

Article

Study of Electroless Nickel Plating on Super Duplex Stainless Steel for Lithium-Ion Battery Cases: Electrochemical Behaviour and Effects of Plating Time

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Abstract: With increasing demand for Li-ion batteries, studies are focusing on enhancing battery performance and safety. However, studies on battery cases remain scarce. Herein, we propose the use of super duplex stainless steel SAF2507, which is a two-phase (austenite + ferrite) steel, for battery casings. Unlike conventional AISI304, SAF2507 maintains its corrosion resistance and strength at high temperatures and precipitates a secondary phase at approximately 975 °C. However, the effects of Ni plating on this secondary phase are not well documented. Therefore, the electroless Ni plating of SAF2507 after secondary-phase precipitation was studied. Briefly, heat treatment at 1000 °C was used to induce precipitation, and the electroless Ni plating behaviour over varying plating periods was analysed using open-circuit potential, potentiodynamic polarisation, and electrochemical impedance spectroscopy measurements. The plating state and corrosion behaviour were examined using scanning electron microscopy. Heat-treated SAF2507 steel with a secondary phase exhibited excellent electroless Ni plating behaviour, which enhances the safety and durability of Li-ion batteries. Furthermore, uniform plating and electrochemical behaviour were achieved after 180 s, suggesting that SAF2507 is superior to AISI304. These findings contribute to the development of safer and more efficient batteries and address the growing demand for Li-ion battery case materials.

Keywords: Li-ion battery case; super duplex stainless steel; secondary phase; electroless Ni plating; electrochemical behaviour



Citation: Shin, B.-H.; Park, J.; Kim, S.; Ok, J.-W.; Kim, D.-I.; Yoon, J.-H. Study of Electroless Nickel Plating on Super Duplex Stainless Steel for Lithium-Ion Battery Cases: Electrochemical Behaviour and Effects of Plating Time. *Metals* **2024**, *14*, 307. <https://doi.org/10.3390/met14030307>

Academic Editor: Zhifeng Wang

Received: 7 February 2024

Revised: 4 March 2024

Accepted: 4 March 2024

Published: 5 March 2024



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1. Introduction

Lithium-ion batteries (Li-ion) are rechargeable power sources for electronic devices, having high energy densities, low self-discharge rates, rapid charging abilities, and long cycle lives [1–3]. These attributes have made Li-ion batteries the preferred choice for electric vehicles and devices, and demand has increased in recent years [4–6]. Considering the integration of Li-ion batteries into daily life, it is crucial to ensure their safety [7–9]. Consequently, research in this field has focused on enhancing the safety of Li-ion batteries. Recent researchers have been conducting studies on temperature measurement and control to enhance the stability of batteries.

Ongoing Li-ion battery research involves the development of high-density and high-performance cathode materials, high-temperature stability, advanced electrolytes, nanoscale battery designs, and sustainable energy materials. For instance, Park focused on enhancing cathode material performance by developing TiC₃ [10]. Khan studied the heating of Li-ion battery phase-change material substrates to achieve temperature control [11], and Kale explored flexible solid-state polymers as Li-ion battery electrolytes [6]. These research trends

collectively aim to enhance the performance and safety of lithium-ion batteries, thereby enabling their application in diverse fields [12–14]. However, research on battery casing materials to enhance the safety of Li-ion batteries remains relatively limited. Improvements in battery materials offer the potential for enhancing stability, necessitating research in this area.

Li-ion batteries consist of a Li electrolyte between the cathode and the anode [1,6,7]. The cathode, which must have a high electrical conductivity, commonly utilises materials of the lithium oxide series, whereas the anode, which serves as the casing, must have high strength and corrosion resistance; thus, stainless steel is frequently used. The prevalent choice for Li-ion casings is austenitic AISI304 stainless steel, which is renowned for its excellent strength (500 MPa) and corrosion resistance [15–17]. However, its high-temperature strength is limited, and it suffers from significant thermal expansion ($16 \times 10^{-6}/^{\circ}\text{C}$), rendering it unsuitable as a battery casing when heat-generating reactions occur [16–18]. Therefore, materials with high-temperature strength and excellent corrosion resistance can enhance the safety of batteries in terms of improved stability.

In contrast, duplex stainless steel (DSS) demonstrates superior strength (800 MPa) and lower thermal expansion ($12 \times 10^{-6}/^{\circ}\text{C}$) at elevated temperatures than AISI304 [19–21]. These characteristics can enhance the safety of Li-ion batteries, making DSS an appropriate candidate for a Li-ion battery casing material. Notably, DSS comprises austenitic and ferritic structures, providing a balance between strength and corrosion resistance [22–24]. Based on the pitting resistance equivalent number (PRE), DSS is categorised into lean (below 20), standard (30 or above but below 40), super (40 or above but below 50), and hyper (50 or above) grades [19,25,26]. Super-grade duplex SAF2507 stainless steel, commonly used in marine plant applications, has a strength of 800 MPa and a lifespan of up to 50 years in seawater. Considering SAF2507's exceptional strength and corrosion resistance, it is a promising candidate for enhancing LIB safety [27–29]. Nevertheless, research investigating DSS and, specifically, SAF2507 as materials for Li-ion batteries is currently non-existent. Therefore, extensive research is required for utilising SDSS as a battery casing.

Notably, SAF2507 shows variations in the austenitic and ferritic fractions after heat treatment temperatures, with the precipitation of secondary phases observed at approximately 375 and 975 °C [20,26,30,31]. Many researchers have conducted studies on the physical properties of these secondary phases to use their advanced properties and to decrease embrittlement. These secondary phases, which predominantly comprise Cr and Mo precipitates, detrimentally affect the strength and corrosion resistance of SAF2507 [32–34]. Given SAF2507's potential as a battery casing material, understanding and mitigating the effects of these secondary phases is crucial. However, research on the utilisation of SAF2507 with precipitated secondary phases as a battery casing material is currently lacking. Therefore, precipitating secondary phases and analysing their electrochemical behaviour is a crucial issue.

In addition, as a battery casing material, SAF2507 should have excellent electrical conductivity [30,35–37]. However, Fe-based materials typically have low electrical conductivity, necessitating surface plating. Nickel, which is known for its excellent reactivity with metals, offers a high plating speed [38–40]. Furthermore, plating can be performed using both electrolytic and electroless methods. Electrolytic plating is fast but may lead to brittleness and high surface roughness. In contrast, electroless Ni plating offers slower deposition but provides uniform quality and low surface roughness [41–44]. However, research on this topic is lacking, and this study aims to address this gap by investigating it.

