

Effect of Current Density and ZrO₂ Concentration on the Microstructure, Mechanical and Tribological Properties of Electro- co-deposited Cu-ZrO₂ Composite Coatings

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Article

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Abstract

The structural, mechanical, tribological, adhesion, and corrosion behavior of Cu-ZrO₂ nanocomposite coatings has been comparatively explored by varying the concentration of ZrO₂ nanoparticles and the current density. Electro-co-deposition films were formed on stainless-steel substrates at the current densities of 3 and 4 A/dm² with the concentrations of ZrO₂ of 25, 50, 75 and 100 mg/L. The phase structure and crystallite characteristics, their morphology and elemental distribution were analyzed using X-ray diffraction, scanning electron microscopy and energy-dispersive spectroscopy. The performance of coatings was assessed by microhardness, adhesion strength, and wear tests. The current density and ZrO₂ content generally increased the incorporation of particles and the performance of the coatings, and increased ZrO₂ content promoted grain refinement and enhanced coating compactness. The best mechanical and tribological properties were obtained when the ZrO₂ coating was deposited with a concentration of 75 mg/L and a current density of 4 A/dm², with the adhesion strength of the coating of 26.8 MPa and minimum wear volume of 2.17 mm³. The results show that the concentration of ZrO₂ and current density both have a significant influence on the coating performance.

1. Introduction

Copper coatings are of great interest to engineers because of its desirable properties of high electrical and thermal conductivity, good surface quality and high application potential for automotive, electrical, marine and industrial components. But the mechanical wear resistance and hardness of copper are relatively low, which limits its service life in severe sliding and corrosive environment. In this context, numerous studies have been done to modify the surface of the copper by composite coating, where a hard second phase is added to the metallic matrix to get a better structure-property relationship. Electrodeposited metal-matrix composite coatings have recently been mentioned as a viable option for achieving a combination of mechanical, tribological and corrosion properties ^{1,2,3}. The ability of control the microstructure of coatings by the composition of the electrolyte and electrical parameters makes electro-co-deposition particularly attractive for the production of metal-ceramic composite coatings, which are relatively simple to produce, relatively cheap and have low requirements for skills and equipment. In the process, suspended ceramic nanoparticles can be pushed towards the cathode and be included within the growing metallic matrix. The subsequent incorporation of particles can, thus, alter the nucleation, grain growth, crystallite size, hardness, wear resistance and corrosion characteristics ^{4,5}. In recent years, it was shown that the electrodeposition parameters including the current density, particle concentration, particle dispersion, and agitation have a significant effect on the incorporation of these nanoparticles and hence on the functional properties of the composite coatings ^{6,7}. Zirconium dioxide (ZrO₂) is mainly used as a ceramic reinforcement due to its high hardness, chemical stability, thermal stability and wear resistance. ZrO₂ nanoparticles can be introduced into the electrodeposited metallic matrix to achieve dispersion strengthening and grain refining effect due to the restriction of grain growth and reduction of the ability of dislocations to move between the nanoparticles. Studies about Ni-, Co-,

and Co-W-based ZrO_2 composite coatings have shown that proper addition of ZrO_2 nanoparticles has improved the hardness, wear property, corrosion property, and compactness of the coating^{8,9,10,11,12}. For instance, ZrO_2 incorporation has been found to result in grain refinement and high electrochemical stability of Ni-Mo- ZrO_2 coatings^{8,9} and concentration-dependent variations in coating morphology and mechanical properties have been reported for Co-W- ZrO_2 and C-Mo-P- ZrO_2 systems^{8,10}. One of the critical parameters, however, is the concentration of nanoparticles in the bath. Too low a concentration can lead to poor particle incorporation, while too high a concentration can cause particle agglomeration and nongrading deposition, causing surface defects and weak regions. In several of the most recent electrodeposited wear or corrosion resistant coatings containing ZrO_2 particles, a maximum volume fraction of particles, which yielded coatings with a denser structure, smaller particle size, enhanced hardness, better wear or corrosion resistance, was found to lead to a reduction in some properties when the particle concentration was increased further¹³. Consistent results have been found for mechanical or ultrasonic agitation to enhance the dispersion of nanoparticles which is relevant for the final coating structure^{11,13}. Nanoparticle concentration along with current density are among the most critical parameters in electro-co-deposition process. Current density variations affect parameters such as cathodic overpotential, nucleation rate, mass transport, reduction of metal ions, electrophoretic movement and entrapment of suspended particles. Thus, with a varying current density, the coating morphology, crystallite size, inclusion of particles, hardness, adhesion, wear and corrosion resistance can vary¹⁴. It has been observed that the study of the electrodeposited coating composition and microstructures in the presence of ZrO_2 particles and other ceramic particles have revealed that the optimum current density has a significant effect on the coating composition and microstructure depending on the nature of the particle, particle concentration and deposition bath^{15,16}. To date, Ni- and Co-based matrices of ZrO_2 -reinforced electrodeposited coatings have been widely studied; however, few studies have systematically explored the effect of ZrO_2 concentration and current density on Cu-based nanocomposite coatings. A previous study on electrodeposited Cu- ZrO_2 coatings showed that ZrO_2 concentration, applied current and stirring conditions affect particle incorporation and coating properties; however, a comparative assessment of various functional properties is still relevant at very closely selected current densities¹⁷. The systems Cu- ZrO_2 have also been investigated with further research for multifunctional surface protection and were shown to possess additional beneficial properties for improving hardness, wear and corrosion protection more recently¹⁸.

Based on this, the present study concentrates specifically on the comparative study of the effect of ZrO_2 concentration and current density on Cu- ZrO_2 nanocomposite coatings. To fabricate the coatings, four different concentrations of ZrO_2 (25, 50, 75 and 100 mg/l) were used with a direct current density of 3 A/dm² and 4 A/dm², with the other deposition parameters held constant. The resulting coatings were systematically characterized with respect to their phase structure, their surface morphology, their incorporation of elements, their microhardness, their resistance of wear, their adhesion strength and their corrosion behavior. The main goal was to find out how the ZrO_2 concentration change affects the

current density, and to determine which would give the best combination of structural, mechanical, tribological, adhesion, and corrosion properties for the deposited material when it is used in the selected deposition condition. Such a comparison approach is aimed at gaining a better understanding of the process-structure- property relationship of Cu-ZrO₂ nanocomposite coatings.

2. Material and Method

2.1 Materials and Electrolyte Preparation

All the chemicals and reagents used in the experimental investigation were analytical grade. The main electrolyte used was copper sulphate (CuSO₄) and the reinforcing phase used were ZrO₂ nanoparticles with an average particle size of 30–50 nm, provided by Nano Research Lab. The electrolyte was prepared by dissolving 100 g of CuSO₄ in 400 mL of deionized (DI) water and then making up a final electrolyte volume of 500 ml. Throughout the deposition process, the pH of the electrolyte bath was kept at 5.0 ± 0.1 by adding sulphuric acid and the bath temperature was kept at $34 \pm 1^\circ\text{C}$. Different coatings of Cu-ZrO₂ nanocomposite layers were synthesized with different concentrations, i.e., 25, 50, 75 and 100 mg/L of ZrO₂ nanoparticles added to the electrolyte. A certain amount of ZrO₂ was poured into 500 mL of electrolyte, and a magnetic stirring was performed to disperse the ZrO₂. The suspension was then treated with ultrasound at 15 kHz and 500 W for 1 h to ensure uniform distribution and aggregation-free dispersion of nanoparticles. The magnetic stirring was performed continuously for 60 min at 300 rpm in the electrodeposition process. The two different DC current densities used were 3 and 4 A/dm² with all other deposition parameters kept constant, and the influence of the current density was investigated. The aim of the experimental study was, therefore, to investigate the characteristics and performance of the produced Cu-ZrO₂ nanocomposite coatings as a function of the concentration of ZrO₂ in the electrolyte and current density. The summary of principal electrolyte and deposition parameters are shown in Table 1.

