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Investigating electrodes for the extraction and utilisation of copper from spent etching solutions

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ABSTRACT

Today, reagent-based technologies for metal removal from water often fail to achieve the necessary purification efficiency for reuse and result in the accumulation of toxic sludge. This article aimed to test a regenerator for restoring the properties of etching solutions used in printed circuit boards, evaluate the performance of electrodes in depositing dense copper precipitates during etching solution regeneration, and explore the use of recovered copper in repair processes. During the study, an electrochemical process was employed to deposit a copper layer onto the regeneration unit's electrodes, along with mechanical testing of the electrodes for separating the removed product from the spent solution. This article investigated the electrochemical regeneration of etching solutions in the printed circuit board etching process, leading to the formation of dense copper deposits on electrodes (regenerators) made of copper, stainless steel, and titanium. The mechanical separation of the recovered copper material reveals that titanium cathodes yield superior results, making titanium the preferred electrode material for regenerator design. Additionally, the findings confirm that the copper deposited on titanium cathodes meets the quality standards required for reuse after remelting in the electrical engineering industry. The copper recovered from spent etching solutions, when combined with a bronze coating applied to the copper layer, improves adhesion and can be used in repair work. Additionally, it enhances the antifriction properties of the friction pair. The article identified suitable materials for use as regenerator electrodes in removing copper from spent etching solutions. It also examined the correlation between electrode surface finish quality and the detachment force of copper deposits in the regenerator, considering the surface roughness of titanium electrodes. The proposed methods and regeneration scheme for the used etching solution enable the creation of integrated equipment that incorporates a regeneration unit, with the primary component being a system (electrolyser) where the copper extracted from the used etching solution will be deposited onto the designated electrode. This study facilitates the development of a technological process for recycling aqueous etching solutions, helping to minimise environmental impact.

Keywords: regeneration, etching solution, titanium, copper, cathode, dense deposits.

INTRODUCTION

The use of electroplating in today's manufacturing carries the risk of the accidents related to waste storage and the direct execution of technological processes. Various factors, including technical, technological, and organisational others, contribute to the development of unfavourable situations. Storage of waste and technological processes can be hazardous to the environment, affecting humans, and wildlife fauna. At the same time, deploying waste in the production of construction materials is not always sensible or economical [1]. Discharging and storing spent etching solutions, also in the form of sludge, on the premises of enterprises leads to environmental pollution and requires significant expenses for their maintenance and disposal [2, 3].

In turn, environmental pollution worsens the condition of both surface and underground water horizons [4–6]. Calculations and practical experience in etching substrates (printed circuit board bases) show that up to 0.5 kg of copper can be dissolved into the solution from 1 m² of material. A modern etching line uses 100 litres of etching solution to process 10 kg of copper. This leads to accumulating up to hundreds of tons of waste annually at enterprises, in the form of water-containing substances [17, 18]. Further transfer of pollutants occurs through food chains, with humans at the top of these chains [7–10]. Pollution from industrial buildings and residential rubbish damages the environment and causes climate change [11, 12]. Storing etching solution waste as sludge in unprepared containers on company premises results in negative consequences. Over time, the land beneath these facilities and the surrounding environment will become unusable for decades [17, 18].

Chemical adjustment of etching solutions, a traditional method, generates large volumes of wastewater containing heavy metals. These pollutants pose serious risks to soil, groundwater, plant ecosystems, and humans, who occupy the top of the food chain [13, 15, 16]. This section outlines the task of testing the regeneration unit for recovering the etching solution, focusing on extracting etched copper as dense precipitates. It aims to explore and identify suitable electrode materials for this purpose as well as significantly reduce the amount of sludge produced and stored on company premises. This will contribute to improving the environmental conditions in the areas where printed circuit boards are produced.

