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Studies of Coins of Medieval Volga Bulgaria by Neutron Diffraction and Tomography

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Abstract—The phase composition and spatial distribution of chemical components in the volume of coins of medieval Volga Bulgaria are studied using the neutron-diffraction- and neutron-tomography methods. Two coins belonging to different time periods of this medieval state are studied: a Samanid multidirham dating from the first half of the 10th Century and a silver dirham dating from the period of the reign of the Bulgarian emir Bulat-Timur. It is established that both coins consist of a copper-silver alloy. In the Samanid multidirham, the average volume contents of copper and silver are found to be about 50%. Minor spatial variations in the chemical composition are found in the volume of the multidirham under study. It is established that the volume average silver content in the Bulat-Timur dirham is about 95%.

Keywords: methods of nondestructive testing, coins of Volga Bulgaria, neutron diffraction, neutron tomography, cultural heritage

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INTRODUCTION

Detailed investigations of the physical and chemical properties of ancient coins are one of the most important directions in the nondestructive testing of objects of cultural heritage [1, 2]. On the one hand, numismatic material contains precious information about the economic and commercial development of ancient civilizations and states; on the other hand, coins are convenient model objects [3–6] for investigations of corrosion and crack-formation processes that occur in found copper or bronze, silver or gold objects. It should be noted that the experimental data obtained in such investigations are of great importance for the development of a methodology for the restoration and conservation of precious archeological finds [7, 8] and are an invaluable material for identification of the authenticity of the most precious artefacts.

Investigations of ancient coins by means of modern nondestructive methods are related not only to the experimental recording of internal defects and the detection of corrosion creepage [3, 6], but also to detailed study of variations in the chemical and phase content inside the objects under investigation [9–13]. It is known that the chemical composition of numismatic material may give important information about deposits of silver and gold ore [12–14] of which the coins are made, their conformity with a particular historic period or the features of coining, and detection of fakes [15]. Nondestructive X-ray fluorescence analysis, electron microscopy, and metallography methods recognized in archeological and restoration scientific communities have a significant limitation concerning the depth of penetration into the thickness of the coins under investigation [16–19]. Data on the chemical composition obtained by means of the above-mentioned methods correspond only to the surface layers of the material, which may be critical when investigating coins made of copper-silver alloy [16, 20]. It is characteristic of such coins to have the maximum silver content on their surface [11, 16], which is associated with certain processes that occur during initial ore melting [21], the peculiarities of coining [22] or with the processes of liquation of the silver-enriched phase of a two-component alloy [11, 16]. Therefore, data on the chemical composition obtained from the coin surface can be cardinally different from the distribution of chemical components over the entire volume [23–26]. In this case, it seems to be justified to use nondestruc-

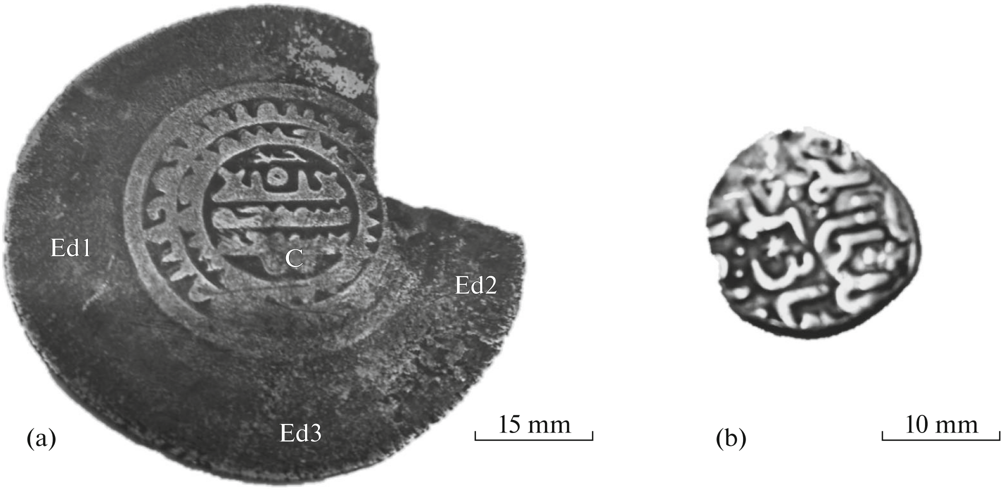

Fig. 1. Photographs of the investigated coins of ancient Volga Bulgaria: the Samanid multidirham (a) and the Bulat-Timur dirham (b). For the images of each coin, the corresponding scales are presented. On the photograph of the multidirham, symbols "Ed1", "C", "Ed2", and "Ed3" refer to the parts of the coin for which the neutron-diffraction data were obtained.

tive structural-diagnostics methods with a high penetrability into the width of metal objects. Such methods include neutron diffraction [9–11], which provides information on the phase composition of the object under investigation, and the neutron radiography and tomography methods, which allow the pattern of the spatial distribution of internal components, including the chemical composition, to be obtained [3, 6, 11, 24].

