

Article

The Preparation and Properties of Thick Bronze Coating Using Friction-Assisted Jet Electrodeposition

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Abstract: In this study, a novel friction-assisted jet electrodeposition technology was applied to prepare bronze coating on 40CrNiMoA structural steel surfaces. The bronze electrode was designed with internal flow channels and nozzles, and the friction brushes made of alumina and silicon carbide were connected to the electrode surface. It was reported that the quality and deposition rate of the thick bronze coatings were significantly improved with friction-assisted jet electrodeposition. The roughness and microstructures were refined, and the deposition rate was up to 100 $\mu\text{m}/\text{h}$ when the current density was 8 A/dm^2 . In addition, the chemical composition was related to current density as the content of tin in the bronze coating made with FJED decreased at a high current density. Moreover, the grain structures were $\alpha\text{-CuSn}$ in the solid-solution phase and the average grain size of FJED coatings was refined at a current density of 8 A/dm^2 .

Keywords: bronze coating; jet electrodeposition; friction assisted; combined processing



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1. Introduction

Bronze is a copper-tin alloy with 7 to 15 wt.% tin [1]. This material has good properties, like high hardness, wear, and corrosion resistance [2–4]. According to different properties, bronze is used for coins, pipes, valves, gears, etc. [5]. Thus, bronze coatings are widely applied to marine equipment, aerospace industry, and heavy machinery [6–8].

With the development of the aerospace industry, a higher requirement was proposed for the aviation engine plunger fuel pump [9]. Furthermore, key frictional pairs are vital parts in determining the performance of hydraulic pumps. Bronze coatings have been prepared on the frictional surfaces to improve wear resistance [10,11]. However, traditional processing methods cannot meet the requirements of high-speed fabricating thick bronze coatings [12]. Diffusion welding is a rapid additive preparation method, especially suitable for bonding between different metals. However, cracks, pores, and a low production rate limit the application of diffusion welding in the aerospace industry. Moreover, traditional electrodeposition is a widely used and long-standing coating manufacturing technology due to its simple structure and low cost. For the efficient preparation of inner hole coatings, traditional electrodeposition is clearly limited by the distribution of electric field lines.

Nowadays, thermal and kinetic spraying methods, electro-spark deposition, and electroplating are used to fabricate bronze coating on metal materials [13–15]. A few papers have also investigated the high-speed preparation of bronze coatings with jet electrodeposition. L.N. Bengoa et al. prepared bronze coatings with up to 22 wt.% tin on a cylindrical surface from PSA-based electrolyte using traditional electrodeposition (TED) but the current density was up to 3.75 A/dm^2 and the surface was rough while the thickness was only 50 μm [16]. A. A. Kasach et al. used improved pulsed electrodeposition to fabricate bronze coatings from sulfate electrolyte but the processing was also at a low current density of about 2 A/dm^2 with many pores on the surface [17]. R. Balaji et al.

doped PTFE particles in cyanide electrolyte and obtained a more wear-resistant surface [18]; however, the current density was still low at $1\sim5\text{ A/dm}^2$ with many copper nodules on the surface [19–21].

As a new electrodeposition method, jet electrodeposition has a high flow rate and can achieve high efficiency [22–24]. Moreover, in order to improve the properties of the deposited layer to meet higher requirements, the combination of jet electrodeposition and physical field has been paid more attention [25,26]. According to the application mode of the physical field, physics-assisted electrodeposition technology can be summarized into a non-contact external force field and a contact external force field [27]. The contact external force field has many advantages in enhancing compactness, removing impurities, creating a uniform electric field, and refining grains [28]. There have been many studies of friction-assisted electrodeposition (FJED) that show that it is feasible to prepare thick bronze coatings with high surface quality using FJED [29,30]. Therefore, FJED was used to prepare bronze coatings with 7–15 wt.% content of tin to improve the deposition rate.

In this study, the electrodes were re-designed and the frictional brushes were connected with the electrodes to remove impurities or hydrogen bubbles on the substrates. The standard cyanide electrolyte was used, and the high-quality thick bronze coatings were manufactured at a high current density. Thus, the electrodeposition time was deep cut. Scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), and X-ray diffraction (XRD) were applied to characterize and analyze the surface morphology, chemical composition, and grain structure. Results showed that FJED had excellent adaptability of the electrolyte and the comprehensive performance of coating manufacturing was improved. Overall, FJED provided a new way to fabricate high-quality thick alloy coating with high efficiency.