In this study, we investigated the electroless Ni plating of SAF2507 following the precipitation of secondary phases. Heat treatment at 1000 °C was employed to induce precipitation, and the electroless Ni plating behaviour over different plating periods was electrochemically analysed. The plating state and corrosion behaviour of SAF2507 were examined by field-emission scanning electron microscopy (FE-SEM, SUPRA 40VP system, Zeiss, Land Baden-Württemberg, Germany) and atomic force microscopy (AFM, NX-10, Park System Corp., Suwon, Republic of Korea). Electrochemical analysis (Potentiostat,

Versa Stat 3.0, AMETEK, Inc., Commonwealth of Pennsylvania, Berwyn, IL, USA) included open-circuit potential, potentiodynamic polarisation curves, and electrochemical impedance spectroscopy measurements. This study contributes to the understanding of electroless Ni plating on SAF2507 with precipitated secondary phases, providing insights into its potential applications as a battery casing material.

2. Materials and Methods

2.1. Materials

The key material utilised in this study was super-grade duplex SAF2507 stainless steel (manufactured by POSCO SS, Changwon, Republic of Korea) having a PRE number of 42. The compositions are listed in Table 1 [19,45]. SAF2507 was obtained by casting in an electric furnace, followed by extraction of a portion of the molten metal for air cooling [46,47]. The primary constituents of SAF2507 were analysed using inductively coupled plasma mass spectrometry (ICP-MS, Thermo Fisher Scientific, Waltham, MA, USA). PRE relies on the content of Cr, Mo, and N as strengthening elements for the passive layer of stainless steel. Each composition interacts differently with the passive layer. Cr and Mo, having atomic sizes comparable to Fe, stabilise the formation of the passive layer. Conversely, N, with its smaller atomic size, acts as an interstitial element, enhancing the passive layer as a strengthening agent.

$$\text{PRE} = 1 \text{ wt.\% Cr} + 3.3 \text{ wt.\% Mo} + 16 \text{ wt.\% N} \quad (1)$$

Table 1. Chemical composition of (a) cast SAF2507 by ICP-MS and (b) austenite and ferrite on annealed SAF2507 by EDS.

		Element (wt.%)								
		C	N	Mn	Ni	Cr	Mo	Cu	Fe	PRE
(a)	Total	0.01	0.27	0.8	6.8	25.0	3.8	0.2	Bal	41.9
(b)	Austenite	0.01	0.51	0.9	7.9	23.3	3.2	0.2	Bal	42.0
	Ferrite	0.01	0.05	0.8	5.5	26.6	4.4	0.2	Bal	41.9

To precipitate secondary phases, while preventing the high-temperature cracking caused by phase transformation, solution heat treatment was conducted at 1100 °C [34,47–49]. Table 1 (row (b)) lists the contents of the austenite and ferrite phases in SAF2507 after solution heat treatment. The major constituents were analysed using energy-dispersive X-ray spectroscopy (EDS, SUPRA 40VP system from Zeiss, Land Baden-Württemberg, Germany), and phase percentages were calculated using a proportional approach because of potential inaccuracies in the nitrogen measurements [19,20,50,51]. In steel materials, ferrite can accommodate nitrogen (N) up to 0.05%, with the remainder being dissolved or precipitated in austenite. Hence, in the absence of separate precipitation, nitrogen is dissolved up to 0.05% in ferrite, while the rest is absorbed into austenite.

$$N_r = \text{chemical composition of } N_{\text{Total}} \text{ wt.\%} - \text{Ferrite}_{\text{VF}} \times 0.05 \text{ wt.\%} \quad (2)$$

Here, N_r is the chemical composition of N in austenite, and $\text{Ferrite}_{\text{VF}}$ indicates the volume fraction of ferrite.

2.2. Heat Treatment Conditions

The material was subjected to heat treatment, including casting, solution heat treatment, and heat treatment, for secondary-phase precipitation [19,28,45,46,52]. A schematic of the heat treatment conditions is shown in Figure 1.

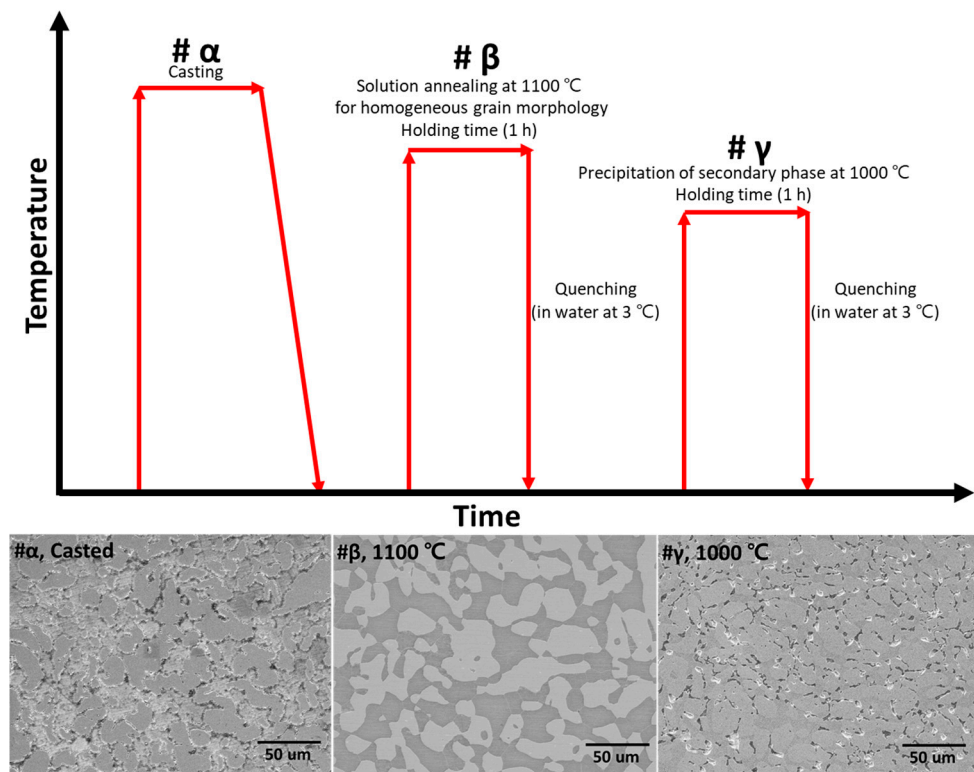


Figure 1. Schematic of heat treatment conditions for secondary-phase precipitation on SAF2507.

During casting, the uneven cooling rate led to the formation of irregularly shaped austenite as a result of air cooling after casting. The untreated SAF2507 thus contained irregular austenite boundaries, which resulted in the precipitation of secondary phases and subsequent material cracking.

