



Multifunctional nanosilver-reinforced biocomposite coatings for metallic surface protection

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Stainless steel 316L is widely used for biomedical implants because of its high mechanical strength, corrosion resistance, and durability; however, its relatively bioinert surface requires modification to improve biological interaction and osseointegration. In this study, multifunctional Ag/PMMA–NiO multilayer coatings are developed on 316L stainless steel. An electroless Ag interlayer is first deposited, followed by electrostatic spray deposition (ESD) of PMMA/NiO composite coatings containing 4, 6, and 8 wt.% NiO. The coatings are characterized by X-ray diffraction (XRD), FESEM/EDS, thickness, surface roughness, wettability, and Vickers microhardness measurements. The Ag coating exhibited an average thickness of 59.1 μm , while the multilayer thickness increased to 86.6, 88.5, and 89.6 μm for 4, 6, and 8 wt.% NiO, respectively. FESEM revealed dense, continuous, and relatively homogeneous coatings, while EDS confirmed the presence of C, O, Ag, and Ni; the Ni content increased from 3.3 to 3.5 wt.% between the 6 and 8 wt.% NiO coatings. Surface roughness decreased from $0.740 \pm 0.016 \mu\text{m}$ for bare 316L to $0.320 \pm 0.016 \mu\text{m}$ after Ag deposition and to 0.255 ± 0.008 , 0.235 ± 0.008 , and $0.245 \pm 0.008 \mu\text{m}$ for 4, 6, and 8 wt.% NiO, respectively. The contact angle decreased from 98.573° for uncoated 316L to approximately 58.948° and 60.948° for the 6 and 8

wt.% NiO coatings. Microhardness increased from 351 ± 3 HV to a maximum of 506 ± 4 HV for the 6 wt.% NiO coating. Overall, the 6 wt.% NiO/94 wt.% PMMA composition provided the most favorable combination of surface smoothness, wettability, and mechanical performance.

The average thickness of the nanosilver coating is approximately 59.1 μm , whereas the average thicknesses of the multilayer composite coatings are 86.6 μm for the 4% NiO-containing coating, 88.5 μm for the 8% NiO-containing coating, and 89.6 μm for the other reported NiO composition. Morphological examination further demonstrated that the deposited layers are uniform, homogeneous, continuous, and essentially free of cracks. Moreover, the coated surfaces exhibited high wettability, suggesting improved surface characteristics that may promote favorable interactions between the implant surface and the surrounding biological environment.

Keywords: 316L stainless steel; Silver nanoparticles; Electroless deposition; Electrostatic spray deposition; Biocomposite coating.

1. INTRODUCTION

The development of advanced orthopedic implants has become increasingly important in biomedical engineering because of the growing demand for materials capable of restoring or replacing damaged bone tissue and improving patients' quality of life. Aging, trauma, and disease may progressively reduce the structural integrity and mechanical performance of natural bone, thereby increasing the need for surgical intervention and implant replacement [1]. Consequently, biomaterials used for orthopedic applications must satisfy both biomechanical and biological requirements. From a mechanical perspective, an implant should possess adequate strength, stiffness, wear resistance, fracture toughness, fatigue resistance, and corrosion resistance. At the same time, it should exhibit favorable biological characteristics, including low cytotoxicity, good biocompatibility, suitable surface properties, and the ability to promote osseointegration. Ideally, the biomechanical behavior of an implant should be compatible with that of the surrounding bone tissue to minimize stress concentration and avoid damage to adjacent tissues [2].

Osseointegration, which refers to the direct structural and functional connection between living bone and the surface of an implant, is a key factor governing the long-term stability and clinical success of orthopedic devices. Effective osseointegration improves implant fixation, decreases the risk of loosening, and may reduce the likelihood of revision surgery. Metallic biomaterials are widely used in orthopedic and dental applications because of their favorable strength, toughness, manufacturability, and corrosion resistance. Among these materials, 316L stainless steel has received considerable attention for both implantable and non-implantable medical devices because of its good mechanical properties, corrosion resistance, availability, and acceptable biocompatibility [3].

Despite these advantages, bare 316L stainless steel still exhibits several limitations when exposed to physiological environments. Prolonged contact with body fluids may promote localized corrosion and the release of metallic ions, while its relatively bioinert surface may provide insufficient interaction with surrounding bone tissue. In addition, bacterial adhesion and subsequent biofilm formation can increase the risk of implant-associated infection. Therefore, surface modification of 316L stainless steel is often required to improve its corrosion resistance, antibacterial behavior, surface bioactivity, and ability to support tissue integration. One promising strategy is the application of protective and functional coatings containing metallic nanoparticles, ceramic phases, polymers, or hybrid biocomposite constituents [4].

Corrosion is one of the principal factors affecting the long-term performance of metallic implants. Body fluids contain water, dissolved oxygen, chloride ions, proteins, and other ionic species that create an aggressive electrochemical environment. Under these conditions, deterioration of the protective passive film on the implant surface may result in localized corrosion and the release of metallic ions into the surrounding tissues. Such degradation may also reduce fatigue resistance and compromise the structural integrity of the implant. Excessive release of metallic species, particularly nickel- and chromium-containing ions, may produce undesirable biological effects. Therefore, improving the barrier characteristics of the implant surface is essential for enhancing both corrosion resistance and biological safety [5].

Austenitic stainless steels represent one of the most widely used categories of stainless steel because of their favorable mechanical properties, corrosion resistance, and broad engineering applicability. In biomedical applications, 316L stainless steel provides sufficient mechanical strength to withstand substantial physiological loads. However, its relatively limited intrinsic bioactivity restricts direct interaction with bone tissue. Furthermore, prolonged exposure to physiological fluids may lead to corrosion-related degradation and ion release. Surface engineering is therefore an effective approach for improving the interfacial behavior of 316L stainless steel without compromising its desirable bulk mechanical properties [6].

Silver nanoparticles have attracted considerable interest as functional coating materials because of their strong antimicrobial activity and useful physicochemical properties. Nanosilver can inhibit a broad spectrum of microorganisms, making it particularly attractive for reducing the risk of implant-associated infections. In addition, silver-containing coatings can provide an interfacial barrier between the metallic substrate and the surrounding physiological environment. Depending on coating composition, morphology, and microstructure, silver-based layers may also contribute to improved corrosion behavior, surface stability, and overall biological functionality. Therefore, the incorporation of nanosilver into biomedical coatings represents a promising strategy for producing multifunctional implant surfaces [7, 8].

Among the available surface modification techniques, electroless deposition is particularly attractive because it can produce relatively uniform coatings on substrates with complex geometries without requiring an external electrical power source. Electroless nanosilver coatings can provide continuous and adherent surface layers with improved protective and functional characteristics. In parallel, electrostatic spray deposition has emerged as a versatile method for applying polymeric and composite coatings. This technique offers several advantages, including relatively rapid deposition, efficient use of coating material, reduced processing requirements, and the possibility of producing uniform composite layers [9, 10].

Poly(methyl methacrylate) (PMMA) is a promising polymeric matrix for biomedical coating applications because of its chemical stability, processability, relatively low density, and established use in medical materials. However, the performance of pure PMMA can be further improved through the incorporation of functional ceramic nanoparticles. Nickel oxide (NiO) is of interest because its incorporation into a polymeric matrix may modify the coating microstructure, hardness, adhesion, wettability, and surface functionality. Accordingly, the combination of PMMA with NiO nanoparticles may provide a composite coating capable of enhancing both the mechanical and surface-related biological properties of 316L stainless steel [11,12].

The performance of biomedical coatings is strongly influenced by their structural uniformity, phase composition, surface morphology, elemental distribution, thickness, adhesion strength, hardness, roughness, and wettability. Dense and crack-free coatings with homogeneous particle distribution are generally desirable because structural defects, pores, and discontinuities can facilitate penetration of physiological fluids toward the metallic substrate and accelerate localized corrosion. Surface roughness and wettability are also particularly important because they influence protein adsorption, cell attachment, spreading, and subsequent bone–implant interactions [13,14].

Despite the advantages of nanosilver and polymer–ceramic coatings, comparative investigations of these two coating strategies on 316L stainless steel remain limited, particularly with respect to their combined structural, mechanical, and surface characteristics. A systematic comparison can therefore provide a better understanding of how coating composition and deposition technique influence the functional performance of the metallic substrate [15, 16].

Accordingly, the present study investigates and compares nanosilver and PMMA/NiO composite coatings deposited on 316L stainless steel using electroless deposition and electrostatic spray techniques. The work focuses on evaluating the crystalline structure, surface morphology, elemental composition, coating thickness, microhardness, adhesion strength, surface roughness, and wettability of the coated samples. The overall objective is to improve the surface performance and osseointegration-related characteristics of 316L stainless steel while retaining the favorable mechanical properties of the underlying metallic substrate.

