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Metallurgical investigations on 17th century Maratha Shivrai copper coins

ABSTRACT

The technical and numismatic complexities of Maratha Shivrai copper coins have been well documented. However, more information still needs to be on the chemical, mineralogical, microstructure composition, and metallurgical process employed for coin minting. Five Shivari coins (17-18 century CE) were studied under ED-XRF, XRD, and cross-sectional analysis by SEM-EDX. Scientific investigations reveal that the Maratha practiced excellent cupellation techniques to remove most ore impurities. The ED-XRF data on the surface and interior indicate that the coin comprises pure copper, and copper content varies between 97.73% to 100%. The coin surfaces are free from ore impurities like sulphur, Lead, tin, phosphorus, etc., and the black stains observed are due to copper mineralization detected through XRD analysis. There is a slight iron concentration gradient on the coin surfaces (4.48%) vis-à-vis the inner core (0.14%). Cross-sectional analysis revealed many cracks and cavities in the coin interior due to copper mineralization threatening its stability if not taken care of. The archaeo-metallurgical studies of the coin interior reveal that significant impurities like Lead, sulphur, tin, iron, etc., are present in traces due to excellent polling, and Shivrai is a single-phase copper coin.

***Keywords**: Shivrai copper coins, Maratha dynasty, Cupellation, Metallography, Corrosion*

1. INTRODUCTION

The main focus on research regarding the conservation of cultural heritage relates to finding history and archaeology, recording material losses and intensity of degradation, surface conditions, and materials defects, changes in physio-chemical and phase composition of the materials, investigations on microstructures, and monitoring corrosion process for undertaking remedial measures [1,2]. Archaeological and cultural heritage objects are treasured due to their distinctiveness and antiquity; therefore, is looked-for to study them by contemporary non-destructive methods [3]. It is well known that chemical and metallurgical analyses are potent tools supporting archaeological research in every field. Based on the investigation of analytical data of historical objects, one can precisely identify the complexity of materials for better characterization.

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With some exceptions, metals were primarily sourced for the minting coins that conformed to the technological development of that period and bore the standard design and numismatics of that particular dynasty. Ancient numismatics are regarded most valuable and cultural artistic finds that played a significant role in the technological development of the past. Therefore, dating the ancient coins to their authenticity, iconography, provenance, and metallurgical process practiced for their products plays an essential role in archaeological research [4].

The earliest coins in India were punch marked. The design of coins gives information about the particular civilization, political stability, and religious belief and helps in finding the genealogy of the rulers. Further, it helps in the chronological study of history for the proper documentation of Indian chronology and to understand the development of metal technology in different periods. The coins could also be an excellent source to understand the metal technology of a particular dynasty [5]

Copper is one of the most essential metals in the antiquity. As records of the metal copper, the literature survey shows the use of bronze objects initially followed by copper-arsenic, copperantimony, copper-tin, etc. [6]. Copper alloy, particularly bronze, has been used for millennia in producing different tools and artifacts because of their specific properties, such as good formability, corrosion resistance, and colour reflection [7,8]. Copper is the earliest metal used in antiquity, and many problems associated with it have been the identification of finds of copper antiquities that have been reported in India from the Harrapan period [9,10]. The analysis of slags from Harrapan sites lead to the identification of types and locations of ores and their metallurgical processes [11]

The copper pyrites are chalcopyrite's ($CuFeS_2$) and bornite ($CuFeS_4$), the two most important ores in ancient India, mainly used for extracting copper metal. Compared to iron, copper is a less helpful metal, and mainly, it was sourced for the fabrication of coins and alloys like brass, bronze, etc. In India, the metallurgical process for the extraction of copper was practiced from the earliest Harrapan period to the end of the 17th Century for many applications. The punch-marked copper coins of the Kushan Dynasty (1st -3rd CE), the old Mytra Dynasty of Ayodhya (100CE – 350CE), and many rulers of Northern India minted copper coins during that period (1st Century BC). Ancient copper coins of the Gupta Dynasty (4th – 6th CE) and Kushan Kings have been excavated in many locations in the Ganga River belt of Northern India. In southern India, copper coins of Sat Vahana and Khat Rapa Dynasty (2nd – 6th BC) have been found during excavations. In medieval India, copper was utilized as one of the primary metals for coinage, along with silver and gold. Copper slowly oxidizes by oxygen to form Cuprite (Cu_2O) which changes to Tenorite (CuO). [12,13]

The difficulties in the determination of the origin of metals are due to several problems, such as very scarce deposits of copper ore, high temperature required for smelting, corrosion of the metal during burial, changes in trace elements composition, and easy remelting and reuse of copper for fabricating other objects [14].

