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Electropolishing parameters optimization for enhanced performance of nickel coating electroplated on mild steel☆,☆☆

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ABSTRACT

The purpose of this work is to investigate the influence of electropolishing process on performance parameters of nickel coating electroplating on Q235A mild steel as substrate, including the surface roughness, thickness, elastic modulus and hardness. The contribution of magnetic stirring was first studied. On the basis of this, we explored the effect rules of electropolishing parameters on nickel coating using orthogonal test. The result showed that nickel coating became smoother and thicker by magnetic stirring in electropolishing process. The current density in electropolishing process significantly influenced the thickness, elastic modulus and hardness of Ni coating, while it showed little influence on the coating surface roughness. The above three parameters were also affected by polishing time and the temperature of polishing slurry. With the other condition remaining the same, elastic modulus and hardness of nickel coating reached their peaks after electropolishing with a current density of 30 A/dm^2 in electrolyte at 65 °C for 15 min.

1. Introduction

Q235A mild steel is well known for its good performance and balanced properties of strength, plasticity and weldability. It is widely used in fabrication of mechanical components and abrasive tools in less strict situation. However, the steel is susceptible to pitting corrosion in natural environment because of its low abrasive resistance and chemical stability. Compared to the steel, nickel has higher chemical stability, drawability and plasticity, which makes it much harder and more resistant to abrasion and corrosion as coating material. Notice that the standard electrode potential of nickel is −0.25 V, which is slightly higher than that of iron, −0.44 V [1], and the standard electrode potential of nickel can be even higher after surface passivation. When exposed in air, especially in humid environment, the surface of nickel layer will be oxidized gradually and form a relatively dense oxide layer to prevent the oxidation process from going further. The film makes nickel quite often to be used as wear resistant or anticorrosive material for applications such as surgical instruments plating, surface dressing in printing industry, electroform of plastic abrasive tools and even battery shells.

The shells of traditional rechargeable batteries or alkaline batteries are made of nickel-coated mild steel for the purpose of high corrosion resistance [2]. In order to obtain the production such as battery shells, the most advanced approach nowadays can be divided into two steps. First, pure nickel is plated onto the surface of mild steel. After that, deep drawing is directly employed to nickel-coated steel to obtain the production shells. The advantages of this process are high output ratio and low cost, but the coated layer must be of extremely high integrity. Unfortunately, defects including but not limited to cracks, gauffer or exfoliation are always inevitable in deep drawing process. Ideal nickel coating should be of high strength, toughness, hardness and corrosion resistance to produce further improvement on the antiwear and anticorrosion property of the surface of base material. The performance of Ni coating is affected by several experimental factors, such as pretreatment to substrate [3], the composition of electrolyte [4,5], current density [6–9], contact pressure [10,11], temperature [12–14], pH value [15], stirring process [16] addictive and so on [17–22].

The pretreatment process to substrate includes cleaning, oil removal and surface polishing. The most common approaches used to polish the surface of substrate are electropolishing, chemical polishing and mechanical polishing. Electropolishing is an anodic dissolution phenomenon using an electrochemical reaction to remove electrode metal. Without cutting force and cutting heat, electropolishing process will not cause plastic deformation or leave residual stress on the surface of workpiece like those caused by traditional mechanical polishing process. The surface roughness is dependent on the local charge densities

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and the mass transfer mechanism [23]. As a result, the surface roughness lasts longer after electropolishing than mechanical polishing. Besides, electropolishing is an isotropic process, which means that the variation of polishing quality from one direction to another is almost negligible. This brings a huge difference from mechanical polishing process in terms of surface roughness and geometry. The aim of this work is to analyze the influence law of electropolishing process on the surface roughness, thickness, elastic modulus and hardness of nickel coating plated later. The result provides technical support for the preparation of nickel coating with high intensity, hardness and corrosion resistance.

2. Experiment

2.1. Sample preparation and process flow

As shown in Fig. 1, 60 mm × 10 mm × 3 mm Q235A mild steel blocks were used as sample substrates. The main chemical constituent of Q235A mild steel was 0.14–0.22% C, 0.30–0.65% Mn, ≤0.50% S, ≤0.45% P and ≤0.30% Si and so on. For each sample substrate, coated area was set to be 20 mm × 10 mm with the rest part sealed by insulating material. The 10 mm × 5 mm test area was confined to the shadow part in Fig. 1a in the middle of coating layer. Process flow: distilled water rinsing–electropolishing–distilled water rinsing–chemical deoiling–distilled water rinsing–electrochemical deoiling–distilled water rinsing–nickel electroplating–distilled water rinsing–drying.