2. Materials and Methods

2.1. Pre-Preparation of Steel Substrates

40CrNiMoA structural steel was used as the substrate in this work. The composition of the structural steel is shown in Table 1. The diameter of the substrate was 65 mm, and the substrate surface roughness was machined to $Ra\ 0.8\ \mu\text{m}$ for a better binding strength of the coating. The pre-preparation was conducted before deposition to improve the bonding strength. As shown in Figure 1, the substrate was ultrasonically cleaned in a P-3 solution for 10 min. Then, the substrate was washed in deionized water and dried in a dryer. Subsequently, the steel disk was electrolyzed in sodium hydroxide solution for 5 min, and washed in deionized water. Finally, the substrate was activated in a dilute sulfuric acid solution, washed in deionized water, and dried for the next processing.

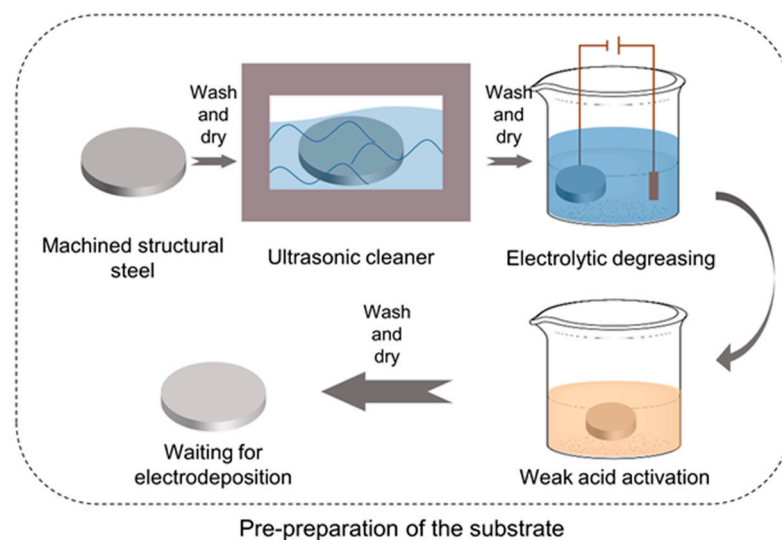


Figure 1. The diagram of the pre-preparation of the substrate.

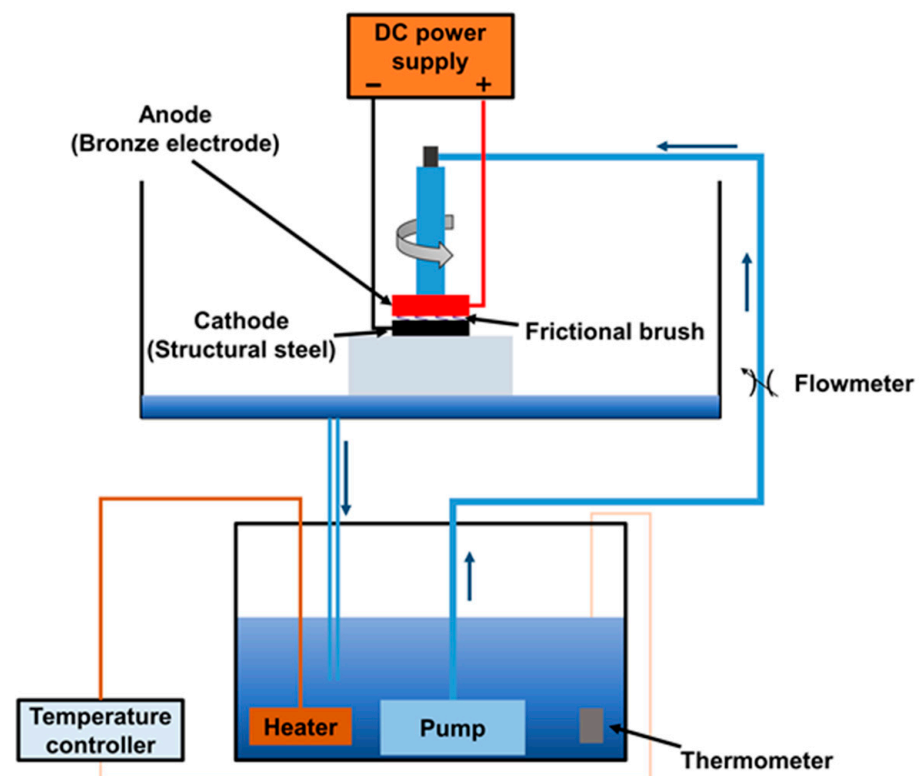
Table 1. The composition of the structural steel 40CrNiMoA.