For copper-based coins, many unreliable analytical data have been reported in the literature [15]. If the coins' exposed surfaces are compositionally different from their inner core, inaccurate results may be obtained by employing surface analysis techniques of XRF, PIXE, EPMA, etc. [16,17]. Based on the archaeometric investigation; it has been reported that copper coins are mostly devoid of any concentration gradient [18]. The concentration gradient is primarily observed in leaded bronzes due to forming islands of lead-rich phases if in contact with chemically inactive/fewer active phases. Therefore, removing the thin surface layer from the coin exposes the inner layer that is representative of the entire composition [18] with some exceptions.

In some cases, certain impurities in the ores may cause problems of removal or remelting of old coins may give rise to inhomogeneity. Lead and copper are invariably present in significant quantities in all brittle coins, and other elements are in traces [19]. However, lead up to the concentration of 0.4% in the copper-lead system does not show any sign of embrittlement of the material [18]. There is also a correlation between the degree of embrittlement and the amount of Lead in the coin [18-20]. Due to inefficient cupellation in the past, it was impossible to remove the lead content from the ores resulting in embrittlement in the coins.

With the modern technical advancement, several sophisticated analytical techniques have now been developed for the accurate analysis of ancient objects that is very high in sensitivity precision and are non-destructive.

Metallographic techniques undertake the microstructural studies of ancient coins by cutting thin sections of metallic objects and examining the polished section for gaining information like metallic composition, micro-structure, method of fabrication, extant corrosion, etc. As applied the examination of metallic objects through metallographic techniques was practiced in the first few decades of the 20th Century. Now the techniques is of great help to conservators, metallurgists, corrosion scientists, archaeologists, etc. [21].

The technical and numismatic complexity of the Shivrai coins have been well documented [22], but there needs to be more chemical composition, coin inhomogeneity, impurities, and corrosion characteristics. Therefore, additional investigations must be performed using SEM-EDX on a cross-section of a specific fragment of the coins. The details of the coins' morphological features and chemical composition were obtained through the scanning electron microscope coupled with an energy-dispersive X-ray spectrometer (SEM-EDX). The SEM-EDX data was compared to the one obtained by XRF to get better information and to visualize the core compositional difference from the exterior of the Shivrai coins. In addition to cross-sectional analysis and elemental compositions, the corrosion features and surface behaviour were also studied through X-ray diffraction (XRD). This has helped to indicate the degradation of materials for the coins.

The main objective of this investigation is to determine the composition and microstructure of the coins to understand the techniques of their manufacturing and analyse the corrosion products.

2. HISTORICAL BACKGROUND OF COINS

Ancient India is known for its gallants, and many warriors have demonstrated their bravery in defending their motherland. The great Maratha King Maharaja Shivaji is one of the most acknowledged warriors of Indian history with a remarkable personality in the 17th Century. After his coronation on 6th June 1674, he assumed the title of a king and became the first Chattrapati of the Maratha empire. The rule of Maratha was a dramatic turn in the history of India. Shivaji Maharaj was the first Maratha king to establish Hindavi Swaraj in the 17th Century and was the first ruler to start Rajshakha (Royal Era). Spread across the Deccan plateau; they had a long-lived empire from 1664-1819 AD. The presence of Mughals started declining due to the Maratha power. He understood the importance of currency for the stability and well-being of his empire and minted coins Hon and Shivrai during his reign. Hon is a gold coin, and Shivrai is a copper coin. The Shivrai Maratha coins remained in circulation till the end of the 19th Century, mostly in the western part of India [21,22]. Around 150 different kinds of Shivrai extant have been reported to exist to date [22]. Before 1830, Shivrai was valued at 1/74 to 1/80 of a rupee [23]. In 1885, during the British rule over India, the local revenue collectors were asked to collect all Shivrai coins and deposit them in the treasury. It was with the purpose to eliminate strong native currency rivals and to bring new piece valued at 1/64 of a rupee.

