Fabrication of lead-plated copper negative grid for high-capacity lead-acid battery

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ABSTRACT

This paper presents the results of an investigation into the properties of a lead-plated layer on the negative electrode grid of a foreign-manufactured high-capacity lead-acid battery. The material composition, the plating layer's thickness and structural morphology were all ascertained using SEM and EDX analysis techniques. This analysis served as a benchmark for developing a lead plating technology on expanded copper mesh using a lead fluoroborate bath. The findings have provided a technological regime for producing a lead plating layer using the electrolytic plating method that satisfies the structural morphology and thickness technical requirements of the foreign-made grid sample.

Keywords: Lead-acid battery; Lead plating; Fluoroborate plating solution; Copper mesh plating.

1. INTRODUCTION

Lead-acid batteries are widely used as power sources with various voltages, sizes, and weights, serving numerous applications in daily life and industry, such as lighting, starting engines, powering transport vehicles, providing backup power, storing solar and wind energy, supplying energy for submarines when submerged, and acting as emergency power sources on nuclear vessels, etc. [1, 2]. With hundreds of millions of units produced worldwide each year, lead-acid batteries still hold the largest market share among all electrical energy storage devices today. This is attributed to their superior characteristics, such as stable manufacturing technology, readily available raw materials, high recyclability (over 95%), and a cost per unit of electrical energy ($/Wh) that is 2 to 3 times cheaper than other common batteries. However, the drawbacks of lead-acid batteries include low material utilization efficiency and a relatively short lifespan. Therefore, research has focused on increasing energy density and extending battery life. These are the major directions in the development of lead-acid battery technology [4, 5]. Typically, a lead grid is used as the electrode framework in commercial lead-acid batteries. The lead grid serves as both a support frame for the active material paste and a current collector. It significantly impacts the total weight of the battery, the charge-discharge efficiency and the battery lifespan [9, 10]. Due to the high specific gravity of lead (11.3 g/cm^3), lead-acid batteries are heavy, resulting in a low energy density (30–50 Wh/kg). This energy density is much lower than that of Lithium-ion batteries (100–300 Wh/kg) [5]. Consequently, research has focused on replacing lead grid with lighter materials, aiming to reduce weight and increase energy density. Various materials such as lead-antimony alloys, lead-calcium alloys, lead-calcium-aluminum alloys, polymer meshes, aluminum alloy meshes, and lead-plated copper meshes have been investigated. Compared to conventional lead mesh, lighter-than-lead materials have shown some improvement in mechanical strength, energy density, and internal resistance [1, 3, 5, 6]. This study was conducted with the aim of creating a lead plating layer on a copper mesh that was of comparable quality to the lead plating layer on the negative electrode grid sample from a foreign-manufactured high-capacity lead-acid battery.

2. EXPERIMENTAL

2.1. Chemicals, materials, and equipment

The primary chemicals used in the experiments included: $Pb(OH)_2.2PbCO_3$; Na_2CO_3; NaOH;

Na_3PO_4; Na_2SiO_3; H_2SO_4 (d = 1.84 g/cm^3); HNO_3; H_3BO_3; HF; Gelatin (PA grade, China); HBF_4 was synthesized from H_3BO_3 and HF; $Pb(BF_4)_2$ salt was synthesized from $Pb(OH)_2.2PbCO_3$ and HBF_4; Lead electrodes (Vietnam). The copper mesh used for the study was an expanded mesh of M1 grade copper alloy (South Korea) with a thickness of 0.5 mm, with geometric size as described in figure 1. Equipment included a 12V-10A DC rectifier and a magnetic stirrer with a hotplate. The reference negative electrode grid was taken from a foreign-manufactured high-capacity battery.