ANALYSIS OF RECENT RESEARCH AND PUBLICATIONS

A detailed review of patent and technical literature demonstrates that addressing the problem of spent solutions involves transitioning to a production process based on a closed cycle in a single technological operation [17, 19, 20]. However, to solve this complex problem, it is vital to investigate the material and design of the electrode system in regenerators. For example, the alkaline etching line for printed circuit boards includes loading, etching, lighting, rinsing, monitoring, hot drying and unloading modules, equipped with a regeneration module connected by a pipeline system to the etching module, and a control system [17]. In other words, it is a complex of equipment united into a single system. The key component of such a line with the restoration of the etching property of the spent solution is the regenerator, in which the main working part consists of electrodes, and the removal of copper in the form of powders or dense sediments depends on their operation [17, 21]. There have been few works describing the electrode system of regeneration units. Still, some works address the problems of metal extraction simultaneously with the regeneration of solutions [22]. In recent years, there have been studies focused on electrode management, examining the relationship between electrode consumption and current density in various solutions [21].

Currently, there is a lack of research related to the process of extracting solid sediments and their suitability for further use after industrial processing. Existing equipment for electrochemical regeneration of iron-copper-chloride etching solutions for printed circuit boards ensures stable and productive operation. Nevertheless, it only provides copper extraction in a powdered form, which is a significant drawback, since it can lead to short-circuits of the electrode system due to the accumulation of copper powder sediment in the lower part of the regeneration unit [17]. Furthermore, it is essential to note the increased entrainment of the etching solution from the regeneration unit during the recovery operation of etching properties (in conditions of copper powder extraction). According to the authors' research, this entrainment can exceed 5–6 times the solution entrainment from the boards and can reach values of 0.3 l/m².

The above highlights that the research aimed at developing a regeneration technology for extracting dense copper precipitates from used etching solutions in printed circuit board manufacturing is not being conducted by either companies or research organisations [17, 18]. However, addressing this issue presents researchers with significant challenges. These challenges include analysing the composition of the solution suitable for regeneration while ensuring the required etching speeds and identifying as well as studying the electrode materials and the cleanliness of their surfaces.

Thus, the article sought to present the results of research and tests on the electrodes for the recovery of dense copper sediments during the regeneration of etching solutions and create environmentally friendly equipment. This equipment aims to significantly reduce the discharge of spent solutions into wastewater treatment plants and waste storage sites (landfills) [17, 23].

MATERIALS AND RESEARCH METHODS

The research was conducted using an alkaline ammonia copper chloride solution, used in the etching of printed circuit board blanks. The process of purifying wastewater from copper ions and the reuse of the aqueous solution (regeneration) were carried out on an electrolyser (regenerator) with electrodes made of copper grade M1, stainless steel 12CrI8Ni9Ti and titanium grade VT1-0 with a thickness of 3 mm.

The schematic diagram of the installation (the technological scheme of the aqueous solution regeneration device) used for the research is presented in Fig.1. The scheme operates as follows: A pipe system connects the etching machine to the electrolyser (regenerator) 1. An ejector 2 is connected to the regenerator, which, along with pump 3, controls the pH of the solution within the required limits to maintain a constant etching capacity of the regenerated solution. The solution is subsequently pumped to the etching machine using pump 4.

A significant drawback of the etching and regeneration scheme is the possibility of ammonia gas release at the anodes and the lack of a specific device to extract this release and use it for potential oxidation of the regenerated solution. To avoid such an effect, the possibilities of increasing the surface area of the anodes were considered, which could be achieved by using carbon fibre materials. However, due to the high current loads required for addressing regeneration problems and a significant increase in current-carrying capacity issues, this solution was deemed difficult to implement under industrial conditions. The problem has