3. MATERIALS AND METHODS

3.1. Visual appearance

To investigate Shivrai coins of Marathas, a very small piece of coin was sectioned. Five coins numbered as MS1 to MS5 were studied for this work.

The coins were first photographed for its obverse and reverse view, and the same is shown in (Figure-1). An examination of the inscriptions reveals that Shri/Raja/Shiva is inscribed in three lines on the obverse side, and Chattrapati is inscribed in two lines on the reverse side in Devanagari Script. The coins are not perfectly circular in shape. The thickness of the coins is around 4mm, and die-struck technology was adopted for minting. The Maratha Shivrai coins were mainly minted during the period 1674 to 1830. The physical parameters of the coins, like diameter, weight, and thickness, are listed in Table-1. A greenish-black colour layer with some corrosion product was observed on both sides of the coin.

Figure 1. Obverse and reverse view of Maratha Shivral copper coins

Slika 1. Pogled na avers i revers bakarnih novčića Maratha Shivral

For microstructure examination, very small samples were sectioned from two coins using a slow-speed diamond cutter to study the surface topography of the coins. The sectioned part was cold-mounted in a fast-setting resin that sets in a time span of 30 minutes. On setting, the part was mechanically polished through different grades of silica carbide/emery paper of 400 to 2000 grade., cloth polished with alumina powder, and distilled water to get a mirror finish. Subsequently, the sectioned part was coldly polished using alumina powder and distilled water to get a mirror finish. The polished surface of the coin was chemically etched with the help of 100 ml distilled water, 30 ml hydrochloric acid, and 10 gm ferric chloride so as to reveal the microstructure of copper.

Table 1. Physical parameters of shivrai copper coins

Tabela 1. Fizički parametri šivrai bakarnih kovanica

Sr. No.	Diameter (cm)	Weight (gm)	Thickness (mm)
MS1	1.8	8.50	3.8
MS2	1.9	8.65	3.86
MS3	2.0	9.15	3.92
MS4	1.9	9.10	3.89
MS5	1.9	9.23	4.01

3.2. Portable X-Ray fluorescence (XRF)

X-ray fluorescence (XRF) has extensively been used as an analytical technique for the determination of coin composition. Due to its simplicity, accuracy, and ease of operation, XRF is the most versatile technique for the analysis of coins. The elemental composition was determined using the μ-XRF (Bruker Artax Model 200). The scans were recorded at different points on the obverse and reverse sides of the coin at a live time of 30 s. The instrument was operated at a maximum operating voltage of 50kV, and the current of the molybdenum X-ray tube was kept at 700μA. At least 3 measurements were taken for each point and data averaged.

3.3. X-Ray diffraction (XRD)

The surface impurities and minerals/corrosion products observed in the coin surface were characterized using non-destructively by X-Ray Diffractometer (Model: Rigaku Smart Lab) with a PhotonMax high-flux 9kW rotating anode X-ray source coupled with a HyPix-3000 high energy resolution 2D multidimensional semiconductor detector. XRD data were collected for the 2θ range from 5 to 120^o with a scan rate of 2 /min. The peaks were identified by JCPDS software. Although XRD analysis was carried out for all the coins, due to the similarity of results, spectra of coins MS2 to MS5 are reported for this study.

3.4. Scanning electron microscope – Dispersive X-Ray spectrometry (SEM-EDX)

Scanning electron microscopy (SEM) coupled with energy-dispersive X-ray spectrometry (EDX) was chosen to analyse the microstructure of artifacts since the X-rays do not alter the basic structure of the object. SEM helps in visualization of the surface morphology and topology of the object with the help of the secondary electrons. EDX analysis which is based on X-ray fluorescence, is the oldest and most widely used technique for elemental analysis of ancient coins due to its non-destructive nature, the possibility to analyse a large number of elements in a wide concentration range, fast analysis, excellent analytical parameters, etc. The EDX analysis is based on the excitation of the elements using an incident electron beam, followed by the emission of a characteristic X-ray radiation with a specific wavelength that allows the identification of each element; the specific intensity of the emitted X-ray allows the determination of the element concentration.

The coins MS1 and MS2 were sectioned, and the sectioned part of the coin was analysed under energy dispersive X-ray analysis (EDX) based on a field emission scanning electron microscope (FE-SEM) and FEI Nova Nano SEM 450 field emission scanning electron microscope (FE-SEM) with a Bruker XFlash 6I30 EDX analyser used to study sectional microstructure.

