

TESTING AND CHARACTERIZATION OF ANTIMICROBIAL COPPER COATINGS DEPOSITED ON STEEL SUBSTRATES

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The high incidence of nosocomial infections in medical units requires the adoption of advanced measures to control and limit their spread. In this context, the study and optimization of intrinsically bactericidal coatings, such as copper, is a highly topical research direction. In this work, copper deposits on three different metallic substrates were investigated: structural steel S235 and austenitic stainless steels 304L and 316L, in order to establish the optimal correlations between surface properties and antimicrobial efficiency. The experimental methodology included profilometric roughness determinations, structural analyses by optical microscopy (qualitative and quantitative), adhesion evaluation by scratch tests and wettability determinations by the sessile drop method, correlated with the calculation of the surface free energy (SFE), both for the substrates and for the deposited layers. By correlating the experimental data, it was determined that the best antimicrobial properties and the best layer adhesion were recorded in the case of the 316L stainless steel substrate sample, copper coating.

Keywords: antimicrobial copper coating, steel substrate. scratch test, roughness measurements, wettability, contact killing, arc spraying

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1. Introduction

The global problem of healthcare-associated infections represents a pressing public health emergency, affecting many patients annually [1]. In hospital settings, patients and healthcare workers are routinely exposed to high-touch surfaces, predominantly fabricated from stainless steel or aluminum alloys. These materials permit the persistence of clinically significant pathogens, such as

methicillin-resistant *Staphylococcus aureus* (MRSA), vancomycin-resistant *Enterococcus* (VRE), and multidrug-resistant (MDR) Gram-negative organisms for periods ranging from days to months [1].

Copper-based surfaces exhibit continuous and self-sustaining antimicrobial activity through multiple interrelated mechanisms, including controlled ion release, disruption of membrane integrity, and induction of oxidative stress via reactive oxygen species (ROS), that achieve persistent microbial inactivation without external intervention [2, 3]. Integration of such coatings onto stainless steel and aluminum substrates constitutes a technically and economically viable strategy for the passive and continuous reduction of surface microbial burden [4, 5].

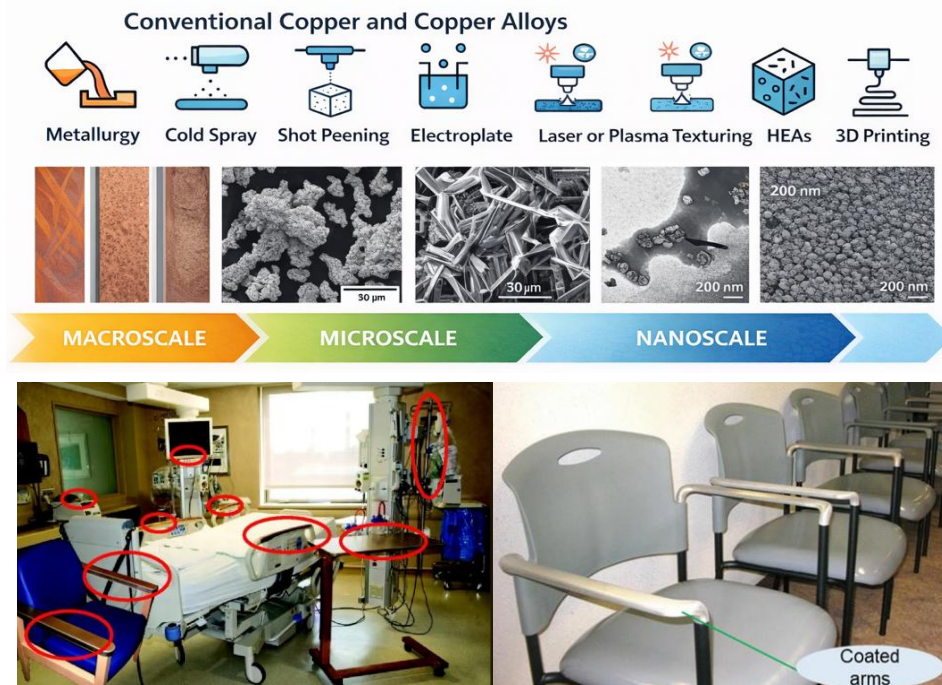


Figure 1. Schematic presentations of the methods for obtaining copper coatings in healthcare settings [1].

