Influence of ultrasonic in low thermal expansion Fe-Ni electrodeposition process for micro-electroforming

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ABSTRACT

The electrochemical mechanism of Fe-Ni electrodeposition under ultrasonic was investigated by electrochemistry methods. Linear scanning voltammetry and cyclic voltammetry were used to show that the deposition process changed from the diffusion control under static conditions to an electrochemical control under ultrasonic conditions. Chronoamperometry curves showed that the Fe-Ni deposit occurred by a mechanism that instantaneous nucleation is followed by three-dimensional growth under charge transfer control. Chronopotentiogram indicated that because of the intensity of the ultrasound stripping effect, high ultrasonic power is unsuitable for electroforming Fe-Ni alloy, and a high current density is also not appropriate. Thus, the optimum parameters for Fe-Ni electrodeposition under ultrasonic conditions are ultrasonic power between 80 and 100 W (power density 0.28–0.35 W/cm²), and a current density lower than 10 mA/cm² with temperature 323 K and pH 3. Experiments were performed to verify that the Fe-Ni masks prepared by ultrasonic-assisted electroforming had a good surface quality. The increase in ultrasonic power can obtain a larger grain size, thus got a low thermal expansion coefficient and a high hardness. Therefore, ultrasonic-assisted electrodeposition technology provides an effective and practically feasible manufacturing method for OLED Fe-Ni mask preparation.

1. Introduction

Fe-Ni alloys have been used in industrial applications for more than a hundred years due to their soft magnetic properties and low coefficient of thermal expansion (CTE) [1,2]. Industrial applications such as integrated circuit (IC) packages, glass seals, organic light-emitting diode (OLED) shadow masks, composite material-forming molds, etc. are typical applications because of their low degree of thermal expansion, while applications such as read-write head magnetic memory, magnetic actuators, magnetic headers for hard disks, and in magnetic shielding are based on their soft magnetic properties [1–4]. Electroforming technology is an important process for extending the wide application of Fe-Ni alloys, such as the complex surface electroforming of Fe-Ni molds, the electroplating of Fe-Ni pins, the electroforming of Fe-Ni packages in electronic packaging, and the UV-LIGA electrodeposition of OLED Fe-Ni mask [5]. However, studies have reported that Fe-Ni alloy prepared by electroforming technology often companies with a poor surface quality (hydrogen pores and nodules), and possess a higher CTE than that prepared by the traditional metallurgical methods [6,7]. These are not conducive to the application of Fe-Ni alloy. Therefore, it is necessary to find a good auxiliary method for electrodeposition of iron-nickel alloys.

Ultrasonic-assisted electrodeposition has been experimentally demonstrated to affect the surface quality, grain size, and mechanical properties of electrodeposited metals such as nickel, silver, and tin [8–10]. Based on these, ultrasound was introduced in the electrodeposition process of Fe-Ni alloy in this study to explore electroforming OLED masks with required properties, such as good surface quality, low thermal expansion coefficient, etc. The effect of ultrasound on the electrodeposition process of Fe-Ni alloy was investigated from the perspective of microscopic electrochemistry. The results of the electrochemical experiments provided the optimum parameters for Fe-Ni electrodeposition and then an OLED Fe-Ni alloy mask with required properties was electroformed successfully.

2. Experimental details

2.1. Mechanism of ultrasound-assisted electrodeposition

Fe-Ni electrodeposition is a typical anomalous co-deposition, with the Fe²⁺ ions being preferentially adsorbed on the cathode surface and
the metal anions are reduced to the atomic state [11]. The process of Fe-Ni co-deposition was assumed to occur via the following reactions (M stand for Fe$^{2+}$ and Ni$^{2+}$):

$$M^{2+} + OH^- = MOH^{-} + e^- = MOH_{ad}^{-} + e^- = M + OH^-$$

(1)

$$2H^+ + 2e^- = H_2$$

(2)

As shown in Fig. 1a), the hydrogen made by hydrogen evolution reaction on the cathode will adhere to the cathode surface until it grows up and leaves. And that may cause the formation of pores on the electrodeposited surface. In addition, as the reaction progresses, there will be a delayed transmission of metal ions from the body of the liquid to the cathode surface and this will induce the increase of the diffusion layer thickness $\delta$.