In this study, the results of structural investigations of ancient coins from the territory of Volga Bulgaria [26, 27] are presented. The presence of abundant numismatic material found in the course of the archeological excavations of towns and settlements [28] is an important indicator of the economic and commercial development of this medieval state. The convenient position of Volga Bulgaria at one of the central trade routes led to the creation of economic preconditions for the formation of commodity-money relations in the Volga region both within it and with neighboring regions, which resulted in the development of local coining and the wide usage of coins minted in other regions. The study of silver coins of Volga Bulgaria by means of new methods can make it possible to reveal the peculiarities of the chemical composition and the technology of their production to reveal the laws of the Middle Volga region and other regions of that age [13, 29].

The numismatic material is selected with consideration for the peculiarities of money turnover in this region. The coins for analysis were taken from the reserves of the Archeological Museum of the Republic of Tatarstan; they belong to the two most active periods of development of commodity-money relations in the region and the maximum circulation of metal coins in trade relations. The Samanid multidirham of the period of Al-Amir as-Said Abu Salikh I bin Nuh khan, dating from the first half of the 10th Century [30], characterizes the premongolian period of development of trade relations of Volga Bulgaria. The second coin, the silver dirham from the Karatun treasure minted in 1361–1367, during the reign of Bulat-Timur, who was the Golden Horde emir of Bulgaria, represents another period of active coin circulation in the Volga region.

These coins are different in size, which allows determination of the limits of informativeness of the used method at the current stage. The difference between the neutron scattering and absorption cross sections for copper and silver makes it possible to study the phase composition and reconstruct its spatial distribution in these coins over their entire volume.

EXPERIMENTAL

Photographs of the investigated coins are presented in Fig. 1. The Samanid dirham is a large coin with a diameter of about 52 mm. It has a triangular chip missing. On the head side of the coin, one can distinguish legend remnants written in kuphic script with a circular ornamental pattern. The coin thickness does not exceed 0.8 mm. Its mass is 7.764 g.

The silver dirham of the Bulat-Timur period is a well-preserved whole coin with a diameter of ~17 mm. On the head side and reverse of the coin, one can clearly see the legend written using kuphic script. The coin thickness is uneven: on one side it reaches 0.6 mm, while at the opposite thin side, it is about 0.2 mm. The coin mass is 0.930 g.

Studies of the crystal structure and phase composition of the coins were carried out by means of neutron diffraction using a special diffractometer DN-6 [31] at the IBR-2 high-flux reactor for investigating microsamples. The diffraction spectra were measured at

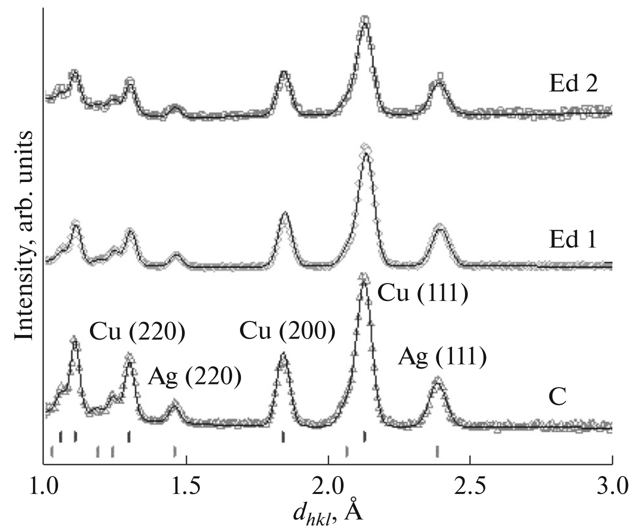

Fig. 2. Neutron-diffraction spectra of the multidirham obtained for different parts of this coin. The experimental points and the profile calculated according to the Rietveld method are presented. The vertical strokes refer to the calculated positions of the diffraction peaks for the cubic phase of copper (the upper row) and silver (the lower row). The spectra correspond to particular parts of the investigated coin (see the designations in Fig. 1 and in the text). Several diffraction reflections and the corresponding Miller indices for the copper and silver phases are indicated.