2. EXPERIMENTAL PART

2.1. *Electroless deposition of nanosilver on 316L stainless steel*

An experimental setup consisting of a magnetic stirrer equipped with a heating system is employed for nanosilver deposition. The system enabled controlled heating of the electroless bath over the range of 0–100 °C. A glass beaker is positioned on the heating plate, while a laboratory stand is used to support the 316L stainless steel specimen and thermometer during deposition. Continuous magnetic stirring is applied to maintain bath homogeneity, improve mass transfer, and minimize particle agglomeration or sedimentation. The stirring rate is selected experimentally to provide sufficient agitation without destabilizing the electroless bath.

Rectangular 316L stainless steel specimens with dimensions of $20 \times 20 \times 2 \text{ mm}^3$ are used as substrates. Prior to coating, the specimens are mechanically ground using 400-, 500-, and 600-grit emery papers to remove surface oxides, corrosion products, contaminants, and irregularities. The specimens are then thoroughly rinsed with distilled water.

Surface degreasing is carried out by immersing the specimens in an alkaline cleaning solution maintained at 60–80 °C for 10–20 min. The cleaning solution contained $30 \text{ g.L}^{-1} \text{ Na}_3\text{PO}_4$, $8 \text{ g.L}^{-1} \text{ Na}_2\text{SiO}_3$, $25 \text{ g.L}^{-1} \text{ Na}_2\text{CO}_3$, and $25 \text{ g.L}^{-1} \text{ NaOH}$. After alkaline cleaning, the specimens are rinsed thoroughly with water. Surface activation is subsequently performed by immersion in a dilute HCl solution for 30 s, followed by sequential rinsing with cold water and deionized water.

The electroless silver bath is prepared using two separately prepared solutions that are mixed immediately before deposition. The silver-containing solution consisted of $3 \text{ g.L}^{-1} \text{ AgNO}_3$, 60 mL

NH₄OH, and 2.1 g.L⁻¹ Na₂S₂O₃ dissolved in approximately 450 mL of distilled water. The reducing solution is prepared using 0.2 g.L⁻¹ C₄H₆O₆, 2.25 g.L⁻¹ C₆H₁₂O₆, and 5 mL C₂H₅OH in approximately 450 mL of distilled water. After preparation, the two solutions are combined to form the final electroless deposition bath.

The pH of the bath is adjusted to approximately 12, and the deposition temperature is maintained within the range of 45–50 °C. The activated 316L stainless steel specimens are immersed in the freshly prepared bath for 35–45 min under continuous magnetic stirring. These conditions are selected to promote uniform silver deposition over the exposed substrate surface.

Following deposition, the coated specimens are removed from the electroless bath and thoroughly rinsed with distilled water to eliminate residual chemical species. The samples are subsequently dried using an air dryer before further characterization.

The deposited nanosilver coatings are evaluated using X-ray diffraction (XRD) to identify their crystalline phases and structural characteristics. Vickers microhardness testing is used to assess the mechanical behavior of the coatings, while contact-angle measurements are performed to examine surface wettability and related surface characteristics.

2.2. Preparation of PMMA/NiO composite coatings by electrostatic spray deposition

PMMA/NiO composite coatings are prepared using polymethyl methacrylate (PMMA) as the polymeric matrix and nickel oxide (NiO) nanoparticles as the reinforcing phase. Three different PMMA compositions, namely 96:4, 94:6, and 92:8 wt.%, are selected to investigate the effect of NiO content on the structural, mechanical, and surface properties of the resulting coatings.

The required amounts of PMMA and NiO powders are weighed according to the selected compositions and mechanically blended for approximately 30–40 min using a powder-mixing system. This mixing step is performed to promote uniform dispersion of the NiO nanoparticles throughout the PMMA matrix and to minimize particle agglomeration. The resulting homogeneous powder mixtures are subsequently used as feedstock for the coating process.

The PMMA/NiO composite powders are deposited onto the prepared 316L stainless steel substrates using an electrostatic spray deposition (ESD) system. The deposition apparatus consisted of a metallic collector used to support the substrate and a stainless-steel spray nozzle connected to a positive-polarity high-voltage power supply (COLO CL 668W/30 kV/PN). During deposition, the applied voltage is maintained at 25 kV, while the nozzle diameter is fixed at 0.8 mm. The distance between the spray nozzle and the substrate surface is maintained at 20 cm. The spraying time is fixed at 35 s, and the nozzle is oriented at an angle of approximately 45° with respect to the substrate surface. These operating parameters are maintained constant for all specimens to obtain reproducible coating conditions and to control the thickness and uniformity of the deposited composite layers.

Following electrostatic deposition, the coated specimens are thermally treated in an electric furnace at 160 °C for 1 h. This treatment promoted consolidation of the PMMA matrix, improved cohesion between the deposited particles, and facilitated the formation of a continuous composite coating on the

316L stainless steel surface. After thermal treatment, the specimens are allowed to cool naturally to room temperature prior to subsequent structural, morphological, mechanical, and surface characterization. The influence of NiO concentration on the characteristics of the PMMA/NiO coatings is subsequently evaluated by comparing the 4, 6, and 8 wt.% NiO-containing specimens.

2.3. Characterization of the coated specimens

The structural, morphological, compositional, surface, mechanical, and tribological characteristics of the uncoated and coated 316L stainless-steel specimens are systematically investigated using complementary characterization techniques.

The crystalline structure and phase composition of the deposited coatings are investigated using an X-ray diffractometer (D8 ADVANCE, Bruker AXS, Germany) equipped with Cu K α radiation ($\lambda=1.5406$ Å). The diffraction patterns are recorded over an appropriate 2θ range to identify the crystalline phases associated with the electroless Ag interlayer and NiO reinforcement and to examine changes in crystallinity after formation of the PMMA/NiO composite coatings.

Surface morphology, particle distribution, coating continuity, and microstructural characteristics are examined using field-emission scanning electron microscopy. A Nova NanoSEM 450 FESEM (FEI/Thermo Fisher Scientific, USA) and a MIRA3 FESEM (TESCAN, Czech Republic) are employed for high-resolution surface imaging. The instruments are capable of operation at accelerating voltages up to approximately 30 kV and provide high-magnification imaging suitable for evaluating particle morphology, agglomeration, pores, surface defects, and coating homogeneity. Cross-sectional FESEM observations are also used to examine the multilayer structure.

Elemental composition is determined using an X-MaxN energy-dispersive X-ray spectroscopy detector (Oxford Instruments, UK) coupled to the FESEM system. EDS analysis is used to identify and semi-quantitatively evaluate the major elements present in the coating system, particularly C, O, Ni, and Ag, as well as possible contributions from the underlying 316L stainless-steel substrate. The EDS results are used together with FESEM observations to assess the incorporation and distribution of NiO within the PMMA matrix and the presence of the electroless Ag interlayer.

The thickness of the deposited layers is additionally evaluated using a Dektak XT stylus profilometer (Bruker, USA). The instrument provides nanometre-to-micrometre vertical measurement capability and is used to quantify the thickness of the Ag layer and the total PMMA/NiO multilayer coating. Cross-sectional microscopic measurements are used as complementary observations where appropriate.

Surface roughness is measured using a TR200 portable surface roughness tester (TIME Group Inc., China). The instrument provides an R_a measurement range of approximately 0.005–16 μm and is employed to evaluate changes in surface topography resulting from Ag and PMMA/NiO deposition. Five measurements are collected at different positions on each specimen, and the results are expressed as the mean $\pm$ standard deviation (SD).

Surface wettability is evaluated using a DSA25 Drop Shape Analyzer (KRÜSS GmbH, Germany). Static contact-angle measurements are performed using deionized water by the sessile-drop technique over an angular measurement range of approximately 0–180°. The water droplet is allowed to remain on the specimen surface for 60 s before the contact angle is recorded. These measurements are used to

compare the hydrophilic behavior of bare 316L stainless steel, the Ag-coated surface, and the different PMMA/NiO composite coatings.

The mechanical response of the coating systems is assessed using an HMV-G Micro Vickers Hardness Tester (Shimadzu Corporation, Japan). Vickers microhardness measurements are performed under an applied load of 9.8 N (1 kgf) with a dwell time of 30 s. Three indentations are made at different positions on each specimen, and the microhardness values are reported as the mean $\pm$ SD to evaluate the influence of Ag deposition and NiO reinforcement on resistance to localized plastic deformation.