Table 1
Electrolyte composition and electro-co-deposition parameters

Parameter	Value
Electrolyte volume	500 ml
ZrO ₂ nanoparticles	25, 50, 75, and 100 mg/L
Current density	3 A/dm ² and 4 A/dm ²
pH	5.0 ± 0.1
Bath temperature	34 ± 1 °C
Agitation speed	300 rpm
Ultrasonication	15 kHz, 500 W, 1 h
Deposition time	60 min
Cathode	Stainless steel, 25 × 25 × 2 mm
Anode	Cu rod, 5 mm diameter

2.2 Substrate Preparation and Electro-co-deposition

A stainless steel plate (25×25×2 mm) was used as a cathode while a copper rod, 5 mm diameter, was used as an anode. The stainless-steel substrates were mechanically polished prior to electro-co-deposition to acquire a clean and relatively smooth surface using sequentially 320, 800, and 2500 grit abrasive papers. The polished substrates were then degreased and cleaned with acetone and DI water to remove any surface contaminants, oxides and oils from the surface. The electro-co-deposition procedure for Cu-ZrO₂ is presented in the schematic in Fig. 1. During deposition, the electrolyte where ZrO₂ nanoparticles were prepared was continuously stirred and kept in this electrolyte. The deposition time was fixed at 60 min for both deposition currents 3 and 4 A/dm². The coated samples were subsequently removed from the electrolyte, rinsed with DI water and dried before characterisation.

2.3 The characterization of the Cu-ZrO₂ coatings

The surface morphology and elemental composition of the coatings deposited were investigated by an EVO18 Carl Zeiss scanning electron microscope (SEM) system with energy-dispersive spectroscopy (EDS). Identification of the phase and its structural characteristics were carried out by a Anton Paar XRDynamic 500 X-ray diffractometer (XRD) with a scanning rate of 0.1°/s. Microhardness was measured with Banbros HM 200 Micro Vickers hardness tester. A load of 25 g was applied and a dwell time of 14 s was used. At each coating surface multiple indentations were taken at different points and the mean value taken for analysis. The tribological performance of the coatings was tested in a Magnus pin-on-disc tribometer. The counter body was made of 5 mm diameter EN31 steel pin. Wear tests were carried

out with a normal load of 5N, at ambient temperature and without lubrication. The radius of wear was 5 mm and the speed of sliding was 300 rpm. Using a digital balance of 0.0001 g precision, the mass loss of the specimens was measured and then a wear volume/rate was calculated based on the measured mass loss and material density. The adhesion strength of the coatings was tested by a PosiTest Pull-Off Adhesion Tester. Before testing, the surfaces to be coated were polished and cleaned with acetone. The aluminium dollies (diameter 20 mm) were cemented by a high strength epoxy glue to the coating surface and cured for 24 h at room temperature. The strength of the coating pull-off was then tested with a uniaxial tension applied perpendicular to the coating surface until the failure or the pull-off of the coating. The adhesion strength was determined by dividing the maximum load of detachment by the actual area of adhesion.

3. Result and Discussion

3.1. Surface morphology and elemental analysis

3.1.1. Scanning Electron Microscopy:

The surface morphology of the composite Cu-ZrO₂ coatings obtained from the electro-co-deposition process at a current density of 3 A/dm² and 4 A/dm² at various concentrations of ZrO₂ bath (50, 75, 100 and 25 mg/L, marked as (a)-(d), respectively) is shown in Fig. 2 and Fig. 3. The coating obtained at the lowest concentration of particles (25mg/L, Fig. 2a) has a typical coarse-grained, pyramidal-like crystalline copper matrix, which shows distinct grain facets and large inter-grain boundaries. A higher ZrO₂ concentration (50 mg/L in Fig. 2b and 75 mg/L in Fig. 2c) results in a much finer grain structure and a more compact and homogeneous nodular structure. The ZrO₂ nanoparticles in the incorporated state create active heterogeneous nucleation sites in the electro crystallization and also act as grain boundary pinning, which inhibits the continuous lateral growth of Cu grains. The matrix at the concentration of 100 mg/L (Fig. 2d) has a fine texture and particulate clusters appear on the surface with a more dense distribution. Overall, the coatings have a denser and more refined grain packing at the higher current density of 4 A/dm² due to the increased cathodic over potential that lead to a more rapid nucleation rate. Fine ZrO₂ nanoparticles are easily seen and are well-distributed throughout the crystal facets and grain boundaries at 25 mg/L and 50 mg/L (Fig. 3a,b). The coating synthesized using 75 mg/L ZrO₂ (Fig. 3c) has the most compact, defect free and homogeneously refined microstructure, which indicates optimum co-deposition and particle dispersion. However, higher concentrations, up to 100 mg/L (Fig. 3d), cause the particles to agglomerate and cause some irregularities in the surface. At high current densities, electrophoretic migration and entrapment of particles is increased, which can lead to cluster formation if the bath concentration is high, and cause non-uniform matrix growth.

3.1.2. Energy-Dispersive X-Ray Spectroscopy:

The energy-dispersive X-ray spectroscopy (EDS) spectra and corresponding quantitative compositional analyses of the Cu-ZrO₂ composite coatings prepared by the three different ZrO₂ concentrations in the bath: 25, 50, 75 and 100 mg/L and prepared at 3 A/dm² and 4 A/dm² are shown in Fig. 4 and Fig. 5. The successful incorporation of ZrO₂ nanoparticles in the metallic copper matrix under all deposition conditions is evident in the appearance of characteristic peaks of Cu, Zr and O in the XRD pattern. The weight of elemental fractions of Zr and O increase monotonously with increasing ZrO₂ bath concentration for both low and high current densities, from 0.19 wt.% to 0.58 wt.% Zr at 3 A/dm², and from 0.28 wt. % to 0.78 wt. % O. Zr at 4 A/dm². At the same time, the percentage of copper matrix proportionally reduces.

Overall, the particle entrapment efficiency is greater for the composite coatings made at 4 A/dm² than those made at 3 A/dm² at the equivalent concentration. Following Guglielmi's adsorption model, the increased cathodic overpotential at 4 A/dm² increases the electrophoretic attraction and convective mass transport, thus promoting physical adsorption of the charged nanoparticles to the cathode surface before their electrochemical retention by the fast growing copper film.