Therefore, solution heat treatment at 1100 °C was performed, followed by rapid quenching to achieve a uniform austenitic structure. Subsequently, to induce the precipitation of secondary phases, a further heat treatment at 1100 °C was performed for 1 h, followed by rapid quenching at 50 °C/s. Secondary phases precipitated at the boundaries of the austenitic structure and comprised approximately 13% of the total composition.

2.3. Electroless Ni Plating

Before plating, the specimen was subjected to surface polishing using 0.025 μm colloidal silica to reduce the surface roughness to 100 nm or less. Subsequently, to facilitate the formation of a passive layer, the specimen was immersed in deionised water for 1 h. Then, the plating reaction was induced in the electrolyte solution by stirring, as shown in Figure 2.

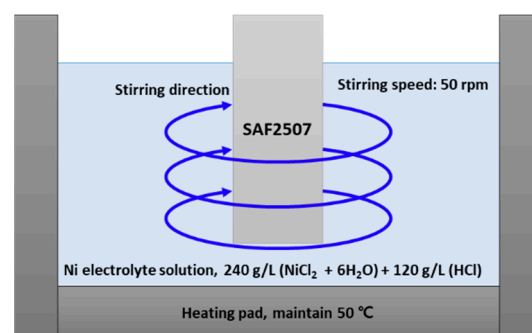


Figure 2. Schematic of the plating bath for electroless Ni plating on SAF2507.

The electrolyte consisted of a mixture of nickel(II) chloride (NiCl_2 , 240 g/L) and hydrochloric acid (HCl, 37 mol%, 120 g/L) [38–40,43]. To minimise the solution's effects on the plating, the pH was maintained at 3.0 to 3.5, and the temperature was constant at 50 °C. The solution was stirred at 350 RPM to prevent the formation of concentration gradients, and the plating duration varied from 0 to 300 s. The post-plating state was analysed using field-emission scanning electron microscopy (FE-SEM, SUPRA 40VP system, Zeiss, Land Baden-Württemberg, Germany), and X-ray diffraction (XRD, D8 VENTURE, Stanford, CA, USA) was employed to identify the formed phases.

The Ni plating behaviour was analysed using FE-SEM, atomic force microscopy (AFM), and XRD. Surface and cross-sectional images were obtained at different plating times using an optical microscope (OM, DSX500, Anyang, Republic of Korea). Changes in the surface roughness and Ni thickness were monitored using AFM [41,42], and phase analysis was achieved using XRD. Surface images were captured using OM after plating, and the changes in the surface roughness (R_a) with respect to plating time were measured at 10 μm intervals in five locations for both the austenite and ferrite phases. This measurement was repeated five times, and the average values were calculated (Figure 2). R_a was estimated using Equation (3):

$$R_a = 1/l \int_0^l |f(x)| dx, \quad (3)$$

where l is the total length of the measurement. The phase analysis was performed using XRD over an area of 100 cm^2 , scanning from 30° to 100°. These techniques allow for a comprehensive analysis of Ni plating behaviour, including its effects on surface morphology, roughness, and phase composition.

2.4. Electrochemical Properties

The surface of the electroless nickel-plated SAF2507 was subjected to electrochemical analysis to discern variations in its electrochemical characteristics. Electrochemical analysis was performed using a potentiostat (Versa Stat 3.0, AMETEK, Inc., Berwyn, PA, USA) in a three-electrode cell configuration. The three-electrode cell consisted of a working electrode (sample specimen, 1 cm^2), counter electrode (Pt mesh, 20 mm $\times$ 20 mm), and reference electrode (SCE, saturated calomel electrode). The electrolyte solution was prepared following ASTM G 61 guidelines with 3.5 wt.% NaCl [19,31,35].

Three different methods were employed to analyse the electrochemical properties with respect to the plating time. Although the galvanic series can be used to calculate the potential of pure metals, it is unsuitable for measuring potential variations in alloys or plating states. Therefore, open-circuit potential (OCP) measurements were performed from 0 to 6000 s to ascertain potential variations during plating [30,35]. Further, potentiodynamic polarisation tests, which measure the change in current density with potential, provided insights into electrochemical behaviour. The voltage measurements ranged from −0.6 to 1.2 V at a scan rate of 0.137 mV/s [31,34]. Electrochemical impedance spectroscopy (EIS) was used to identify the changes in resistance at frequencies ranging from 10^6 to 10^{-3} Hz [53–55]. The obtained values are represented using Bode plots, Nyquist plots, and an equivalent circuit model.

3. Results

3.1. Electroless Ni Plating Behaviour with Time

The sample surface was analysed using FE-SEM with respect to the plating time, and the results are shown in Figure 3. Surface nodules were observed after short plating times, specifically, 0–60 s. In addition, secondary phases were evident from 0 to 120 s but were not observed at 180 s, suggesting stabilisation of the plated layer after this time. With an increase in plating time, the secondary phases were obscured by the Ni plating layer, and differentiation between austenite and ferrite was possible. In these images, austenite appears bright grey, whereas ferrite appears darker grey, allowing for differentiation [19,20,31].