The nature of the copper-based coating is strongly affected by the copper source and the substrate used [2]. There are several techniques to produce copper coatings on metallic surfaces. Amongst them, chemical vapor deposition, physical vapor deposition, electrodeposition, electroless deposition, sputtering, and thermal spray techniques are widely used methods [6, 7]. The properties of copper coatings are influenced by four interdependent factors: (1) the nature and purity of the copper source; (2) the transport behavior of copper species toward the substrate; (3) the nucleation and growth mechanisms at the substrate surface; and

(4) the physicochemical properties of the substrate, including surface energy, crystallographic texture, roughness, and residual stress state [1, 8]. (Figure 1)

Various researchers have clearly demonstrated the long-term antimicrobial efficacy of the thermally sprayed copper coatings in healthcare settings under real-world hospital operational conditions (Figure 1) [2, 3, 9]. Even that, some challenges encountered in the deployment of copper coatings on stainless steel substrates in the term of adhesion strength.

The aim of the present study was to systematically characterize the adhesion strength and microstructure of copper coatings deposited on carbon steel and stainless-steel substrates via thermal spray deposition.

2. Materials and methods

For the experiments, three grades of steel were used as substrates for the subsequent copper deposits: S235 – a general-purpose structural steel (structural low-carbon steel), and 304L and 316L – austenitic stainless steels. The three samples were cut from hot-rolled strips with thicknesses of 4–6 mm, having surface dimensions of approximately 50×50 mm. The coding of the samples is presented in Table 1.

Table 1. Sample coding

Samples Code	Substrate	Coating
S1	S235	Copper
S2	304L	
S3	316L	

The chemical composition of the substrates was determined using a LECO GDS-500A spectrometer.

To produce copper coatings on the steel substrates, the twin-wire arc spraying (arc metallization) technique was selected. This is a versatile method (using two metallic wires melted by an electric arc), recognized for advantages such as rapid solidification, dense and well-adherent coatings, and the absence of substrate deformation. The deposition parameters are listed in Table 2. For the coatings, copper wire with a purity of 99.95% and a diameter of 1.6 mm was used.

Table 2. Deposition Parameters

Arc Voltage [V]	Current Intensity [A]	Air Pressure [bar]	Spray Distance [mm]	Feed Rate [m/min]	Air Flow Rate [l/min]
28	200	6	125	6	400

For copper deposition, the steel samples were grit-blasted using alumina powder with an average particle size of 0.3 mm. Following this operation, the substrates were preheated to 80 °C.

The samples were structurally analyzed by optical microscopy using a Nikon Eclipse MA100N inverted microscope (Nikon Metrology Europe NV, Leuven, Belgium), and coating thickness measurements were also performed. The metallographic preparation of the samples was carried out using a Struers preparation line.

Roughness measurements were performed for both the substrates and the coatings in accordance with ISO 21920, using a contact (stylus) profilometer, Form Talysurf® I-Series PRO Range (Taylor Hobson).

The wettability of the substrates and the deposited coatings was analyzed using the sessile drop method by determining the contact angles, with the aid of a KRÜSS DSA30 drop shape analysis system (A. Krüss Optronic GmbH, Hamburg, Germany). Distilled water, diiodomethane, and ethylene glycol were used as test liquids. Subsequently, the surface free energy was calculated using the OWRK method (Owens, Wendt, Rabel, and Kaelble) through linear regression.

The scratch test was performed in accordance with the EN 1071-3:2005 standard, on different areas of the sample surfaces over a distance of 10 mm, with a load ranging from 0 to 100 N, applied over one minute at room temperature (23 ± 1 °C). The test was carried out using a universal tribometer – the UMT-TriboLab platform (Bruker, Madison, WI, USA).

3. Results and discussion

3.1 Substrates characterization

- chemical composition

It was experimentally determined for each substrate and included in Table 3. For comparison, the compositions of the standardized grades of the steels used are also presented.

By comparatively analyzing the compositional data, it can be considered that the three steels used in the experiments fall within standardized specifications.