scattering angles of $2\theta = 90°$. For these scattering angles, the diffractometer resolution at the wavelength $\lambda = 2$ Å was $\Delta d/d = 0.025$. The characteristic time of measuring was 30 min for one spectrum. For additional measurements of different parts of the multidirham, a cadmium collimator with a slit diameter of 5 mm was used, which made it possible to study the phase composition of several parts of the coins: the center of the coin is indicated by symbol C in Fig. 1a and the three edge parts are indicated by Ed1, Ed2, and Ed3. Analysis of the diffraction data was performed by the Rietveld method using the FullProf code [32].

The copper and silver spatial distributions in the coins were investigated by neutron radiography and tomography using a specialized experimental station [33, 34] at channel 14 of the IBR-2 pulsed high-flux reactor. Due to the different degrees of attenuation of intensity of the neutron beam [35] in the process of its passage through components of different chemical compositions, this method makes it possible to get information on the phase distribution throughout the volume of the investigated material with a micro-scale spatial resolution [11, 36]. In the neutron-tomography experiments, the volume reconstruction of the inner construction of the studied coin is performed using a set of separated radiographic projections obtained at different angular positions of the specimen with respect to the neutron-beam direction. The three-dimension (3D) model obtained by this method is a data array consisting of 3D voxels [35], which characterize the degree or the coefficient of attenuation of the neutron beam at a particular point of the specimen under study. The voxel size in the neutron-radiography experiment was $52 \times 52 \times 52$ μm. In the radiographic experiment, the conversion of neutrons to visible light detected by a CCD video camera was carried out by means of the plate of a ^{6}LiF/ZnS scintillator with a thickness of 0.1 mm produced by RC TRITEC Ltd (Switzerland). The tomographic experiments were facilitated by a HUBER system of goniometers with a minimum rotation angle of up to 0.02°. A high neutron flux at the specimen under study determines a short exposure time of 10 s for recording one neutron image. The neutron data obtained in this experiment were corrected to exclude background noise and normalized to the incident neutron beam using the ImageJ software package [37]. Tomographic reconstruction from the set of angular projections of the investigated objects was performed using the H-PITRE code [38]. Visualization and analysis of the obtained 3D data were carried out using the VGStudio MAX 2.2 software package developed by Volume Graphics (Heidelberg, Germany).

Table 1. Volume contents of copper and silver obtained from the neutron-diffraction data for different parts of the multidirham

Part of the coin	Copper content, %	Silver content, %
C	49.5(2)	50.4(2)
Ed1	51.0(2)	48.9(2)
Ed2	50.4(2)	49.5(3)
Ed3	51.9(2)	48.1(3)

RESULTS AND DISCUSSION

Neutron Investigations of the Samanid Multidirham

Figure 2 shows the neutron-diffraction spectra obtained for different parts of the coin under study: the edge of the coin opposite the chip (indicated “Ed1” in Fig. 1a), the coin center (“C”), and the region in the immediate vicinity of the chip (“Ed2”). The obtained diffraction spectra correspond to the following two phases of the coin material: the cubic phase of silver and the cubic phase of copper belonging to the $Fm3m$ space group. By analyzing the relative intensities of the neutron diffraction peaks obtained by the Rietveld method, the corresponding volume contents of silver and copper in the coin material were revealed (Table 1). It is seen that the coin material is an alloy that consists of nearly identical volume fractions of silver and copper. Taking into account the copper and silver densities, it is possible to evaluate the average mass fraction of silver in the coin—about 58.9%, or ~4.5 g of the total mass of the coin.