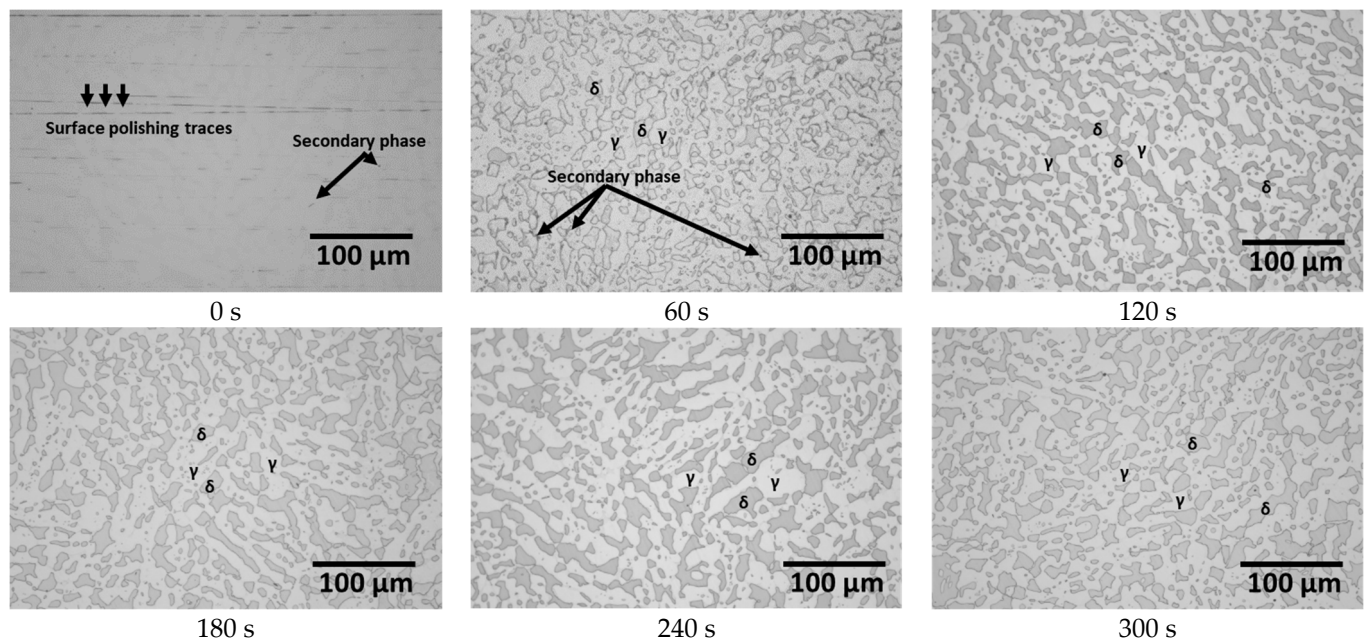


Figure 3. Surface images and volume fractions of austenite and ferrite with electroless Ni plating time after heat treatment at 1000 °C of super duplex SAF2507 stainless steel.

XRD analysis allowed for the examination of the phases and assessment of the growth of the plated layer over various Ni plating times [38–40,44], and the results are shown in Figure 4. Secondary phases were observed until the plating time reached 300 s. However, the intensity of the austenite (111) reflection increased, which coincided with those of Ni and SAF2507. After 60 s, there was an evident increase in the intensities of the austenite (111) and ferrite reflections. Although the reflections arising from the secondary-phase nodules were weak for all plating durations, they were visible for up to a plating time of 300 s. A change in intensity shows with the electroless Ni plating time. Austenite intensity increased to 3800 counts at 300 s from 3000 counts at 0 s, and ferrite intensity decreased to 1500 counts at 300 s from 3200 counts at 0 s.

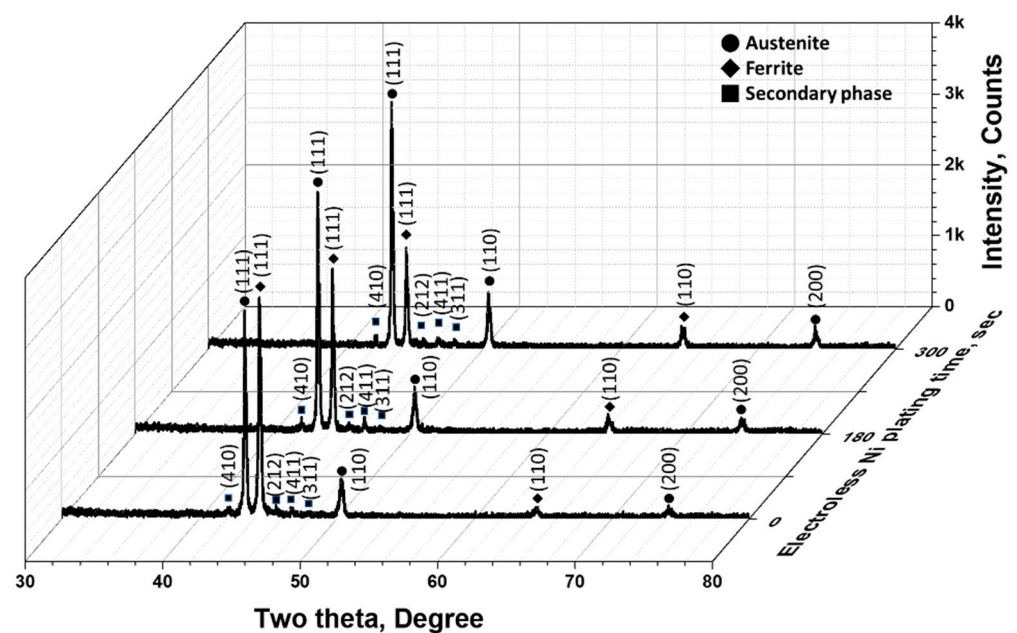


Figure 4. Plot of XRD intensity (counts), 2θ (degrees), and electroless Ni plating time curve showing XRD patterns of SAF2507 at different electroless Ni plating times.

These results reveal that the surface state changed with the plating time and confirm an increase in the Ni plating layer over time [43,44,56]. As the plating time increases, the initial nodules and secondary phases become uniformly plated, making it difficult to distinguish them [45,51]. In addition, the base structure became visible, allowing the differentiation of ferrite and austenite. Notably, the secondary phases appear to be similar to those of ferrite, indicating comparable plating rates. If the secondary phases had the same plating rate as that of austenite, the proportion of austenite would have increased. However, owing to the slower plating rate of the secondary phases, the proportion of ferrite increased. Further, observation of the base structure revealed a higher proportion of ferrite compared with the substrate for up to 300 s of plating [39,42,56]. Therefore, it can be concluded that the Ni plating is influenced by the base structure, and an increase in the thickness of the Ni plating layer likely amplifies this effect.

In the XRD patterns of samples treated at 40–45 °C, the intensities of the peaks corresponding to the secondary phase were low. As shown in Figure 2 and Table 2, the secondary phase grew along the grain boundaries and did not form a flat surface morphology after grinding. The precipitation of the secondary phase reduced the intensity of the XRD reflections and that shows in the drop in intensity from 140 counts to 170 counts at 0–180 s. This reduction in peak intensity as a result of the secondary-phase morphology persisted after nickel plating. Even after increasing the plating time to 300 s, there was no significant increase in the intensities of the secondary-phase peaks. However, the presence of a thin plating layer did not reduce the intensity of the secondary phase, and as the thickness of the plating layer increased, the XRD peak intensity decreased.

Table 2. XRD results with respect to electroless Ni plating time of super duplex stainless steel SAF2507.