3.1.3. X-Ray diffraction:

The X-ray diffraction (XRD) patterns of Cu-ZrO₂ composite coatings were synthesized from the different concentrations of ZrO₂ (25, 50, 75, and 100 mg/L) for the current densities of 3A/dm² and 4A/dm² and presented in Fig. 6(a) and Fig. 6(b), respectively. The major diffraction peaks appearing at $2\theta \sim 43.30$, 50.40 and 74.10 are directly related to the crystal planes (111), (200) and (311) of face-centered cubic (fcc) metallic copper matrix. Further, the characteristic reflection peaks observed at $2\theta \approx 30.20$, 35.20 and 60.20 are indexed to the (111), (200) and (220) lattice planes of ZrO₂, respectively, and it is revealed that the ZrO₂ nanoparticles are successfully co-deposited and physically entrapped in the growing copper matrix without any secondary intermetallic phase formation or detectable impurities. The relative peak intensities of the ZrO₂ phase clearly increase with the increase of the ZrO₂ concentration in the electrolyte from 25 mg/L to 100 mg/L, indicating an increase in the volume fraction of the embedded reinforcement, across both current densities. At the same time, the Cu diffraction peaks show measurable peak broadening (increase in the full width at half maximum, FWHM) and a slight decrease in intensity. This broadening suggested a progressive decrease in the average crystallite size and increase in micro-strain, which supported the grain refinement seen in the SEM micrographs. The coatings prepared at 4 A/dm² when compared to the 3 A/dm² for the same bath concentrations show stronger ZrO₂ peak intensity as a ratio of the copper matrix. Higher cathodic over potential will promote higher mass transport and convective-electrophoretic entrapment, leading to higher incorporation of nanoparticles. The preferred growth orientation in all conditions is still the close-packed Cu (111) plane, suggesting that the grain dimension refinement due to the inclusion of particles does not change the intrinsic preferred growth texture of Cu lattice with the face-centered cubic structure.

3.2 Hardness:

Increasing the ZrO_2 concentration, the microhardness of the nanocomposite coatings was found to rise from 78 HV at 25 mg/L up to 91, 99 and 117 HV at 50, 75 and 100 mg/L, respectively, for ZrO_2 concentration at 3 A/dm². The same upward trend was also found at 4 A/dm², where the values of 80, 94, 104 and 125 HV, respectively were obtained (Fig. 7.). The hardness increases with increasing ZrO_2 content, which can be explained by the presence of hard ceramic nanoparticles in the Cu matrix. The uniformly dispersed ZrO_2 particles are obstacles to dislocation motion and plastic deformation, which also help to grain refinement the electrodeposited Cu matrix. Moreover, an increase in the current density from 3 to 4 A/dm² led to an increase in the hardness at various ZrO_2 concentrations of about 2.6%, 3.3%, 5.1% and 6.8% for 25, 50, 75 and 100 mg/L respectively. This improvement might be correlated with higher nucleation rate and better incorporation of ZrO_2 nanoparticles during the electrodeposition process. The microhardness was the highest of 125 HV was obtained at 100 mg/L ZrO_2 and 4 A/dm², while the lowest microhardness of 78 HV was obtained at 25 mg/L and 3 A/dm². In conclusion, the findings indicate that the presence of ZrO_2 particles and the current density play significant roles in enhancing the strength of the Cu matrix.

3.3 Wear Performance:

As the concentration of ZrO_2 increased, the wear resistance of the Cu- ZrO_2 nanocomposite coating improved (Fig. 8.), with the wear loss of the coating reducing gradually as the amount of ZrO_2 increased. The wear loss was 34.4% lower at 100 mg/L compared to 25 mg/L, with a decrease in wear loss from 3.72 to 2.44 mm³ at 3 A/dm². This also occurred at 4 A/dm² where the wear loss reduced from 3.45 mm³ to 2.38 mm³ (around 31.0%). The lowest amount of wear loss was measured at ZrO_2 of 75 mg/L and 4 A/dm² (2.17 mm³) which indicated that there was the optimum particle incorporation, optimum matrix strengthening and optimum microstructural refinement. This slight wear increase at 100 mg/L and 4 A/dm² might be related to the agglomeration of nanoparticles and generation of small defects which serve as weak areas during sliding. In general, the effect of reinforcement of ZrO_2 has been seen to significantly improve the wear resistance of electrodeposited Cu coatings, of which the 75 mg/L at 4 A/dm² seems to give the best wear performance.

3.4 Adhesion Strength

Figure 9. shows the adhesion strength of the Cu- ZrO_2 nanocomposite coatings dependence with ZrO_2 concentration was clearly observed as well as dependence with the applied current density. The adhesion strength was enhanced with increasing ZrO_2 concentration in the range of 25–100 mg/L at 3 A/dm², supporting that it is the bonding between the reinforcement and the matrix that is being improved. The adhesion strength rose from 18.52 MPa at 25 mg/L to the maximum value of 26.80 MPa at 75 mg/L and then decreased slightly to 24.55 MPa at 100 mg/L. This better adhesion at moderate

ZrO₂ content can be explained by the better incorporation of particles, to increase the surface roughness, and to improve the mechanical interlocking between coating and substrate. The higher current density also favored adhesion up to 75 mg/L which may be related with the higher deposition rates and the more compact coating. The decrease of adhesion at 100 mg/L and 4 A/dm², however, may result in significant incorporation of ZrO₂ and agglomeration of the particles, creating weak adhesion points and defects. The adhesion was interestingly highest at the lowest coating, 3 a/dm², at this current density, which could allow more uniform particle distribution at the highest concentration due to lower deposition rate. Overall, the optimum adhesion strength (26.80 MPa) was obtained at 75 mg/L ZrO₂ and 4A/dm². This shows that to get a maximum adhesion strength between the coating and the substrate, it is important to balance the particle concentration and deposition current density.

4. Conclusion

The present study systematically investigated the effect of ZrO₂ nanoparticle concentration and applied current density on the microstructural, mechanical, tribological, and adhesion characteristics of electro-co-deposited Cu–ZrO₂ nanocomposite coatings. The principal conclusions are summarized as follows:

- SEM/EDS confirmed the incorporation of ZrO₂ into the Cu matrix. Increasing ZrO₂ concentration promoted grain refinement and coating densification, with 75 mg/L producing the most compact and homogeneous microstructure. Further addition to 100 mg/L caused nanoparticle agglomeration.
- XRD confirmed the presence of Cu and ZrO₂ phases without detectable secondary phases. Increasing ZrO₂ content resulted in peak broadening, indicating reduced crystallite size and increased microstrain.
- Microhardness increased with ZrO₂ concentration, reaching a maximum of 125 HV at 100 mg/L and 4 A/dm², due to nanoparticle strengthening and restricted dislocation movement.
- Wear resistance improved significantly with ZrO₂ incorporation. The lowest wear loss of 2.17 mm³ was obtained at 75 mg/L ZrO₂ and 4 A/dm².
- The maximum adhesion strength of 26.80 MPa was achieved at 75 mg/L ZrO₂ and 4 A/dm², whereas higher concentration caused a slight reduction due to particle agglomeration.
- Overall, 75 mg/L ZrO₂ at 4 A/dm² provided the best balance of microstructural refinement, wear resistance, and adhesion, demonstrating its suitability as an optimum deposition condition for Cu–ZrO₂ nanocomposite coatings.