Minor variations of about several percent in the relative volume fractions of silver and copper for different

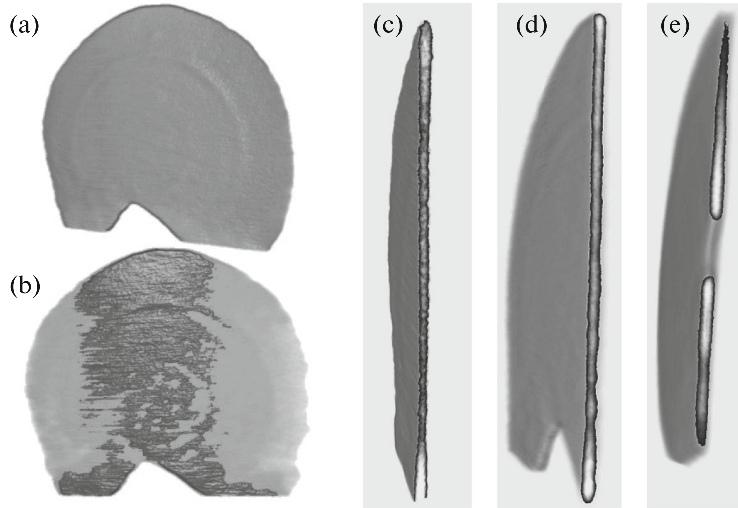

Fig. 3. 3D model of the investigated multidirham reconstructed from the neutron-tomography data (a) and the part of the coin with a higher silver content separated from the three-dimensional data (b). The lighter parts correspond to the maximum coefficient of attenuation of the neutron beam or to regions with a high silver concentration. The darker regions are characterized by a lower total-neutron-absorption cross section, which corresponds to regions with a high copper content. Longitudinal virtual sections of the reconstructed 3D model of the investigated multidirham corresponding to parts "C" (c) and "Ed1" (c), and part "Ed3" (d) of the coin and the transverse virtual section of the 3D model of the multidirham in the region near the chip "Ed2" (e) are presented.

parts of the investigated coin are evidence of relative homogeneity of the phase-component distribution throughout the coin volume. To study the spatial distribution of copper and silver over the coin volume in more detail, neutron-tomography experiments were performed. The attenuation coefficient for a neutron beam with an average wavelength of neutrons $\lambda \sim 2$ Å for silver is $\Sigma_{Ag} = 0.79$ cm^{-1} [39]. This value is higher by nearly one order of magnitude than the corresponding coefficient for copper: $\Sigma_{Cu} = 0.09$ cm^{-1}. In this case, one can expect the formation of a high radiographic contrast in the neutron radiography and tomography experiments.

Figure 3a shows a 3D model of the investigated coin reconstructed from the neutron-tomography data. As a result of analysis of the obtained 3D model, taking into account the small thickness of the coin and the effects associated with higher neutron absorption on the coin surface because of patina and external pollution, it was revealed that the distributions of the corresponding volumes with a large content of silver and those characterized by a dominant content of copper are uneven. It should be noted that, according to the neutron-diffraction data, the difference in the relative volumes of these parts of the coin does not exceed several percent (Table 1). To estimate the relative volumes occupied by silver and copper components, the procedure of segmentation of the 3D neutron data was performed [40, 41], as a result of which regions of the coin characterized by a higher silver content were separated. The spatial distribution of these regions over the coin volume is shown in Fig. 3b. It was established that the entire volume of the 3D model of the coin is formed by 59868460 voxels, which corresponds to a volume of 8417.98 mm^3. The calculated volume of the coin enriched with silver consists of 32232288 voxels, which corresponds to 4532.11 mm^3 or to 53.8% of the entire volume of the coin. Despite the fact that the separation of the silver and copper phases in the neutron-tomography method has an exclusively evaluative character, the values obtained by this method are in good agreement with the data obtained in the neutron-diffraction experiments (Table 1).

Several transverse and longitudinal virtual sections of the 3D model of the multidirham, which were reconstructed from the neutron-tomography data, are presented in Figs. 3c–3e. It is seen that the maximum neutron-beam attenuation and, thus, the dominant silver content are observed for regions of the coin located near the chip and in the upper part "Ed1" of the coin. The corresponding edges of the coin, one of which corresponds to region "Ed3", are characterized by a higher copper content (Table 1).