Tribological performance is evaluated using a TRB³ pin-on-disc tribometer (Anton Paar, Switzerland). Wear testing is conducted under an applied normal load of 10 N and a total sliding distance of 500 m. The wear-volume loss, specific wear rate, and coefficient of friction (COF) are determined for uncoated 316L stainless steel, electroless Ag-coated 316L, and the PMMA/NiO composite coatings containing 4, 6, and 8 wt.% NiO. These measurements are used to determine the influence of the NiO concentration on the frictional and wear resistance of the multilayer coating system.

Together, these characterization techniques enabled comprehensive evaluation of the relationships among phase composition, morphology, elemental distribution, coating thickness, surface roughness, wettability, microhardness, and tribological performance of the Ag/PMMA–NiO multilayer coatings deposited on 316L stainless steel.

3. RESULTS AND DISCUSSION

3.1. Surface morphology and elemental analysis by FESEM/EDS

Field-emission scanning electron microscopy (FESEM) is employed to investigate the surface morphology, coating continuity, particle distribution, and microstructural characteristics of the deposited layers. Energy-dispersive X-ray spectroscopy (EDS) is additionally used to identify the elemental constituents of the coatings and assess the incorporation of the different coating components. Figure 1 presents the FESEM micrographs and corresponding particle-size distributions of the investigated coatings. Figure 1(a) shows the morphology of the electrolessly deposited Ag coating on the 316L stainless steel substrate, whereas Figure 1(c) and Figure 1(e) show the PMMA/NiO composite coatings containing 4 and 6 wt.% NiO, respectively, deposited over the Ag interlayer. The corresponding particle-size distributions are presented in Figure 1(b), Figure 1(d), and Figure 1(f), respectively. The Ag layer therefore acts as an intermediate functional layer between the 316L stainless steel substrate and the outer PMMA/NiO composite coating [17-20].

The FESEM image of the electroless Ag coating, Figure 1(a), reveals extensive coverage of the stainless-steel substrate by a relatively dense and continuous layer. The surface consists of closely distributed nanoscale features with a heterogeneous but compact morphology. No extensive cracking or large uncovered regions are observed, indicating effective deposition of Ag over the substrate. Image-based particle-size analysis, Figure 1(b), indicates a relatively broad nanoscale distribution, reflecting the coexistence of fine particles and locally agglomerated surface features. The estimated mean equivalent feature size is approximately 151 nm, although this value should be considered an image-analysis estimate because of the limited separation between individual nanoscale features [21].

Following deposition of the PMMA/NiO composite layer, a pronounced change in surface morphology is observed. The 4 wt.% NiO coating, Figure 1(c), is characterized by predominantly rounded and nearly spherical polymer-rich particles distributed over the underlying Ag layer. The particles exhibit

different sizes and are closely packed, producing a continuous composite layer with limited interparticle voids. NiO-containing regions appear to be embedded within or distributed between the PMMA-rich particles, indicating that the ceramic reinforcement is successfully incorporated into the polymer matrix. The corresponding particle-size distribution, Figure 1(d), shows a relatively broad distribution with an estimated mean particle diameter of approximately 37.3 μm . The broad distribution is attributed to the coexistence of individual particles and partially coalesced or aggregated features produced during electrostatic spray deposition and subsequent thermal treatment [22].

For the coating containing 6 wt.% NiO, Figure 1(e), the surface remains largely covered by rounded and interconnected particles; however, the morphology becomes comparatively more heterogeneous. A greater number of smaller particles and locally fused particle clusters can be observed. The increased NiO content may alter powder charging, particle transport, packing behavior, and polymer flow during thermal consolidation, thereby affecting the final surface morphology. The particle-size distribution shown in Figure 1(f) exhibits an estimated mean diameter of approximately 32.4 μm , which is slightly lower than that obtained for the 4 wt.% NiO coating. This change suggests that increasing the NiO fraction may promote the formation or retention of finer surface features while simultaneously increasing the number of particle-particle contact regions [23].

Although localized clustering is observed, particularly in the NiO-containing coatings, no severe large-scale agglomeration or extensive coating delamination is evident. The overall morphology indicates satisfactory consolidation and adhesion of the composite coatings. The thermal treatment at 160 °C appears to promote softening and consolidation of the PMMA matrix without completely eliminating the original particulate morphology. Consequently, the coatings retain a textured and relatively rough surface. Such surface characteristics are important for biomedical coatings because surface topography and roughness can affect wettability, protein adsorption, cell attachment, and subsequent bone–implant interactions [24].

Cross-sectional measurements further demonstrated the successful buildup of the multilayer coating system. The average thickness of the electroless Ag layer is approximately 59.1 μm . For the complete Ag/PMMA–NiO multilayer coatings, the average thickness increased to approximately 86.6 μm for 4 wt.% NiO, 88.5 μm for 6 wt.% NiO, and 89.6 μm for 8 wt.% NiO. Thus, a progressive increase in coating thickness is observed with increasing NiO content. This behavior may be associated with increased solid loading, enhanced powder accumulation during electrostatic spraying, and changes in the packing characteristics of the PMMA/NiO particles.

EDS analysis provided additional evidence for the incorporation of the constituent elements within the deposited coatings. The detection of Ag originates from the electroless silver interlayer, whereas the simultaneous presence of Ni and O is consistent with the incorporation of NiO into the PMMA-based composite coating. The carbon signal is mainly associated with the PMMA polymeric matrix, while oxygen may originate from both the polymer and the NiO reinforcement. Because EDS provides elemental rather than phase identification, the presence of NiO should be interpreted in conjunction with the corresponding XRD results [25].

Overall, the combined FESEM, particle-size distribution, thickness, and EDS analyses demonstrate the successful fabrication of multilayer Ag/PMMA–NiO coatings on 316L stainless steel. The coatings exhibit extensive surface coverage, relatively uniform particle distribution, good continuity, and effective incorporation of the ceramic reinforcement. Increasing the NiO content modifies the particle-

size distribution and surface morphology while producing a gradual increase in the overall coating thickness. These microstructural changes are expected to influence the mechanical behavior, wettability, adhesion, and biological response of the coated 316L stainless steel substrate.

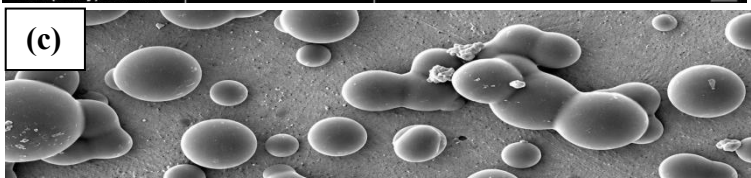
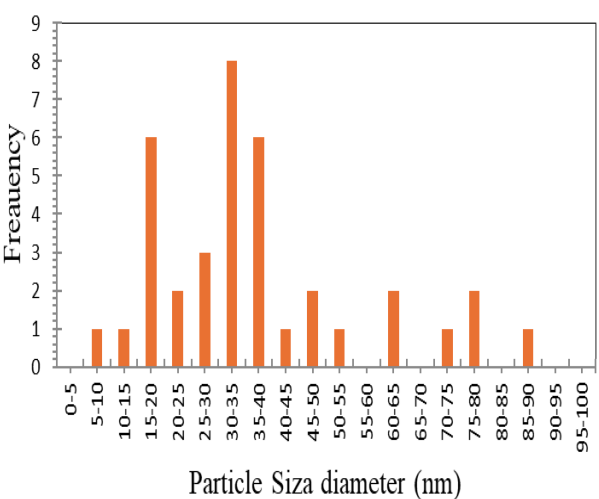
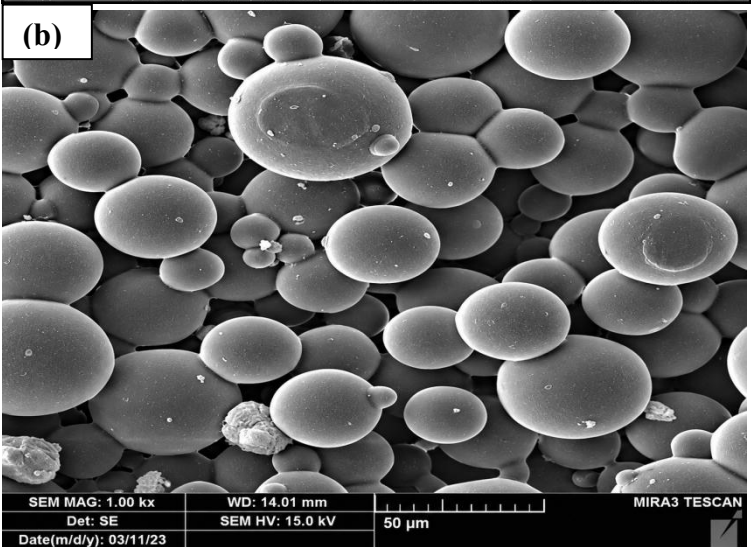
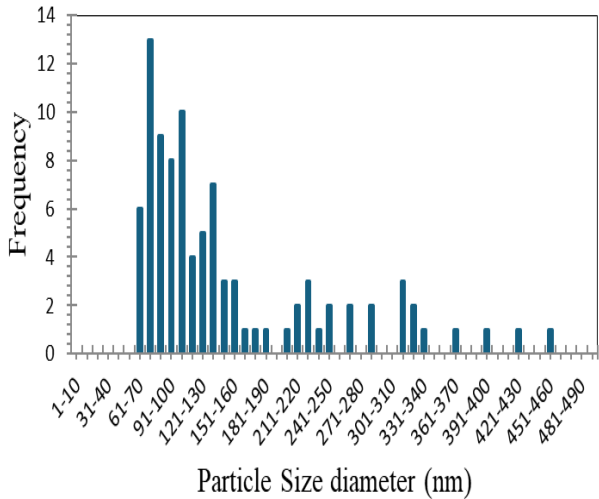
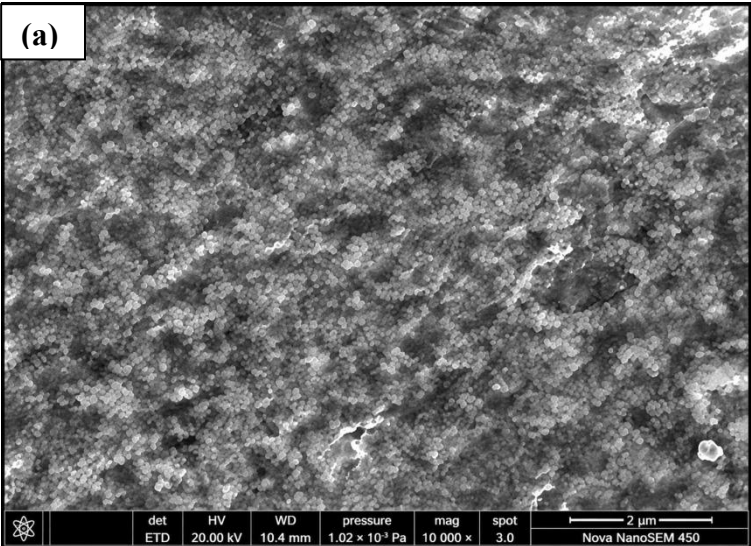