Declarations

Data and materials availability

Data and materials that have been generated and/or analyzed during the course of the current study are available and can be requested from the corresponding author upon reasonable request.

Competing interests

The authors declare that they have no competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Author Contribution

Krishna Kumar Sharma contributed to conceptualization, data collection, data analysis, data interpretation, and manuscript preparation. Premanand Singh Chauhan and Anupma Agarwal advised the research methodology, critically reviewed the manuscript, and helped interpret and discuss results. Anshul Gangele supervised the work, gave methodological advice, made critical revisions to the manuscript, and gave the final consent to the submitted version.

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Data Availability

Data and materials that have been generated and/or analyzed during the course of the current study are available and can be requested from the corresponding author upon reasonable request.

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Figures

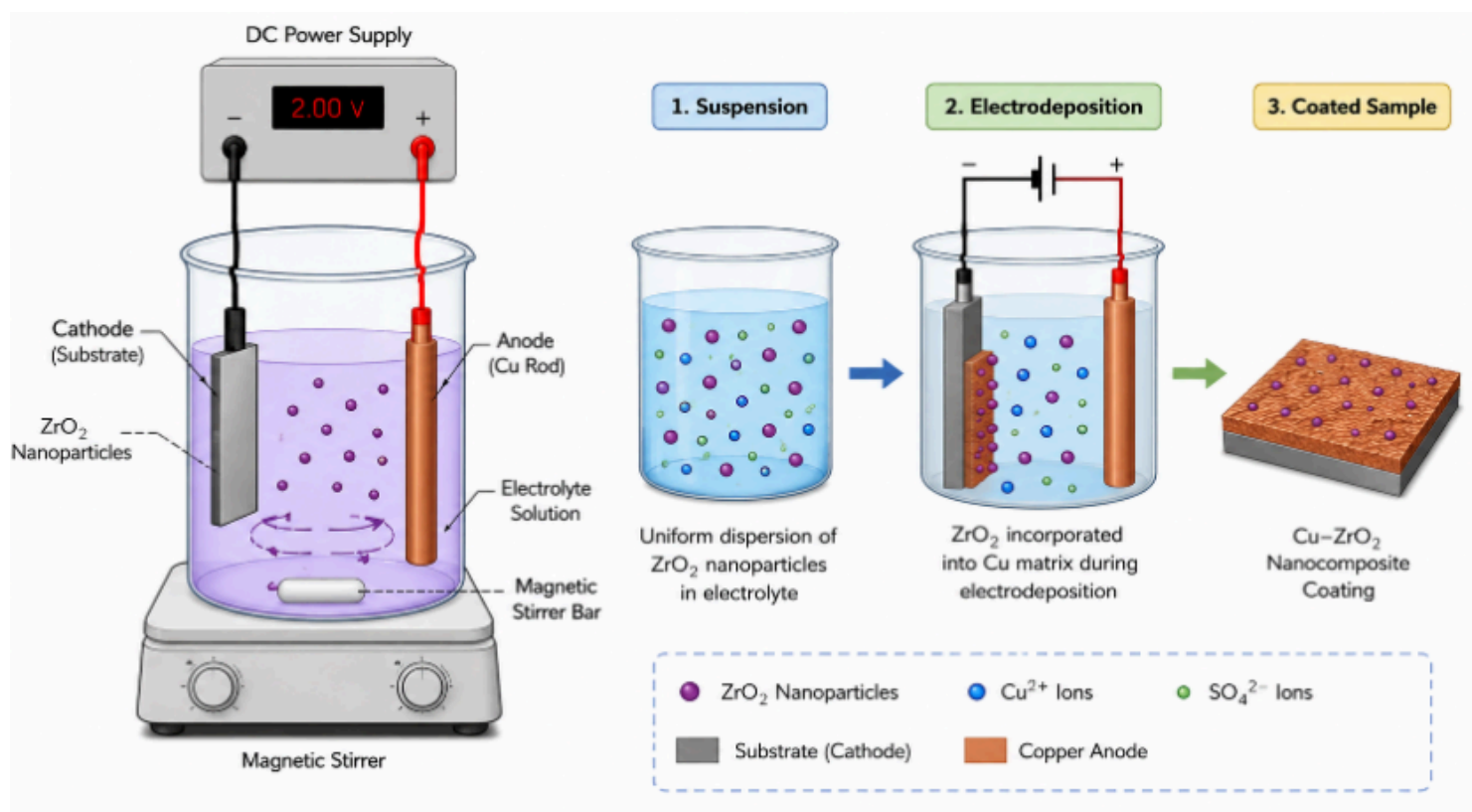


Figure 1

Experimental Setup of Cu-ZrO₂ nanocomposite coating by electro-co-deposition method.