The mechanisms of the formation of an uneven spatial distribution of the silver and copper components over the multidirham volume are not clear. The neutron-tomography data point out that the variation in the chemical-component composition of the coin (Figs. 3c and 3d) has, apparently, a volume character, which is not characteristic of silver liquation processes that have an exclusively surface character [11, 16]. At the same time, in the regions near the chip, one can observe that the silver component is dominant, which may be associated with additional chemical treatment

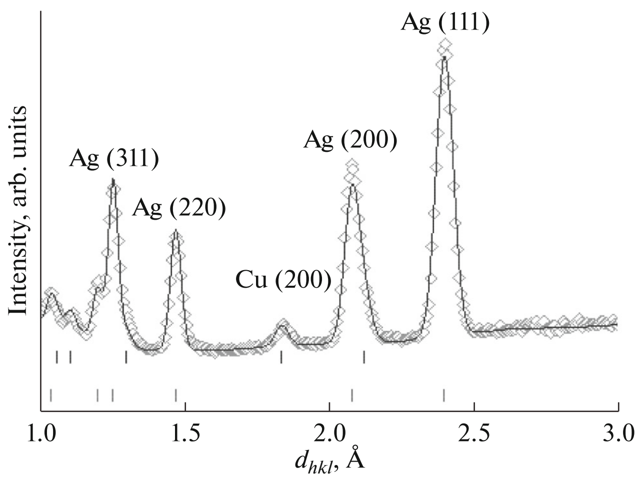

Fig. 4. Neutron-diffraction spectrum of the Bulat-Timur silver dirham. The experimental points and the profile calculated according to the Rietveld method are presented. The vertical strokes refer to the calculated positions of the diffraction peaks for the cubic phases of copper (the upper row) and silver (the lower row). The diffraction reflections and the Miller indices for the copper and silver phases corresponding to them are indicated.

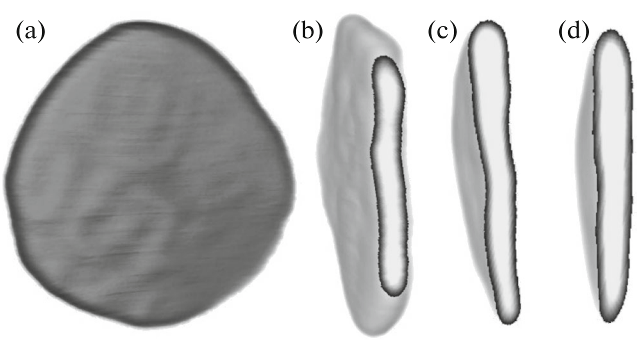

Fig. 5. 3D model of the Bulat-Timur silver dirham reconstructed from the neutron-tomography data (a). Longitudinal virtual sections of the reconstructed 3D model that correspond to the thin edge of the coin (b), the coin center (c), and the thick edge of the coin (d) are presented.

of the coin [42] by other researchers. We can suppose that the volume variation in the chemical composition of the multidirham may be related to the peculiarities of the copper-silver ore composition [13, 29] or to the processes of its melting when it underwent coining.

Neutron Investigations of the Silver Bulat-Timur Dirham

The small size of this coin did not allow us to scan its different regions; therefore, neutron-diffraction data were obtained for the entire volume of the coin under study. Figure 4 shows the neutron-diffraction spectrum of the Bulat-Timur coin. It is seen that for this dirham, the silver phase is dominant over the entire coin volume. One can observe only a weak reflection at $d_{hkl} \sim 1.8$ Å, which corresponds to the diffraction peak (200) of the cubic phase of copper. Analyzing the neutron-diffraction data, we calculated the volume content of silver and copper in the dirham material: 94.8(3)% of silver and 5.2(1)% of copper. The calculated mass of copper in the coin is 0.04 g; that of silver is 0.89 g.

To study the internal structure of the dirham, neutron radiography and tomography experiments were performed. Figure 5 shows a 3D model of the Bulat-Timur dirham reconstructed from the neutron-tomography data and several virtual transverse sections of the coin. The neutron data point out that silver is evenly distributed in the coin material. The spatial localization of the copper phase was not established because of its low concentration.

CONCLUSIONS

Nondestructive structural diagnostic methods—neutron diffraction and neutron tomography—were used to investigate coins belonging to different epochs of development of medieval Volga Bulgaria. It is established that both studied coins consist of a copper-silver alloy. However, the Samanid multidirham from the 10th Century is characterized by a very high copper content—on average, about 50% of the entire volume of the coin material. The spatial distribution of silver and copper in this coin is uneven, which can be associated with both the peculiarities of the initial ore and with the processes that occur during its coining. No outcrops of a high silver concentration on the coin surface, which are characteristic of the liquation processes, were revealed. As distinct from the multidirham, the dirham of the age of the reign of Bulat-Timur (the end of the 14th Century) consists almost completely of silver. The volume content of copper in this coin is extremely low—5.2%. The data on this coin composition are in good agreement with the results of investigations of the Golden Horde coins of that epoch [43].

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