Figure 1 FESEM micrographs and corresponding particle-size distributions of the deposited coatings: **(a)** electrolessly deposited Ag coating on 316L stainless steel, **(b)** particle-size distribution of the Ag coating, **(c)** PMMA/4 wt.% NiO composite coating deposited over the Ag interlayer, **(d)** particle-size distribution of the PMMA/4 wt.% NiO coating, **(e)** PMMA/6 wt.% NiO composite coating deposited over the Ag interlayer, and **(f)** particle-size distribution of the PMMA/6 wt.% NiO coating.

3.2. *Elemental composition of the PMMA/NiO multilayer coatings*

Figure 2 presents the EDS spectra of the PMMA/NiO multilayer coatings deposited over the electroless Ag interlayer, corresponding to 6 wt.% NiO [Figure 2(a)] and 8 wt.% NiO [Figure 2(b)]. In both spectra, the principal detected elements are C, O, Ag, and Ni, which are consistent with the expected constituents of the PMMA/NiO/Ag multilayer coating system.

For the coating containing 6 wt.% NiO, Figure 2(a), the quantitative EDS analysis shows approximately 55.3 wt.% C, 29.8 wt.% O, 11.6 wt.% Ag, and 3.3 wt.% Ni. The dominant carbon signal originates mainly from the PMMA polymer matrix, since carbon constitutes the principal backbone of poly(methyl methacrylate). The relatively high oxygen content is associated with both the ester functional groups of PMMA and the oxygen-containing NiO reinforcement. The detection of Ni confirms the incorporation of the nickel-containing reinforcement into the composite coating, whereas the Ag signal originates predominantly from the underlying electroless silver layer [26].

The spectrum exhibits a strong C peak in the low-energy region and a pronounced O peak at approximately 0.5 keV. The Ni signal is represented by the low-energy Ni-L line and additional higher-energy Ni-K peaks in the region of approximately 7.5–8.3 keV. Ag is identified principally by its characteristic L-series peaks around 3 keV. The simultaneous detection of C, O, Ni, and Ag therefore verifies that the EDS interaction volume contains contributions from both the outer PMMA/NiO layer and the underlying Ag interlayer [27].

A distinctly different elemental contribution is observed for the 8 wt.% NiO coating, Figure 2(b). The corresponding EDS analysis gives approximately 60.5 wt.% Ag, 21.0 wt.% C, 15.1 wt.% O, and 3.5 wt.% Ni. Compared with the 6 wt.% NiO coating, the measured Ni concentration increases slightly from 3.3 to 3.5 wt.%, which is qualitatively consistent with the higher nominal NiO content. However, the difference detected by EDS is substantially smaller than the difference between the nominal powder compositions. This is expected because EDS provides a localized compositional measurement and its results depend on the selected analysis area, coating thickness, particle distribution, electron-beam penetration depth, and the relative contribution of the underlying layers.

A particularly important feature of Figure 2(b) is the substantial increase in the Ag signal, from 11.6 wt.% in Figure 2(a) to 60.5 wt.% in Figure 2(b). At the same time, the measured carbon fraction decreases from 55.3 to 21.0 wt.%, while oxygen decreases from 29.8 to 15.1 wt.%. This change should not necessarily be interpreted as a bulk compositional transformation of the entire coating. Instead, it strongly suggests that the analyzed region in Figure 2(b) received a greater contribution from the underlying Ag interlayer. Such behavior may result from local variations in the thickness of the PMMA/NiO top layer, differences in surface coverage, local porosity, particle packing, or penetration of the electron beam through the polymer–ceramic layer into the Ag-rich intermediate coating.

The strong Ag signal in Figure 2(b) is therefore consistent with the multilayer nature of the coating. Since the outer PMMA/NiO layer is deposited over an electroless Ag coating, the EDS interaction volume can sample the underlying silver depending on the local thickness and density of the composite layer. The absence, or very low intensity, of characteristic Fe and Cr peaks from the 316L stainless steel substrate is also noteworthy. This indicates that the deposited Ag and PMMA/NiO layers provide substantial coverage of the stainless-steel surface and largely suppress the direct elemental contribution from the metallic substrate within the analyzed regions [28].

The Ni peaks detected in both spectra provide evidence for successful incorporation of the Ni-containing reinforcement. Nevertheless, EDS alone cannot establish that nickel is present specifically as crystalline NiO. EDS confirms the simultaneous presence of Ni and O, whereas identification of the NiO crystalline phase should be supported by the corresponding XRD results. Similarly, the measured oxygen concentration cannot be assigned exclusively to NiO because PMMA itself contains oxygen in its ester groups [29]. The comparison between the two compositions can be summarized as follows:

Table 1 Quantitative EDS elemental composition (wt.%) of PMMA/NiO multilayer coatings containing 6 and 8 wt.% NiO deposited over the electroless Ag interlayer on 316L stainless steel.

Element	6 wt.% NiO coating	8 wt.% NiO coating	Observed change
C	55.3 wt.%	21.0 wt.%	Strong decrease
O	29.8 wt.%	15.1 wt.%	Decrease
Ag	11.6 wt.%	60.5 wt.%	Strong increase
Ni	3.3 wt.%	3.5 wt.%	Slight increase

The slight increase in the detected Ni content with increasing nominal NiO loading supports the successful addition of more Ni-containing reinforcement. However, the very large change in the Ag contribution indicates that local coating thickness and sampling depth strongly influence the EDS results. Therefore, the EDS percentages should be interpreted primarily as local surface/compositional measurements rather than as exact bulk compositions of the entire multilayer system.

Overall, Figure 2 confirms the successful formation of the intended Ag/PMMA–NiO multilayer architecture. Carbon and oxygen are associated predominantly with the PMMA matrix and oxide reinforcement, Ni indicates incorporation of the Ni-containing ceramic phase, and Ag confirms the presence of the electroless silver interlayer. Together with the FESEM observations, the EDS results support successful deposition and integration of the PMMA/NiO composite coating over the Ag-modified 316L stainless steel substrate.