4. RESULT AND DISCUSSION

4.1. XRF Analysis

The chemical composition of the coins was obtained by ED-XRF, and the data is listed in Table-2. To investigate surface inhomogeneity, a few microns thick flake was removed mechanically from the obverse side of all coins, and ED-XRF analysis was performed on the exposed surface (Table-2). The copper content on the obverse sides of the coins varied between 97.78% to 98.6% for the five numbers of Shivrai coins. On the reverse side, the copper percentage varied between 97.73 to 99.20, indicating copper was the main constituent for the coins. After removal of a flake from the exterior surface of the coins, the copper percentage varied between 99.10 to 100%. Except for coin number MS1, none of the coins showed the presence of chloride on the outer surface. In coin MS1, the chloride content of 0.43% was detected on the obverse surface. Calcium was detected from the outer surfaces of the coins MS1, MS2, and MS4 in the range of 0.26% to 0.67%. probably due to external contamination or as an impurity. Iron is present as an impurity on the outer surfaces of all the coins, and iron content varied between 0.79% in coin MS5 to 4.48% in coin MS4 (Table-2). There is some degree of surface enrichment of iron on the outer surfaces of the coins since ED-XRF measurements taken after the removal of the metal flake showed iron content in the coin interior varying between 0.75 to 0.80%. The cross-sectional analysis by SEM-EDX of the coin MS1 and MS2 have shown iron content of 0.14% to 0.35%, respectively, in the inner core (Table-3). No sulfur was observed on the outer surfaces of the coins, while traces are present in the inner core observed through the cross-sectional analysis. The coin is also free from any lead contamination though traces were detected in the inner core. Sulfur might have lost from the external areas due to its high volatility. Lead (Pb) is commonly found in archaeological copper and alloys. Sometimes it was added intentionally, but most of the time, it is found naturally with ore of copper. During ore processing, it gets extracted in copper and visible in archaeo-materials. The solubility of Lead in copper is very low (about 0.007%), which is why it does not affect the solidification structure and remains present as a globule in the copper matrix. The Lead has a negative effect on the physical properties of the final product, but it makes the molten metal less viscous and facilitates casting. Its presence in traces in the Shivrai coins may be considered as an ore impurity which was not eliminated during smelting.

Table 2. Composition of shivrai copper coins (weight %)

Tabela 2. Sastav šivrai bakarnih kovanica (težinski %)

S. No	Measurement Side	Cu	Fe	Ca	Cl	S	K	Sn	Pb	P
MS1	Obverse	97.91	1.32	0.33	0.43					
	Reverse	97.73	2.26	-	-	-	-	-	-	-
	After Flake removal	99.21	0.78	-	-					
MS2	Obverse	98.61	1.38	-	-					
	Reverse	98.91	0.82	0.26	-	-	-	-	-	-
	After Flake removal	100	-	-	-					
MS3	Obverse	98.19	1.80	-	-					
	Reverse	98.70	1.29	-	-	-	-	-	-	-
	After Flake removal	99.26	0.73	-	-					
MS4	Obverse	97.78	2.20	-	-					
	Reverse	94.83	4.48	-	-	-	-	-	-	-
	After Flake removal	99.24	0.75	0.67	-					
MS5	Obverse	98.33	1.66	-	-					
	Reverse	99.20	0.79	-	-	-	-	-	-	-
	After Flake removal	99.19	0.80	-	-					

4.2. XRD Analysis

X-ray Diffraction studied all the coins to determine the corrosion products and surface behavior of the coins. XRD pattern of four coins (MS2, MS3, MS4, MS5) are presented in **Figure-2**. From XRD data, it is observed that cuprous oxide (Cu2O) and cupric oxide (CuO) are invariably formed on coins. The cuprous oxide was detected as a significant corrosion compound in the coins. In coins MS2 and MS4, the presence of Cu2O was observed, and from the X-ray diffractogram, it appears that CuO is dominant in coin MS4 as it shows intense surface corrosion compared to the rest of the coins. The presence of cuprous oxide could be due to air oxidation of copper and some black spots of tenorite (CuO) resulting from the transformation of the cuprite. Visual appearance also gave greenish coloration on the outer surfaces at isolated spots. Due to burial or poor storage conditions and exposure to oxygen or moist air, cuprite, the first widely occurring alteration mineral of copper, is formed. Cuprite plays a decisive role in the protection of copper antiquities; however, an increase in oxygen pressure breaks the conformity of the cuprite layer and provokes dissolution. The formation of copper oxide (CuO) provided resistance, and probably for this reason, the coins are very little corroded. Overall, the Shivrai coins are single-phase copper alloys, as evidenced by investigative studies.