C	Cr	Ni	Mo	Mn	Si	P	S
0.37–0.44	0.60–0.90	1.25–1.75	0.15–0.25	0.50–0.82	0.17–0.37	≤0.035	≤0.035

Unit: wt.%.

2.2. Friction-Assisted Jet Electrodeposition

The schematic diagram of FJED is shown in Figure 2. The electrolyte was stored in the solution tank, and heated using a controlled heater. Then, the electrolyte was transported and injected into the cathode surface with a submersible pump (LP-548, Zhejiang Jialu Aquatic Products Co., Ltd., Zhoushan, China). The flow rate was adjusted using a flowmeter. Finally, the electrolyte flowed back into the solution tank. The bronze anode and the 40CrNiMoA cathode were placed face to face with a 1 mm interval between the two electrodes. The anode with frictional brushes was rotated via a motor to rub against the cathode surface.

**Figure 2.** The schematic diagram of friction-assisted jet electrodeposition.

According to the classic theory of cathodic reduction, metal ions were reduced to metal atoms, and metal atoms were deposited on the cathode surface. Moreover, the electrolyte flow was accelerated by the jet electrodeposition. Thus, the concentration polarization was weakened, and the deposition rate was increased. In this work, the design of the electrode was changed using flow field simulation to improve the homogeneity of electrolyte flow on the processing surface. The frictional brush was connected with the anode surface to remove hydrogen bubbles and dendritic microstructures. The key processing parameters are listed in Table 2. In addition, a standard cyanide electrolyte was used in this work, and the main composition included copper ions, tin ions, KCN, KOH, and other additives.

Table 2. The key parameters of friction-assisted jet electrodeposition.

Parameters	Values
Current density	8, 10, 20 A/dm ²
Electrodes interval	1 mm
Electrolyte flow rate	150 L/h
Reynolds number	9000
Electrolyte temperature	60 ± 5 °C
Electrolyte pH	10 ± 0.1
Electrode rotation rate	4 r/min
Electrode position time	1, 5 h

2.3. Characterization

The field emission scanning electron microscope (FE-SEM, Zeiss Ultra Plus, Oberkochen, Germany) was applied to characterize the surface morphologies of the bronze coating. The chemical composition and distribution of the bronze coating were analyzed with energy dispersive spectroscopy (EDS, Oxford Instruments X-MAX, Abingdon, UK). The grain structures and grain sizes were characterized with X-ray diffraction (XRD) on the X-ray diffractometer (SmartLab 9 kW, Rigaku, Tokyo, Japan).

3. Results and Discussion

3.1. Surface Morphology

The surface morphologies of the bronze coatings fabricated with different parameters are shown in Figure 3. In Figure 3a,b, rough grain structures were shown on coatings fabricated using TED and jet electrodeposition (JED). Compared with the coating prepared with TED, more gaps were shown on the JED coating. The rate of deposition was improved by the higher electrolyte flow and current density but the roughness and porosity were increased by the high current density. Thus, the FJED was introduced to improve the coating qualities. As shown in Figure 3c–f, lower roughness and porosity were observed on the coatings prepared using FJED. When the current density was 10 or 20 A/dm², the interval between globular cells sharply decreased. However, a protrusion of approximately 200 µm formed on the FJED coating in Figure 3d. With the decrease in the current density, the coating surface morphology was significantly improved in Figure 3e. The globular cells were approximately 5 µm, and the interval between globular cells was invisible on the coating fabricated at 8 A/dm². The internal morphology of the machined coating is shown in Figure 3f. There were no cracks, pores, or defects inside the coating.