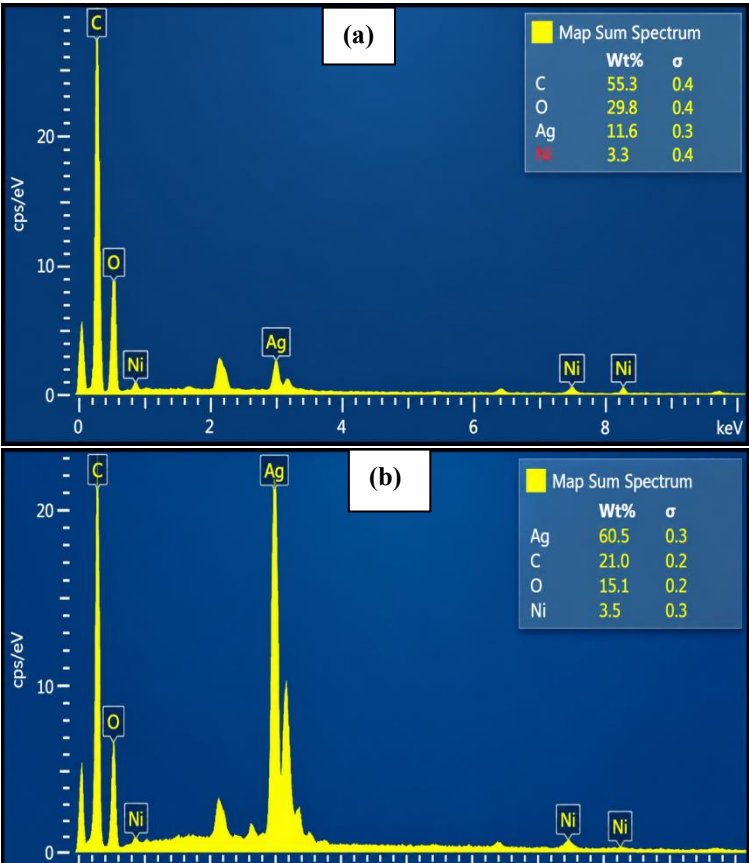


Figure 2 EDS spectra and quantitative elemental compositions of PMMA/NiO multilayer coatings deposited over the electroless Ag interlayer: **(a)** PMMA/6 wt.% NiO coating and **(b)** PMMA/8 wt.% NiO coating.

3.3. X-ray diffraction

Figure 3 presents the X-ray diffraction patterns of the electroless Ag coating and the PMMA/NiO composite coatings deposited over the Ag interlayer on 316L stainless steel. The diffraction pattern of the Ag-coated specimen exhibits characteristic reflections attributable to crystalline metallic silver. The principal Ag reflections are observed in the regions associated with the face-centered cubic (fcc) structure and can be indexed to the (111), (200), (220), and (311) crystallographic planes. These reflections confirm the successful formation of a crystalline Ag layer on the stainless-steel substrate.

For the PMMA/NiO multilayer coatings, the diffraction patterns contain contributions from the crystalline inorganic phases together with the broad background associated with the predominantly amorphous PMMA matrix. The broad diffuse features observed in the PMMA-containing coatings are characteristic of the limited long-range molecular ordering of the polymer. In contrast, the sharper reflections originate mainly from the crystalline Ag interlayer and NiO-containing phase [30].

The incorporation of NiO into the PMMA matrix modifies the relative intensity and shape of the diffraction peaks. Compared with the coating containing the lower NiO fraction, the sample with 6 wt.% NiO exhibits a noticeable increase in the intensity of several crystalline reflections, suggesting an increased contribution from the ceramic phase and/or differences in the degree of particle exposure within the polymer matrix. Slight variations in peak position may also arise from residual stress, interfacial strain, differences in coating thickness, or local structural distortion within the multilayer coating system. However, such small peak shifts should not be attributed exclusively to tensile stress unless supported by quantitative lattice-parameter or residual-stress analysis [31].

The persistence of the characteristic Ag and Ni-containing reflections after deposition of the PMMA/NiO composite layer indicates that the crystalline inorganic phases remain structurally stable during electrostatic spray deposition and subsequent thermal treatment. At the same time, the broad amorphous contribution from PMMA remains evident, demonstrating that the polymer matrix largely retains its non-crystalline nature.

The decrease in the apparent intensity of some peaks after formation of the PMMA-based composite layer may be related to attenuation of the incident and diffracted X-rays by the polymeric coating, changes in coating thickness, and differences in the distribution of the ceramic particles. Therefore,

variations in peak intensity should be interpreted in conjunction with the FESEM/EDS observations rather than considered as direct evidence of a phase transformation.

No additional diffraction peaks attributable to newly formed reaction products are evident within the detection limits of the measurement. This suggests that Ag, NiO, and PMMA are primarily combined through physical incorporation and interfacial interaction rather than through the formation of a new crystalline compound. Thus, the XRD results support the successful fabrication of a hybrid multilayer coating in which crystalline Ag and NiO phases are incorporated within or beneath a predominantly amorphous PMMA matrix.

The XRD analysis confirms the coexistence of the crystalline metallic Ag interlayer, the NiO-containing ceramic reinforcement, and the amorphous PMMA phase in the multilayer coating system. The preservation of these individual structural features indicates that the deposition and thermal-treatment procedures did not cause substantial crystalline phase degradation, while changes in diffraction intensity with NiO content reflect differences in the relative contributions and distribution of the coating constituents [32].

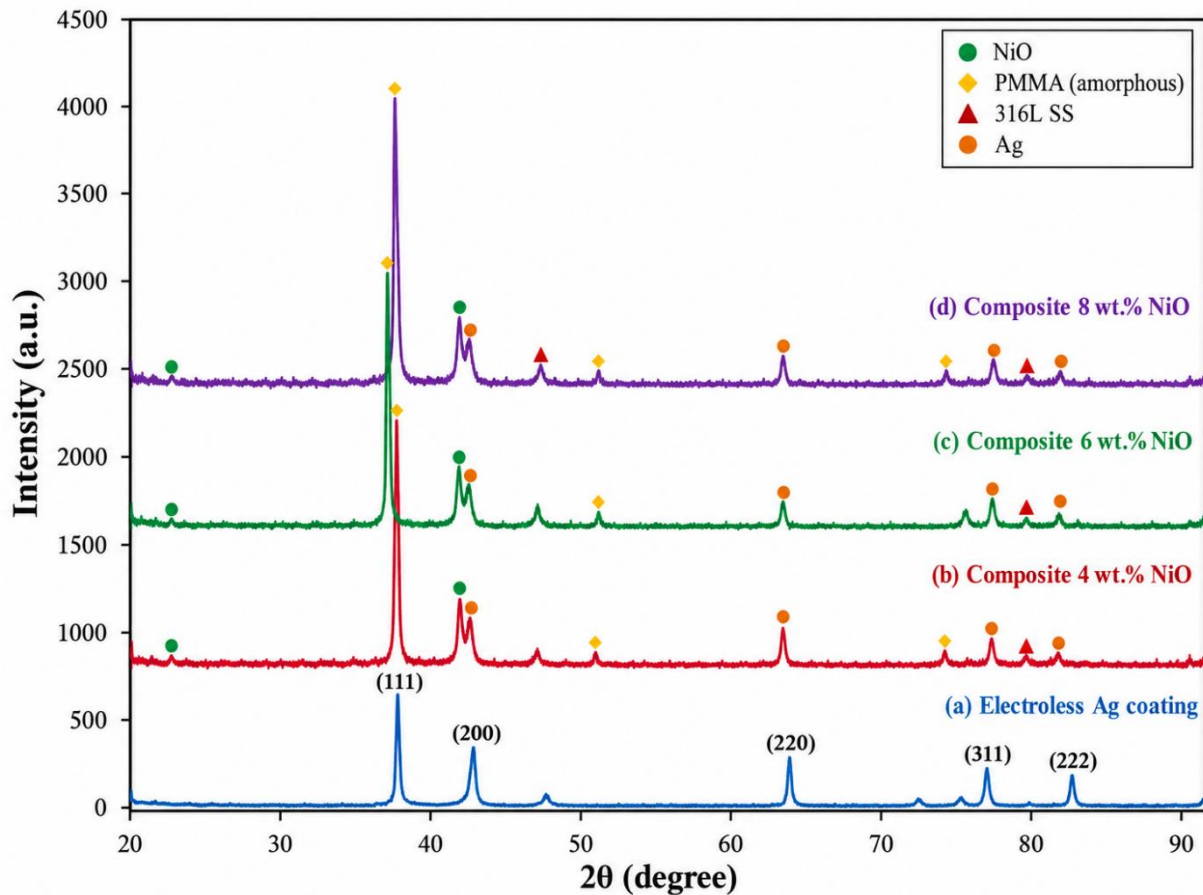


Figure 3 X-ray diffraction (XRD) patterns of (a) electrolessly deposited Ag nanoparticles on 316L stainless steel, (b) PMMA/NiO composite coating containing 4 wt.% NiO and 96 wt.% PMMA, (c) PMMA/NiO composite coating containing 6 wt.% NiO and 94 wt.% PMMA, and (d) PMMA/NiO composite coating containing 8 wt.% NiO and 92 wt.% PMMA.

