Study on the influence of technological parameters on the formation of a chemical nickel plating layer on fiberglass/polyamide composite

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ABSTRACT

This study investigates the effects of temperature, plating time, and plating area-to-solution volume ratio on the formation of chemical nickel plating layers on fiberglass/polyamide composites. The experimental results demonstrate that optimal plating conditions are achieved at temperatures ranging from 75 to 80 °C. A minimum plating duration of 45 minutes is required to ensure adequate layer quality, characterized by electrical resistance ≤ 1.5 Ω and layer thickness ≥ 6 μm. Furthermore, the optimal plating area-to-solution volume ratio is determined to be within the range of 2.5:1 to 3.0:1 (dm²/L). These findings provide critical process parameters for achieving consistent and reliable chemical plating performance on composite substrates.

Keywords: Chemical nickel plating; Fiberglass/polyamide composite.

1. INTRODUCTION

Currently, composite materials are among the advanced materials that are widely used in the manufacture of weapons. The proportion of composite materials used to manufacture components of tactical weapons is increasing. The fiberglass/polyamide composite materials are a typical material of this type, with a density of only ~ 1.2 g/cm^3. In many types of instruments and controlled flying weapons, the shell structure is often conductive material to facilitate the elimination of eddy currents and reduce interference to internal electronic components [1]. When the shell material is made of an alloy, the overall mass will be very large, which is not favorable for maneuverability and flight time is limited. The application of fiberglass/polyamide composite materials to manufacture these parts helps to significantly reduce the mass. However, the fiberglass/polyamide composite materials are non-conductive materials. To create conductivity, chemical plating methods are often applied to deposit a nickel layer ≥ 6 μm thick [2]. To ensure reliability, the nickel plating layer must meet the following main criteria: the plating layer resistance must be ≤ 1.5 Ω and after thermal shock testing in an air environment from -(50 ± 3) °C to +(50 ± 3) °C for 3 cycles, with each temperature maintained for 4 hours, the plating layer must not peel off at any location on the surface compared to the initial time [2]. In this study, the effects of temperature, time and plating area/solution volume ratio on the formation of a chemical nickel plating layer on glass fiber/polyamide composite will be investigated.

2. EXPERIMENTS AND RESEARCH METHODS

2.1. Research equipment and supplies

The equipment used includes: a Sandblasting equipment made in China, a 300 °C drying oven made in China, a Struers sample cutter with cutting speed from 100-400 rpm made in Denmark, and a magnetic stirrer with heating made in China.

Chemicals used in the experiment: Al_2O_3 (20-40 μm, PA, Germany), Na_2CO_3 (PA, Germany), Na_3PO_4 (PA, Germany), Na_2SiO_3 (PA, Germany), NaOH (PA, Germany), $KMnO_4$ (PA, Germany), $SnCl_2$ (PA, China), HCl (PA, China), Sn kim loại (China), $PdCl_2.6H_2O$ 99% (Sigma-Aldrich,

Singapore) $NiCl_2.6H_2O$ (PA, China), NaH_2PO_2 H_2O (PA, Germany), $PbCrO_4$ (PA, China), $Na_3C_6H_5O_7.H_2O$ (PA, Germany), NH_4Cl (PA, Germany), NH_4OH (PA, Germany), distilled water, clean tap water.

2.2. Experiment

The fiberglass/polyamide composite sample used had dimensions of 100 × 20 × 2.5 mm, and the surface was prepared before chemical plating according to the following steps [1, 2]:

Table 1. *Technological steps for surface preparation before plating [1, 2].*

Technology step	Ingredients	Conditions
Degreasing	NaOH 15g/L, Na_2CO_3 20g/L, $Na_3PO_4.12H_2O$ 20g/L, Na_2SiO_3 10g/L	60 ºC, 15 ÷ 20 minutes
Create roughness	Al_2O_3 20 ÷ 40 µm	Spray pressure 8 atm, spray distance 10 ÷ 12 cm, spray angle 90°, speed 2 cm/s
Etching	$KMnO_4$ 130 g/L, NaOH 55 g/L	70 ºC, 10 minutes
Sensitization	$SnCl_2.2H_2O$ 50 g/L, HCl 65 ml/L, Sn 1÷ 2 g/L	Room temperature, 10 minutes
Activation	$PdCl_2.2H_2O$ 2 g/L, HCl 2 ml/L	Room temperature, 8 minute

