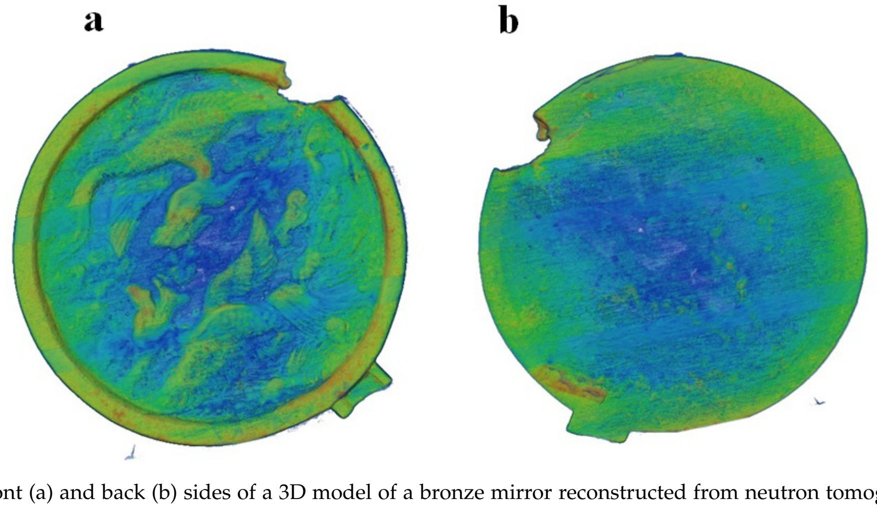

Figure 3. Front (a) and back (b) sides of a 3D model of a bronze mirror reconstructed from neutron tomography data.

The total reconstructed volume was 67316 mm^3. Examples of neutron tomographic sections in different spatial planes of a bronze mirror are shown in

Figure 4. A detailed analysis of these sections revealed that the rim on the edge of the mirror was made of a completely different material. This can be observed in the 70/1960 section, where the boundary of two different media is clearly visible for which the neutron attenuation coefficient is different. If the rim and the mirror were cast together, then the design would be solid and it would not be possible to distinguish the contrast, and in this case we can distinguish the border of two different alloys. Figure 5 a-b shows a close-up section of 90/1960 and a selected three-dimensional volume of the rim and handle of the mirror, the volume of which occupies 25.6% of the total volume of the mirror. Also, based on the comparison of the neutron attenuation coefficient, it can be argued that the copper content in the rim of the mirror is relatively higher than in the bases of the mirror.

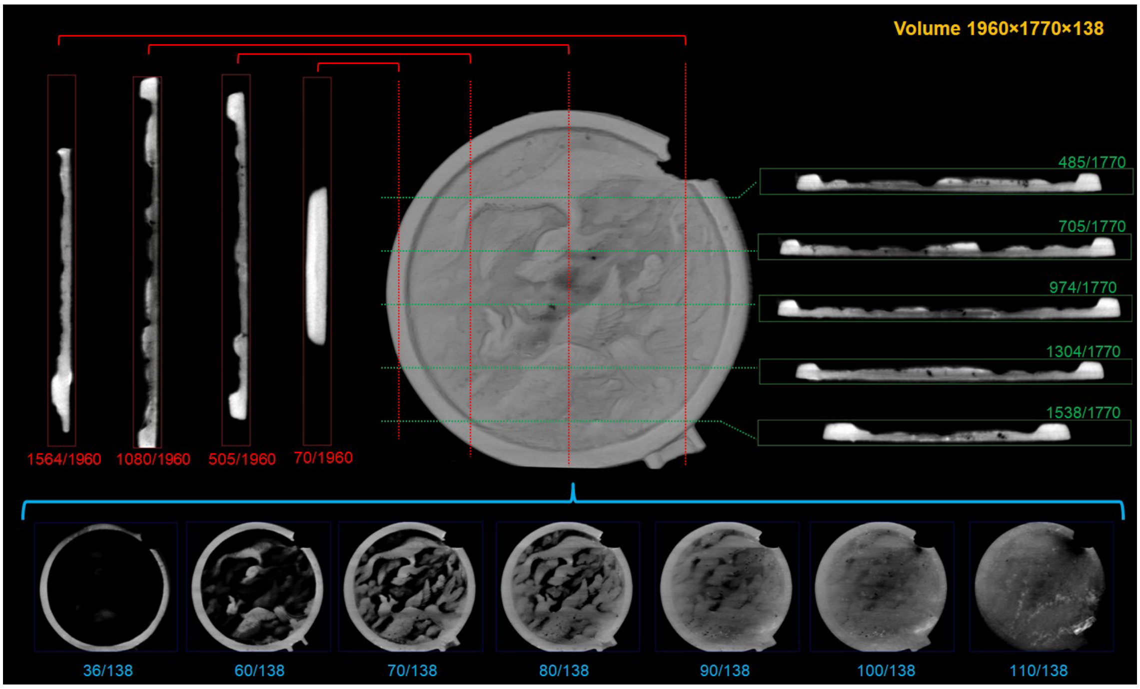

Figure 4. Volumetric visualization of a bronze mirror with virtual sections. The total volume is $1960 \times 1770 \times 138$ voxel^3 . The position of the slices is indicated with lines of different colors. The bright regions are highly attenuating regions within the mirror.