As shown in Fig. 1b), the introduction of ultrasound can produce an ultrasonic cavitation effect. It can cause a certain pressure difference in the liquid, and the pressure difference will promote the rapid precipitation and explosion of hydrogen on the surface of the cathode.
Therefore, it can effectively solve the pore defects on the electrodeposited surface. Furthermore, the explosion of cavitation bubbles which are produced by ultrasonic caviation can form the jets of liquid with a high velocity leading to mass-transport enhancement [12]. This provides a guarantee for the continuity of the electrochemical reaction. Eventually, ultrasonic caviation decreases the thickness of the diffusion layer δ, which has an impact on the electrochemical process.

2.2. Electrodeposition experiments

An electrochemical workstation (Zahner Instruments Inc., Germany) with a three-electrode system was used to make the electrochemical measurements. As shown in Fig. 2, the counter electrode was a platinum plate with a surface area of 4 cm². A saturated calomel electrode (SCE) was used as the reference electrode. The working electrode was a square block of SS304 stainless steel with an effective area of 1 cm² whose other faces were insulated with polytetrafluoroethylene. Before each measurement, the surface of the working electrode went through fine pre-treatments, including polishing and rinsing with absolute ethanol and distilled water. The electrolyte was composed of 0.06 mol of NiSO₄·6H₂O, 0.03 mol of FeSO₄·7H₂O, sodium saccharin (1 g/L), and ascorbic acid (0.8 g/L) at a pH of 2–5 adjusted with sulfuric acid (H₂SO₄) or sodium hydroxide (NaOH). The solution was prepared with distilled water and analytically pure reagents. The measurements were conducted in an ultrasonic cleaning bath (Ningbo Shuangli Instruments Company, China) at 303–333 K (30–60 °C), with an ultrasonic power rating of 0–120 W with the frequency of 40 kHz.

3. Results and analysis

3.1. Effects of different parameters on the polarization curve

Linear scanning voltammetry (LSV) was performed at pH 3, 323 K, ultrasonic power of 0 W, 20 W, 40 W, 60 W, 80 W, 100 W, and 120 W (power density 0 W/cm², 0.07 W/cm², 0.14 W/cm², 0.21 W/cm², 0.28 W/cm², 0.35 W/cm², 0.42 W/cm² respectively), where 0 W represents static conditions. The scan rate was 20 mV/s⁻¹.

As shown in Fig. 3, under static conditions (0 W), when the potential is increasing to –600 mV, the current begins to increase, reach a peak value at –780 mV, and subsequently decline. The partially enlarged drawing Fig. 3b illustrates that it is only a small increase in the total reaction current. The results of component testing proved that the reduction reaction of metal cations did not occur on the electrode surface within the potential range from 0 to –1.1 V. This was due to the noble precipitation potential of H⁺ ions compared to the metal ion Fe²⁺ and Ni²⁺ so that hydrogen evolution occurred preferentially with increasing electrochemical potential. However, the local pH value near the surface of the cathode soared because of hydrogen evolution, and that favored the formation of hydroxide species FeOH⁺ and NiOH⁺. The hydroxide species might reduce the number of hydrogen reduction sites and result in an overpotential for hydrogen evolution. As the cathode surface was mainly covered by the hydroxide species, hydrogen evolution was retarded, and anomalous co-deposition of Fe-Ni was initiated at –1.1 V, as shown in Fig. 3.

As the co-deposition proceeded, the ions on the cathode surface were gradually consumed. Fig. 3c shows that, as the potential was scanned to –1.6 V, due to the delay in the mass transfer (as shown in Fig. 1a), the reaction current reached the cathodic peak current i_p. Subsequently, the reaction current increased as a result of water electrolysis when the potential was scanned to –1.8 V. The cathodic peak current i_p can be expressed as [13]:

\[
i_p = \frac{zFDCE}{\delta}
\]

where z is the number of electrons gained and lost in the electrode reaction, F is the Faraday constant, C₀ is the total concentration of reactants and δ is the thickness of the diffusion layer. The cathodic polarization curves of the Fe-Ni electrodeposition obtained at different scan rates are shown in Fig. 4. When the scan rate increased from 20 to 100 mV/s⁻¹, the potential of the cathodic peak i_p gradually shifted in the negative direction. This denotes that the electrode reaction is irreversible. The relationship between i_p and the square root of the scan rate u¹/² is linear, as shown in the inset to Fig. 4. This means that Fe-Ni electrodeposition is diffusion-controlled in the static conditions, and the anomalous co-depositions are suppressed. Perhaps due to the effect of the nucleation and growth processes, the fitted line did not pass through the origin of the coordinate system.