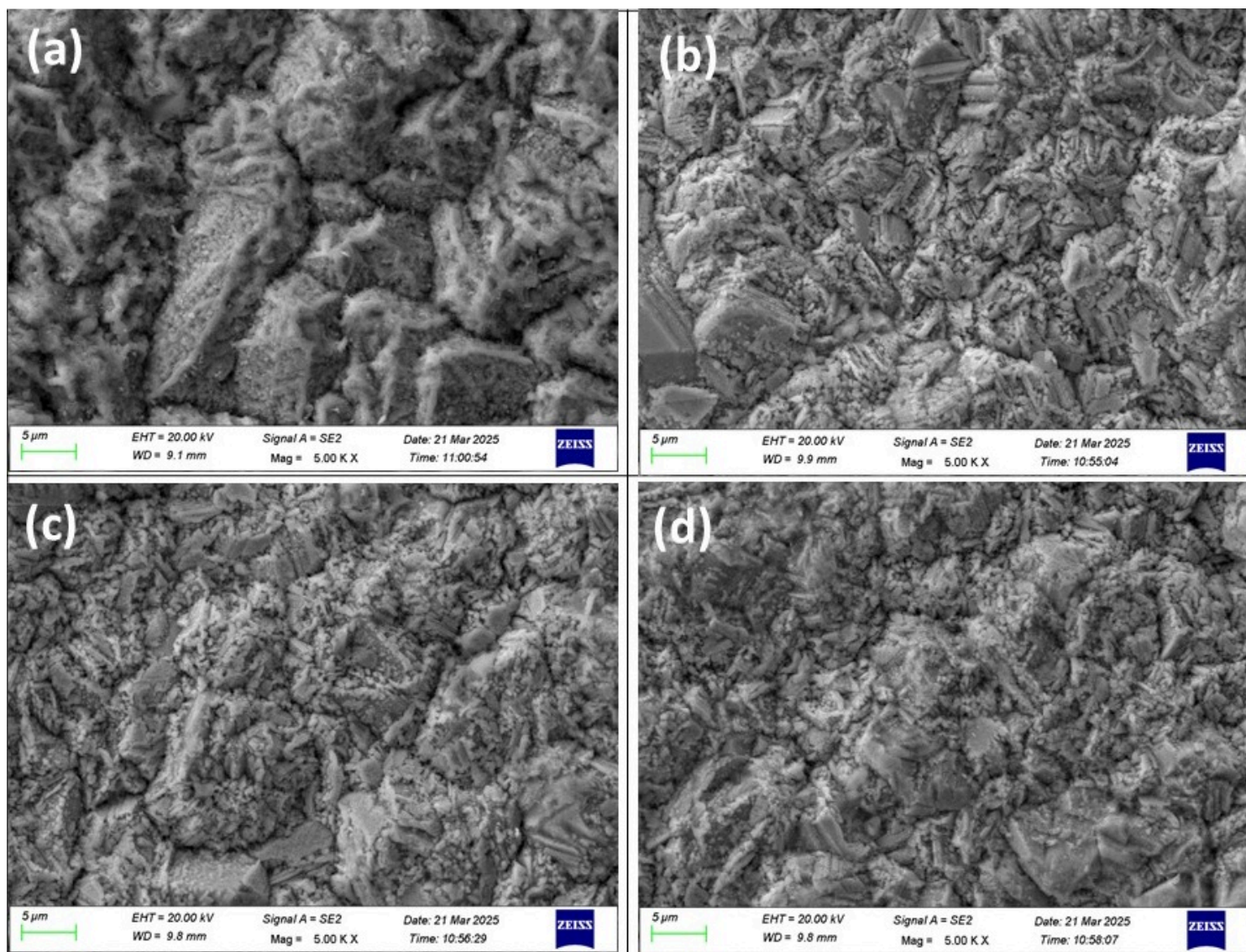


Figure 2

The SEM images(a-d) of the Cu-ZrO₂ nanocomposite coatings with a ZrO₂ concentration of 25, 50, 75, and 100 mg/L at 3 A/dm² current density.

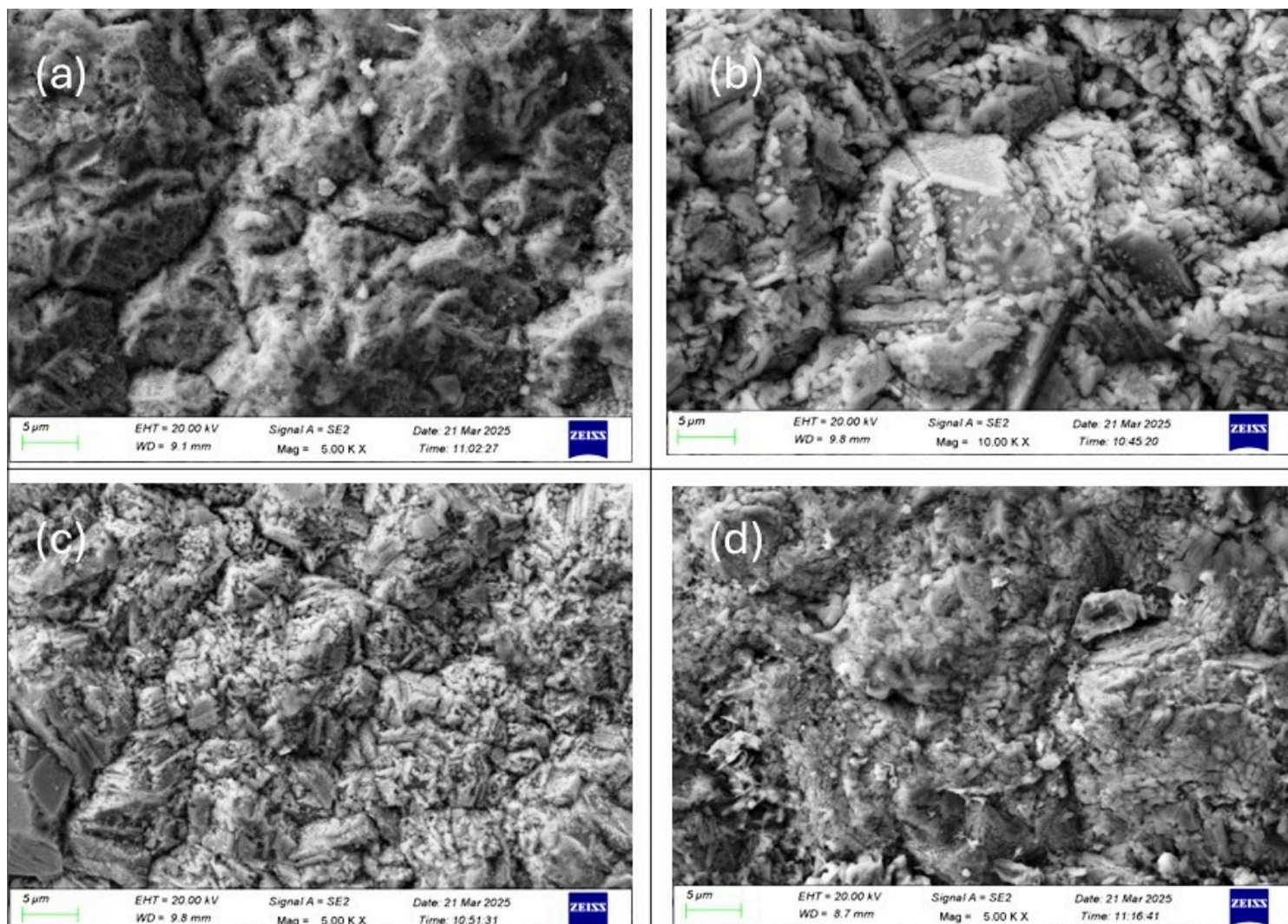


Figure 3

The SEM images (a-d) of the Cu-ZrO₂ nanocomposite coatings with a ZrO₂ concentration of 25, 50, 75, and 100 mg/L at 4 A/dm² current density.