Table 3. Chemical composition of the substrates

Sample substrate	Chemical composition								
	C	Mn	Cr	Ni	Mo	Si	P	S	Fe
S1	0,20	1,20	-	-	-	0.30	0.040	0.035	Bal.
S235 (SR EN 10025-2)	≤0.17	≤1.40	-	-	-	<0.35	<0.035	<0.035	Bal.
S2	0.1	1.9	18.0	8.0	-	0.71	0.040	0.042	Bal.
304L (ASTM A240)	≤0.08	≤2.00	17.5- 19.5	8.0- 10.5	-	≤1.00	≤0.045	≤0.030	Bal.
S3	0.04	1.80	18.0	10	2.6	0.63	0.040	0.038	Bal.
316L (ASTM A240)	≤0.03	≤2.00	16.0- 18.0	10.0- 14.0	2.0- 3.0	≤0.75	≤0.045	≤0.030	Bal.

- **optical microscopy**

Figure 2 presents the microstructural images of the three substrates, recorded at a magnification of 500x.

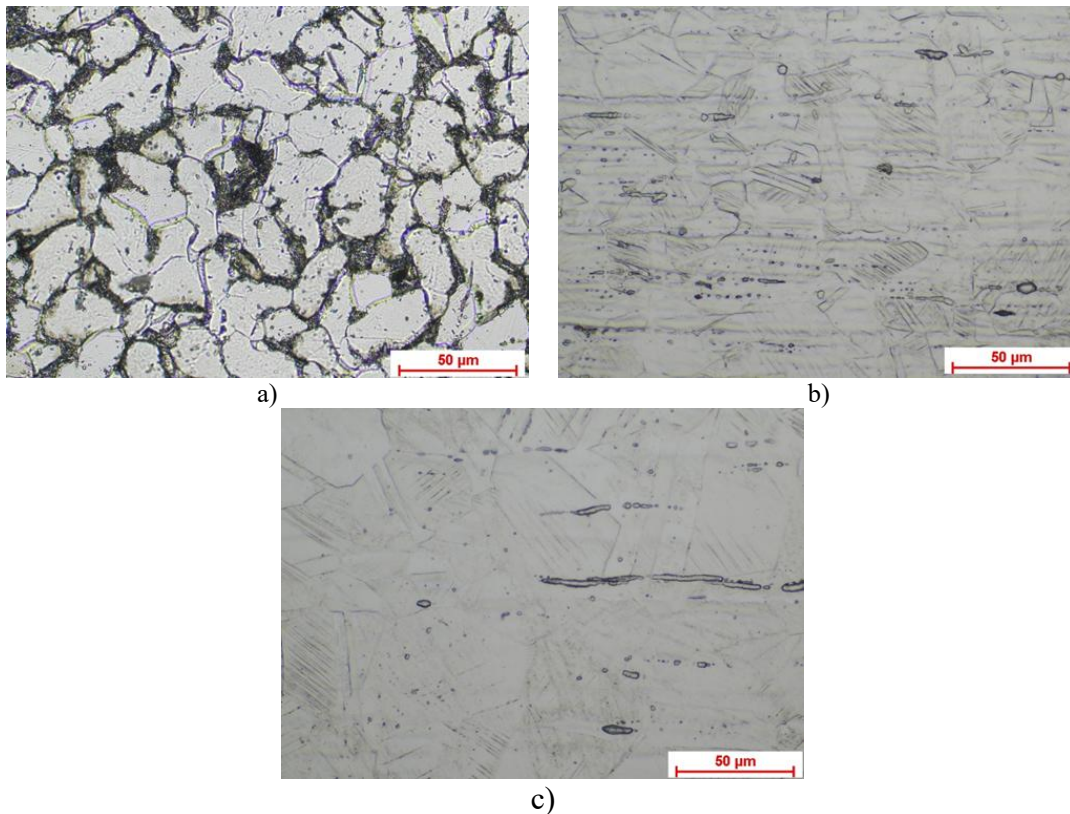


Figure 2. Optical micrographs of samples substrates: **a)** S1 – NITAL 2% reagent **b)** S2 – Marble's reagent, **c)** S3 – Marble's reagent

In Figure 2(a), a ferrite–pearlite structure can be observed, with ferrite as the predominant phase, characteristic of S235 steel. In some areas, small networks of tertiary cementite can also be identified, which are relatively common in low-carbon steels. In Figures 2(b) and (c), austenitic grains with twins—both annealing twins and mechanical twins—can be observed, together with inclusions aligned along the main hot deformation direction, particularly sulfide-type inclusions, most likely manganese sulfides. Given the well-known high susceptibility of austenitic stainless steels to work hardening [10], the mechanical twins were formed mainly during the grinding and polishing stages of sample preparation.