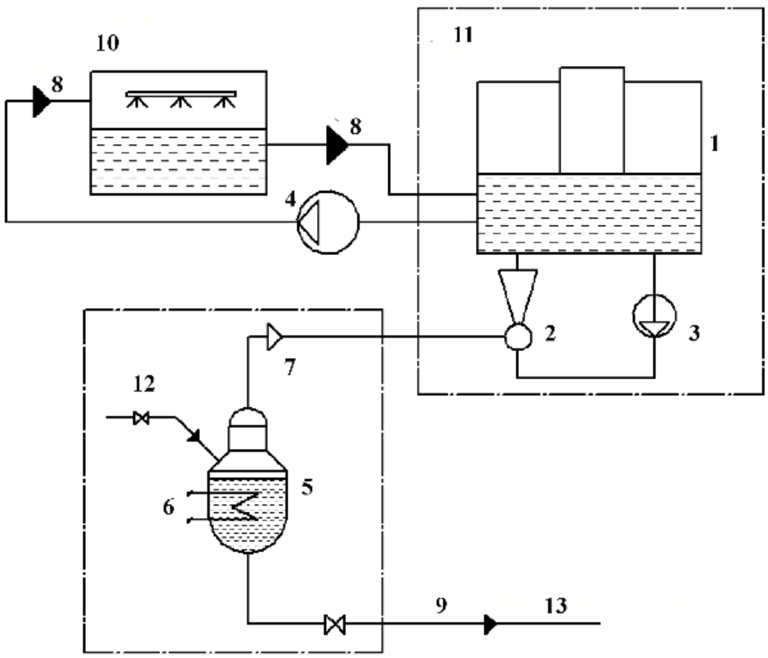

Figure 1. The technological scheme of the aqueous solution regeneration device: 1 – electrolyser; 2 – ejector; 3,4 – pump; 5 – capacity; 6 – electric heater; 7 – electromagnetic valve; 8, 9 – stop valves; 10 – etching machine; 11 – regenerator; 12 – inlet pipe of 25% ammonia solution; 13 – inlet pipe for supplying a 5% ammonia solution to the rinsingmodule

been solved by adjusting the solution composition (optimised using factorial experimental methods) for etching while maintaining the desired etching rate and regeneration limits to obtain dense copper precipitates.

For the proposed regeneration scheme of the spent etching solution, only solid electrodes were used in the industry. The most widely used electrodes for similar purposes are platinum and graphite. Therefore, within this research, they served as a basis for comparison. In an attempt to replace expensive platinum, the possibility of using a titanium electrode for analysis was investigated. The corrosion behaviour of titanium in the etching solution was studied beforehand. A sample of titanium wire with a length of 100 mm and a diameter of 0.7 mm (0.2104 g) showed no change in mass after 115 hours of exposure to the etching solution when weighed on analytical scales. This allowed using a titanium electrode in the etching solution as a working electrode for the research.

Titanium offers high specific strength and good plasticity, even though its electrical and thermal conductivity are quite low. It has excellent corrosion resistance, nearly as good as platinum, due to the formation of a passive dense film on its surface. Steel grade 12CrI8Ni9Ti is resistant to corrosion and chemically active compounds, boasts high mechanical strength, and is easy to machine.

Copper grade M1 is distinguished by its exceptional plasticity, corrosion resistance, and high thermal and electrical conductivity.

The electrodes (cathodes) used in this research were made of copper (M1 grade), stainless steel 12CrI8Ni9Ti (this steel is quite resistant to the aggressive action of etching solutions) and titanium alloy VT1-0 with a thickness of 3 mm. Some disadvantages of 12CrI8Ni9Ti steel include relatively low hardness (HV 140–170 after hardening) and high viscosity, which complicates its mechanical processing. Titanium alloys are high-strength materials at normal and elevated temperatures and have a stable oxide film on their surface, which serves as a protective barrier. Graphite electrodes were used as the cathodes.

The printed circuit board mechanically supports and electrically connects electronic components using conductive traces, contact pads and other elements etched from copper sheets laminated onto a non-conductive substrate. In the etching machine, copper is etched on the substrate following a specific pattern. During the etching process of printed circuit boards, the etching solution becomes saturated with copper compounds, which can reduce the etching speed of subsequent boards and result in incomplete etching of the components on those boards. After fulfilling its primary function, the spent etching solution is directed to the regenerator, where the etching solution is restored, and copper is recovered on the cathodes. The pH of the solution is adjusted in the regenerator. After restoring the etching characteristics, the solution is sent back to the etching machine. The cathodes in the regenerator are made from copper (M1 grade), stainless steel 12CrI8Ni9Ti or titanium alloy VT1-0 with a thickness of 3 mm. They are installed in the regenerator to determine the best characteristics of copper precipitation in dense layers and to find the ways to remove the copper sediment material and weigh it.