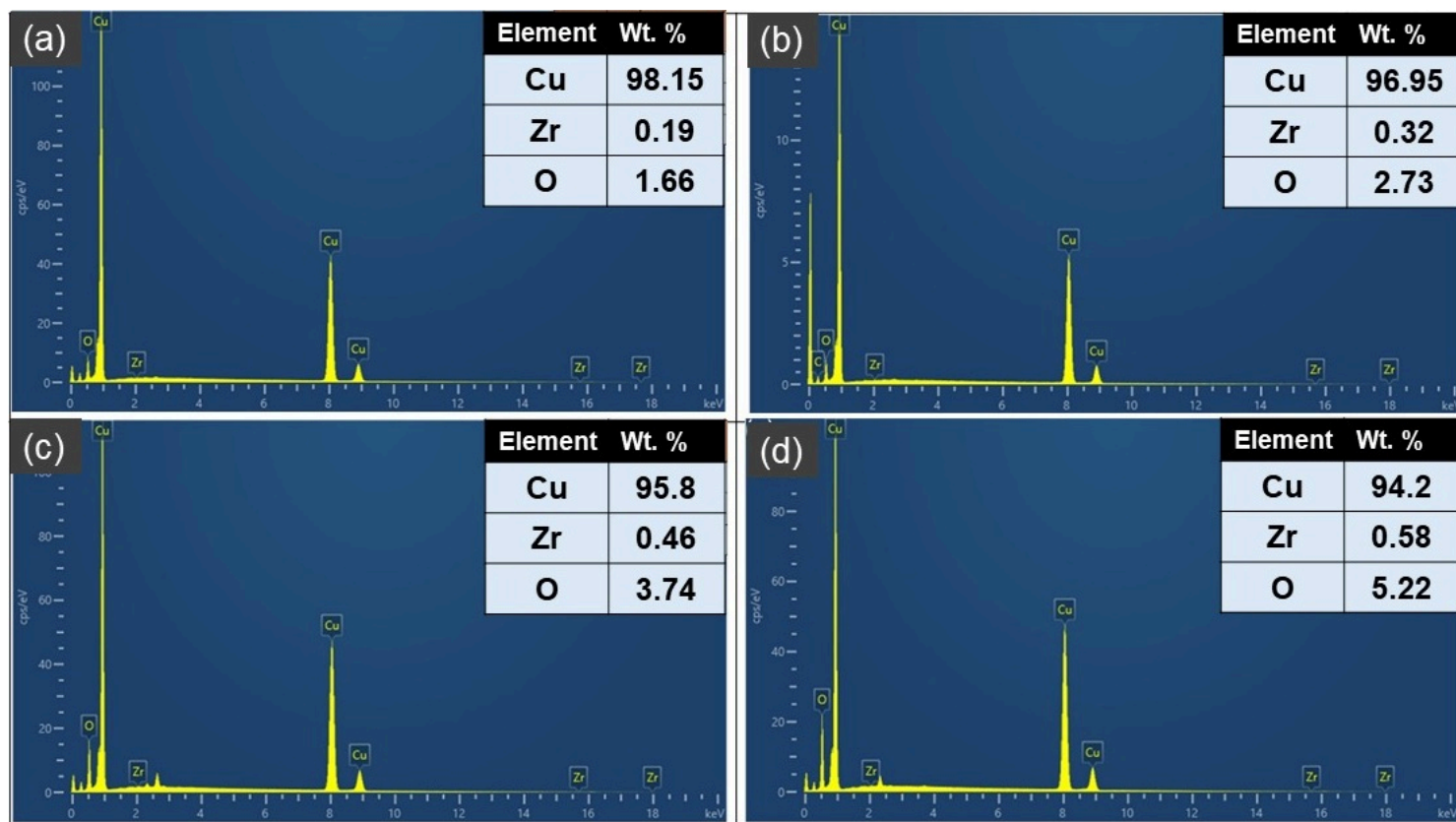


Figure 4

EDS spectra and elemental compositions of Cu-ZrO₂ composite coatings prepared at 3 A/dm² with ZrO₂ concentrations of: (a) 25 mg/L, (b) 50 mg/L, (c) 75 mg/L, and (d) 100 mg/L.

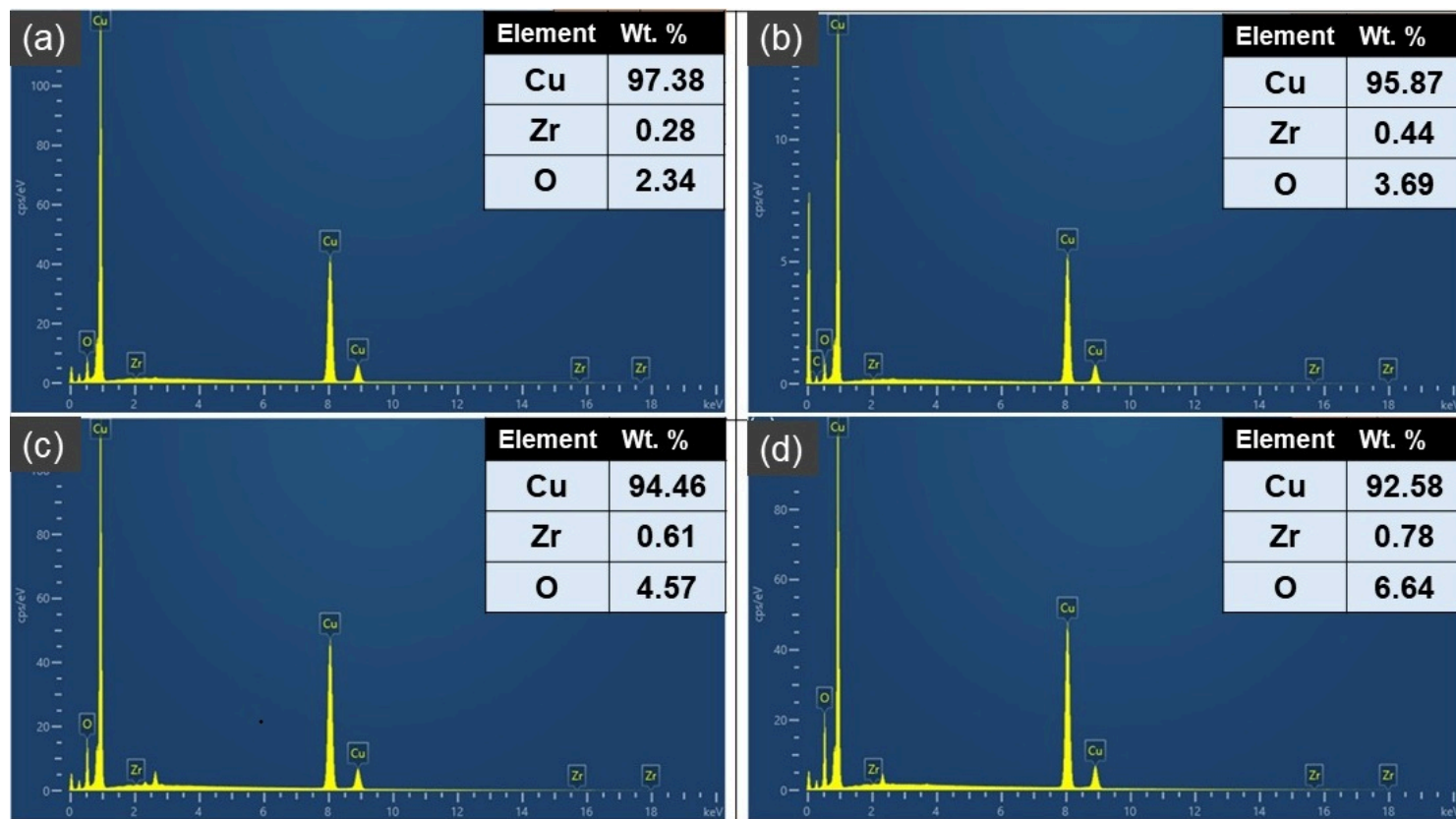


Figure 5

EDS spectra and elemental compositions of Cu-ZrO₂ composite coatings prepared at 4 A/dm² with ZrO₂ concentrations of: (a) 25 mg/L, (b) 50 g/L, (c) 75 mg/L, and (d) 100 mg/L.