Intensity	0 s	180 s	300 s
Austenite (111) plane at 42°	3000 ± 30	3700 ± 33	3800 ± 35
Ferrite (111) plane at 44°	3200 ± 15	2300 ± 21	1500 ± 25
Secondary-phase (410) plane at 41°	150 ± 29	170 ± 31	170 ± 32
Secondary-phase (212) plane at 46°	160 ± 34	160 ± 44	150 ± 40
Secondary-phase (411) plane at 47°	150 ± 25	180 ± 32	160 ± 31
Secondary-phase (311) plane at 48°	140 ± 31	160 ± 34	150 ± 32

Next, the surface roughness and thickness variations of the plating layer were measured using AFM, and the results are shown in Figure 5 and Table 3. The surface roughness gradually decreased owing to the growth of the electroless nickel plating layer. The influence of Ni on the growth of the plating layer decreased with the increase in plating time because of the interaction of the plating layer with SAF2507, and the thickness of the nickel-plated layer increased with the increase in nickel plating time. The thickness of the Ni layer varied depending on the main phase. On the austenite phase, a 60 nm nickel layer formed after plating for up to 300 s, whereas on the ferrite phase, a 43 nm nickel layer formed within the same period. This difference highlights the varying reactivities of austenite and ferrite. Non-electrolytic nickel plating exhibits higher reactivity towards austenite than toward ferrite. However, the influence of this grain structure is expected to decrease with increasing plating time.

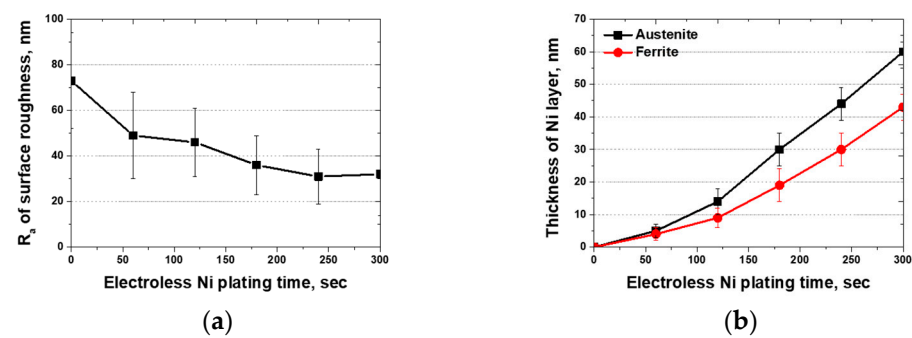


Figure 5. AFM analysis of surface structure with respect to electroless Ni plating time of super duplex stainless steel SAF2507: (a) R_a (nm) vs. electroless Ni plating time (s) curve, and (b) thickness of Ni layer (nm) vs. electroless Ni plating time (s).

Table 3. AFM results with respect to electroless Ni plating time of super duplex stainless steel SAF2507.

		0 s	60 s	120 s	180 s	240 s	300 s
Ra of surface roughness, nm		73 ± 21	49 ± 19	46 ± 15	36 ± 13	31 ± 12	32 ± 18
Thickness of Ni layer (nm)	Austenite	0	5 ± 2	14 ± 4	30 ± 5	44 ± 5	60 ± 5
	Ferrite	0	4 ± 2	9 ± 3	19 ± 5	30 ± 5	43 ± 4

3.2. Effect of Electroless Ni Plating Time on Electrochemical Properties

Analysis of the electrochemical characteristics of the Ni plating layer provides insights into the subtle features of the layer. Although the potential of a material can be determined using a galvanic series, alloys or post-treated states remain more challenging to analyse [2,42,57]. Therefore, OCP measurements with respect to the plating time were employed, and the results are shown in Figure 6. As shown, the OCP decreased from 0.0 to -0.3 V, before rising again to approximately -0.2 V. These potential variations reflect the influence of the plating layers.

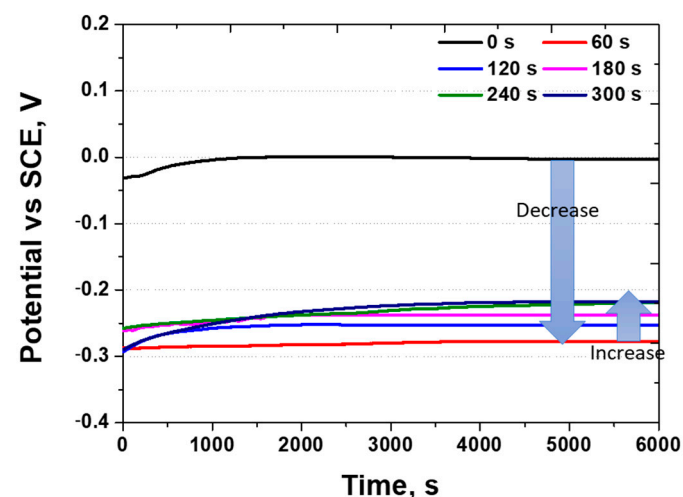


Figure 6. Potential (V) vs. time (s) curve, OCP curves with respect to electroless Ni plating time after heat treatment at 1000 °C of super duplex SAF2507 stainless steel in 3.5 wt.% NaCl.

The initial OCP (0.0 V) before plating represents that of SAF2507. At the onset of plating, the OCP decreased significantly, reaching its lowest value at 60 s, indicating galvanic corrosion arising from the potential difference between the secondary phases and

the Ni plating layer. Subsequently, the OCP gradually increased, approaching the potential of Ni (-0.22 V).

Thus, these changes in the OCP effectively mirrored the variations in the Ni plating layer, allowing us to monitor its growth. Furthermore, the influence of secondary phases on the OCP became apparent. Notably, the uneven plating of the secondary phases for 60 s induced galvanic corrosion [57–59]. As time progressed, the potential gradually increased, nearing -0.22 V after 240 s of electroless Ni plating. Therefore, short plating times resulted in a decreased potential, because the uneven plating layer caused galvanic corrosion between the SAF2507 and the Ni plating layer. As the plating time increased, the potential increased, indicating the formation of a more uniform plating layer that stabilised the surface conditions.

Potentiodynamic polarisation tests measure changes in current density with respect to voltage, providing insights into electrochemical behaviour [17,58–60]. The results are shown in Figure 7 and listed in Table 4 [40,42]. The potentiodynamic polarisation curve of SAF2507, for which secondary phases were precipitated before plating (electroless plating time of 0 s), exhibited a dual-loop characteristic attributed to the dual reactivity of the secondary phases during activation polarisation. This feature was observed up to 60 s of electroless Ni plating, when the influence of the secondary phases vanished because of the formation of the Ni plating layer.