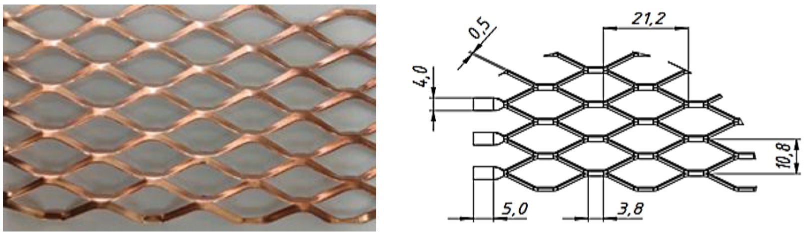


Figure 1. *The expanded copper mesh used for the negative grid and its geometric size.*

2.2. Sample preparation and research methods

2.2.1. Sample preparation

The copper mesh sheets were cut to a size of 100 x 50 mm and degreased in a solution containing 30 g/L NaOH, 25 g/L Na_2CO_3, 50 g/L Na_3PO_4, and 5 g/L Na_2SiO_3 at a temperature of 60 °C for 5 minutes. Afterward, the sheets were rinsed with water and activated in a 20% H_2SO_4 solution at room temperature for 1 minute. The lead plating process was performed in an electroplating bath with the composition of 450 g/L $Pb(BF_4)_2$, 30 g/L free HBF_4, 20 g/L free H_3BO_3, and 1 g/L Gelatin, at room temperature. The current density was varied from 1 to 5 A/dm^2, and the plating time was varied from 20 to 240 minutes.

2.2.2. Analysis methods

The thickness of the plating layer was determined using an Axiovert 40MAT equipment in accordance with the ASTM B487-20 standard. The material composition was evaluated using Energy-Dispersive X-ray Spectroscopy (EDX). The surface morphology of the plated layer was examined using Scanning Electron Microscopy (SEM) on a JEOL JSM-6490 device.

3. RESULTS AND DISCUSSION

3.1. Analysis of the foreign-manufactured negative electrode grid

3.1.1. Chemical composition of the grid

A negative electrode grid sample from a foreign-manufactured high-capacity lead-acid battery was analyzed to determine the base material and outer coating composition. The EDX analysis of the grid's base material revealed copper (Cu) peaks (figure 2), whereas the outer layer showed lead (Pb) peaks (figure 3). This indicates that the negative electrode grid of this kind of battery is made of copper mesh that has been covered with a layer of metallic lead.

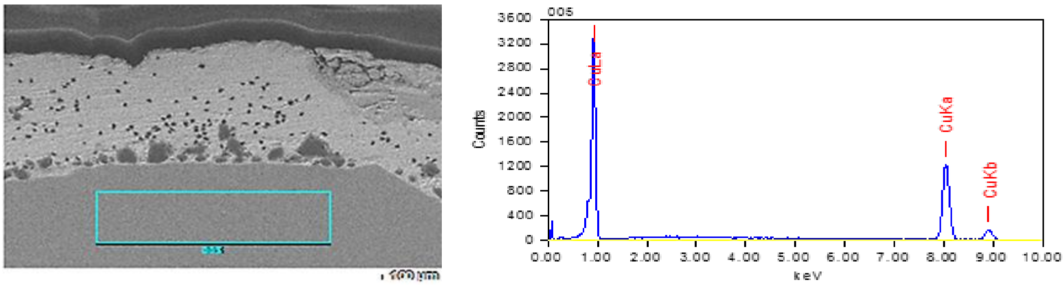


Figure 2. *EDX analysis results of the base material of the negative electrode grid in a commercial lead-acid battery.*

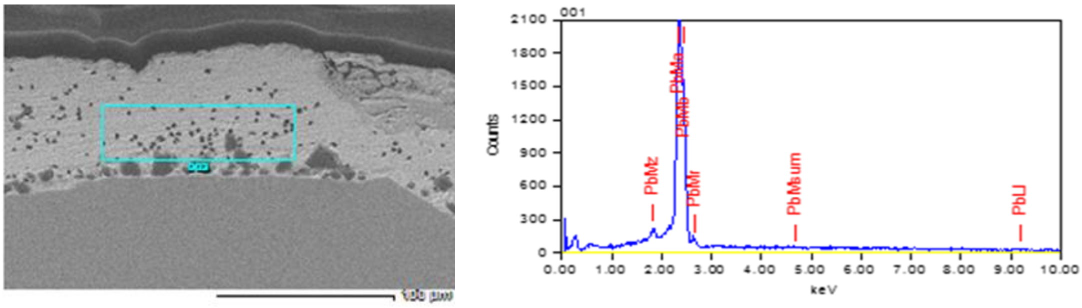


Figure 3. *EDX analysis of the outer coating of the negative electrode grid in a commercial lead-acid battery.*