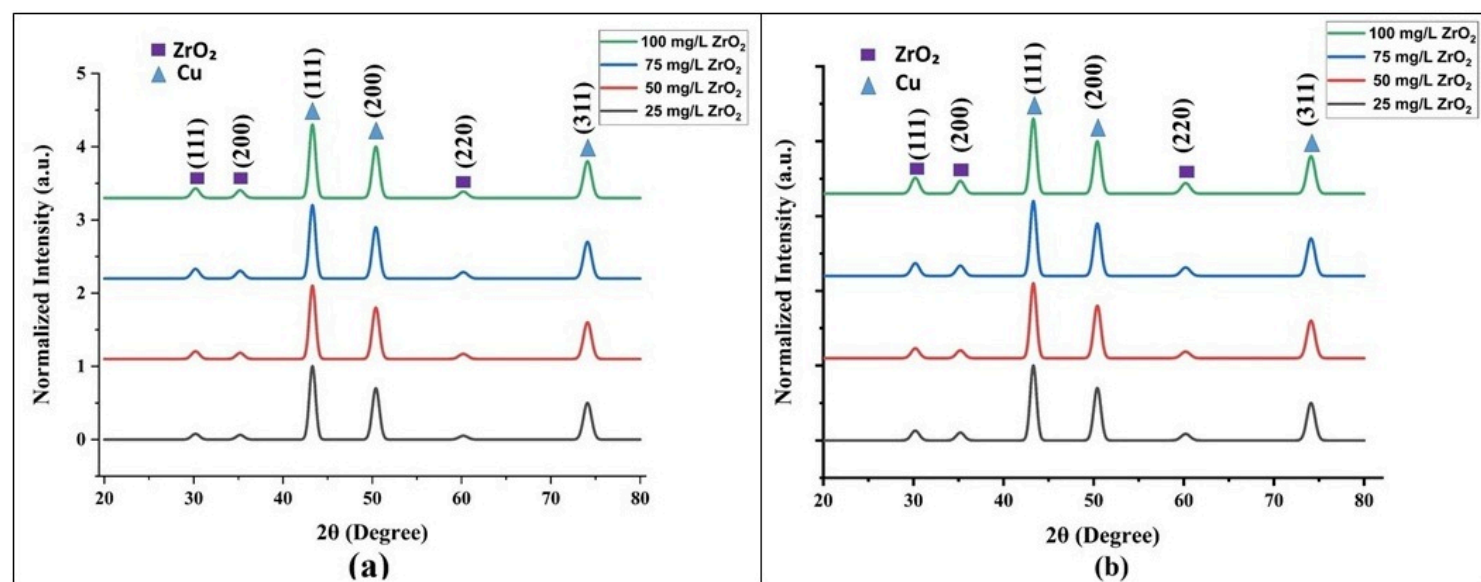


Figure 6

X-ray diffraction (XRD) patterns of electro-co-deposited Cu-ZrO₂ composite coatings synthesized at different ZrO₂ concentrations (25, 50, 75, and 100 mg/L) under current densities of: (a) 3 A/dm² and (b)

4 A/dm².

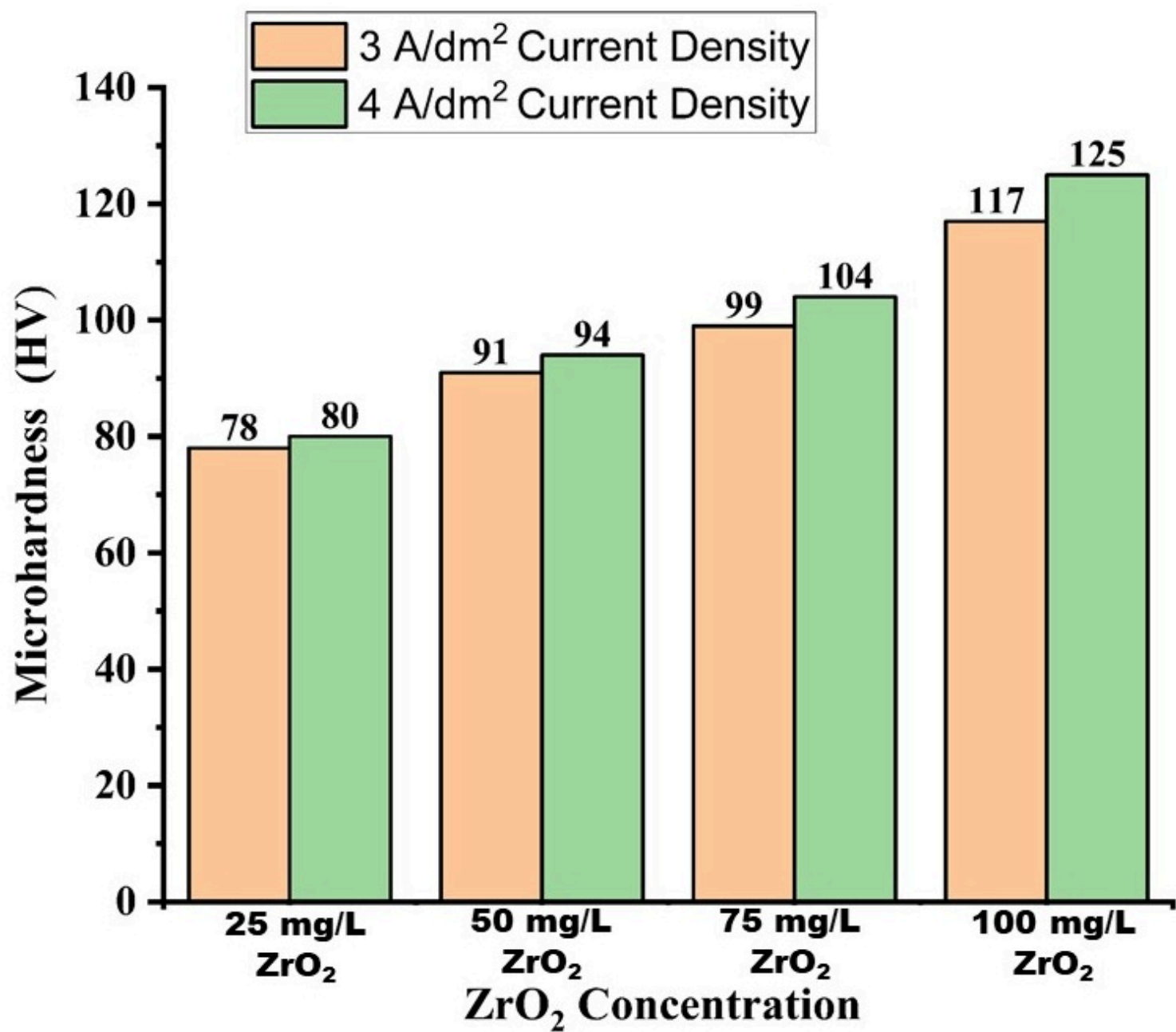


Figure 7

Microhardness of Cu-ZrO₂ composite coatings at 3 A/dm² and 4 A/dm² with varying ZrO₂ concentrations.