Table 3. Elemental composition by energy dispersive X- Ray analysis (Wt %)

Tabela 3. Elementarni sastav pomoću energetsko disperzivne rendgenske analize (Wt %)

S. No.	Cu	C	O	Fe	Ca	S	K	Sn	Pb	P
MS 1	81.39	10.55	7.44	0.14	0.10	0.11	0.10	0.15	0.10	0.02
MS 2	77.80	14.58	6.54	0.35	0.30	0.20	0.13	0.10	0.12	0.04

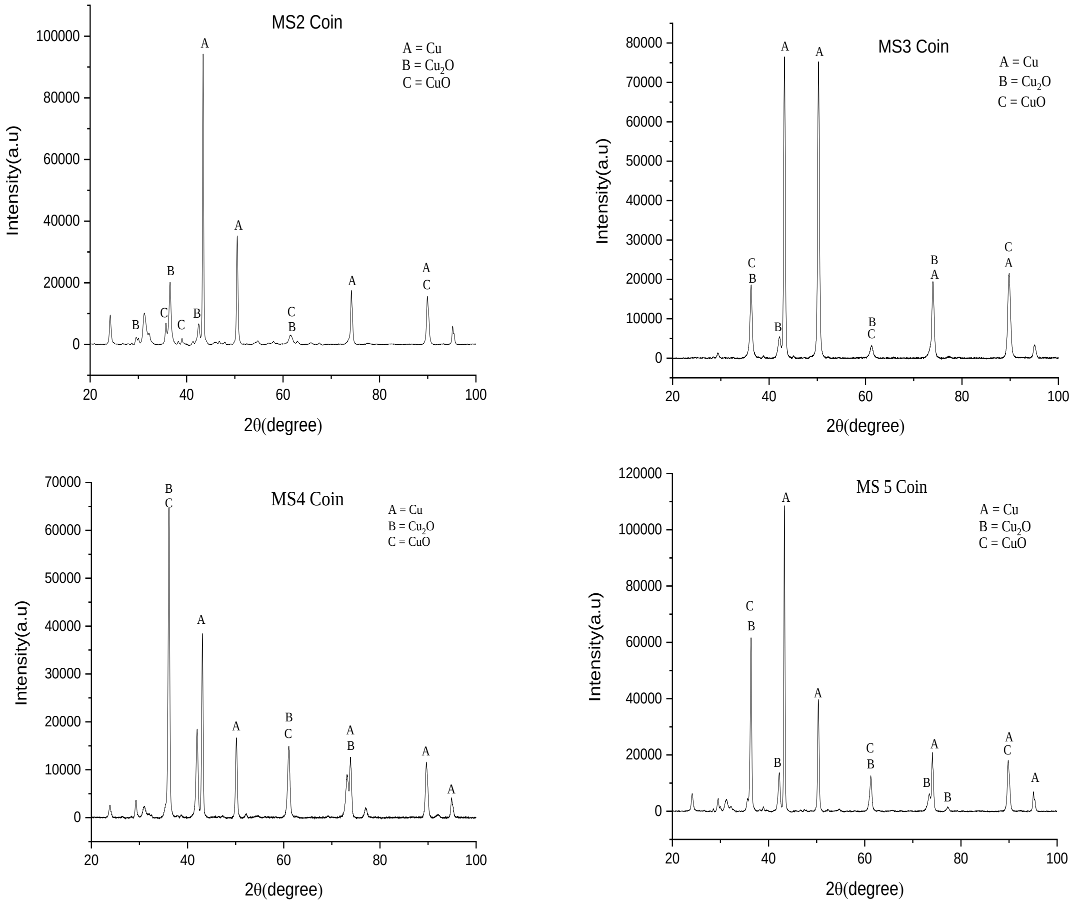
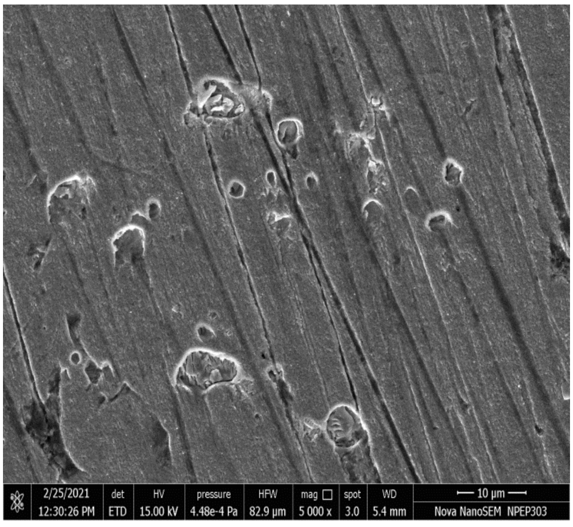