3.1.2. Plating thickness

Since the electrode grid of a lead-acid battery operates long-term in a highly corrosive sulfuric acid electrolyte, the copper mesh must be protected with a lead layer. The thickness of this lead coating is crucial for protecting the grid, enhancing the battery's lifespan and ensuring stable operation over time. The thickness of the lead plating on the copper mesh of a negative grid sample in the foreign-manufactured battery was examined. Measurements were taken at two locations: the inner mesh area and the outer edge of the grid (figure 4). The analysis results are presented in figures 5 and 6, and tables 1 and 2.

The results in table 1 showed that at the outer edge of the grid, the average plating thickness was 95.31 µm; at the inner area of the grid, the average thickness was 54.06 µm. Since the outer edge constitutes a very small fraction of the total surface area, the thickness of the inner plating is the critical baseline for determining the required lead coating thickness for corrosion protection. A lead plating thickness of 20÷30 µm on a copper mesh grid was sufficient for protection and increased durability, but may not guarantee longevity for long-term use (over 10 years) [4]. For aluminum grids, a lead plating thickness of about 40 µm was suggested [5]. For polymer-based grids, a conductive metal layer of 15 µm and a protective lead layer not exceeding 100 µm were used [3].

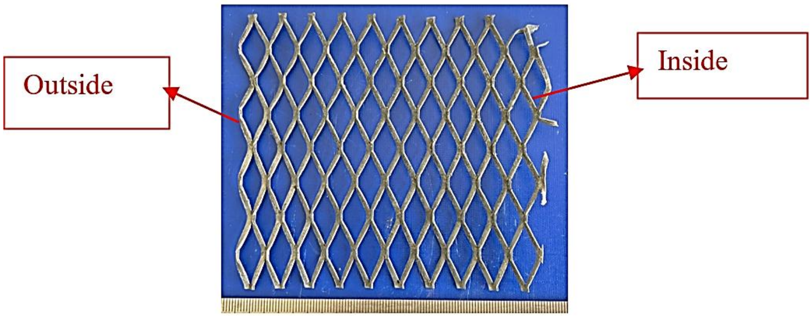


Figure 4. *The electrode grid sample and the locations for analysis.*

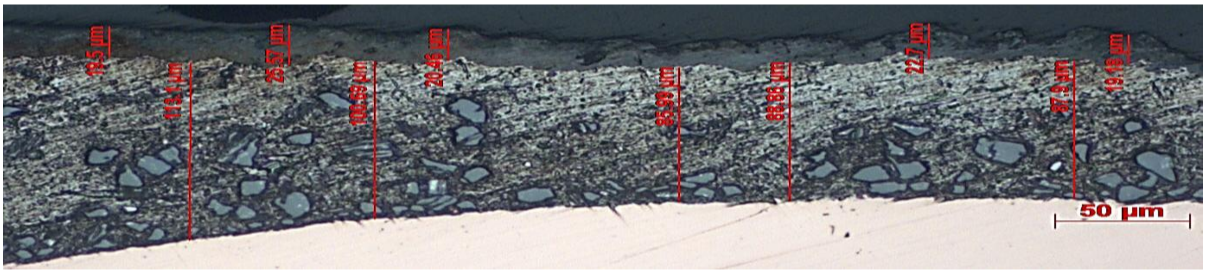


Figure 5. *Cross-sectional micrograph for measuring the thickness of the lead coating at the outer region of the sample, 200x.*

Table 1. *Thickness measurement results of the plating layer at the outer region of the sample, in µm.*

Measurement No.	1	2	3	4	5	Average value
Plating layer thickness, µm	113.10	100.69	85.99	88.86	87.90	95.31