Fix the composition of the plating solution (researched to optimize the composition) as follows: $NiCl_2.6H_2O$ 45 g/L; NH_4Cl 45 g/L; $Na_3C_6H_5O_7.H_2O$ 45 g/L; $NaH_2PO_2.H_2O$ 20g/L; $PbCrO_4$ 0,002 g/L; pH = 7,5 ÷ 9 [1, 2]. Plating temperature is carried out in various modes: 70 ºC, 75 ºC, 80 ºC, 85 ºC, 90 ºC. For each temperature regime, conduct a survey of the thickness, resistance of the plating layer and the stability of the plating solution at different time intervals of 15 minutes, 30 minutes, 45 minutes and 60 minutes. The ratio of plating area/solution volume in the fixed temperature survey is 2:1. In the surveys of the influence of plating area/solution volume ratio, the levels of 2:1; 2.5:1; 3:1; 3.5:1; 4:1 will be studied to select the optimal ratio. Finally, the optimal plating mode will be carried out to comprehensively check the technical parameters of the plating layer for verification.

2.3. Research methods

Using research methods to investigate the properties of the plating layer, including: mass weighing method on PA214 electronic analytical balance (USA); conductivity and resistance measurement method on DLRO-10 device (UK) according to ASTM B193-16; optical microscopy method on Axio Image A2M optical microscope (Germany) according to ASTM E3-11; scanning electron microscopy method SEM on JMS 6610LV-JED2300 device, JEOL, Japan; thermal shock resistance test method on Operon deep freeze cabinet (Korea) and drying oven (China). The thermal shock test cycle was carried out according to the technical acceptance conditions of the Russian Federation 9П516.00.00.000TY: ambient temperature from -(50 ± 3) ºC to +(50 ± 3) °C in 03 cycles, at each temperature kept for 4 hours.

3. RESULTS AND DISCUSSION

3.1. Effect of temperature and time on electroless nickel plating layer on fiberglass/polyamide composite surface

The effects of temperature and time on the thickness and resistance of the plating layer are shown in table 2. For the plating process carried out at 70 °C, no self-decomposition phenomenon was observed even when plating was carried out for a long time. This proves that the plating solution is very stable when plating at 70 °C. However, the plating speed is relatively slow, or in

other words, the plating efficiency is not high. After 45 minutes, the plating layer thickness only reached 4.632 μm, the resistance reached 1.024 Ω, and it took 60 minutes to reach the thickness of 6.041 μm. When plating at 75 °C, the plating solution is also very stable. In 45 minutes of plating, a plating layer of about 7.412 μm was created, and the resistance of the plating layer reached 0.108 Ω. The second batch was plated, adding chemicals and plating at this temperature, and the solution was still very stable. Similarly, plating at 80 °C, the plating speed is higher than that at 75 °C, and the solution is also quite stable. After 60 minutes of plating, the plating layer thickness reached 11.364 μm, and the plating layer resistance reached 0.086 Ω. After adding the second plating solution (the same content as when adding at 75 °C), the plating solution is still stable.

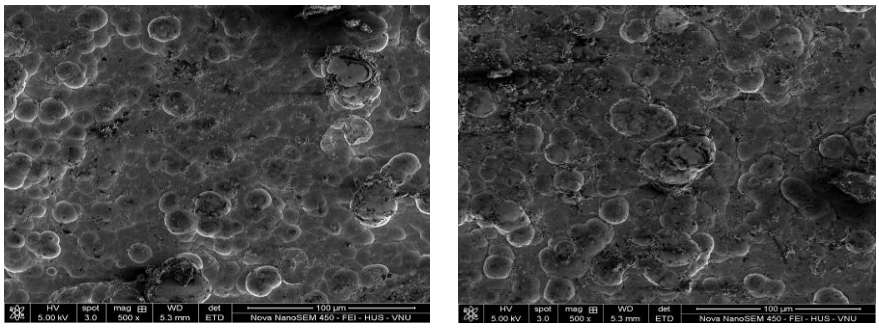
Table 2. *Thickness C (μm) and resistance T (Ω) of the chemical nickel plating layer at different times corresponding to different plating temperatures and times.*