Figure-2. XRD diffractogram of shivrai copper coins MS2 to MS4

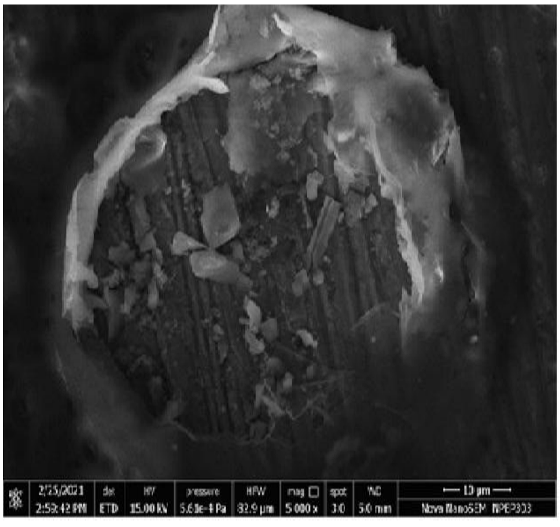
Slika 2. XRD difraktogram šivrai bakarnih kovanica MS2 do MS4

4.3. SEM-EDX Analysis

The combination of instrumental techniques always provides better information about the coin composition and metallurgical process and answers any queries besides indicating the types of ores exploited for the minting of coins, refinement degree of ores, etc. The knowledge of the original composition also discriminates the coins between the original and the fake. The Shivrai coins MS1 and MS2 were subjected to cross-sectional analysis, and the result of SEM photomicrographs are presented in Figure-3 (A&B), taken at various magnifications.



a)



b)

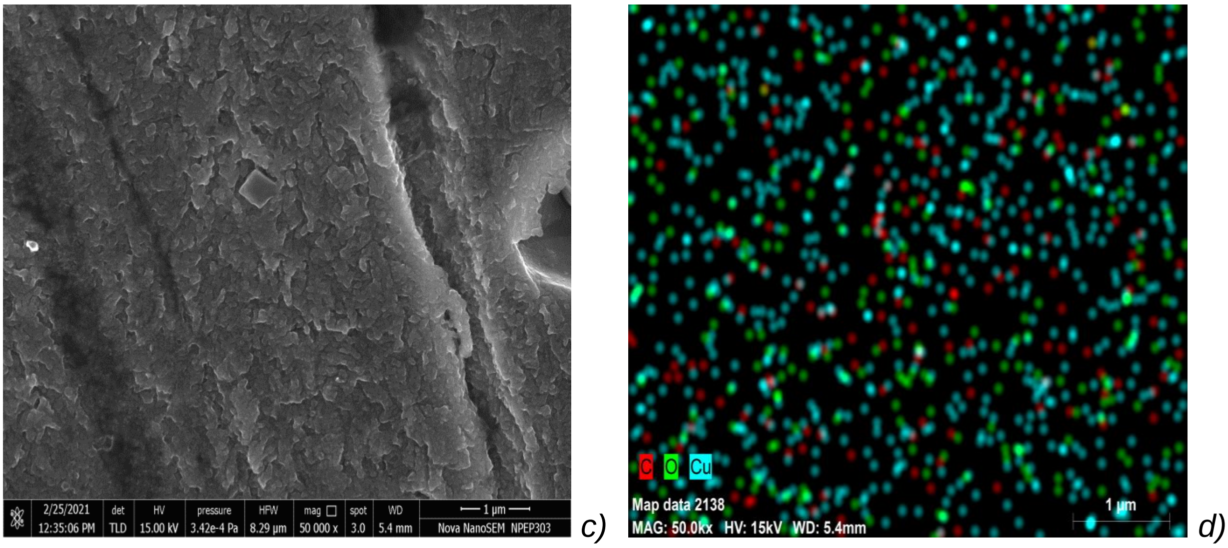


Figure 3A. SEM Photomicrograph Showing microstructure of coin (MS1) at magnifications (a) 5000X, (b) 5000X, (c) 50000X and elemental mapping in (d)