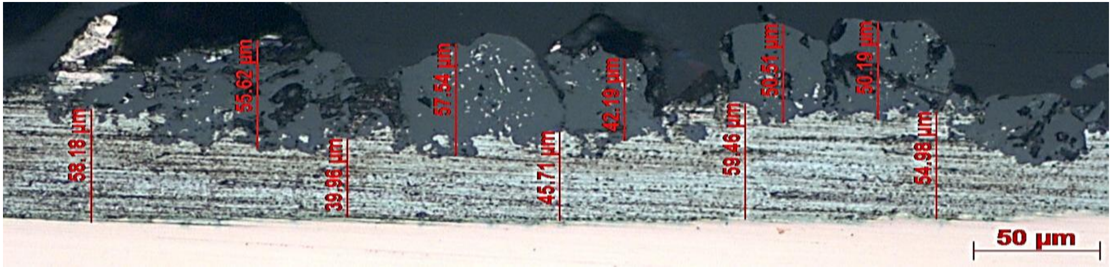


Figure 6. *Cross-sectional micrograph for measuring the thickness of the plating layer at the inner region of the sample, 200x.*

Table 2. *Thickness measurement results of the plating layer at the inner region of the sample, in µm.*

Measurement No.	1	2	3	4	5	Average value
Plating layer thickness, µm	58.18	49.96	45.71	59.46	54.98	54.06

3.1.3. Morphology of the plating layer

In addition to sufficient thickness, the structure of the lead plating is also critical for protecting the copper grid. A dense, fine-grained structure with no pinholes or defects is required. Figure 7 shows an SEM image of the lead-plated surface on the foreign-manufactured grid sample. At 500x magnification, the surface appeared highly uniform, dense, and free of any pores, cracks, or pinholes. This ensures excellent corrosion protection for the copper grid in the sulfuric acid environment over a long period.

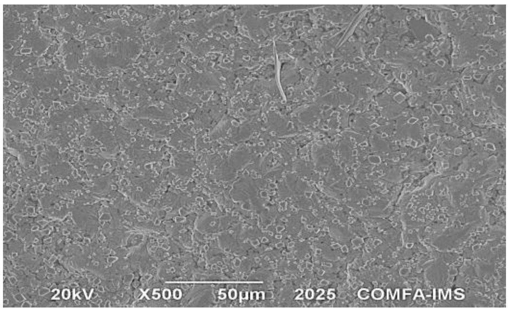


Figure 7. *SEM surface micrograph of the lead (Pb) coating on the foreign-manufactured negative electrode grid.*

3.2. Study of lead coating on copper mesh via electroplating

The analysis of the negative electrode grid's properties of a foreign-manufactured high-capacity lead-acid battery indicated that the lead coating on the copper mesh must meet specific criteria to ensure the grid's durability. Among the most critical properties of the lead coating are its thickness and microstructure. These properties are dependent on the plating solution's composition and the process operating conditions. Within the scope of this paper, using a lead fluoroborate solution with a pre-determined composition (as described in section 2.2.1), this study focuses on investigating two key parameters: current density and plating time, in order to achieve a lead coating on copper mesh with quality comparable to the foreign-manufactured benchmark.

3.2.1. Effect of current density on the coating properties

Plating experiments were conducted at varying current densities from 1 to 5 A/dm^2 for a fixed time of 120 minutes at room temperature. At low current densities (1÷2 A/dm^2), the lead coating was matte and not bright, but the surface was uniform and dense, with a thickness of less than 30 µm. When the current density increased to 3 ÷ 4 A/dm^2, the coating became bright, uniform, and of good quality, with a thickness exceeding 55 µm. Further increasing the current density to 5 A/dm^2 resulted in a rough coating with dendritic growth at the edges and a non-uniform surface. SEM images of the surfaces plated at different current densities are shown in figure 8.