The research on the regeneration system with copper, steel and titanium cathodes was conducted in the following manner:

1. Preparing the working surface of the cathode and ensuring its cleanliness.
2. Preparing the working surface of the anode.
3. Weighing the cathode plate before and after the electrolysis.
4. Separating the dense copper sediment from the cathode plate.
5. Determining the quantity and quality of the dense copper sediment on the cathodes of the regeneration system.
6. Determining the yield of dense copper sediment based on the current.
7. Determining and assessing the etching rate of substrate samples while simultaneously regenerating the etching solution.

The operation of the installation was carried out under the conditions described below. Control weighings of the cathode were conducted every 30 minutes. To perform this operation, the working surface of the cathode was cleaned with a soft brush to remove excess elements, and it was weighed to determine the quantity of dense copper sediment and calculate the yield based on the current. Subsequent research on the physical and mechanical properties of the electroplated coatings was conducted using the following methodology. The research was conducted under the following conditions:

- substrates for printed circuit boards: copper-plated fibreglass (FR-4) laminates;
- etching rate for printed circuit board substrates: 25–35 µm/min;

- flow rate of the spent etching solution: 1.5 cm/s;
- temperature of the solution within the range of 35–40 °C (but not exceeding 40 °C);
- cathode current density for direct current: 15–19 A/dm².

Quality control of the obtained copper (foil) was conducted based on the following parameters: appearance, thickness and porosity. If the thickness of the deposited copper was less than 50 µm, the porosity of the foil was determined by applying a filter paper method. Filter paper, sized 2 × 2 cm, was placed on the surface of the copper deposit that was still attached to the corresponding base. The filter paper was soaked in a solution containing the following components per litre: $K_3Fe(CN)_6$ – 10 g and NaCl – 5 g. Care was taken to ensure no air bubbles were trapped between the copper surface and the paper. The paper was left in contact with the copper deposit for 20 minutes. The presence of pores reaching the base resulted in the formation of stains on the filter paper. Papers with imprints of pores in the form of dots and stains were removed, rinsed with distilled water and cleansurfaces were divided into square centimetres. The number of pores was counted separately in each square, and the results were averaged. The authors also conducted measurements of the foil thickness. The obtained results allowed them to assess the quality of the copper foil (its appearance, uniformity in thickness, its adhesion to the form), calculate the copper yield by current, the rate of copper foil growth and the specific electrical energy consumption. The copper thickness was measured using a micrometre with an accuracy of 0.01 mm. However, this level of precision is not essential, as the extracted copper is intended for melting and repurposing as an electrical material. The average thickness of the metal deposit was calculated using the formula below [21]:

$$\delta = \frac{\tau \cdot B_c \cdot K_e \cdot i_c}{d_m \cdot 10^2} \quad (1)$$

where: τ – the duration of regeneration (electro deposition), hrs; d_m – density of the metal coating, g/cm³; B_c – cathodic current efficiency (as a fraction); k_e – the electrochemical equivalent of the metal (copper) coating, g/(A·hr); i_c – average cathodic current density, A/dm².

One of the negative phenomena that limits the maximum working current density and leads to uneven metal deposition on the cathode's surface is the non-uniformity in the distribution of current density across the cathode's surface. Avoiding this phenomenon during the growth of significant metal layers is an important technological challenge, because current density localised in creases can lead to the formation of coarse crystalline dendritic or dispersed metal deposits. The formation of such deposits is undesirable due to the reduction in the overall purity of the cathodic metal. Additionally, the formation of dendrites can lead to short circuits between the cathode and anode, increasing unproductive metal losses.