Regarding the higher number of inclusions in sample S2, this can be correlated with its higher sulfur and silicon content, as shown in Table 3.

- roughness measurements

The measured parameters, Ra, Rq, and Rsk, are presented in Table 4.

Table 4. Surface roughness parameters of the substrates

Substrate	Ra (μm)	Rq (μm)	Rsk
S235	1.4033 $\pm$ 0.039	1.9355 $\pm$ 0.057	0.2147 $\pm$ 0.029
304L	0.9079 $\pm$ 0.021	1.5337 $\pm$ 0.022	0.3983 $\pm$ 0.055
316L	1.3317 $\pm$ 0.042	1.6700 $\pm$ 0.051	-0.0158

It can be observed that the highest Ra value is recorded for the unalloyed steel, as it exhibits slightly lower hardness and a lower tendency for work hardening compared to the other substrates.

Regarding the austenitic stainless steels, 304L shows the lowest Ra value, of 1.1979 μm . The explanation lies in the low value of stacking fault energy (SFE), which favors the phase transformation from austenite (γ) to martensite (ϵ or α') [11, 12]. Thus, in the substrate of sample S2, metastable austenite is present, which partially transforms into martensite during blasting. This martensite is rich in dislocations and leads to a rapid increase in hardness from the early stages of blasting. The effect is the formation of a less rough surface. In contrast to the substrate of sample S2, 316L has a higher stacking fault energy (SFE), exceeding 30 mJ/m^2 [13], which inhibits phase transformation but promotes deformation by twinning, a process similar to that occurring in TWIP steels. Although hardening is also significant in this case, it occurs more gradually, allowing a higher surface roughness to be obtained.

Obviously, a similar variation is also observed for the Rq parameter.

For the substrate of sample S3, the Rsk parameter is close to zero (but with a slightly negative value), indicating an almost symmetric topography, characterized by a balanced distribution of peaks and valleys (asperities and pits) relative to the mean line of the profile. A positive value of this parameter for S235

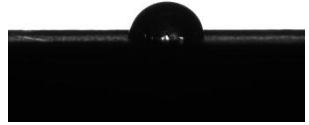
can be correlated with its lowest hardness compared to the other two (high-alloyed) steels, which led to a higher volume of protrusions relative to depressions.

The relatively high value of the skewness of the roughness profile recorded for 304L steel may suggest a non-uniform, highly localized phase transformation, which resulted in the formation of pronounced protrusions in certain areas.

- wettability measurements

The measured values of the contact angle and the subsequently calculated free surface energy (FSE) are presented in Table 5.

Table 5. Wettability measurements of the substrates

Substrate	Contact Angle (°)	Surface Free Energy (mN/m)
S235	52.66±1.64 	65.8±0.98
304L	79.4±1.97 	58.31±1.01
316L	82.73±1.98 	56.46±0.93

The evaluation of substrate wettability is relevant for obtaining an adherent coating and a pore-free interface, and it also represents an important indicator of the degree of surface degreasing prior to deposition. In this regard, it can be stated that in all cases the contact angles are lower than 90°, meaning that the substrates are hydrophilic [14].

The formation of a non-porous, compact Cr₂O₃ layer—further reinforced by MoO₂ in the case of the S3 substrate—explains the relatively large difference between the contact angle measured for S235 (covered with a porous layer of iron oxyhydroxides/oxides) and those measured for the stainless steels.

3.2 Layers Characterization

The thickness measurements of the coatings for the three samples are illustrated in Fig. 3(a)–(c) and summarized in Table 6.