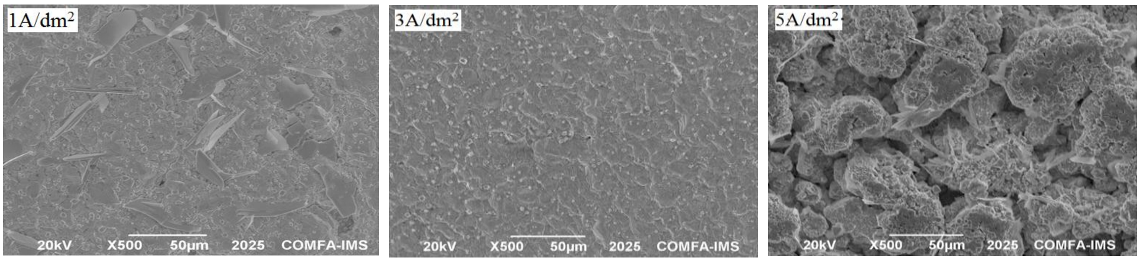


Figure 8. *SEM images of the lead coatings on the copper mesh at different current densities.*

The SEM images (500x magnification, figure 8) showed that at a low current density of 1 A/dm^2, the deposition rate of Pb^{2+} ions was slow, resulting in an uneven surface with poor leveling. Increasing the current density to 3 A/dm^2 improved the leveling of the crystalline layer, resulting in a more uniform and brighter surface. At 5 A/dm^2, the deposition rate was much faster, leading to rapid crystal growth. This resulted in larger crystals, a more porous layer, and a non-uniform surface with rough and dendritic forms at the corners and edges. Therefore, to obtain a lead plating layer with sufficient thickness and a satisfactory surface, a current density of 3÷4 A/dm^2 should be used.

3.2.2. Effect of plating time on the coating's structure and thickness

Experiments were conducted at a fixed current density of 3 A/dm^2 with plating times varying from 20 to 240 minutes. The quality of the lead coating over time is shown in table 3. After 20 minutes, the coating was matte and gray. Increasing the time from 30 to 150 minutes produced a smooth, even, and bright gray coating, indicating stable growth. After 240 minutes of plating, the surface became rough, with dendritic growth at the edges of the plated sample.

Table 3. *Effect of plating time on the quality of the lead coating.*

No.	Plating Time (min)	Coating quality
1	20	Dull, gray coating
3	30	Even, smooth, bright coating
4	60	Even, smooth, bright coating
5	90	Even, smooth, bright coating
6	120	Even, smooth, bright coating
7	150	Even, smooth, bright coating
8	240	Rough coating with dendrites at the edge

SEM images of the platings' surfaces at different plating times (figure 9) revealed changes in the coating structure. At short durations, the layer was thin and not yet fully dense or uniform. In the range of 60 to 150 minutes, the coatings exhibited a very uniform, dense, smooth, and pore-free structure with excellent leveling, comparable to the foreign-manufactured sample. At the plating time of 240 minutes, the surface became rough and porous, which is detrimental to protecting the grid in the sulfuric acid environment. Therefore, to achieve a high-quality coating, the plating time should be between 30 and 150 minutes.

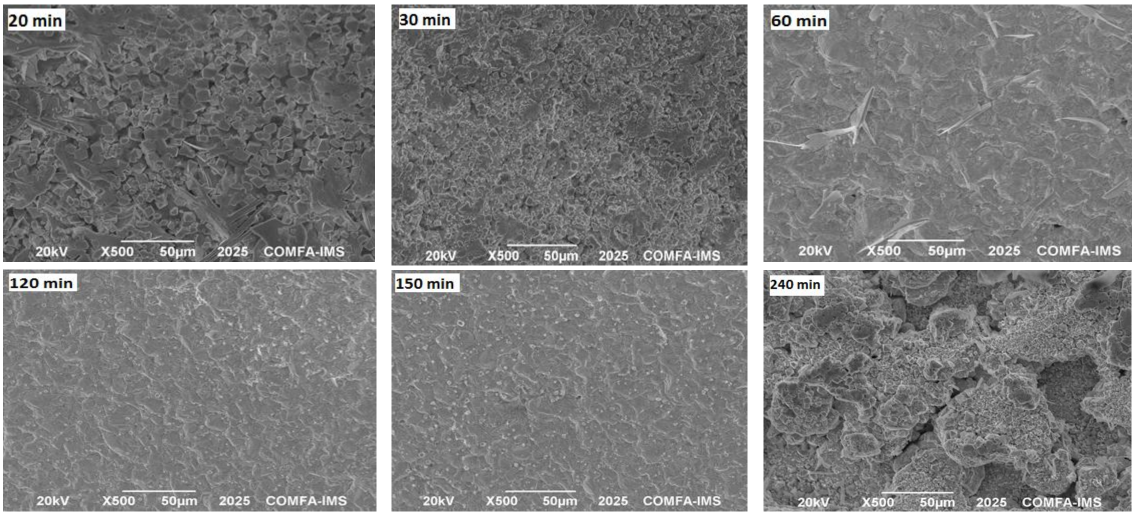
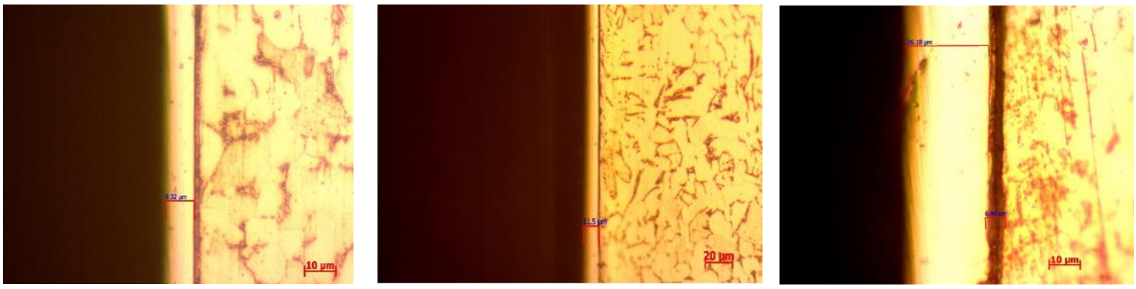