3.4. Contact angle

The wettability of the uncoated and coated specimens is evaluated by measuring the static water contact angle using deionized water. Surface wettability is an important parameter for biomedical materials because it influences protein adsorption, cell attachment, interfacial interactions, and, consequently, the biological response of the implant surface. In general, a water contact angle lower than 90° indicates a hydrophilic surface, whereas values greater than 90° are characteristic of hydrophobic behavior. Improved hydrophilicity is generally desirable for implant coatings because it can facilitate interaction between the material surface and the surrounding physiological environment. Figures 4 show representative contact-angle measurements obtained for the investigated specimens after placing a deionized-water droplet on the surface for 60 s. The uncoated 316L stainless steel exhibited a relatively high contact angle of approximately 98.57° , indicating comparatively hydrophobic behavior. In contrast, the application of the Ag and PMMA/NiO coatings significantly decreased the water contact angle, demonstrating that surface modification improved the wettability of 316L stainless steel [33,34].

The PMMA/NiO composite coatings exhibited greater wettability than the uncoated 316L substrate. This behavior may be attributed to changes in surface chemistry, roughness, particle distribution, and the presence of polar oxygen-containing groups within the PMMA matrix together with the NiO ceramic phase. Increasing the NiO fraction modifies the surface topography and the number of high-energy surface sites, which can enhance the interaction between the coating and water molecules. Consequently, the deposited droplets spread more extensively over the composite-coated surfaces than over bare 316L stainless steel.

Among the investigated composite compositions, the coatings containing 6 wt.% NiO/94 wt.% PMMA and 8 wt.% NiO/92 wt.% PMMA exhibited contact angles of approximately 58.95° and 60.95° , respectively. These values confirm the hydrophilic nature of the composite surfaces and represent a substantial reduction relative to the uncoated 316L specimen. The 6 wt.% NiO coating therefore exhibited slightly greater wettability than the 8 wt.% NiO coating, as indicated by its lower contact angle.

The improvement in wettability may be beneficial for biomedical applications because hydrophilic surfaces generally promote rapid interaction with biological fluids and may support favorable protein adsorption and cell–material interactions. In addition, surface wettability can influence bacterial attachment; however, bacterial adhesion depends on several factors, including surface chemistry, charge, roughness, microbial species, and environmental conditions, and therefore cannot be predicted solely from the contact-angle value [35].

The contact-angle measurements demonstrate that deposition of the Ag/PMMA–NiO multilayer coating converts the relatively hydrophobic 316L stainless steel surface into a more hydrophilic surface. The improved wettability, particularly for the 6 wt.% NiO-containing composite, suggests enhanced surface characteristics that may favor biological interaction and osseointegration-related performance.

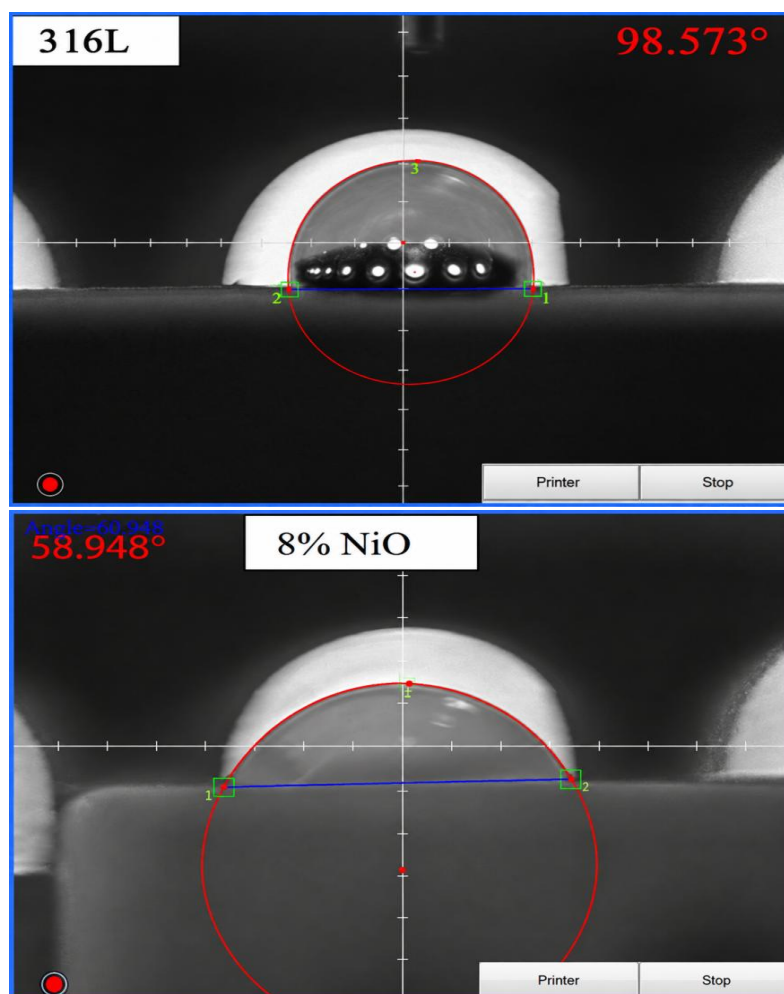


Figure 4 Representative water contact-angle images of (a) uncoated 316L stainless steel and (b) PMMA/NiO composite coating containing 8 wt.% NiO deposited over the Ag interlayer.

3.5. Roughness test

Surface roughness is measured using a TR200 surface roughness tester, and the arithmetic average roughness, R_a , is used to evaluate the effect of the different coatings on the surface characteristics. Five measurements are considered for each specimen, and the corresponding mean values are summarized in Table 2.

Table 2 Surface roughness (R_a) measurements of uncoated 316L stainless steel, Ag-coated 316L, and PMMA/NiO composite-coated specimens with different NiO contents.

Sample	Ra-1 (μm)	Ra-2 (μm)	Ra-3 (μm)	Ra-4 (μm)	Ra-5 (μm)	Mean Ra (μm)
316L SS	0.73	0.75	0.74	0.76	0.72	0.740
Ag-coated 316L	0.31	0.33	0.32	0.34	0.30	0.320
PMMA/4 wt.% NiO	0.25	0.26	0.255	0.265	0.245	0.255
PMMA/6 wt.% NiO	0.23	0.24	0.235	0.245	0.225	0.235
PMMA/8 wt.% NiO	0.24	0.25	0.245	0.255	0.235	0.245

The surface-roughness results presented in Figure 5 demonstrate a clear modification of the 316L stainless-steel surface after successive coating treatments. The uncoated 316L substrate exhibited the highest mean roughness, with an Ra value of approximately $0.740 \pm 0.016 \mu\text{m}$. After electroless deposition of the Ag layer, the surface roughness decreased markedly to $0.320 \pm 0.016 \mu\text{m}$, indicating that the deposited silver layer partially filled or covered the original surface irregularities and produced a comparatively smoother surface. A further reduction in roughness is observed after deposition of the PMMA/NiO composite coatings. The mean Ra values are approximately $0.255 \pm 0.008 \mu\text{m}$, $0.235 \pm 0.008 \mu\text{m}$, and $0.245 \pm 0.008 \mu\text{m}$ for the coatings containing 4, 6, and 8 wt.% NiO, respectively. Among the investigated compositions, the 6 wt.% NiO/94 wt.% PMMA coating exhibited the lowest surface roughness, suggesting that this composition provides a more uniform and compact surface morphology. When the NiO content is increased from 6 to 8 wt.%, a slight increase in Ra is observed. This behavior may be associated with increased particle accumulation, localized agglomeration, or reduced homogeneity of the ceramic phase within the polymer matrix at higher NiO loading. Such microstructural changes can generate small surface protrusions and irregularities, resulting in a modest increase in roughness. The results indicate that both Ag deposition and subsequent PMMA/NiO composite coating significantly smooth the original 316L surface. The relatively small variation among the composite-coated specimens further suggests that NiO concentration influences the final surface topography, with 6 wt.% NiO providing the most favorable surface uniformity under the present processing conditions [36,37].