The morphologies of the coating cross-sections are shown in Figure 4. The coating thickness was further determined with the EDS mapping spectrum and line scanning spectra in Figure S1. In Figure 4a, the thickness of the bronze coating prepared with TED for 1 h was merely 15 µm. In Figure 4b–d, the thicknesses of the coating prepared using FJED were 350 µm, 80 µm, and 150 µm, respectively. Some small cracks and pores were detected on the internal side of the TED coating, while the deposition time was 1 h. The cross-sections of coatings prepared using FJED were smooth and compact, which implies that the frictional brushes remove the large grain cells and hydrogen bubbles during processing. The deposition rate of the processing could be approximately calculated according to thickness and deposition time, and the formula is shown in Equation (1):

$$R_d = T_c / t \quad (1)$$

where R_d is deposition rate, T_c is coating thickness, and t is deposition time, respectively. Thus, the deposition rates of the processing were 15 µm/h (TED at 1.5 A/dm²); 70 µm/h (FJED at 8 A/dm²); 80 µm/h (FJED at 10 A/dm²); 150 µm/h (FJED at 20 A/dm²). It was noted that the deposition rate of FJED at 8 A/dm² was a valid value because turning reduced the actual thickness of the coating. The typical deposition rate of the coating prepared using FJED at 8 A/dm² was 100 µm/h. The deposition rate was lower at the

beginning of the processing because the interval of the electrodes was relatively wide. The wider the interval of electrodes, the lower the deposition rate. However, the deposition rate of FJED at any current density was much higher than that of TED.

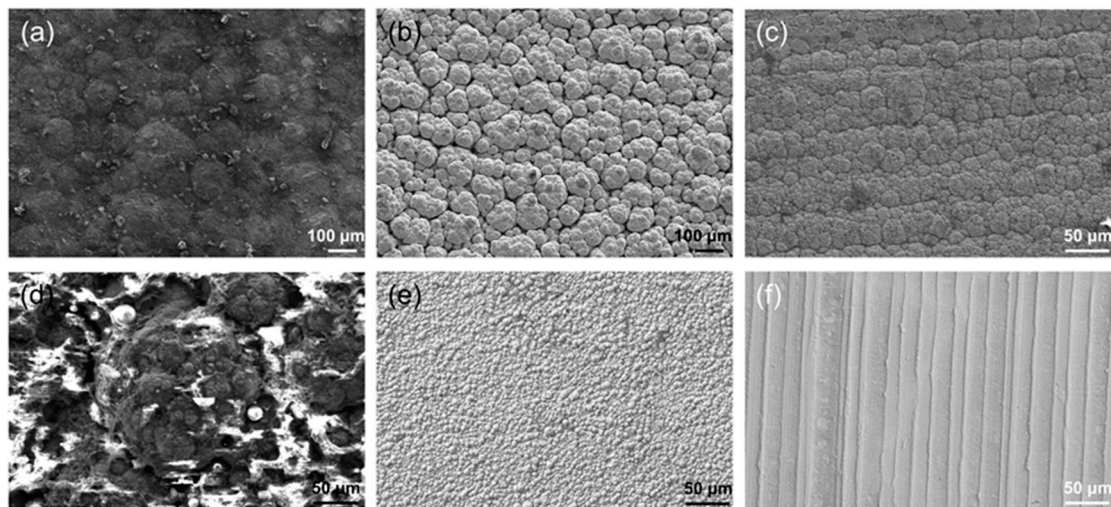


Figure 3. The surface morphologies of the bronze coatings prepared using (a) TED at 1.5 A/dm^2 , (b) JED at 10 A/dm^2 , (c) FJED at 10 A/dm^2 , (d) FJED at 20 A/dm^2 , (e) FJED at 8 A/dm^2 , and (f) FJED at 8 A/dm^2 with the post-processing of turning.