Slika 3A. SEM fotomikrografija koja prikazuje mikrostrukturu novčića (MS1) pri uvećanjima (a) 5000X, (b)5000X, (c) 50000X i mapiranje elemenata u (d)

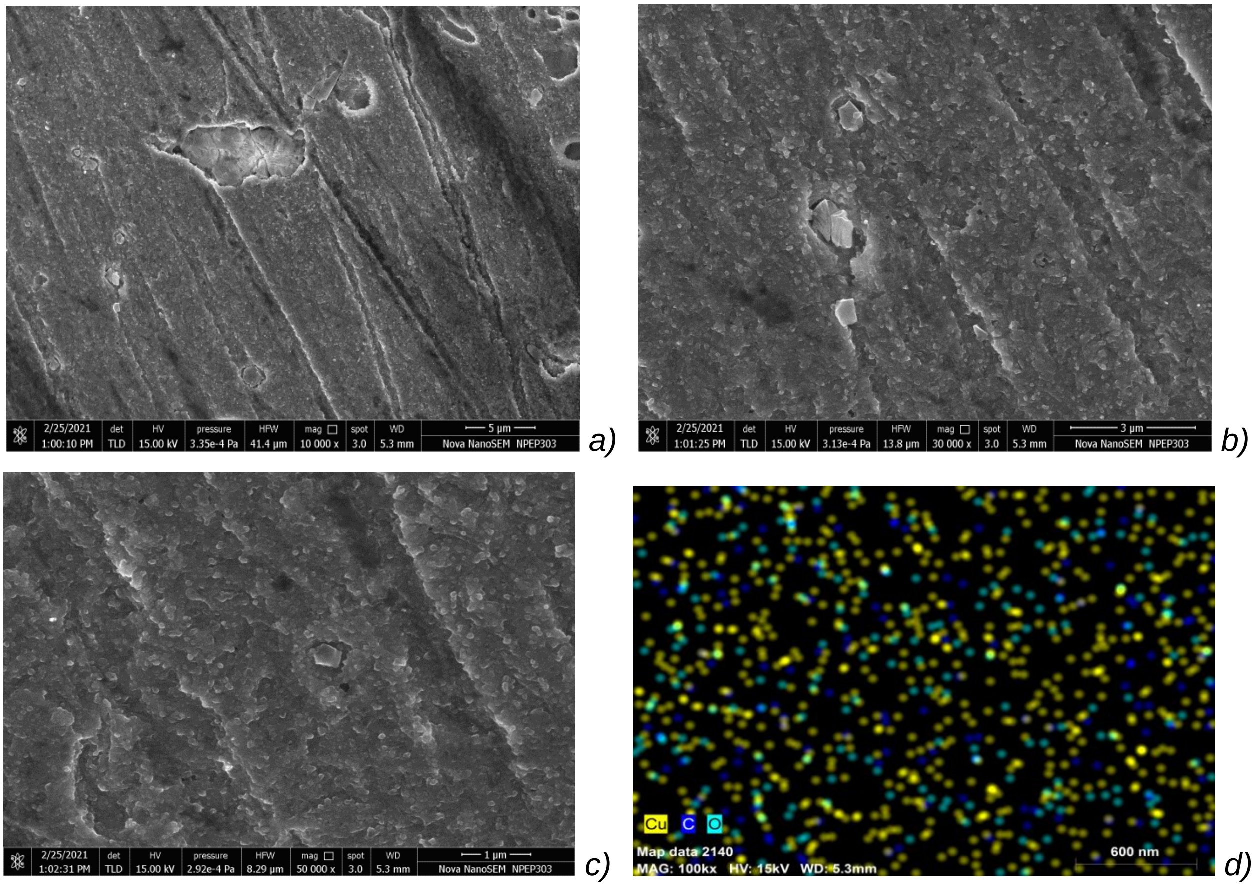


Figure 3B. SEM Photomicrograph Showing microstructure of coin (MS2) at magnifications (a) 10000X, (b) 30000X, (c) 50000X and elemental mapping in (d)