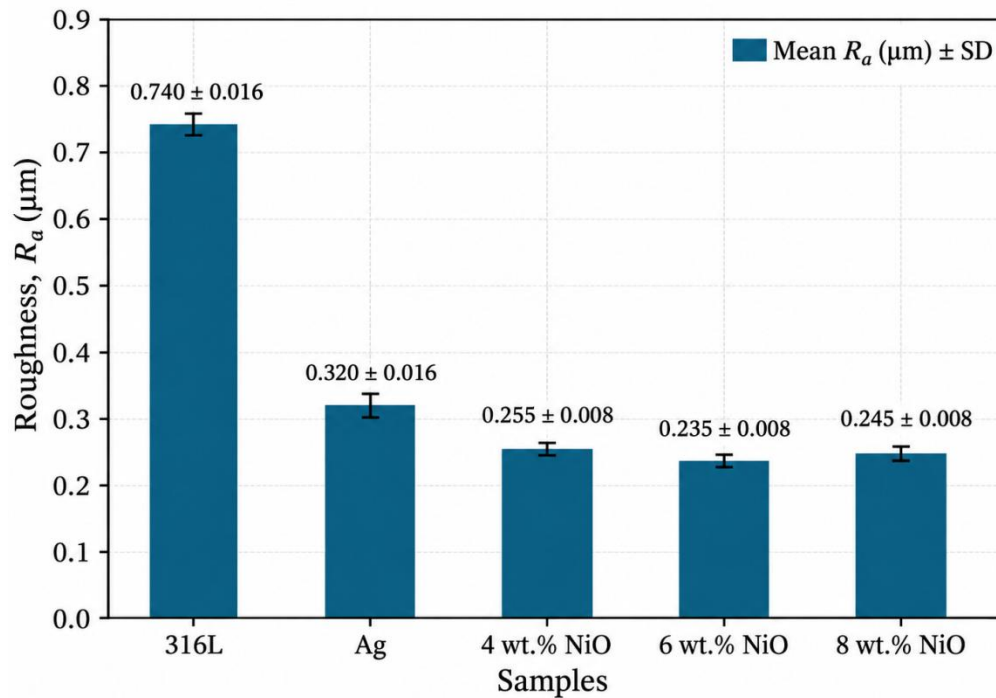


Figure 5 Surface roughness of uncoated 316L stainless steel, electroless Ag nanocoating, and PMMA/NiO composite coatings containing different NiO concentrations.

3.6. Vickers microhardness test

The Vickers microhardness of uncoated 316L stainless steel, electroless Ag-coated 316L, and PMMA/NiO composite coatings is evaluated to determine the influence of surface modification and NiO reinforcement on the mechanical response of the coated system (Figure 6). Measurements are performed at three different locations on each specimen using an applied load of 9.8 N and a dwell time of 30 s. The results are expressed as mean $\pm$ standard deviation (SD), and the error bars in Figure 6 represent ± 1 SD. The measured values are 351 ± 3 HV for uncoated 316L stainless steel, 384 ± 3 HV for the electroless Ag coating, 487 ± 4 HV for PMMA/4 wt.% NiO, 506 ± 4 HV for PMMA/6 wt.% NiO, and 485 ± 4 HV for PMMA/8 wt.% NiO.

The uncoated 316L stainless steel exhibited the lowest microhardness, with a mean value of 351 HV and an SD of 3 HV, giving an error-bar range of approximately 348–354 HV. After electroless deposition of the Ag layer, the microhardness increased to 384 ± 3 HV, corresponding to an approximate range of 381–387 HV and an increase of about 9.4% relative to the uncoated substrate. This improvement indicates that the Ag interlayer modifies the surface mechanical response and increases resistance to localized indentation.

A more pronounced enhancement is observed after deposition of the PMMA/NiO composite coatings. The PMMA/4 wt.% NiO specimen exhibited a mean hardness of 487 ± 4 HV, corresponding to an

approximate error-bar range of 483–491 HV. Increasing the NiO content to 6 wt.% further increased the microhardness to 506 ± 4 HV, with a corresponding range of 502–510 HV. This represents an increase of approximately 44.2% compared with uncoated 316L stainless steel and about 31.8% compared with the Ag-coated specimen.

The 6 wt.% NiO/94 wt.% PMMA coating therefore exhibited the highest microhardness among the investigated samples. Its relatively small SD of 4 HV also indicates good repeatability among the three indentation measurements. The enhanced hardness can be attributed to effective incorporation and dispersion of the comparatively rigid NiO ceramic phase within the PMMA matrix, which restricts localized plastic deformation and improves the load-bearing capability of the composite layer.

When the NiO concentration is increased further to 8 wt.%, the mean hardness decreased slightly to 485 ± 4 HV, corresponding to an approximate range of 481–489 HV. This modest reduction may be associated with localized particle agglomeration, reduced dispersion uniformity, increased interfacial heterogeneity, or less effective consolidation of the composite coating at the higher ceramic loading. Such microstructural irregularities can create locally weaker regions despite the increased nominal NiO content [38, 39].

The low SD values of 3–4 HV for all specimens indicate relatively consistent microhardness measurements. The results confirm that both the Ag interlayer and the PMMA/NiO composite coatings significantly enhance the surface hardness of 316L stainless steel, with the PMMA/6 wt.% NiO coating showing the highest mean microhardness of 506 ± 4 HV under the present processing conditions.

Table 3 Vickers microhardness results of the samples, an electroless, and the composite coating (PMMA-NiO) produced by ESD.

Sample	HV-1	HV-2	HV-3	Mean HV
316L stainless steel	348	351	354	351
Electroless Ag coating	381	384	387	384
PMMA/4 wt.% NiO	483	487	491	487
PMMA/6 wt.% NiO	502	506	510	506
PMMA/8 wt.% NiO	481	485	489	485

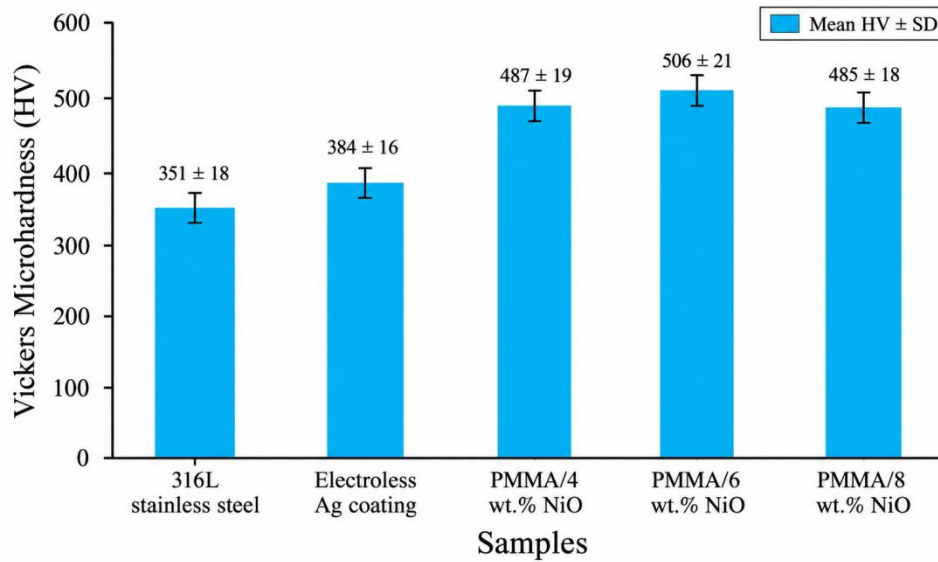


Figure 6 Vickers microhardness of uncoated 316L stainless steel, electroless Ag-coated 316L, and PMMA/NiO composite coatings produced by electrostatic spray deposition (ESD).

3.7. Tribological performance and wear behavior

The tribological performance of uncoated 316L stainless steel, electroless Ag-coated 316L, and PMMA/NiO composite coatings is evaluated in terms of wear-volume loss, specific wear rate, and coefficient of friction (COF). The corresponding numerical values are summarized in Table 4, while the comparative results are illustrated in Figure 7(a–c). For the present dataset, the wear behavior is considered under a pin-on-disc configuration with an applied normal load of 10 N and a total sliding distance of 500 m. The specific wear rate, W , is calculated according to [40, 41]:

$$W = \frac{V}{F \times L} \quad (1)$$

where V is the wear-volume loss (mm^3), F is the applied normal load (N), and L is the sliding distance (m). As shown in Table 4 and Figure 7(a), uncoated 316L stainless steel exhibited the highest wear-volume loss of $0.420 \pm 0.021 \text{ mm}^3$, indicating the poorest resistance to material removal among the investigated specimens. The corresponding specific wear rate is $(8.40 \pm 0.42) \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, as shown in Figure 7(b), while the COF reached 0.62 ± 0.03 in Figure 7(c). The comparatively high wear and friction of the bare substrate can be attributed to direct interaction between the stainless-steel surface and the counterbody, resulting in localized plastic deformation, microcutting, and formation of wear debris.