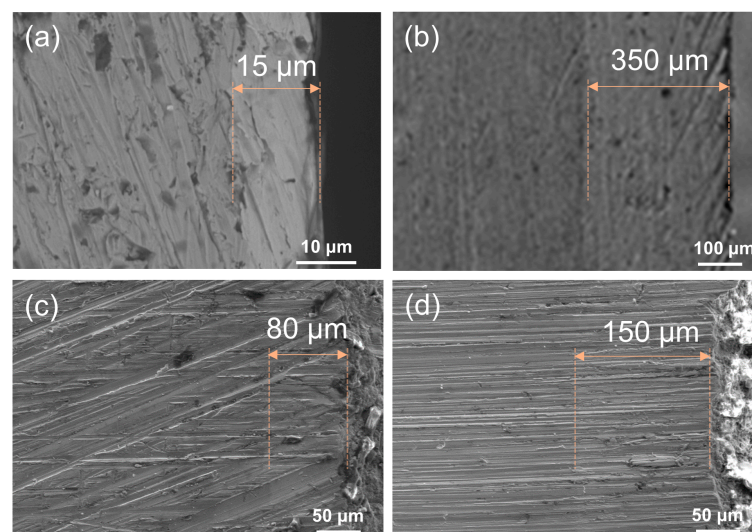


Figure 4. The cross-sectional morphologies of the coatings prepared using (a) TED at 1.5 A/dm^2 for 1 h; (b) FJED at 8 A/dm^2 for 5 h with the post-processing of turning; (c) FJED at 10 A/dm^2 for 1 h; (d) FJED at 20 A/dm^2 for 1 h.

3.2. Chemical Composition

Figure 5 shows the EDS spectra of the bronze coatings. The content of tin in the bronze coating vitally influenced the hardness and brittleness. In Figure 5a, 13.24 wt.% tin was detected on the bronze coating fabricated using TED at 1.5 A/dm^2 . As shown in Figure 5b,c, the concentrations of tin on the FJED bronze coatings were decreased to 10.30 wt.% and 11.26 wt.%, respectively. When the current density was increased to 20 A/dm^2 , it further weakened the deposition effect of tin on the substrate steel, and the concentration of tin was barely 6.30 wt.%.

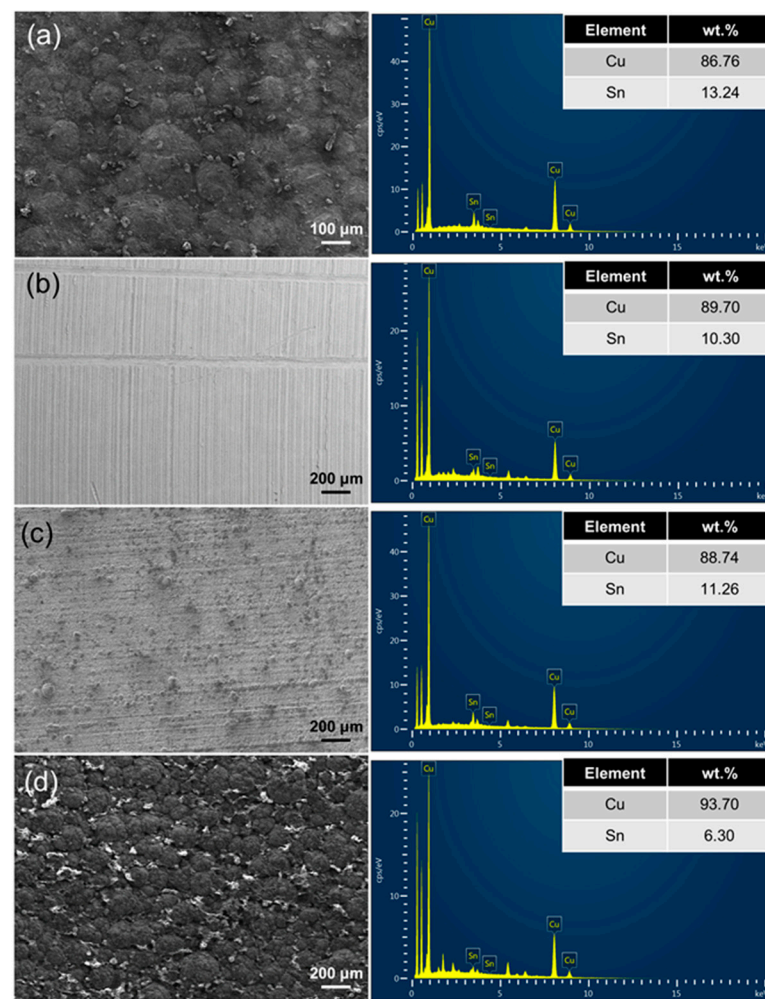


Figure 5. The EDS spectra of the coatings prepared using (a) TED at 1.5 A/dm^2 ; (b) FJED at 8 A/dm^2 with the post-processing of turning; (c) FJED at 10 A/dm^2 ; (d) FJED at 20 A/dm^2 .