As presented in Fig. 3a, the reaction current obtained under ultrasonic conditions was higher than that obtained under static conditions within the same range of scanning potential from –400 mV to –2.1 V. With increasing ultrasonic power from 0 to 80 W, the start potential of Fe-Ni co-deposition shifted from –1.1 V to –1 V. This indicates that the reduction reaction of iron and nickel ions was promoted in ultrasonic conditions. The promotion was also reflected in the slowing of the decrease in current at –1.6 V, and the current finally stabilized (straight line section). As described in Fig. 1b, ultrasonic caviation promoted the diffusion of ions in the solution and reduced the ion concentration changes which is caused by insufficient diffusion. Hence, the Fe-Ni co-deposition process changed from the diffusion control under static conditions to an electrochemical control under ultrasonic conditions with the scanning potential range.

Due to the effect of depolarization of ultrasound, the overpotential η under ultrasound is smaller than that under static conditions and decreases with the increasing of ultrasonic power. Eqs. (4)–(6) describe the relationship between the nucleation probability w, the nucleation formation energy A, and the polarized overpotential η [10].

\[
A = \frac{16F^2V^2}{3\varepsilon^2F^2\eta^4}
\]

\[
zF\eta = \frac{2eV}{h_i}
\]

\[
w = B\exp\left(-\frac{h_i}{\eta}\right)
\]

With decreasing polarized overpotential η, the nucleation formation energy A and the critical nucleation radius h_i both increase, and the nucleation probability w decreases. The effect of ultrasonic depolarization increases both the grain size and the probability of crystal growth and reduces the nucleation rate. In addition, the stripping effect generated by ultrasonic power can peel the newly formed nuclei with
potential nuclei, and increase the grain size. Thus, the grain size of the Fe-Ni mask poor adhesion to the electrode surface, promote the growth of existing nuclei, and increase the grain size. Thus, the grain size of the Fe-Ni mask is increasing with the increase of ultrasonic power.

Nevertheless, it is important to mention that the polarization overpotential \( \eta \) calculate from Fig. 3 was \( \eta_{40W} < \eta_{20W} < \eta_{60W} < \eta_{120W} < \eta_{200W} \). This is because an ultrasonic power greater than 80 W is thought to have strong ultrasonic stripping effects [14–15]. The strong ultrasonic cavitation hinders the adsorption of metal cations on the surface of the cathode, which will increase the polarization over-potential. Therefore, factor the statement above into considering, an ultrasonic power between 80 and 100 W (0.28–0.35 W/cm²) is better suited to Fe-Ni alloy electrodeposition.

Fig. 5 was the LSV curves obtained at pH 3, an ultrasonic power of 80 W, and temperatures of 303, 313, 323, and 333 K at a scan rate of 20 mV·s⁻¹. At temperature 303 K, the current increased slightly and the co-deposition of Fe-Ni began at ~ 1.1 V. When the temperature increased from 303 to 333 K, the start potential of Fe-Ni deposition shifted in the positive direction, and the reaction current gradually increased. This is because the speed of ions mass transfer was low due to the lack of movement power of ions at a low temperature of 303 K. With the increase of temperature, the activity of ions increases, and this increased the ability of metal cations to capture electrons. Hence, a high temperature is beneficial to co-deposition. However, taking into account that iron ions are easily oxidized at high temperatures, 323 K should be used for the electrodeposition process.

Fig. 6 shows the LSV curves obtained at a scan rate of 20 mV·s⁻¹, an ultrasonic power of 80 W, a temperature of 323 K, at pH 2, 3, 4, and 5. At pH 2, the high concentration of H⁺ ions promoted hydrogen evolution, which is beginning at the potential ~ 0.5 V and accompanied a large reaction current. With an increase in the pH from 2 to 5, the initial potential of hydrogen evolution moved to a more negative value of ~ 0.7 V. Eqs. (1) and (2) indicates that the existence of OH⁻ in the solution is important for the reduction reactions of Fe²⁺ and Ni²⁺ [11]. As the pH increased, the concentration of OH⁻ is increasing. Therefore, the start potential of co-deposition changed from ~ 1.4 V at pH 2 to ~ 1.05 V at pH 5. Nevertheless, studies have proved that the mechanical properties of the deposited layer will decrease due to the formation of a Fe(OH)₃ precipitate from the oxidation of Fe²⁺ under conditions of high pH [16]. Therefore, pH 3 is optimal for comprehensive consideration.