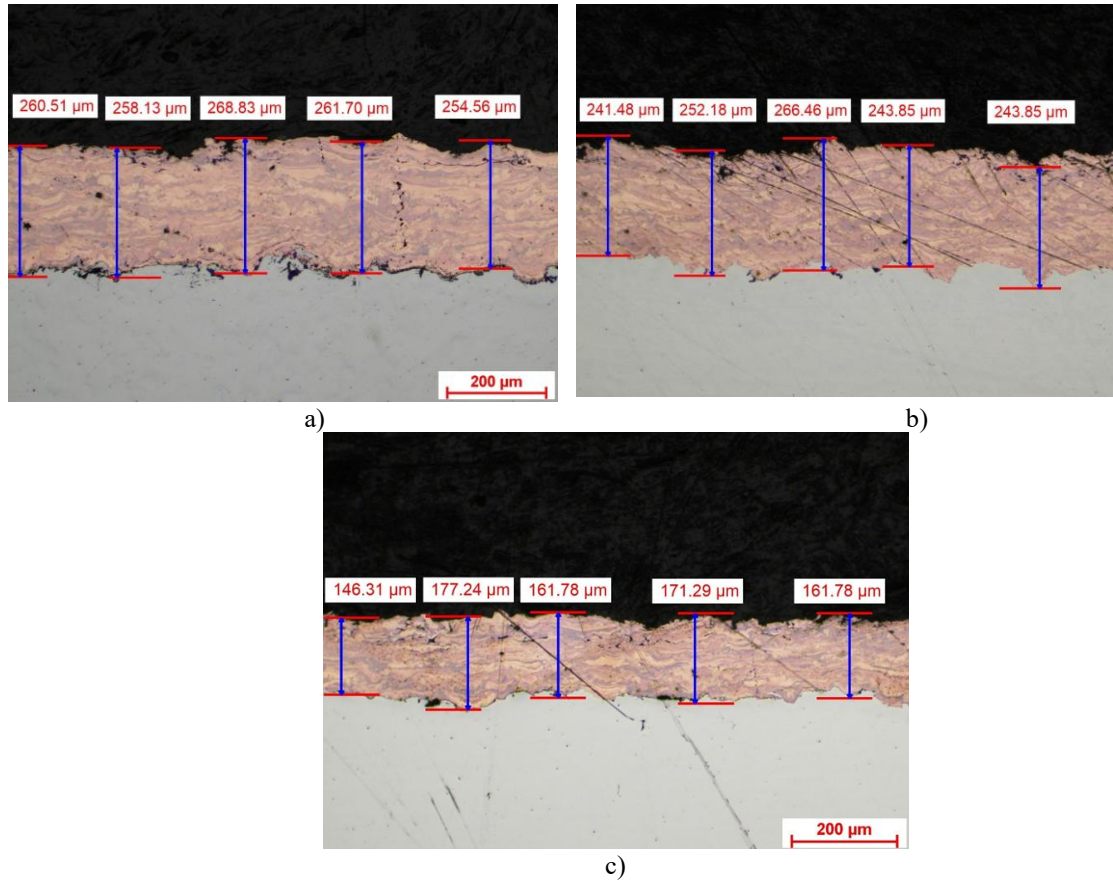


Figure 3. Quantitative optical microscopy images of the unetched samples:
a) – S1, b) – S2, c) – S3

Table 6. Thickness of the copper coatings	
Sample	Average coating thickness [μm]
S1	260.746
S2	249.564
S3	163.68

The smallest coating thickness is obtained for sample S3, due to the highest contact angle recorded (82.73 ± 1.98). Poor wettability leads to material losses during deposition through the rebound of liquid droplets, resulting in the formation of a coating with reduced thickness.

In Figure 3(a), pronounced porosity can be observed at the interface, most likely as a result of the rapid dehydration of oxyhydroxides present on the surface of the S1 substrate when in contact with the molten copper droplets. The liquid metal has a sufficiently high temperature to cause their decomposition. The transformation of the resulting water into vapor is accompanied by a significant increase in volume. This leads to the initiation of microcracks, in addition to the pores formed due to the entrapment of vapor by the solidified copper.

A lower porosity, inherent to the deposition process, can also be observed in Figures 3(b) and 3(c).

-roughness measurements

The roughness parameters measured for samples S1, S2, and S3 are presented in Table 7.

Table 7. Surface roughness parameters of the samples

Samples Code	Ra (μm)	Rq (μm)	Rsk
S1	3.1160 $\pm$ 0.048	4.0113 $\pm$ 0.061	0.3271 $\pm$ 0.030
S2	3.3394 $\pm$ 0.053	4.3367 $\pm$ 0.072	0.4087 $\pm$ 0.051
S3	3.2257 $\pm$ 0.067	4.1130 $\pm$ 0.068	0.5021 $\pm$ 0.024

The roughness of the copper coatings deposited on the three steels is comparable in terms of the measured parameters, and the relatively low values indicate optimal control of the deposition parameters. As a result, post-deposition finishing by grinding/polishing will require minimal effort. The positive Rsk coefficients indicate a higher volume of protrusions, i.e., more sharp peaks distributed above the mean line—an unfavorable profile for antibacterial applications, as the contact between bacteria and copper is reduced. To improve the biocidal character and, respectively, the antimicrobial properties, it is necessary to reduce the roughness and obtain a more favorable profile through polishing.