It was noted that the chemical composition of the bronze coating prepared using electrodeposition was related to the current density. In the regular co-deposition, the metal with higher potential (copper in this work) was easier to deposit on the surface at a high current density, and the content of metal with higher potential was increased with the improvement of the metal ions in the electrolyte. Furthermore, the current density was improved to increase the content of the metal with lower potential (tin in this work) due to the local concentration polarization of the electrolyte. The potential of tin was -0.1375 V , and the potential of copper was 0.3419 V . However, the content of tin decreased when the current density was higher, as seen in Figure 5. This implies that the local concentration polarization did not occur during the process of FJED. When the current density was high, the copper ions were easier to be deposited on the cathode. In the process of TED, the concentration of the copper ions was sharply reduced at the high current density. Then, low-potential hydrogen and tin were deposited separately and formed detrimental hydrogen bubbles and tin atoms. Thus, it was difficult to achieve the efficient and high-quality preparation of low-tin bronze coatings with TED. Comparatively, the electrolyte was continuously injected on the cathode surface to replenish the copper ions in the process of FJED, and the copper atoms were further deposited. On the one hand, the deposition rate of copper did not decrease without concentration polarization at a high current density; on the other hand, the content of tin was increased by reducing the current density. Overall, the chemical composition of bronze coatings can be tuned with the electrolyte flow and current density.

The EDS mapping spectrum of the coating surfaces and cross-sections are shown in Figure 6. In Figure 6a,b, copper and tin were evenly distributed on the bronze coating surfaces. However, some dark areas were shown on the TED coating surface because defects existed in the concaves between grain cells. In addition, the FJED coating showed that the interior of the coating was completely filled with copper and tin. In Figure 6c, the cross-section of the TED coating showed that the interface between the coating and the steel substrate was not smooth. It was believed that the transition layer was formed at the interface. Copper and tin atoms were diffused into the iron lattice, and the iron lattice was distorted. Chen et al. reported that bronze coatings on bare steel surfaces accelerate the formation of infiltrated cracks from the bronze coating to the steel [31]. However, the interface between the bronze coating and steel substrate was sharp and smooth. There was no copper detected in the steel area, and no iron detected in the bronze area. In the distribution of tin, a part of the faint tin peaks was displayed in the steel area. That did not mean that tin infiltrated to the steel substrate. Due to the low content of tin in the bronze coating, the background peaks in the steel substrate were displayed in the tin mapping spectrum. Thus, infiltrated cracks were eliminated with FJED.

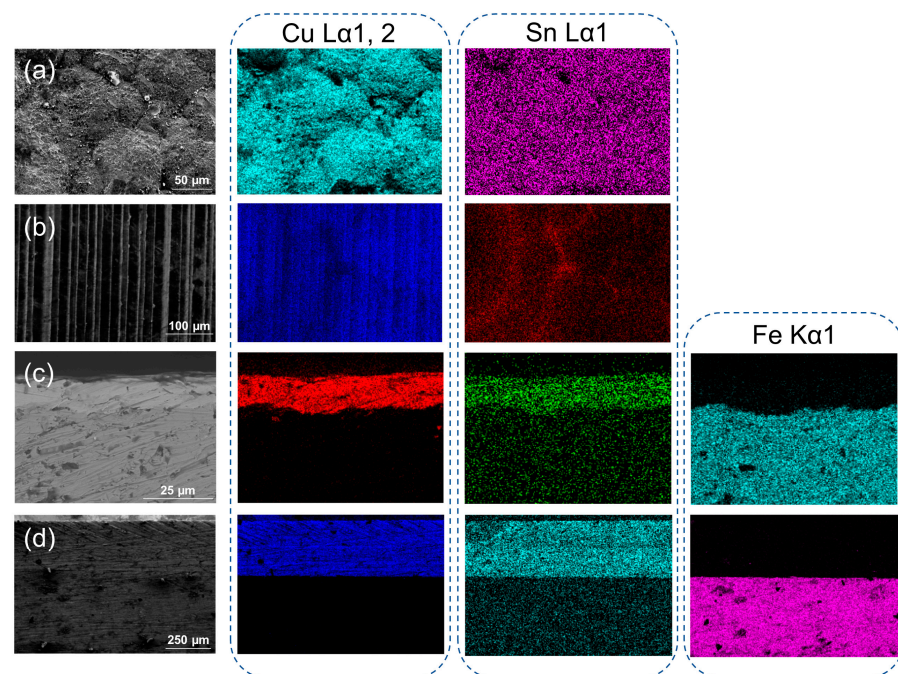


Figure 6. The EDS mapping spectrum of the coating surfaces and cross-sections prepared using (a,c) TED at 1.5 A/dm^2 for 1 h; (b,d) FJED at 8 A/dm^2 with the post-processing of turning.