In the regeneration unit (bath), the current density across the cathode surface can be non-uniform, both vertically and horizontally. The non-uniform distribution of current density horizontally is mainly caused by design flaws in the electrolyser. The reasons for the non-uniformity of vertical current density distribution and, consequently, metal deposition are as follows:
- stratification of the working solution by concentration in the vertical direction;
- possible non-uniform distribution of potential in the height of large-sized forms due to the ohmic resistance of the conductive layer material;
- the plating and electrochemical deposition of copper on the cathodes were conducted under the same conditions for all three types of electrodes: copper, stainless steeland titanium.

RESULTS AND THEIR DISCUSSION

There is a lack of comprehensive studies aimed at significantly reducing the amount of waste (sludge) stored on company premises. This article addresses this issue by examining the solution and electrodes, leading to the extraction of copper from the used etching solution as a dense layer.

Consequently, the authors found that the surface of the deposited copper was clean and free from electrolyte residues, and there were no dendritic or porous copper deposits. This indicates the purity of the copper coating on the cathode, which can be used as an electrical material after re-melting in production and for processes related to copper layer deposition.

The separation of the copper layer (foil) was carried out after using copper, steel and titanium as electrodes with identical geometrical dimensions. It appeared that it was practically impossible to detach the deposited copper from a copper electrode through simple methods due to the bonding process. As for the steel electrode, due to the significant adhesion between steel and copper, such an electrode cannot be proposed for use. A copper layer on a steel electrode provides adhesion strength of up to 30 MPa. The best electrode that allows the detachment of deposited copper from the spent solution is titanium. It was noted that the detachment effort was lower the cleaner the surface of the titanium electrode, as shown in Table 1.

The separation of the extracted copper raw material revealed that the cathodes made of titanium VT1-0 produce better results (during manual bending of the cathode plate, the copper is easily separated, simply peeling off due to internal stresses), which identifies titanium as the primary element for use in the regenerator design. For broader utilisation of the extracted copper from the spent etching solution, additional research was carried out on the copper coating of a component in a vehicle (trolleybus). In particular, the research was conducted on the coating of the pivot bearing socket of the trolleybus, a crucial component that enables the continuous manoeuvring of the vehicle in the required direction. The intensive wear of the bearing socket (with wear levels reaching 20–25 μm) is caused by substantial friction forces and elevated temperatures. Components with such wear are typically restored, and plasma spraying with bronze powder is employed for this purpose.

This coating method is quite complex and labour intensive within the production process. The procedure is carried out according to the following steps:

1. Surface activation and the creation of significant roughness (mechanical processing to apply a "torn" thread pattern).
2. Thermal activation of the surface (heating to 150 °C).
3. Plasma spraying.

Plasma spraying was performed using a Kyiv-7 plasma gun with bronze powder Br.OTsS5-6-5. The powder particles have a granulated size range of 40 to 100 microns. Argon is used to atomise the molten metal.

The process results in the formation of micropores. To reduce porosity and improve the uniformity of the sprayed coating, the particle size is minimised. However, extremely fine particles (with a fraction size of less than 10–20 microns) often prove unsuitable for coating formation. The durability and performance of the coating depend not only on the properties of the applied alloy but also to a significant extent, on the adhesive forces between the contacting surfaces – "steel-bronze". As practice has shown, the adhesive contact between the steel surface and the sprayed bronze was insufficient, leading to the premature failure of the restored journals due to delamination and destruction of the applied coating. Fig. 2 and 3 contain the examples of the bearing socket damages.

Safety in passenger transport is the most critical aspect of its operation, and, as such, constant monitoring and replacement of worn-out bearing parts are essential to prevent accidents. As it was mentioned above, the replacement process (bronze powder plasma spraying) requires the use of specialised tools, which makes it expensive due to the utilisation of energy-intensive and costly equipment. Additionally, plasma spraying is carried out on a specially prepared surface with specific roughness requirements. The process involves significant heating of the part (the bearing), making it a rather labour-intensive technology. Its reliability is largely determined by the