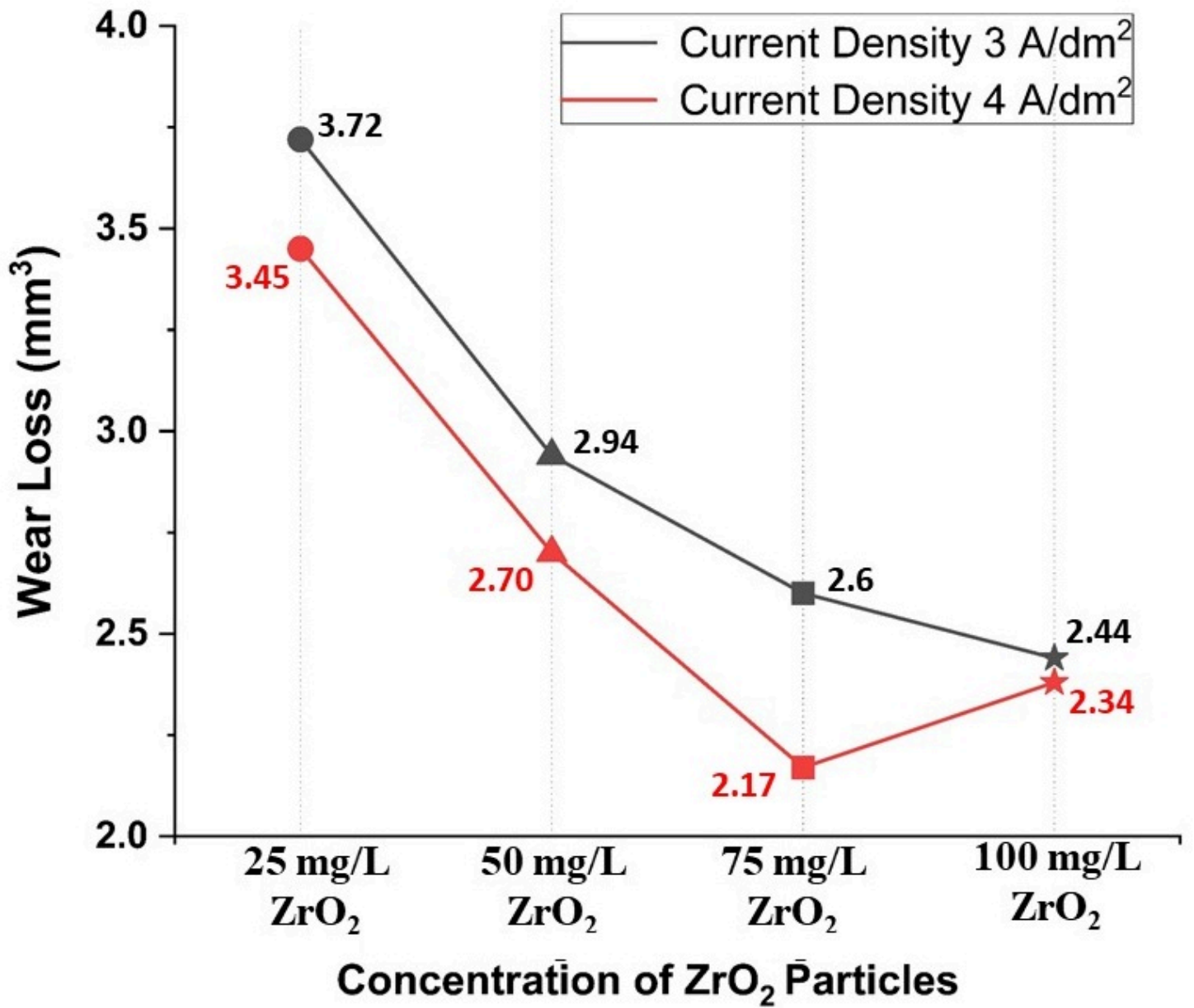


Figure 8

Wear loss of Cu-ZrO_2 composite coatings at 3 A/dm^2 and 4 A/dm^2 with varying ZrO_2 particle concentrations.

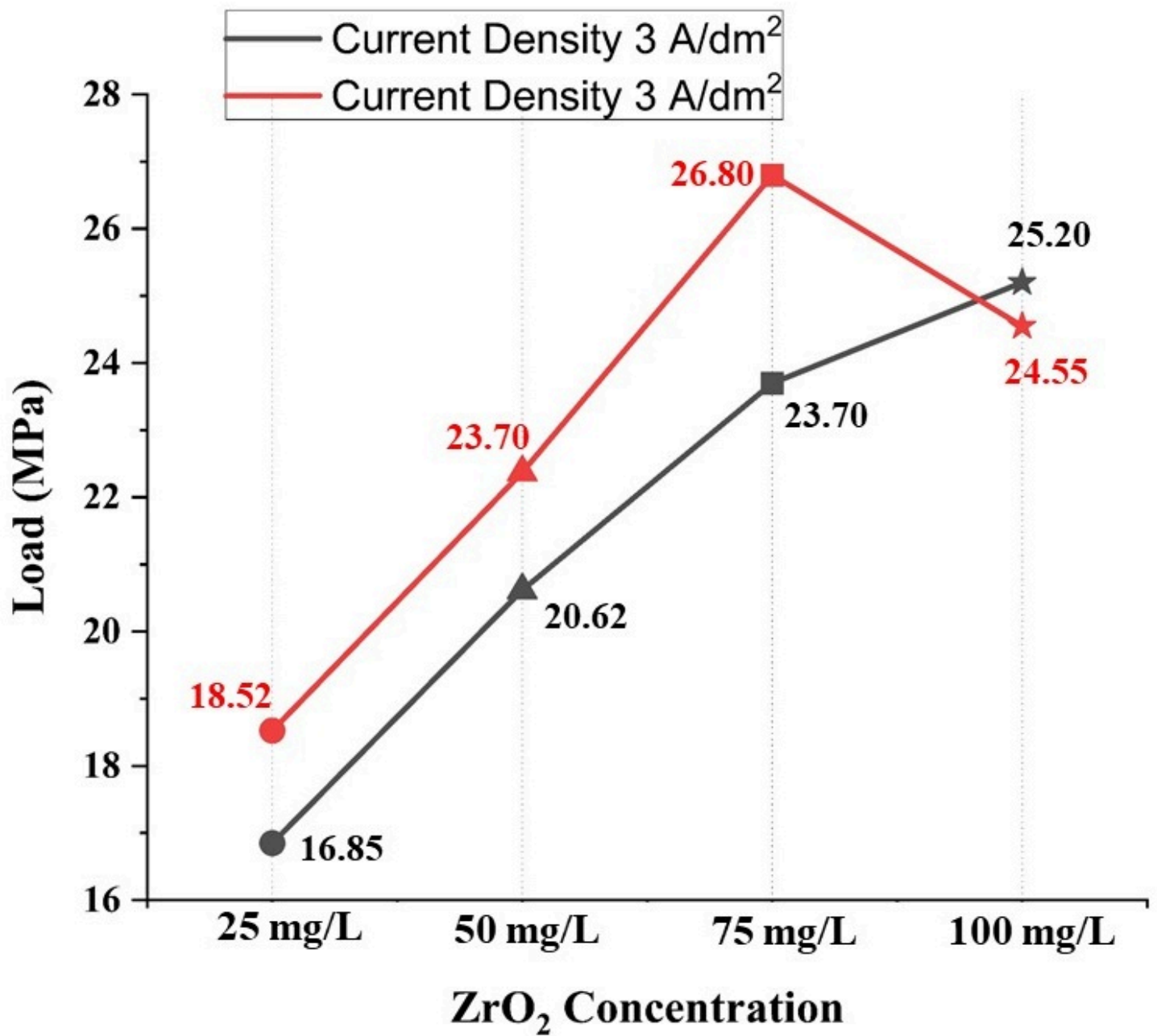


Figure 9

Adhesion strength (Critical Load) of Cu-ZrO₂ composite coatings at 3 and 4 A/dm² with varying ZrO₂ particle concentrations.