3.3. Grain Structure

The XRD patterns of the bronze coatings prepared using TED and FJED are shown in Figure 7. The grain structures and grain sizes were characterized to compare the difference between TED and FJED. The iron element of the steel substrate was not detected with XRD because the coating was too thick to be penetrated. The main chemical phase was the α -CuSn solid-solution phase in two coatings [32]. The crystal planes were (1 1 1), (2 0 0), (2 2 0), and (3 1 1). The 2θ angles of diffraction peaks were 42.8° , 49.9° , 73.1° , and 88.7° , respectively. Thus, the main phase was not affected by the different processing or current density. However, the crystal plane (2 0 0) sharply decreased on the FJED coatings. This implied that the main crystal plane was transferred to (1 1 1) on the FJED coatings, and the crystal plane (2 0 0) was suppressed when using FJED processing.

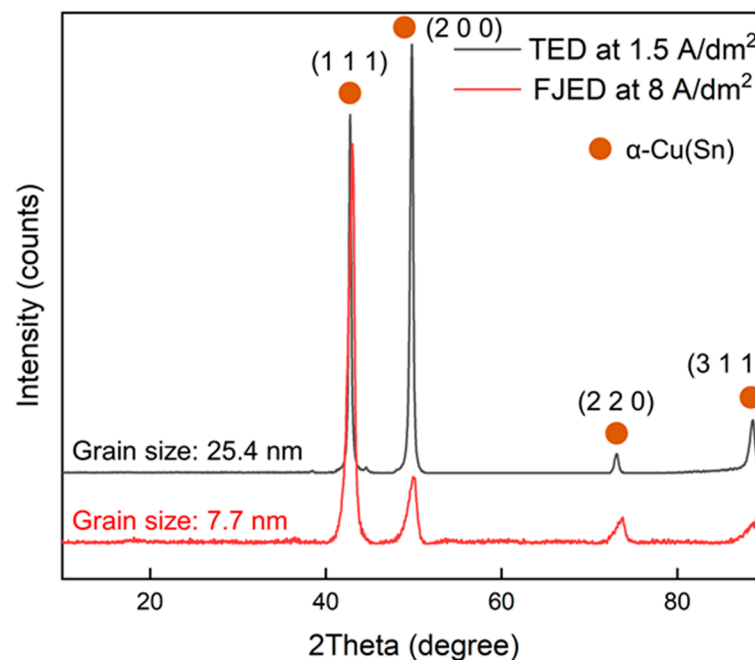


Figure 7. XRD patterns and grain sizes of the coatings prepared with TED and FJED.

Additionally, every diffraction peak of the FJED coating was broadened compared with the TED coating. It was reported that the grain refinement can be reflected by diffraction peak broadening [33]. The broadening of diffraction peaks was generally accompanied by a decrease in intensity. The smaller the grain size in the sample, the lower the intensity of the diffraction peak but the peak becomes wider. The average grain sizes of the two coatings were analyzed and calculated with XRD analysis software (MDI Jade 6.5) and Debye–Scherrer’s formula, as shown in Figure 7. The average grain sizes were both less than 100 nm; thus, implying that the nanocrystalline bronze coating was prepared on the steel substrate. However, the grain size of the FJED coating was less than that of the TED coating. It was believed that high current density can promote grain refinement. When the current density was increased, the overpotential was increased on the cathode, resulting in an increase in the nucleation rate and a slowdown in grain growth. It also corroborated that the surface morphology of the FJED coating was refined with an appropriate increase in current density.