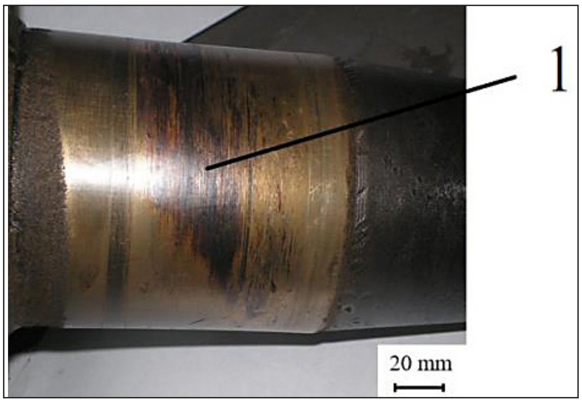

Figure 2. The seating area of the internal bearing of the vehicle where extensive oxide films are formed– zone 1

Table 1. The peel force of the deposited copper in the regenerator, depending on the roughness of the titanium electrode surface

Electrode surface roughness, R_a, μm	0.1	0.2	0.3	0.4
Peel force, P, g	1700	2500	2700	3400

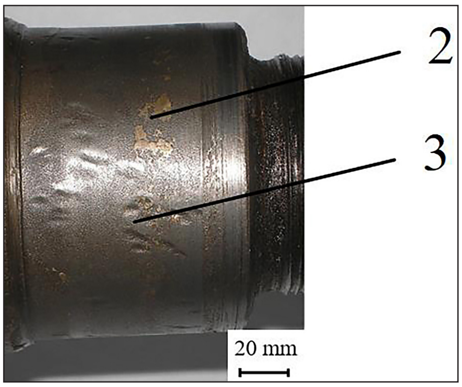

Figure 3. The area with the detachment of the bronze coating–zone 2, the seating area of the outer bearing with operational damage–zone 3

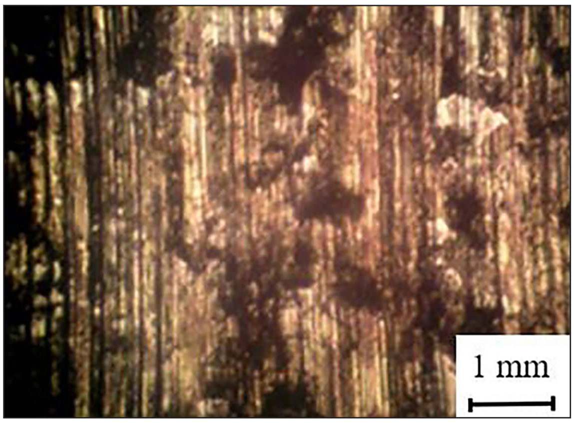

Figure 4. Surface after wear tests with the applied copper layer

adhesion strength between the bronze coating and the steel surface, which may not always be sufficient to ensure the performance of the parts under the challenging operating conditions of electric transport.

The authors improved the adhesive strength of the coating by applying a copper layer through galvanic deposition on the worn surface. This process was carried out using a recycled etching solution. The copper-plating of the bearing is achieved by conducting the process with the following parameters:

a) current density during copper-plating: 0.8–12 A/dm²;
b) bath solution temperature: 40–50 °C.

The thickness of the applied copper coating is up to 0.15 mm, with subsequent spraying of a layer of bronze on it to provide a durable, wear-resistant contact. When paired with steel, copper has a strong adhesion effect. The coatings with a copper layer were tested for wear and the comparative results indicated that the wear of the coating with the copper layer was approximately twice as low as that of the samples without a copper layer. Friction track width measurements (a) were made using a PMT-3 microhardness tester, with an accuracy of 0.005 mm.

The results of the experiment are presented in Table 2 (Sample No. 1 – the tested journal surface with a bronze coating applied over a copper layer, Sample No. 2 – the tested journal surface without a copper layer).

Figures 4 and 5 show the surfaces after conducting comparative wear tests, from which it can be concluded that the working surface with a copper layer is a better option for making production decisions. The wear test results, shown in Table 2 and Figures 4, 5 indicate that the bronze coating applied over the copper layer enhances the strength and adhesion to the part surface, thereby improving the antifriction properties of the friction pair.