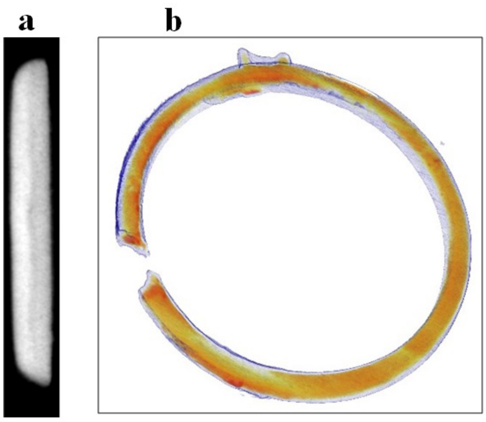

Figure 5. a) Vertical section (90/1960) taken from the rim of the mirror and b) a separate three-dimensional image of the rim of the mirror.

For a bronze mirror, the corrosion layer in the front surface was cleaned by the restorers, but a small part of the corrosion of the ornaments left (especially on the wings of dragons) is not visible by tomography. The backside of the bronze mirror (see Figure 1-b) has areas of thick green corrosion (presumably copper carbonate hydroxide) along with areas of red corrosion that are thought to be cuprite. Figure 6 shows a section of the rear side of the mirror and shows the distribution of the corrosion layer. The corrosive layer is marked with yellow lines for clarity, the area where the museum number of the mirror is written using a correction fluid is marked in red. The entire volume of these corrosion is 0.3% of the total volume of the mirror. The thickness of these corrosion layers does not exceed 400–500 μm.

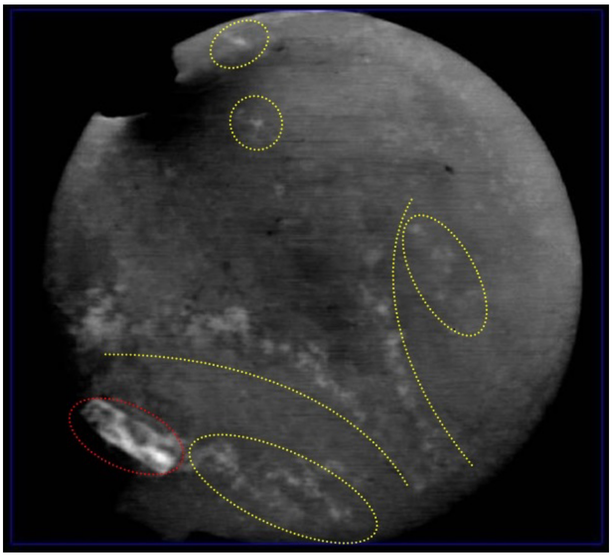

Figure 6. Vertical section (110/138) of a 3D model of a bronze mirror. Yellow lines and circles indicate the region where the corrosion layer is spread.

Another important point observed from neutron tomography data is the presence of pores in the bronze mirror structure. Pores are small holes formed during metal casting due to the presence of gases. The presence of pores can adversely affect the mechanical properties of a metal product, since such defects inside the structure are less resistant to external influences. Figures 7 a-b-c show the mirror section (80/138), the volumetric representation of the pore distribution in the 3D model, and the distribution of separated pores and voids. Depending on the volume or size, the pores were painted in different colors. It can be seen from picture 7 a-b, that the pores are localized under the ornaments and drawings of the mirror, mainly under the wings of the fish-dragon. It is obtained from the processes of secondary casting on the basis of mirrors and the application of various figures and ornaments. Figure 6 d-e shows a histogram of the volume and equivalent diameter of a bronze mirror. The entire pore volume is 0.05% of the total volume of the bronze mirror, most of the pores have a volume of less than 1 mm^3, and only 3 pores with a volume of more than 1 mm^3 are observed. The equivalent pore diameter ranges from 0.2 to 1.8 mm.