3.4. Mechanism of FJED

The mechanism of FJED is shown in Figure 8. The electrolyte was injected on the cathode surface through the internal flow channel and anode nozzles. The flexible frictional brushes were driven by the anode to rotate and rub against the cathode surface. When the current density increases, the metal ions in the electrolyte are rapidly consumed, and a small amount of hydrogen ions in the solution will be reduced to hydrogen by the electric field and attach to the cathode surface. In traditional electrodeposition processes, hydrogen bubbles attached to the cathode surface cannot detach and are covered by a new layer, resulting in porosity defects inside or on the surface of the coating. Because the current density of FJED was higher than that of TED, hydrogen bubbles and irregular grain cells were easily formed on the cathode surface. In addition, the impurities were distributed in the electrolyte and contaminated the cathode surface. TED was unable to increase the current density due to the formation of these by-products. During the process of FJED, hydrogen bubbles, irregular grain cells, and impurities were also generated but flexible frictional brushes removed these by-products. Then, these impurities were taken away by the high-speed electrolyte. Compared to JED, the high-speed electrolyte not only

replenished metal ions but also took away impurities scraped off by the brushes. Finally, the cathode surface was constantly deposited and refreshed.

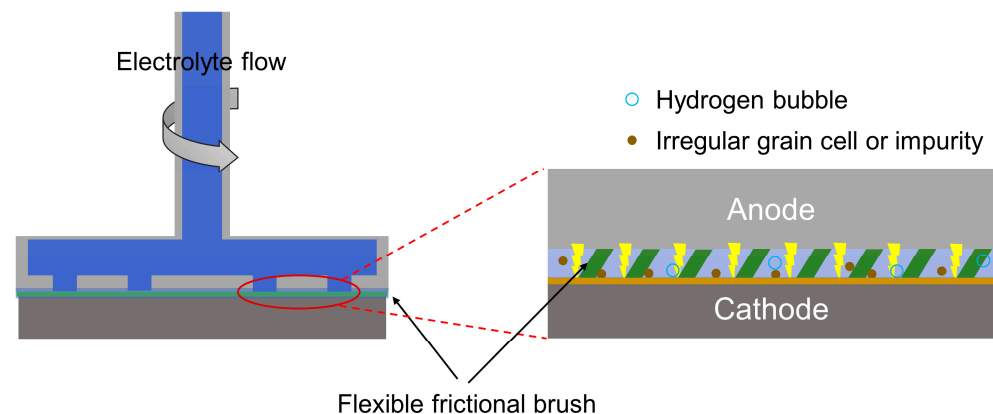


Figure 8. The schematic diagram of the mechanism of FJED.

4. Conclusions

In this study, it was feasible to manufacture high-quality bronze coatings using friction-assisted jet electrodeposition at the deposition rate of 100 $\mu\text{m}/\text{h}$, and the composition of tin was controlled at approximately 10 wt.%. The friction brushes made of alumina and silicon carbide can reduce the surface defects of the coating caused by cathodic hydrogen evolution and remove impurities on the cathode surface. The high-quality low-tin bronze coatings, with a thickness of 500 μm , were efficiently prepared at the deposition rate of 100 $\mu\text{m}/\text{h}$. The surface morphology, chemical composition, and grain structure were characterized by the prepared bronze coatings. The main conclusions are as follows:

1. The quality and deposition rate of thick bronze coatings were improved by FJED. When the current density was 8 A/dm^2 , the roughness and microstructures on the coatings were the best among the four sets of the samples (TED coating prepared at 1.5 A/dm^2 ; FJED coatings prepared at 8, 10, and 12 A/dm^2), and the deposition rate of FJED was up to 100 $\mu\text{m}/\text{h}$, which was approximately 6 times that of TED;
2. The chemical composition of the bronze coating was related to current density. As the current density increased, the tin content gradually decreased. Due to the rapid replenishment of metal ions by the high-speed electrolyte, the content of tin in the coating prepared using FJED at 12 A/dm^2 decreased to 6.3 wt.%. In addition, the distribution of copper and tin on TED and FJED coatings was uniform. However, infiltrated cracks detected on the TED coating were eliminated by FJED;
3. The grain structures of TED and FJED bronze coatings were $\alpha\text{-CuSn}$ in the solid-solution phase. The average grain size of FJED coatings was refined to 7.7 nm at the current density of 8 A/dm^2 .

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/coatings14010150/s1>, Figure S1: (a) The EDS mapping spectrum of the coating cross-section prepared using TED at 1.5 A/dm^2 ; the EDS line scanning spectra of coatings prepared using FJED at (b) 8 A/dm^2 , (c) 10 A/dm^2 , (d) 20 A/dm^2 , respectively.

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