3.2. Cyclic voltammetry study

The cyclic voltammograms (CV) obtained at 323 K, pH 3, an ultrasonic power of 0, 20, 40, 60, and 80 W, and a scan rate of 20 mV·s⁻¹ are presented in Fig. 7.

Under static conditions, the voltammogram obtained by the negative sweep voltage is similar to the polarization curve in Fig. 2. Then, when the negative scanning reaches ~ 2.1 V, the back scan begins. It is worth mentioning that the back scanning line intersects the front scanning line at a point with potential \( E_c \) (~ 1.14 V). The crosspoint implied that Fe-Ni alloy electrodeposition occurred more easily on itself than on the SS304. After that, with the potential scanned to ~ 745 mV, the Fe-Ni deposited on the cathode began dissolving and an anode peak appeared at ~ 150 mV.

With the ultrasonic power increased from 0 to 20 W, the potential required for co-deposition moved in the positive direction. The quantity of charge transferred increased steadily and the cathode peak disappeared. Both of these observations are consistent with ultrasonic streaming increasing the rate of transfer of metal ions to the surface. On the back scan, the initial potential of dissolution, as well as the position of the anodic peak both shifted to a positive value, and the anodic peak current increased. This indicated that more alloys were deposited and the deposited layer became denser. With increasing of ultrasonic power from 20 to 80 W, the move of the initial potential of dissolution was not obvious. It signified that the Fe-Ni co-deposition process was an electrochemical control under ultrasonic conditions.

3.3. Nucleation and growth of Fe-Ni

A potentiostat technique was used to further study the electrocrystallization process and nucleation model of Fe-Ni alloy on SS304.
stainless steel plate. Analysis of the data presented in Fig. 7 indicated that, at a potential of 0 V, no metal deposition occurred under either static or ultrasonic conditions. Thus, 0 V was chosen as the initial potential. When the phase step potential $E_s$ changes from 0.8 V to 1.7 V, the current-time ($I$–$t$) curves obtained under different conditions for Fe-Ni electrodeposition on the cathode are summarized in Fig. 8.

As can be seen in Fig. 8, the transients all exhibited dramatic decreases in current to constant values as $E_s$ was positive over $E_c$ ($E_s \geq E_c + 0.1$ V). This is due to the charging of the double electrode layers, which indicates a lack of crystallization of Fe-Ni at these potentials. When $E_s$ is negative relative to $E_c$, both in Fig. 8a and b, the current increases, which can be attributed to the growth of each independent core (instantaneous growth) and/or the increase in the number of cores (progressive growth). At the time $t_m$, the current reached its maximum value ($I_m$). The more negative the value of $E_s$, the greater the value of $I_m$ and the shorter the time $t_m$ for the current to reach $I_m$. All of this indicates that in static conditions, the electrocrystallization of Fe-Ni alloy on the surface of V, the current–time ($I$–$t$) curves obtained under different conditions for Fe-Ni electrodeposition on the cathode are summarized in Fig. 8.

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![Fig. 8. Current transients of Fe-Ni co-deposition at different step potentials under different conditions: a) static; b) ultrasonic.](image)

![Fig. 9. Nondimensional curves at different potentials under ultrasonic conditions.](image)

![Fig. 10. Chronopotentiogram at various current densities under different conditions: a) ultrasonic; b) static.](image)

### Table 1

| Properties of electrodeposited Fe-Ni alloy mask. | Static conditions with 10 mA/cm² | Ultrasonic conditions at 53 W and 10 mA/cm² | Ultrasonic conditions at 93 W and 10 mA/cm² |
|-----------------------------------------------|---------------------------------|--------------------------------------------|---------------------------------------------|
| Fe content                                    | 57.08 wt%                       | 60 wt%                                     |                                             |
| Grain size                                     | 12.8 nm                         | 16.6 nm                                    |                                             |
| CTE                                           | $5.34 \times 10^{-6}$ /°C        | $4.25 \times 10^{-6}$ /°C                  |                                             |
| Microhardness                                  | 311.6 HV                        | 351.5 HV                                   |                                             |
| Roughness                                      | 0.788 µm                        | 0.469 µm                                   |                                             |
stainless steel conforms to the characteristics of three-dimensional nucleation growth under diffusion control. As the reduction progressed, the concentration of metal cations (Fe$^{2+}$, Ni$^{2+}$) near the cathodic surface continually decreased and was insufficient to maintain the electrochemical reaction. As a result, the current rapidly decays to the limit diffusion current under static conditions, as shown in Fig. 8a. However, as can be seen in Fig. 8b, with the identical potential $E_0$, the decay of current was minimal and even remained stable under ultrasonic conditions. This is because ultrasonic conditions can help the metal cation in the body of the liquid to quickly replenish to the vicinity of the cathode. Thus, the reductive reaction can continue, and the current remained stable. At this point, the electrochemical reaction and the diffusion of the metal cations to the electrode surface were in a dynamic state of equilibrium.