Time / Temperature		15 minutes	30 minutes		45 minutes	60 minutes
70 °C	C (μm)	1.453	3.126		4.632	6.041
	T (Ω)	4.268	1.351		1.024	0.126
75 °C	C (μm)	2.452	5.061		7.412	9.874
	T (Ω)	3.112	0.826		0.108	0.098
80 °C	C (μm)	2.563	5.424		8.418	11.364
	T (Ω)	2.653	0.865		0.107	0.086
85 °C	C (μm)	2.865	3.926	Decomposes in 21 minutes		
	T (Ω)	1.857	1.223			
90 °C	C (μm)	2.942	3.631	Decomposes in 18 minutes		
	T (Ω)	1.386	1.215			

Investigation of the nickel plating process at 85 °C showed the following results: The plating speed is very fast, after only about 15 minutes, according to visual observation, the nickel plating layer covered the entire surface of the sample, the plating layer thickness reached 2.865 μm, and the resistance reached 1.857 Ω. After 21 minutes of plating, the solution showed signs of self-decomposition due to the phenomenon of H_2 gas release not only occurring on the product surface but also occurring in the entire solution. From the beginning of plating until the solution lost its effect, the thickness of the plating layer only reached 3.926 Ω. As for the nickel chemical plating process at 90 °C, H_2 gas release occurred in the first 15 minutes on the sample surface, the plating layer thickness reached 2.942 μm, and the resistance reached 1.386 Ω. However, after only 18 minutes of operation, the H_2 gas release process occurred throughout the entire solution, and it lost its ability to operate due to the self-decomposition reaction. After a total plating time of 18 minutes, the plating layer thickness only reached 3.631 μm.

Thus, it can be seen that when the plating temperature increased, the plating speed increased, but when it increased to 85 °C, the phenomenon of self-decomposition of the solution occurred. The suitable temperature for chemical nickel plating was from 75 ÷ 80 °C, the plating time to ensure the plating layer thickness ≥ 6 μm was greater than 45 minutes. The rate of deposition of the chemical nickel plating layer increases exponentially with temperature, so to achieve high plating speed, the higher the plating temperature, the better [1]. However, when Ni^{2+} is reduced to metal, hypophosphite is oxidized to phosphite and produces H_2 [4, 5]. Therefore, the self-decomposition ability of the solution also increases with increasing temperature. Therefore, it is necessary to control the temperature within a reasonable range to both achieve high plating speed and control the phenomenon of self-decomposition of the solution. The SEM images of the surface of the plated sample at 75 °C and 80 °C with the same time of 45 minutes are shown in Figure 1.

The SEM images show that the characteristic of the plating layer is the stacked crystal clusters. The surfaces of the two samples are different but quite smooth and similar [4, 5].



(a) *(b)*

Figure 1. *The SEM images of the surface of chemical nickel-plated samples at 75 °C (a) and 80 °C (b), time 45 minutes.*

3.2. Effect of plating area-to-solution volume ratio on chemical nickel plating layer on fiberglass/polyamide composite surface

The composition of the plating solution was fixed as presented in section 2.2, and the plating solution temperature was chosen as 75 °C. The ratio of plating area-to-solution volume (dm^2/Liter) was investigated at the levels of 2:1; 2.5:1; 3:1; 3.5:1; 4:1. The test results of plating layer thickness and plating layer resistance are shown in table 3.