2.2. Formula and process

2.2.1. The solution formula and process of electropolishing

The electropolishing solution is prepared with H_2SO_4 (15 mL), H_3PO_4 (70 mL), CrO_3 (6 g) and H_2O (14 mL). Two experimental schemes were available here. One was to compare the difference of the performance between coating layers employed magnetic stirring or not in electropolishing process. The other one was to explore the influence of electropolishing on the final coating layer when magnetic stirring was employed.

2.2.2. The solution formula and process of deoiling

In this study, chemical deoiling was first adopted, followed by electrochemical deoiling. Chemical deoiling is based on the principle that hot potash solution can dissolve oil stain at the surface of workpiece because of saponification and emulsification. In this test, the samples were immersed in the solution at 70 °C for 2 min. After that, electrochemical deoiling was performed in electrolyte when Q235A (−) was electrically charged with a platinum net (+). The current density was 0.2 A, the electrolyte temperature was 70 °C and the deoiling time was 1 min. The chemical composition and content of the solution are given in Table 1.

A simple electrochemical deoiling apparatus is illustrated in Fig. 2. The electrochemically degreasing equipment was mainly assembled by the Smart Temperature Control Magnetic Agitator, the model number of DF-101S, and stabilized DC power supply, the type of WYK-30V3A2A1A.

Table 1
The chemical composition and content of oil removal solution.

Compositions	Chemical deoiling	Electrochemical deoiling
NaOH	65 g/L	15 g/L
Na_2CO_3	17.5 g/L	55 g/L
$Na_3PO_4 \cdot 12H_2O$	17.5 g/L	35 g/L
$Na_2SiO_3 \cdot 9H_2O$	5 g/L	7.5 g/L

2.2.3. The solution formula and process of electroplating

The electroplating solution was made up of analytical reagent that provided by Tianjin Guangfu Fine Chemicals Research Center, which was mixed with $NiSO_4 \cdot 6H_2O$, 240 g/L, $NiCl_2 \cdot 6H_2O$, 20 g/L and H_3BO_3, 20 g/L. In our test, the cathode current density was 1–3 A/dm^2, the pH value was between 3.5 and 5 at a rotation rate of 100 rpm at 50 °C for 70 min. Fig. 3 shows the schematic diagram of electroplating set-up.

2.3. Characterization method

Test equipments were mainly provided by Harbin Measuring & Cutting Tool Group Co., Ltd. The thickness of coating layer was measured by Digital Outside Diameter Micrometer with a resolution of 1 μm, and surface roughness parameter *R*a was tested using 2302A Synthesis Measuring Profilometer made in China. By comparing with surface roughness standard specimen, the *R*a of nickel coating was roughly estimated to be in the range of 0.1–0.2 μm. Based on that, the sampling length, evaluating length and probe moving speed were set to be 0.8 mm, 4 mm and 0.2 mm/s, respectively. The elastic modulus and hardness of coating layer were measured by Nano Indenter XP made in USA. Diamond Berkovich indenter was used in the test, and both of the loading and uploading velocity were 40 nm/s. The indentation depth was 2.5 μm. Pressure was continuously loaded at this depth for 10 s. Measurements of the four parameters were confined to the same region as shown in Fig. 1a. Ten measurements were conducted to obtain an average value for each parameter of each sample. This value was marked as the test result for each sample. After averaging all of these values over eight samples, the final result was obtained. A Japanese-made Microscope OLYMPUS STM6-F10-3 was applied to observe the surface profile of sample before and after electropolishing as well as that of nickel coating.

3. Results and discussion

3.1. Influence of magnetic stirring in electropolishing process on the performance of coating layer

In this test, the temperature of electropolishing solution was 60 °C since electrolyte at 50 to 70 °C leads to the best performance according to literature [24–26]. We analyzed the influence law of current density and polishing time on the thickness and surface roughness parameter *R*a of nickel coating under two conditions, with or without magnetic stirring. Samples were randomly divided into four experimental groups, with two in each of them. Nickel coating was coated on the surface of samples as mentioned in process flow. The sample number and technological parameters of electropolishing are shown in Table 2.

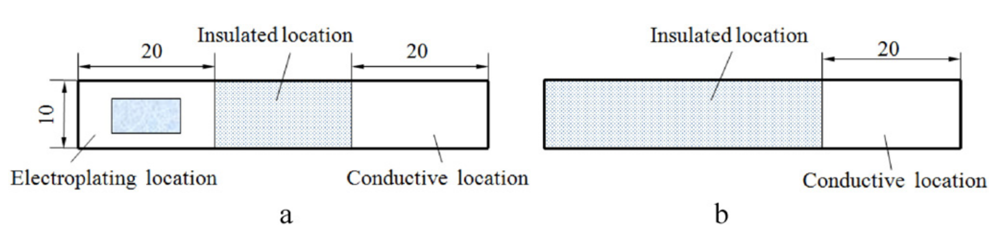

Fig. 1. The schematic diagram of matrix size of the substrate and sealing, plating and conductive location a) front view and b) back face view.