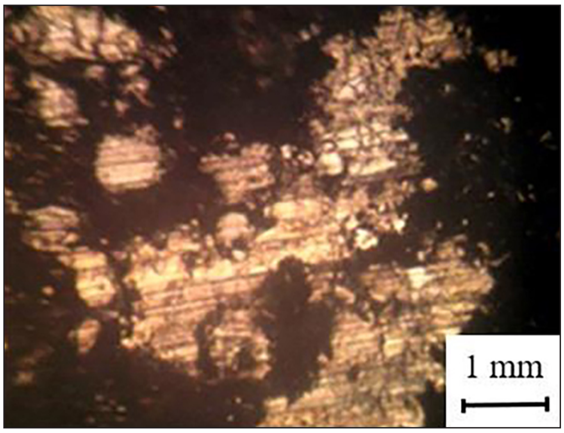

Figure 5. Surface after wear tests with coating without the copper layer

Table 2. Experimental results of journal surface wear testing

Sample	Number of cycles					
	5000	15000	25000	35000	45000	60000
No. 1, *a*, mm	0.125	0.3	0.45	0.56	0.625	0.68
No. 2, *a*, mm	0.31	0.6	0.78	0.85	0.88	0.90

CONCLUSIONS

After conducting research involving the etching of printed circuit boards (PCB) along with regenerating the etching solution simultaneously, the authors of this article proposed to recover copper from the depleted solution and utilise it as a raw material within the electrical industry of the national economic complex. Consequently, it was found that the copper surface was clean, free from electrolyte residues, and there were no deposits. Dendritic growth of a mushroom-like form, as well as deposits of porous copper, is absent.

The obtained results highlight the feasibility of utilizing the regeneration process for spent etching solutions with simultaneous copper recovery in the form of foil. This process offers a significant reduction in waste storage quantities at industrial facilities and waste water discharge into municipal treatment facilities.

By selecting the solution composition through factorial experimentation, an optimal etching speed of 35–38 mm/min was achieved. Combined with the electrode studies, this allowed for the regeneration of the etching solution, producing 28–32 kg of copper during 8 hours of operation on a standard line. The innovation lies in resolving the electrode issues, enabling the practical use of modern etching lines without major structural changes, by supplementing them with regeneration units.

The studies conducted have demonstrated that titanium, and to a lesser extent, steel grade 08X17TM, is the most suitable material for current collectors, offering high chemical and electro chemical resistance.

When the surface roughness of the titanium cathode VT1-0 is increased from 0.1 to 0.4 microns, the detachment force of the copper deposited in the regenerator rises from 1700 to 3400 grams (from an arbitrarily chosen tested area of 25 × 150 mm). Mechanical separation tests of the deposited copper revealed that titanium VT1-0 cathodes provide better results (when manually bending the cathode plate, the copper easily separates), making titanium the preferred material for the electrode in the regenerator design.

The wear resistance of the copper journal was examined on a surface coated with bronze over a copper layer. The findings indicate that the bronze coating on the copper layer significantly improves strength and adhesion to the part's surface, resulting in enhanced anti-friction properties of the friction pair. Comparative tests demonstrated that the wear on the coating with a copper layer is about half that of samples without the copper layer. After 5000 cycles, the friction track width in the coating without the copper layer was 0.31 mm, whereas in the coating with the copper layer, the friction track width reduced to just 0.125 mm.

The study achieved improved adhesion strength of the journal coating by electroplating a copper layer onto the worn surface using spent etching solution. Experiments with electroplated copper recovered from spent etching solution on the worn surface of a trolleybus journal showed that this technology offers an additional use for the recovered material as part of the regeneration process.

The proposed methods and scheme for regenerating spent etching solution allow the creation of equipment complexes in which a regenerator should be used. The primary component of this setup will be an electrochemical cell (electrolyser), where copper extracted from the spent etching solution will be deposited on the corresponding electrode. The research enables the development of a technological process for the reutilisation of aqueous etching solutions and the reduction of their impact on the environment.

Further research can be based on the promising results of the conducted studies and applied in the practical operations of companies. However, it would be beneficial to focus on the continued automation of this process.

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