- wettability measurements

The measured values of the contact angle and the subsequently calculated free surface energy (FSE) are presented in Table 8.

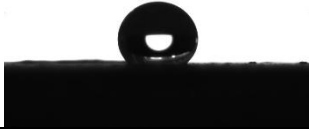
By comparatively analyzing the contact angle values, it can be observed that all samples exhibit hydrophobic coatings. However, the most effective coating from a biocidal standpoint is the one associated with the highest surface free energy, which also correlates with the highest Ra roughness value (sample S3).

Therefore, the contaminated fluid will exhibit better adhesion to the substrate, having sufficient energy to overcome the air pressure within the

microcavities of the copper coating, thus favoring the transition to a Wenzel-type wetting regime.

In the case of sample S1, characterized by the lowest surface free energy, the fluid droplet will not exhibit strong adhesion nor sufficient energy to expel air bubbles from the surface microrelief. As a result, contact with the biocidal material will be significantly reduced, occurring almost exclusively at the sharp peaks of the surface. A similar behavior is also observed for sample S2.

Table 8. Wettability measurements of the coatings

Samples Code	Contact Angle (°)	Surface Free Energy (mN/m)
S1	136.34±1.12 	40.17±1.03
S2	99.45±1.22 	40.79±1.04
S3	92.92±2.36 	45.11±0.83

3.3 Scratch test results

In the scratch test, the critical load (L_c) that caused complete penetration of the coating, with exposure of the steel substrate, was determined. The experimental values are listed in Table 9.

Table 9. Critical load determined in the scratch test

Samples Code	L_c (N)
S1	38±1 ÷ 40±1
S2	18±1 ÷ 20±1
S3	56 ±1 ÷ 58±1

As can be observed from the data in the table, sample S3 exhibits the most adherent and resistant coating, with the highest recorded L_c value. A weaker performance in the test was observed for sample S1, for which the L_c force showed an intermediate value. The least resistant coating was deposited on the

304L steel substrate; a possible explanation is the lowest roughness of the substrate, which did not allow for effective mechanical anchoring of the coating.

Figure 4 presents the scratch test diagrams recorded during the test.