To characterize the nucleation process of Fe-Ni alloy, the data of current–time (I–t) curves were normalized to $(I/I_m)^2–(t/t_m)$ curves and be compared with the Scharifker Hills model [17].

In Fig. 9, at the phase step potential of $-1.1$ V, when $t/t_m \leq 1$, the experimental data is located between the instantaneous nucleation line and the progressive nucleation line. It seems to be closer to instantaneous nucleation. When the potential becomes more negative, the rising part of the curve shows a trend that is similar to instantaneous nucleation. As $t/t_m \geq 1$, there is an obvious deviation between the theoretical curve and the experimental data. This means that the process of Fe-Ni electrodeposition and nucleation does not exactly match the S-H theoretical model, and the result is close to instantaneous nucleation. Therefore, it can be concluded that the Fe-Ni deposit occurred by a mechanism that instantaneous nucleation is followed by three-dimensional growth under charge transfer control in ultrasonic conditions.

Under both static and ultrasonic conditions, the relationship between potential and time is shown in Fig. 10, which was obtained at 323 K, pH 3, and a scan rate of 20 mV s$^{-1}$.

Under ultrasonic conditions, at a low current density of 5, 8, and 10 mA/cm$^2$, metal ions began to reductive and the potential is increasing. After a few seconds, the potential stabilized. Compared to Fig. 10b, the stable potential under ultrasound conditions was lower than that in static conditions, which meant a lower deposition overpotential and benefit the grain growth. When the current density was higher than 10 mA/cm$^2$, the consumption of metal ions on the surface of the cathode was faster, and hydrogen evolution increased. Due to the ultrasonic cavitation effect, the hydrogen bubbles generated by hydrogen evolution were ruptured. The energy generated by the rupture will lead to the instability of the electroforming liquid on the cathode surface, thus reducing the ion depletion layer on the cathode surface, which is detrimental to the electrodeposition of Fe-Ni. In addition, the higher current density also means grain refinement. Hence, for optimum ultrasonic Fe-Ni electrodeposition, the current density should not be lower than 10 mA/cm$^2$.

3.4. Electroforming of the Fe-Ni alloy mask for verification

Based on UV-LIGA (Ultraviolet-Lithography, Galvanoforming, Abformung) technology, with current densities of 10 mA/cm$^2$, at 323 K, pH 3, OLED Fe-Ni alloy masks with a thickness of 20 μm and opening size of 60 μm were electrodeposited at ultrasonic power 93 W, 53 W and 0 W (static conditions) respectively. After electrodeposition, the surface quality and physical and mechanical properties of the OLED Fe-Ni alloy mask were tested according to the application requirements. The surface morphology and iron content of the mask were examined by scanning electron microscopy which was equipped with energy dispersive spectroscopy. The crystalline structure of the mask was investigated by X-ray diffraction (XRD) with Cu K-alpha radiation in 40 kV and 40 mA ranging from 10° to 100°. The mask’s transmission electron microscopy (TEM) was carried out to evaluate the crystallite size. Roughness was the average value measured by the rough meter. The intelligent digital tester was used to test the Vickers micro-hardness of the mask, and the average value of the five tests was taken. A thermal–mechanical analyzer was used to measure changes in the dimensions of the mask with increasing temperature from 20 to 200 °C. The liner average thermal expansion coefficient (CTE) was calculated for different temperature ranges based on Eq. (7):

$$\text{CTE}_{T_1–T_2} = \frac{(l_{T_2} - l_{T_1})}{l_{T_1}(T_2 - T_1)}$$  (7)
where $l_{T_1}$ and $l_{T_2}$ are the lengths of the sample at temperatures $T_1$ and $T_2$, respectively. The following CTE represents the average linear coefficient of thermal expansion between 20 °C and 150 °C. The mask needs to be separated from the cathode for XRD and CTE tests. The performance tests of the electrodeposited Fe-Ni alloys are summarized in Table 1 and Figs. 11-13. Among them, the properties of the Fe-Ni alloy mask electrodeposited under static conditions can not be detected because of peeling.