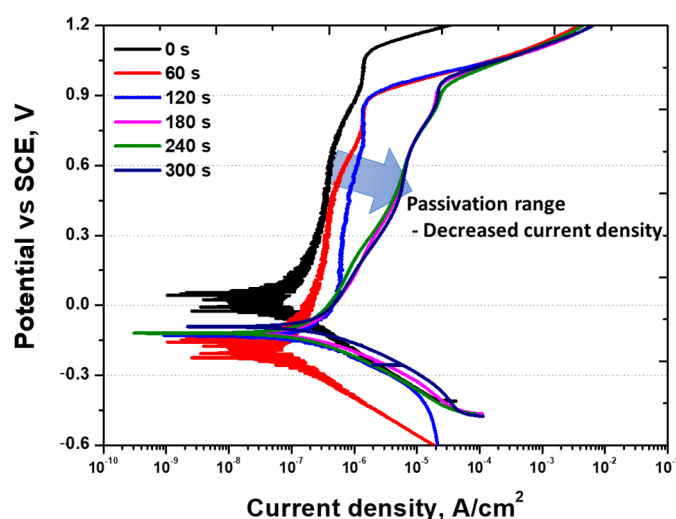


Figure 7. Potential (V) vs. current density (A/cm^2) curve: potentiodynamic polarisation curve with electroless Ni plating time after heat treatment at 1000°C of super duplex SAF2507 stainless steel in 3.5 wt.% NaCl.

Table 4. Potentiodynamic polarisation data of samples with respect to electroless Ni plating time after heat treatment at 1000°C of super duplex SAF2507 stainless steel in 3.5 wt.% NaCl.

	0 s	60 s	120 s	180 s	240 s	300 s
Corrosion potential at active polarisation (V)	0.07 ± 0.02	-0.15 ± 0.02	-0.15 ± 0.02	-0.14 ± 0.02	-0.14 ± 0.02	-0.12 ± 0.02
Corrosion current density at active polarisation (A/cm^2)	$(1 \pm 0.1) \times 10^{-7}$	$(3 \pm 0.5) \times 10^{-7}$	$(4 \pm 0.4) \times 10^{-7}$	$(4 \pm 0.3) \times 10^{-7}$	$(4 \pm 0.3) \times 10^{-7}$	$(4 \pm 0.3) \times 10^{-7}$
Pitting potential (V)	1.05 ± 0.02	0.90 ± 0.04	0.90 ± 0.03	0.91 ± 0.02	0.90 ± 0.02	0.90 ± 0.02

As the electroless plating time increased, the corrosion current density also increased after activation polarisation, because of the growing Ni plating layer [43]. The increase in the current density implies an increase in the corrosion rate, and the upward trend in the slope of the current density in the passivation region indicates an increase in uniform corrosion, which can be attributed to the Ni plating layer. Further, SAF2507, which underwent uniform corrosion after the Ni plating layer, exhibited a pitting potential of 1.05 V, whereas the

Ni-plated SAF2507 had a pitting potential of 0.9 V. Therefore, the Ni plating layer played a role in reducing pitting potential.

Thus, potentiodynamic polarisation tests revealed the Ni plating behaviour with respect to the electroless plating time. For plating times less than 60 s, the plating layer was affected by the substrate microstructure, resulting in a combination of the corrosion characteristics of the substrate and Ni. In particular, the formation of an uneven Ni plating layer facilitated the initiation of galvanic corrosion, thus increasing the corrosion rate. The uniform Ni plating layer, although dominated by Ni-induced corrosion, exhibited diminished corrosion characteristics in SAF2507 when the Ni plating layer was lost. Therefore, electroless Ni plating can create a uniform Ni plating layer on SAF2507, and even in the absence of a plating layer, the electrochemical characteristics of SAF2507 are appropriate for the target use.

Subsequently, the corrosion morphology was examined (Figure 8). Corrosion occurred on the secondary phase, as indicated by the similar shapes of the pitted regions. In other words, the corrosion of SAF2507 originated from the secondary phases, and the effect of the Ni plating layer was minimal [38,39]. However, variations in the form of uniform corrosion were observed.

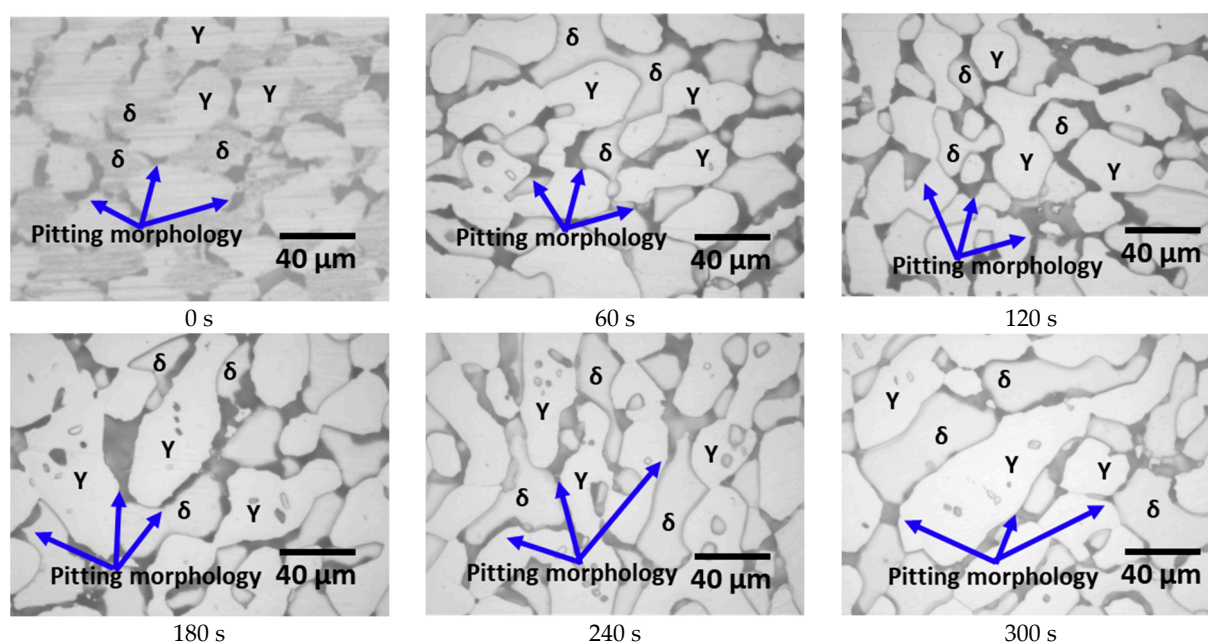


Figure 8. Corrosion morphology with electroless Ni plating time from 0 s to 300 s after heat treatment at 1000 °C of super duplex SAF2507 stainless steel in 3.5 wt.% NaCl from −0.6 to 1.2 V.