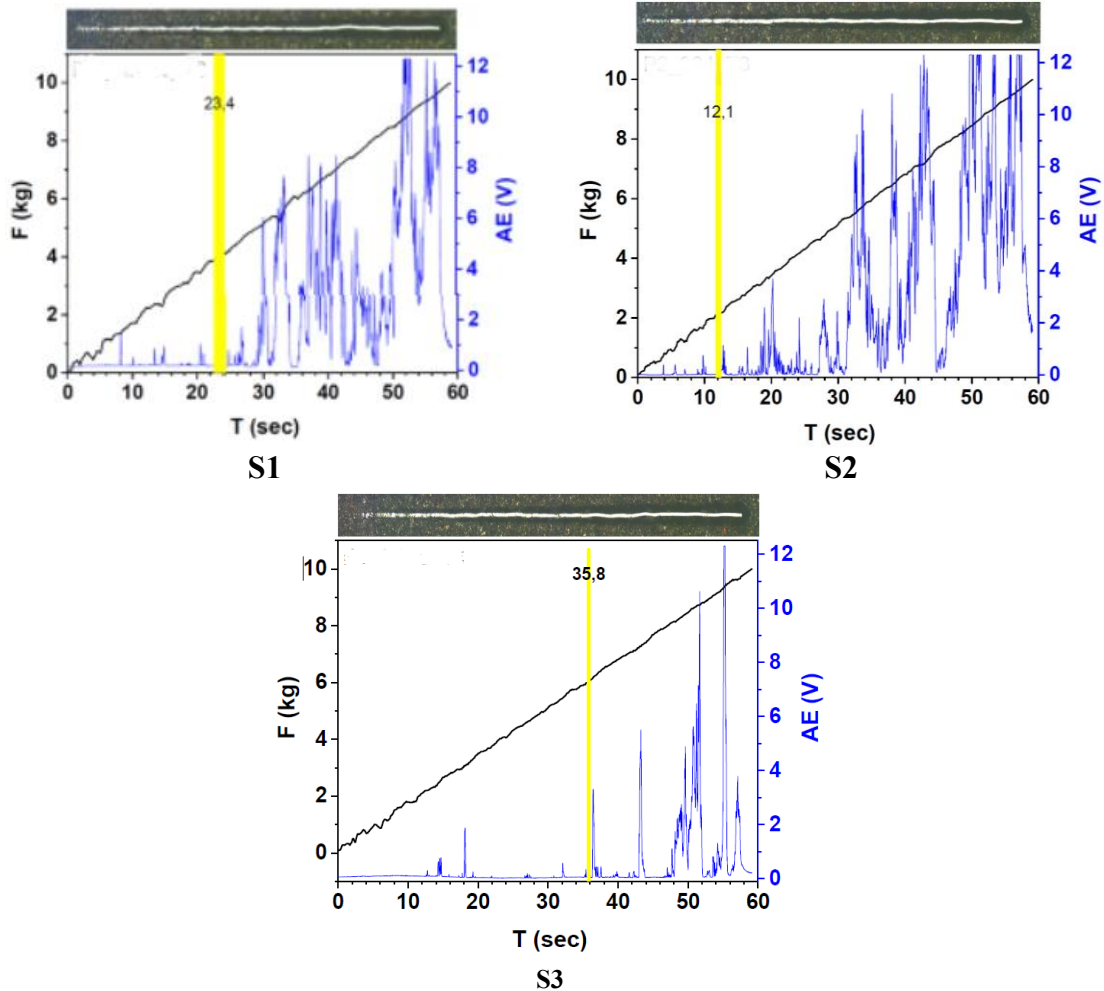


Figure 4. Scratch test diagrams for S1, S2 and S3 samples.

4. Conclusions

Following the correlation of roughness and wettability measurements with the optical microscopy analyses and scratch tests performed on samples S1, S2, and S3 (steel substrates S235, 304L, and 316L, respectively, with copper coatings), the following conclusions can be drawn:

1. Sample S1 exhibits the highest substrate roughness (R_a) and the highest surface free energy (65.8 mN/m), suggesting good adhesion of

the copper coating. However, the lowest coating roughness, combined with a positive surface profile skewness coefficient (R_{sk}) and the lowest surface free energy (40.17 mN/m), results in poor antibacterial behavior, despite the relatively high contact angle. The pronounced porosity at the interface is responsible for the intermediate value of the critical load determined in the scratch test (38–40 N).

2. Sample S2 is characterized by the lowest substrate roughness and intermediate wettability. The deposited copper layer shows the highest roughness which, together with the low surface free energy (40.79 mN/m) and a positive R_{sk} coefficient higher than that of sample S1, also results in reduced contact-killing capability. At the same time, sample S2 recorded the lowest critical load in the scratch test (18–20 N), due to insufficient anchoring of the coating, caused by the low substrate roughness.
3. Sample S3 presents a substrate with intermediate roughness and a slightly negative R_{sk} coefficient, very close to zero. These characteristics, along with lower wettability compared to the other samples (surface free energy of 56.46 mN/m), would have suggested a less adherent copper coating compared to S1. However, the nearly pore-free interface, together with the intermediate substrate roughness, ensured the best coating adhesion. The copper layer is characterized by the highest surface free energy (45.11 mN/m), which, correlated with the lowest contact angle, provides the most effective “contact killing” activity.

Author Contributions

Conceptualization, I.S.; I.A.; methodology, I.S., T.V.T., R.C. and D.A.N.; validation, I.A. and S.C.; investigation, I.S., R.C., D.A.N., T.V.T and R.E.D.; writing—original draft preparation, I.S., D.A.N. and R.E.D.; writing—review and editing, I.S. and D.A.N.; supervision, I.A., S.C.; All authors have read and agreed to the published version of the manuscript.

Data Availability Statement

The data presented in this study are openly available at [repository name] at [DOI or link].

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Conflicts of Interest

The authors declare no conflict of interest.

Artificial Intelligence Disclosure

No artificial intelligence-based or generative tools were used in the preparation of this manuscript.

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