Figure 9. *SEM images of the lead coatings on the copper mesh at different plating times.*

The thickness of the coating on the inner mesh area at different plating times is shown in figure 10. After 120 minutes of plating, the lead coating reached a thickness of 55.08 µm, which is equivalent to the thickness of the lead coating on the foreign-manufactured sample.



Plating time: 20 min (thickness: 9.32 µm). *Plating time: 30 min (thickness: 11.5 µm).* *Plating time: 60 min (thickness: 25.18 µm).*

Plating time: 120 min (thickness: 55.08 µm). *Plating time: 150 min (thickness: 65.79 µm).* *Plating time: 240 min (thickness: 113.39 µm).*

Figure 10. *The thickness of the lead coatings on the copper mesh at different plating times.*

4. CONCLUSIONS

A lead plating layer was successfully created on expanded copper mesh using a plating bath composed of 450 g/L $Pb(BF_4)_2$, 30 g/L free HBF_4, 20 g/L free H_3BO_3, and 1 g/L Gelatin. Under a plating regime with a current density of 3 A/dm^2, 120 minutes of plating, a lead coating with a thickness of 55.08 µm was achieved. The lead coating on the copper grid exhibited a uniform, dense, and pore-free structure. The quality of this lead plating is comparable to that of the coating on the negative electrode grid sample of the foreign-manufactured high-capacity lead-acid battery. This lays the groundwork for the production of high-capacity lead-acid batteries domestically, which could eventually replace imports.

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TÓM TẮT

Nghiên cứu công nghệ tạo lớp mạ chì lên lưới đồng sử dụng làm sườn điện cực cho ắc quy chì axit dung lượng cao

Bài báo trình bày kết quả nghiên cứu khảo sát một số tính chất của lớp mạ chì trên sườn cực âm của ắc quy chì axit dung lượng cao do nước ngoài chế tạo. Đã sử dụng các phương pháp phân tích SEM, EDX để xác định thành phần vật liệu sườn cực, thành phần vật liệu của lớp mạ, chiều dày lớp mạ và hình thái cấu trúc của lớp mạ. Từ đó định hướng nghiên cứu công nghệ mạ chì lên tấm lưới đồng trong dung dịch mạ chì floborat. Kết quả đã xác lập được chế độ công nghệ tạo lớp mạ chì bằng phương pháp mạ điện phân đạt các chỉ tiêu kỹ thuật về chiều dày và hình thái cấu trúc tương đương với mẫu sườn cực do nước ngoài chế tạo.

Keywords: Ắc quy chì axit; Mạ chì; Dung dịch mạ floborat; Mạ lưới đồng.