Slika 3B. SEM fotomikrografija koja prikazuje mikrostrukturu novčića (MS2) pri uvećanjima (a) 10000X, (b)30000X, (c) 50000X i elementarno mapiranje u (d)

The photomicrographs show that the coin is made up of single-phase copper, as no other metal co-exists with it. The SEM photomicrographs for both coins show many micro-cracks at lower magnification, and some corrosion pits are also visible at this magnification. The pit is a localized metal surface corrosion confined to a point or small area that takes the form of a cavity. When metal starts to dissolve locally for many reasons, the local chemical environment tends to change. An individual pit can happen due to the loss of the passive layer. This passive layer may be of copper oxide layer, metal salt layer, or a corrosion inhibitor film. At higher magnification, the SEM micrograph shows a number of pits; some are filled with particles, and some are voids. Cracks are also very clearly visible, and if not taken care of, they may convert into major cracks for the coins affecting their stability. Crystals of CuO are visible at higher magnification in the coins.

The EDX data recorded for the coins MS1 and MS2 is shown in Table-3. The major element is copper and oxygen in the coin. The carbon percentage observed is due to instrumental impurities during measurements. The other elements, like iron, Lead, phosphorus, tin, sulphur, etc., are present in traces in the coin. The EDX data reveal better workmanship in the metallurgical and fabrication techniques of Shivrai coins, as inclusions like sulphur, Lead, phosphorus, iron, etc., could be ideally removed during smelting. Polling during smelting could be performed flawlessly, resulting removal of impurities.

The elemental mapping for the coins is also shown in Figure 3 A&B. The elemental mapping clearly shows the presence of copper and oxygen, and the carbon percentage observed in the mapping is due to instrumental impurities. The SEM photomicrographs of both coins are nearly identical, with pits and cracks.

5. CONCLUSION

The multi-analytical investigations of Shivrai copper coins showed possible smelting of sulphide ore of copper, and better workmanship during smelting removed most of the impurities from the ores. Impurities like tin, Lead, sulphur, phosphorus, etc., are present in traces in the coin. No compositional gradient was observed in the coins except for slight iron enrichment on the coin surfaces. Active corrosion in the form of mineralization of copper into cuprite and tenorite was observed, and the coin interior showed the occurrence of cracks and cavities. The coin hoard needs better storage conditions and immediate conservation measures for stability.

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IZVOD

METALURŠKA ISTRAŽIVANJA BAKARNOG NOVCA IZ 17. VEKA MARATHA SHIVRAI

Tehnička i numizmatička složenost bakarnih kovanica Maratha Šivrai je dobro dokumentovana. Međutim, još uvek je potrebno više informacija o hemijskom, mineraloškom, mikrostrukturnom sastavu i metalurškom procesu koji se koristi za kovanje novčića. Pet Šivari kovanica (17-18 vek n.e.) proučavano je pod ED-KSRF, KSRD i analizom poprečnog preseka pomoću SEM-EDKS. Naučna istraživanja otkrivaju da su Maratha praktikovali odlične tehnike kupelacije kako bi uklonili većinu nečistoća rude. Podaci ED-KSRF na površini i unutrašnjosti ukazuju da novčić sadrži čisti bakar, a sadržaj bakra varira između 97,73% do 100%. Površine novčića su slobodne od rudnih nečistoća kao što su sumpor, olovo, kalaj, fosfor, itd., a uočene crne mrlje su posledica mineralizacije bakra otkrivene KSRD analizom. Postoji blagi gradijent koncentracije gvožđa na površini novčića (4,48%) u odnosu na unutrašnje jezgro (0,14%). Analiza poprečnog preseka otkrila je mnogo pukotina i šupljina u unutrašnjosti novčića zbog mineralizacije bakra koja ugrožava njegovu stabilnost ako se ne vodi računa o tome. Arheo-metalurška istraživanja unutrašnjosti novčića otkrivaju da su značajne nečistoće poput olova, sumpora, kalaja, gvožđa itd. prisutne u tragovima zbog odličnog ispitivanja, a Šivrai je jednofazni bakarni novčić.

Ključne reči: *Šivrai bakarni novčići, dinastija Maratha, kupelacija, metalografija, korozija*

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