Following electroless deposition of Ag, a noticeable improvement in tribological behavior is observed. As reported in Table 4, the wear-volume loss decreased to $0.310 \pm 0.016 \text{ mm}^3$, while the specific wear rate decreased to $(6.20 \pm 0.32) \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$. The COF is also reduced to 0.54 ± 0.03 , as illustrated in Figure 7(c). Relative to uncoated 316L, the Ag layer reduced the wear rate by approximately 26%. This improvement suggests that the electroless Ag layer acts as an intermediate protective barrier and reduces direct mechanical interaction between the stainless-steel substrate and the sliding counterface.

A substantially greater improvement is achieved after deposition of the PMMA/NiO composite coatings. The PMMA/4 wt.% NiO coating exhibited a wear-volume loss of $0.190 \pm 0.011 \text{ mm}^3$, a specific wear rate of $(3.80 \pm 0.22) \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, and a COF of 0.43 ± 0.02 , as summarized in Table 4 and displayed in Figure 7(a–c). Compared with uncoated 316L stainless steel, this represents a reduction in specific wear rate of approximately 54.8%. The improved resistance to sliding wear can be associated with the reinforcing effect of the comparatively rigid NiO particles within the PMMA matrix, which improves load distribution and restricts localized deformation.

The most favorable tribological response is obtained for the PMMA/6 wt.% NiO coating. As clearly observed in Figure 7(a), this composition exhibited the lowest wear-volume loss, $0.140 \pm 0.009 \text{ mm}^3$. Similarly, Figure 7(b) shows that the specific wear rate reached a minimum value of $(2.80 \pm 0.18) \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, while Figure 7(c) demonstrates that the COF decreased to 0.36 ± 0.02 . These values correspond to reductions of approximately 66.7% in wear rate and 41.9% in COF compared with bare 316L stainless steel.

The superior wear resistance of the 6 wt.% NiO coating is consistent with its previously observed high microhardness. Effective dispersion of NiO particles within the PMMA matrix may improve the load-bearing capability of the coating, restrict plastic deformation, and reduce direct penetration of the counterbody into the polymeric layer. The 6 wt.% NiO composition therefore appears to provide a favorable balance between ceramic reinforcement and continuity of the PMMA matrix. At this concentration, NiO can contribute to mechanical strengthening without excessively disrupting coating cohesion.

When NiO concentration is increased to 8 wt.%, a slight deterioration in tribological performance is observed. As presented in Table 4, the wear-volume loss increased to $0.170 \pm 0.010 \text{ mm}^3$, the specific wear rate increased to $(3.40 \pm 0.20) \times 10^{-5} \text{ mm}^3 \cdot \text{N}^{-1} \cdot \text{m}^{-1}$, and the COF increased to 0.40 ± 0.02 . These changes are also evident in Figure 7(a–c). Although the 8 wt.% NiO coating still exhibited substantially better performance than both uncoated 316L and the Ag-coated specimen, its wear resistance is slightly lower than that of the 6 wt.% NiO coating.

The modest deterioration at the highest NiO loading may be associated with localized particle agglomeration, increased interfacial heterogeneity, or reduced continuity of the polymer matrix. Such features may facilitate particle pull-out during sliding, and detached NiO particles may subsequently act as third-body abrasives, thereby increasing both friction and material removal.

The trends presented in Table 4 and Figure 7 demonstrate that surface modification significantly improves the tribological behavior of 316L stainless steel. The wear-resistance performance follows the order:

316L < Ag < PMMA/4 wt.% NiO < PMMA/8 wt.% NiO < PMMA/6 wt.% NiO.

Among the investigated coatings, PMMA/6 wt.% NiO/94 wt.% PMMA exhibited the best overall tribological performance, showing the lowest wear-volume loss, specific wear rate, and coefficient of friction. The agreement between the tribological and microhardness results indicates that improved resistance to localized deformation plays an important role in enhancing the wear resistance of the PMMA/NiO composite coatings.

Table 4 Tribological characteristics of uncoated 316L stainless steel, electroless Ag-coated 316L, and PMMA/NiO composite coatings.

Sample	Wear volume loss (mm ³)	Specific wear rate (mm ³ .N ⁻¹ .m ⁻¹)	COF
316L stainless steel	0.420 ± 0.021	$(8.40 \pm 0.42) \times 10^{-5}$	0.62 ± 0.03
Electroless Ag coating	0.310 ± 0.016	$(6.20 \pm 0.32) \times 10^{-5}$	0.54 ± 0.03
PMMA/4 wt.% NiO	0.190 ± 0.011	$(3.80 \pm 0.22) \times 10^{-5}$	0.43 ± 0.02
PMMA/6 wt.% NiO	0.140 ± 0.009	$(2.80 \pm 0.18) \times 10^{-5}$	0.36 ± 0.02
PMMA/8 wt.% NiO	0.170 ± 0.010	$(3.40 \pm 0.20) \times 10^{-5}$	0.40 ± 0.02

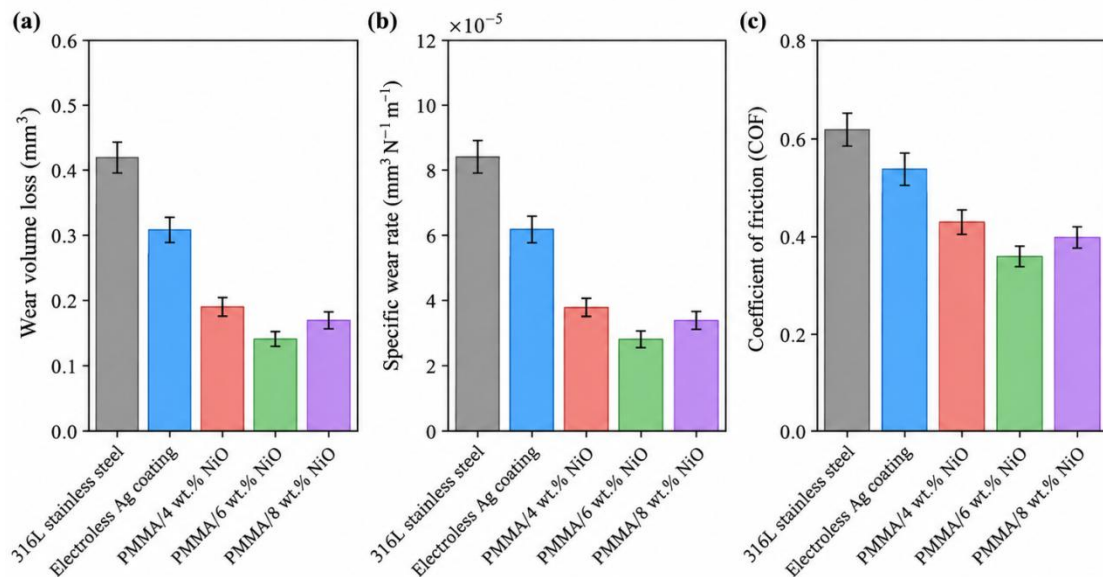


Figure 7 Tribological characteristics of uncoated 316L stainless steel, electroless Ag-coated 316L, and PMMA/NiO composite coatings: **(a)** wear-volume loss, **(b)** specific wear rate, and **(c)** coefficient of friction (COF). Error bars represent the corresponding standard deviations.

4. CONCLUSIONS

The present study demonstrated the successful development of multifunctional multilayer coatings on 316L stainless steel by combining electroless Ag deposition with electrostatic spray deposition of PMMA/NiO composite layers. The electrostatic spray process provided a relatively simple, rapid, and effective approach for producing continuous polymer–ceramic coatings with different NiO contents. FESEM observations revealed dense and well-covered surfaces, while EDS analysis confirmed the presence of Ag, Ni, O, and C associated with the Ag interlayer and PMMA/NiO composite system. XRD analysis further indicated the coexistence of crystalline Ag and NiO-containing phases within the predominantly amorphous PMMA matrix without evidence of major new crystalline reaction products. Surface modification significantly altered the mechanical and interfacial properties of 316L stainless steel. The surface roughness decreased from $0.740 \pm 0.016 \mu\text{m}$ for bare 316L to $0.235 \pm 0.008 \mu\text{m}$ for the 6 wt.% NiO coating. The composite coatings also exhibited improved wettability, with contact

angles substantially lower than that of uncoated 316L. Vickers microhardness increased from 351 ± 3 HV for the substrate to a maximum of 506 ± 4 HV for the PMMA/6 wt.% NiO coating. Consistently, the 6 wt.% NiO composition exhibited the most favorable tribological response, with a wear volume of 0.140 ± 0.009 mm³, wear rate of $(2.80 \pm 0.18) \times 10^{-5}$ mm³ N⁻¹ m⁻¹, and COF of 0.36 ± 0.02 . Overall, the 6 wt.% NiO/94 wt.% PMMA composition provided the most balanced surface, mechanical, and tribological performance, making it a promising coating architecture for further biomedical evaluation.

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