The pre-plated specimen exhibited residual pitting on the surface. In contrast, the Ni-plated specimens exhibited a smoother appearance. In addition, the galvanic corrosion induced by the Ni plating layer increased the corrosion rate, leading to uniform corrosion on the surfaces of both austenite and ferrite, thus reducing surface pitting. In addition, it accelerated the corrosion rate of the secondary phases.

EIS measurements allowed us to observe changes in resistance based on frequency, providing insights into the influence of Ni plating and the passive layers of SAF2507 [22,55,61]. EIS measurements were conducted from 10^6 to 10^{-3} Hz, and the results are presented in Figure 9 using Bode plots, Nyquist plots, and an EIS circuit.

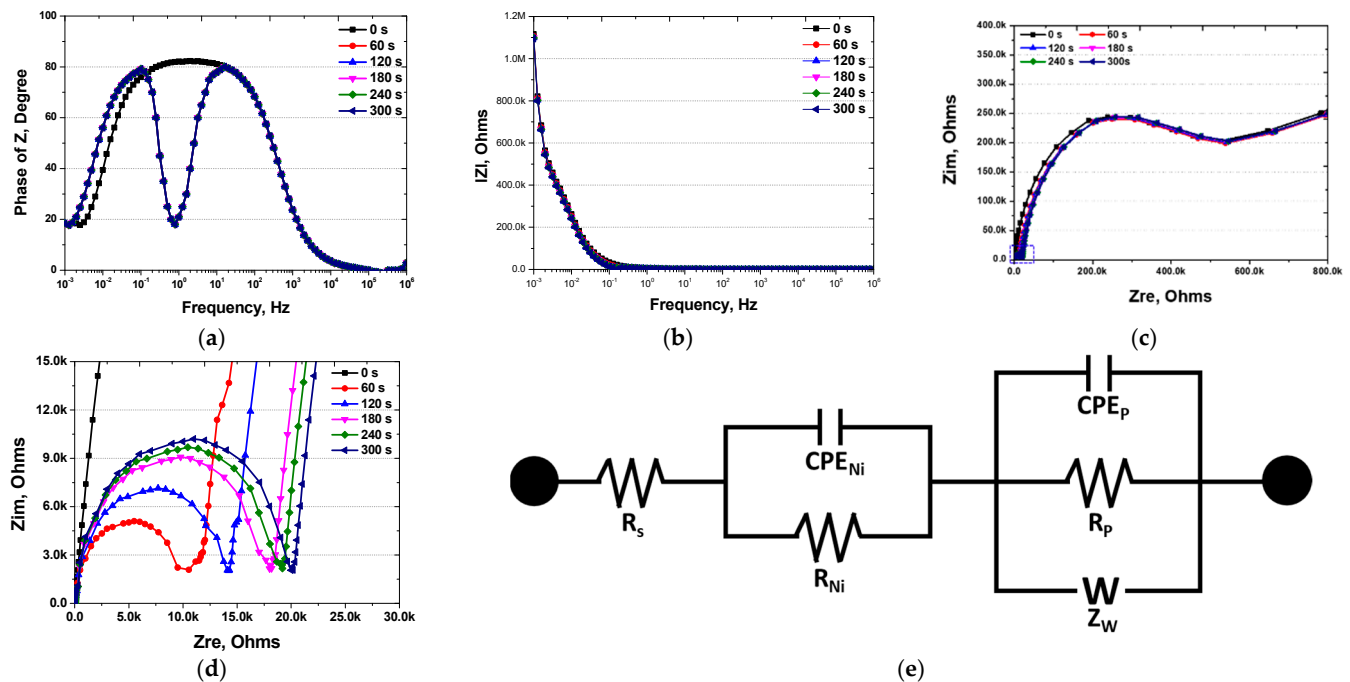


Figure 9. EIS (Bode plot (phase of Z and $|Z|$ vs. frequency curve from 10^{-6} to 10^{-3}), Nyquist plot (Z_{im} vs. Z_{re} curve 0 to 800 kΩ) curves and equivalent circuit with electroless Ni plating time from 0 to 300 s after heat treatment at 1000 °C of super duplex SAF2507 stainless steel. (a) Phase of Z vs. frequency curve, Bode plot from 10^{-3} to 10^6 ; (b) $|Z|$ vs. frequency curve, Bode plot from 10^{-3} to 10^6 ; (c) Z_{im} vs. Z_{re} curve, Nyquist plot from 0 to 800 kΩ; (d) Z_{im} vs. Z_{re} curve, Nyquist plot from 0 to 30 kΩ; (e) EIS circuit with electroless Ni plating time from 0 s to 300 s after heat treatment at 1000 °C.

The Bode plot illustrates the variations in the phase angle and resistance with respect to time as a result of Ni plating, and converting Nyquist plots to Bode plots reveals differences in the initial resistance [29]. The solution resistance (R_s) significantly increased because of the Ni plating layer, and there was a variation in the resistance at different plating times. For example, the resistance of the Ni plating layer (R_{Ni}) during the initial 60 s was 50% of that at 300 s. This indicates a difference in plating rates between 60 and 300 s. The initial plating rate likely demonstrates differences in the reaction because of the presence of surface pits and secondary phases, resulting in a reduction in surface smoothness. Following the Ni plating layer, the resistance of the SAF2507 passive layer (R_p) approached 250 kΩ, which corresponds to the resistance of super duplex stainless steel, indicating the formation of a uniform passive layer of Cr_2O_3 .

4. Discussion

Super duplex SAF2507 stainless steel, having a secondary phase precipitated at 1000 °C, underwent Ni plating. As the plating time increased, the surface pits and secondary phases became indistinguishable after 120 s [38,40,41]. After 180 s, the secondary phase within the plating layer was no longer distinguishable. Considering the proportions, the secondary phase was identified as ferrite, showing a plating rate equivalent to that of ferrite with an increased plating time.

The evolution of plating behaviour over time was confirmed through the surface images and XRD results. The surface images demonstrated stabilisation with increasing plating time. Non-uniform Ni plating layers were observed up to 120 s, followed by the formation of uniform Ni plating layers after 180 s. XRD results revealed differences in the plating state, where an increase in the Ni layer corresponded to growth on the (111) plane of austenite at 42°. Furthermore, an increase in the peak of austenite led to a decrease in the peak of ferrite at 43° on the (111) plane. These phase changes indicate the growth of the plating layer over time. The increase in plating layer thickness as observed by AFM

showed a continuous trend, with a steeper slope after 180 s due to the stabilised growth of the Ni layer. The increase in plating layer thickness was further enhanced by the reduction in grain structure effects associated with the increase in the Ni layer.