Table 3. *Thickness C (μm) and resistance T (Ω) of the chemical nickel plating layer at different times corresponding to different plating solution area-to-volume ratios.*

Time \ Ratios		2:1	2.5:1	3:1	3.5:1		4:1	
15 minutes	C	2.105	2.074	2.035	1.863		1.712	
	T	3.831	3.973	3.984	4.125		4.156	
30 minutes	C	4.983	4.798	4.563	3.836		3.142	Decomposes in 26 minutes
	T	0.928	0.952	1.106	1.285		1.322	
45 minutes	C	7.236	7.194	7.068	5.647			
	T	0.181	0.202	0.231	0.762			
60 minutes	C	9.841	9.658	9.537	6.734	Decomposes in 54 minutes		
	T	0.097	0.116	0.121	0.125			

According to the results in table 3, when increasing the ratio of area-to-volume of plating solution (dm^2/Lit), after 15 minutes, the thickness of the plating layer formed on the sample surface is inversely proportional to the ratio of area-to-volume of solution, the ratio of 2:1 gave the largest thickness of 2.105 μm, the ratio of 4:1 gave the smallest thickness of 1.712 μm. Surveying the resistance of the plating layer after the first 15 minutes gave results proportional to the ratio of area-to-volume of solution, meaning that the higher this ratio, the greater the resistance.

For other time points, the results of the survey of the plating layer thickness and the plating layer resistance also have the same rule as at the 15-minute time point. However, in the case of the plating solution area/volume ratio of 4:1, after 26 minutes of operation, the solution has self-decomposed and lost its ability to function. By the time the solution had self-decomposed, the plating layer thickness had only reached 3.142 μm and the resistance had reached 1.322 Ω. With the technical requirement of plating layer thickness ≥ 6 μm, the case of the ratio of 4:1 does not meet the requirements. In another case, the ratio of 3.5:1 also self-decomposed but at a later time of 54 minutes. At this point, the plating layer reached a thickness of 6.734 μm, although it met the requirement of ≥ 6 μm, the plating solution had self-decomposed, so the plating efficiency was still too low. In cases where the area/volume ratio of the plating solution was from 2:1, 2.5:1 to 3:1 when plating the next batches after adding more solution, it still showed stability.

Thus, with the plating solution area-to-volume ratio from 2:1 to 3:1, the requirements for creating a plating layer ≥ 6 μm and plating layer resistance ≤ 1.5 Ω are met. However, if considering the same, applying a larger plating solution area-to-volume ratio will give a greater plating efficiency. Therefore, the ratio of 3:1 is the most optimal ratio when conducting chemical nickel plating for fiberglass/polyamide composites.

3.3. Plating test results corresponding to the most optimal technology mode

According to the survey results in sections 3.1 and 3.2, the most optimal technological mode when performing electroless nickel plating for fiberglass/polyamide composites is a temperature of 75 °C, a time of 45 minutes and a plating solution area-to-volume ratio of 3:1.

The plating process was carried out according to the optimal technological mode and checking the plating thickness, the results are shown in figure 2. As shown on the metallographic microscope image, the thickness of the chemical nickel plating layer was in the range of 7.63 ÷ 7.96 μm. The result of the plating thickness for this optimal mode ensured the requirement of ≥ 6 μm. Checking the plating resistance on the DLRO-10 device, the results showed that the resistance was in the range of 0.0957 ÷ 0.0960 Ω.