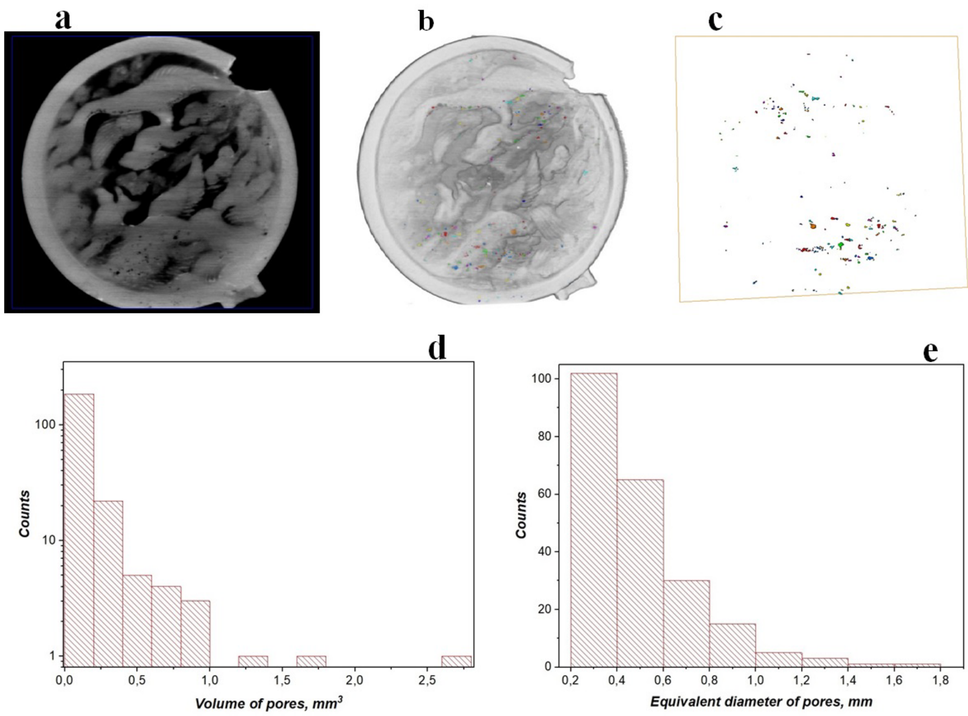

Figure 7. a) Virtual section (80/138) of a three-dimensional model, b) three-dimensional view of a mirror with pores, and c) volumetric distribution of pores and voids of a bronze mirror. The distribution of pores and voids by (d) volume and by (e) equivalent diameter is shown.

Conclusion

Thermal neutron tomography has proven to be a very powerful method for "qualitative" study of the internal structure of a bronze mirror. Using this method, a three-dimensional model was restored, which made it possible to reveal the features and inhomogeneities of the structure of the bronze mirror found in mound 7 of the Akterek burial complex, and shed to reveal the various components and methods used to assemble such a sample. It was also possible to identify and visualize the lines of ornaments, the boundaries of various inclusions, corrosion products with great accuracy, as well as calculate the morphological parameters of the distribution of pores inside the sample.

Acknowledgments

This work was carried out as part of the implementation of the scientific and applied research of the IRN program BR08555277 "Archaeological monuments of the Akterek tract: issues of topography, typology and museumification", funded by the budget program administered by the Ministry of Culture and Sports of the Republic of Kazakhstan in 2020-2022..

References

[1] L. Bertrand et al., Acc. Chem. Res. **54** (2021) 2823–2832. [*CrossRef*]

[2] D. Creagh, D. Bradley, Physical Techniques in the Study of Art, Archaeology and Cultural Heritage (Amsterdam, Netherlands: Elsevier, 2007) 282 p.
[3] K.B. Sihymbaeva et al., KazNU Bulletin: History series **106**(3) (2022) 113–121. [*CrossRef*]
[4] O. Myakisheva et al., Sciences of Europe **3** (2021) 9–15. [*CrossRef*] (In Russian)
[5] O.A. Myakisheva et al., KazNU Bulletin: History series **103**(4) (2021) 183–193. [*CrossRef*]
[6] R. Van Langh et al.,Anal. Bioanal. Chem. **395** (2009) 1949–1959. [*CrossRef*]
[7] S. Kichanov et al., Journal of Imaging **4** (2018) 25. [*CrossRef*]
[8] B.A. Bakirov et al., Eurasian Journal of Physics and Functional Materials **6** (2022) 56–70. [*CrossRef*]
[9] S.E. Kichanov et al., Journal of Surface Investigation **11** (2017) 585–589. [*CrossRef*]
[10] H.A. Aitkul et al., Lingvo-Science **19** (2018) 15–23. (In Russian)
[11] K.M. Nazarov et al., Nuclear Instruments and Methods in Physics Research Section A, **982** (2020) 164572. [*CrossRef*]
[12] B. Muhametuly et al., Journal of Surface Investigation **13** (2019) 877–879. [*CrossRef*]
[13] K.M. Nazarov et al., Eurasian Journal of Physics and Functional Materials **5**(4) (2021) 169–180. [*CrossRef*]
[14] K.M. Nazarov et al., Eurasian Journal of Physics and Functional Materials **5**(1) (2021) 6–14. [*CrossRef*]
[15] N. Kardjilov et al., Materials Today **14** (2011) 248–256. [*CrossRef*]
[16] P. Vontobel et al., Physica B **385–386** (2006) 475–480. [*CrossRef*]
[17] C. A. Schneider et al., Nat. Methods **9** (2012) 671–675. [*CrossRef*]
[18] F. Brun et al., Advanced Structural and Chemical Imaging **3** (2017) 4. [*CrossRef*]