Analysis of the electrochemical properties of the Ni plating layer with respect to time revealed that the OCP and potentiodynamic polarisation curves increasingly resembled those of Ni as the plating time increased. Concerning the OCP, the potential initially induced galvanic corrosion with the secondary phase and Ni, but as the plating time increased to 300 s, the reactions of Ni became apparent.

The potentiodynamic polarisation curve showed a dual loop in the activation polarisation for up to 60 s, followed by an increase in the uniform corrosion rate [20,30,31]. However, after the complete corrosion of Ni, galvanic corrosion occurred in the secondary phase of SAF2507, which decreased the pitting potential. The EIS measurements confirmed the formation of a Ni plating layer [55,61]. The resistance at 60 s was low, indicating the presence of a Ni plating layer, followed by an increase in the resistance.

Despite the uniform appearance of the plating layer in the surface images after 180 s, electrochemical analysis revealed that a uniform Ni plating layer formed after 120 s [30,35]. Furthermore, the EIS results showed the characteristics of the Ni plating at 60 s, whereas the active polarisation in the potentiodynamic polarisation curve contained a dual loop in the plating layer at 60 s, suggesting an insufficient thickness to act as a plating layer and showing the characteristics of the substrate [31]. Therefore, even if a Ni plating layer is formed, its low thickness may compromise the Ni layer and negatively affect its electrochemical properties. These findings were observed through OCP, potentiodynamic polarisation tests, and EIS. Thus, considering the electrochemical characteristics, it became evident that the electroless Ni plating duration should be at least 180 s or longer.

Considering both the surface images and electrochemical characteristics, super duplex SAF2507 stainless steel with a precipitated secondary phase requires a Ni plating time of at least 180 s to achieve the desired surface conditions for post-processing. Furthermore, a Ni plating time of 300 s or more is required to ensure the formation of a uniform Ni plating layer on both austenite and ferrite, to ensure a high surface quality after 180 s. SAF2507, which precipitates secondary phases, forms nickel layers of 100 nm or less up to 300 s. Consequently, electrolytic nickel or copper plating is a feasible strategy for improving the heat resistance and electrochemical properties of SAF2507.

5. Conclusions

After heat treatment at 1000 °C to precipitate a secondary phase, super duplex SAF2507 stainless steel underwent Ni plating for 300 s at intervals of 60 s. The surface images and electrochemical properties were analysed, leading to the following conclusions:

- (1) SAF2507, heat-treated at 1000 °C, precipitated a 13% secondary phase primarily at the ferrite–austenite boundaries. The secondary phase was formed through the precipitation of Cr- and Mo-rich clusters, with the chemical composition of Cr and Mo exceeding 30 and 10 wt.%, respectively. The differences in the chemical composition and lattice structure of the secondary phase influenced the Ni plating. The secondary-phase plating rate was equivalent to that of ferrite and slower than austenite. However, uniform plating layers were formed after 180 s. Examination of the plating thickness via AFM revealed that the plating rate improved from an increase of less than 10 µm to over 15 µm after 180 s. Therefore, uniform plating enhances the plating rate and improves electroless Ni plating ability.
- (2) The secondary phase was homogeneously plated within 180 s of the electroless plating. The plating reaction showed a preference for the (111) plane at 42° of austenite, making the Ni plating layer distinguishable between the austenite and ferrite regions. Considering the phase proportions, the secondary phase exhibited a reaction rate equivalent to that of ferrite, suggesting that it grew together with the ferrite. Therefore, the phases have a greater effect than the material composition on Ni plating on SAF2507. The phase composition affects the plating rate, and since SDSS consists of

both austenite and ferrite, an electroless Ni plating time of over 180 s is required to form a stable plating layer over 20 nm.

- (3) The electrochemical characteristics were uniform after 120 s of electroless Ni plating. In contrast, the uneven Ni plating layer formed after 60 s caused galvanic corrosion with the secondary phase, resulting in a decreased OCP potential from 0.0 V to -0.28 V and activation polarisation. In addition, a dual loop was observed in the potentiodynamic polarisation curves at 60 s. After 120 s, uniform electrochemical properties were evident, showing an increased potential, current density, and reduced pitting potential. The increase in potential approached that of pure Ni after galvanic corrosion, and the increase in current density indicated an enhanced uniform corrosion rate arising from Ni plating. The pitting potential, generated in the secondary phase, decreased from 1.05 to 0.90 V due to increased corrosion after Ni plating.
- (4) SAF2507 precipitated with a secondary phase and showed excellent electroless Ni plating behaviour. Considering the surface conditions and electrochemical characteristics, electroless Ni plating for 180 s (Ni layer thicknesses from 20 to 40 nm and EIS resistance of 20 k Ω) or more is suitable for application as a Li-ion battery case material. Compared to conventional AISI304 stainless steel, SAF2507 offers superior strength and plating characteristics, which can enhance its safety and durability. This study confirms that SAF2507 is a potential replacement for Li-ion battery materials, anticipating the improved safety of Li-ion batteries through future material enhancements.

Supplementary Materials: The following supporting information can be downloaded at: <https://www.mdpi.com/article/10.3390/met14030307/s1>, Figure S1. Heat treatment program (left) and scanning electron microscopy images of cast and heat-treated samples (right). Figure S2. Scanning electron microscopy images of the surface of cast and heat-treated SAF2507. Figure S3. Scanning electron microscopy images of the surface of SAF2507 at different electroless Ni plating times. Figure S4. Potentiodynamic polarisation curves of samples plated for different periods.

Author Contributions: Conceptualisation, B.-H.S. and D.-I.K.; Methodology, B.-H.S.; Validation, J.-W.O., J.P. and B.-H.S.; Formal analysis, J.-W.O., D.-I.K., J.P. and B.-H.S.; Investigation, D.-I.K.; Resources, S.K. and B.-H.S.; Data curation, J.-W.O. and J.P.; Writing—original draft, S.K. and D.-I.K.; Writing—review and editing, S.K., J.-H.Y. and B.-H.S.; Visualisation, B.-H.S.; Supervision, D.-I.K., J.-H.Y. and B.-H.S.; Project administration, J.-H.Y. All authors have read and agreed to the published version of the manuscript.

Funding: This work was supported by grants from the Korea Basic Science Institute (grant number C330320) and the BK21 FOUR program (grant number 4120200513801), funded by the Ministry of Education (MOE, Korea) and the National Research Foundation of Korea (NRF).

Data Availability Statement: The original contributions presented in the study are included in the article and Supplementary Material, further inquiries can be directed to the corresponding authors.

Conflicts of Interest: The authors declare no conflicts of interest.

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