As can be seen in Fig. 11, with the identical current density of 10 mA/cm$^2$, the feature of peeling and pore of the electrodeposited Fe-Ni alloy mask which can be observed obviously in static conditions was completely disappeared in ultrasonic conditions with 53 W and 93 W. As listed in Table 1, the surface quality of mask obtained in ultrasonic conditions was much better than that of in static conditions, with a roughness value of 0.469 ~ 0.788 μm. The XRD patterns in Fig. 12 indicated that the mask electrodeposited in ultrasonic conditions presented double phase compositions with a face-centered cubic (FCC) and a body-centered cubic (BCC) structure. With increasing ultrasonic power, the preferred orientation of the electrodeposited Fe-Ni mask changed from (111) and (200) of FCC to (110) of BCC and (200) of FCC. The previous electrochemical analysis illustrated that the suppression on anomalous co-deposition of Fe-Ni alloy gradually diminished as the ultrasonic power increased. Accordingly, the iron content of the electrodeposited mask is increasing as present in Table 1. The grain size of the electrodeposition mask calculated from Fig. 13 verified the analysis of Fig. 3, in which the grain size increased by increasing the ultrasonic power within a certain range.

The traditional Hall–Petch effect shows that the grain size of metals is inversely proportional to the hardness. However, the result of the hardness test in Table 1 is contradictory with it. This is because the electrodeposited Fe-Ni mask with a grain size of approximately 10 nm belongs to the nanocrystalline. Due to the grain boundary migration, sliding or grain rotation, the crystal materials will be softened when the grain size is reduced below a critical value, and all this has been proved by the experimental measurements and molecular dynamic simulations [18–21]. In this case, the hardness of the material is proportional to the grain size, which is also called the inverse Hall–Petch effect [22,23]. As shown in Table 1, the Fe-Ni mask which is electrodeposited with 93 W has a larger grain size than 53 W, and thus it got a higher micro-hardness 351.5 HV.
The CTE of Fe-Ni mask electrodeposited in the ultrasonic conditions is 4.246 ~ 5.338 x 10^{-6}/°C, which is much lower than that of Fe-Ni alloy prepared by traditional electrodeposition. The Fe-Ni mask electrodeposited at 93 W gets a lower CTE than 53 W. This can be explained from two aspects. On the one hand, the composition of alloy can affect its thermal expansion property. Mask got at the ultrasonic power of 93 W has a higher iron content than others, which is considered to have higher spontaneous volume magnetostriction [24]. So, it exhibits a lower thermal expansion coefficient. On the other hand, the thermal expansion of metals has grain size dependence. Studies on the CTE of nanocrystalline (NC) materials Ni, Cu, Ni-P et al. have confirmed both from theoretical and experimental that there are certain differences between grain boundaries and crystals due to the excess volume at the grain boundary [25–32]. A study on the effects of grain growth and size on the thermal expansion behavior of electrodeposited Fe-Ni alloy proved that this difference seemed to be enlarged in NC Fe-Ni alloy because of the “invar effect” of Fe-Ni alloys affects only the crystallites [15]. The increase in grain size due to the increase of ultrasonic power indicates a rise in the percentage of crystallites. Therefore, the mask prepared by 93 W has a lower coefficient of thermal expansion.

4. Conclusions

In this study, the effect of ultrasound on the electrochemical processes of Fe-Ni alloy was investigated by electrochemical methods. The study revealed that the electrodeposition of Fe-Ni alloy on the surface of stainless steel conformed to a mechanism that instantaneous nucleation is followed by three-dimensional growth under charge transfer control. Due to the ultrasonic effect, the deposition process of Fe-Ni changed from being diffusion-controlled under static conditions to an electrochemical control under ultrasonic conditions. However, because of the strong ultrasonic cavitation effect, both high levels of ultrasonic power and high current densities are unsuitable for electroforming Fe-Ni alloy. Therefore, the optimum parameters for Fe-Ni electrodeposition under ultrasonic conditions are a temperature of 323 K, pH 3, ultrasonic power between 80 and 100 W (power density 0.28–0.35 W/cm²), and a current density lower than 10 mA/cm². The electroforming experiment carried out based on these parameters showed that the Fe-Ni mask with good surface quality and low CTE could be directly prepared by ultrasonic-assisted electroforming. Thus, ultrasonic-assisted electrodeposition technology is particularly suitable for OLED Fe-Ni mask making.

Declaration of Competing Interest

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

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