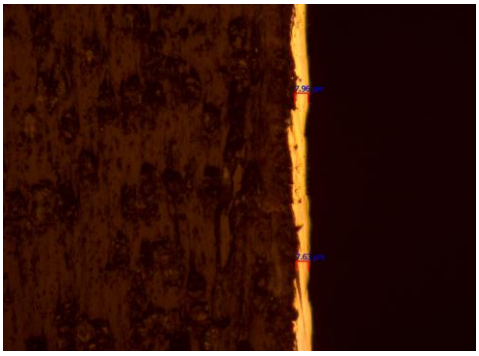
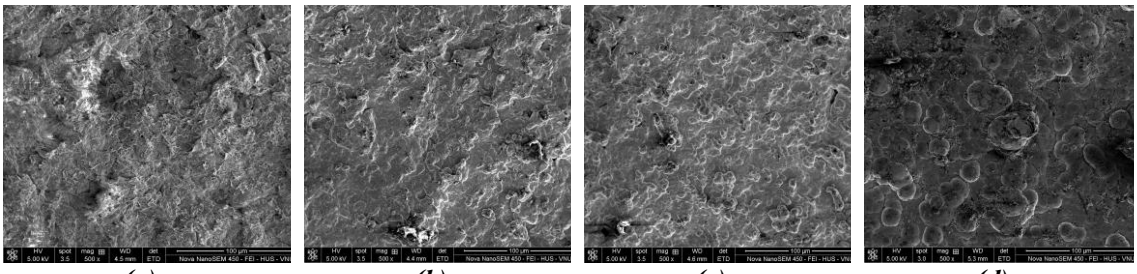


Figure 2. *Optical microscopy image of the cross-section of a plated polyamide composite sample.*

Examined by scanning electron microscopy (SEM) images at 15 minutes, 30 minutes and 45 minutes. The results are shown in figure 3.



(a) ***(b)*** ***(c)*** ***(d)***

Figure 3. *The SEM images of the surface of the nickel chemical plating layer before plating (a), after plating for 15 minutes (b), 30 minutes (c), 45 minutes (d).*

As observed in figure 3, the changes on the surface of the fiberglass/polyamide composite were quite obvious. After the first 15 minutes, the initial crystal nuclei appeared on the surface of figure 3b, but not much, so the surface morphology was still clearly similar to figure 3a. After 30 minutes, the nickel crystal nuclei were quite numerous and the surface morphology no longer copied the surface morphology of the composite but was replaced by a completely new surface morphology. The size of the nickel crystal nuclei was similar to that at 15 minutes. By 45 minutes, the number of nickel crystal nuclei stacked on top of each other forms a thick layer, at this time, the size of the crystal nuclei is larger than at 15 minutes and 35 minutes. The crystal nuclei were quite uniform

and smooth. This research result is similar to the publication of Zhiping Sun or Junjun Huang and colleagues when studying the effect of temperature on nickel coating when plating on poly(ethylene terephthalate) substrate surface, higher temperature has a larger average nickel crystal size [4, 5].

Thermal shock test with ambient temperature from -(50 ± 3) ºC to +(50 ± 3) ºC for 03 cycles, at each temperature held for 4 hours. The results showed that the plating layer did not peel off at any point on the surface.

4. CONCLUSIONS

Influencing factors such as temperature, time and area-to-volume ratio of plating solution directly affect the formation of the plating layer. The suitable temperature for chemical plating of nickel is in the range of 75 ÷ 80 °C. Plating time over 45 minutes ensures that the plating layer thickness will be ≥ 6 μm and the resistance ≤ 1.5 Ω. The optimal area/volume ratio of plating solution is in the range of 2.5:1 ÷ 3:1. Through thermal shock testing according to the standards of the Russian Federation, it was found that the plating layer surface did not peel off at any location compared to the time before the test.

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TÓM TẮT

Nghiên cứu ảnh hưởng của các thông số công nghệ đến quá trình hình thành lớp mạ hóa học niken trên composite nền polyamide cốt sợi thủy tinh

Qua quá trình nghiên cứu ảnh hưởng của nhiệt độ, thời gian và tỷ lệ diện tích mạ/thể tích dung dịch đến quá trình hình thành lớp mạ hóa học miken trên composite nền poliamide có thể rút ra kết luận rằng, nhiệt độ tối ưu từ 75 ÷ 80 °C; Thời gian mạ ngắn nhất là 45 phút để đảm bảo điện trở lớp mạ ≤ 1,5 Ω và chiều dày lớp mạ ≥ 6 μm; Tỷ lệ diện tích mạ/thể tích dung dịch tối ưu nằm trong khoảng 2,5/1 ÷ 3/1 (dm^2/L).

Từ khóa: Mạ hóa học niken; Composite nền poyamide cốt sợi thủy tinh.