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TECHNOLOGIES FOR CHEMICAL MILLING OF ALUMINIUM AND ITS ALLOYS: THE USE OF ALKALINE AGENTS

The problem of improving the efficiency of chemical milling technologies while simplifying the procedure for their implementation remains unresolved. One of the efficient ways to solve this problem is to develop new etching compounds. The purpose of this work was to experimentally determine the optimal conditions for the use of salt additives (NaNO_3 , NaClO_4) as components of alkaline solutions in chemical milling technologies, as well as to develop the foundations for the rational use of waste in these technologies. It is shown that the introduction of a salt additive to an alkaline solution of chemical milling increases the rate of aluminium removal. In this case, the quality of the treated surface is improved, and the amount of evolved hydrogen decreases. The optimal chemical composition of the etching solution is proposed. It is shown that the spent aluminate solutions can be used to obtain the basic aluminium salt $\text{Al}_2(\text{OH})_5\text{Cl}$, which has a high coagulating ability. It has been determined that aluminium hydroxychloride can be used as a precipitating reagent for the decomposition of alkali-aluminate solutions. The scheme of the chemical milling process has been proposed. It provides for the regeneration of the components of the etching alkaline solution and contributes to the rational use of technological resources

Key words: chemical milling, aluminium and its alloys, alkaline solution, salt additive, dissolution speed.

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ТЕХНОЛОГІЇ ХІМІЧНОГО ФРЕЗЕРУВАННЯ АЛЮМІНІЮ ТА ЙОГО СПЛАВІВ: ВИКОРИСТАННЯ ЛУЖНИХ АГЕНТІВ

Підвищення ефективності технологій хімічного фрезерування алюмінію і його сплавів при спрощення процедури їх здійснення залишається актуальною проблемою. Один з ефективних способів її рішення – розробка нових травильних складів. Мета даної роботи полягала в експериментальному визначенні оптимальних умов використання сольових добавок (NaNO_3 , NaClO_4) в якості компонентів лужних розчинів в технологіях хімічного фрезерування, а також в розробці основ раціонального використання відходів в цих технологіях. Показано, що введення сольовий добавки до лужного розчину хімічного фрезерування збільшує швидкість видалення алюмінію. Якість обробленої поверхні поліпшується, а кількість водню, що виділяється, зменшується. Запропоновано оптимальний хімічний склад травильного розчину. Показано, що з відпрацьованих алюмінатних розчинів можна отримати основну сіль алюмінію $\text{Al}_2(\text{OH})_5\text{Cl}$, яка має ефективну коагулюючу здатність. Встановлено, що гідроксихлорид алюмінію може бути використаний в якості реагента-висаджувача при декомпозиції алюмінатних лужних розчинів. Запропоновано схему процесу хімічного фрезерування. Вона забезпечує регенерацію компонентів травильного лужного розчину і сприяє раціональному використанню технологічних ресурсів.

Ключові слова: хімічне фрезерування, алюміній та його сплави, лужний розчин, сольова добавка, швидкість розчинення.

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ТЕХНОЛОГИИ ХИМИЧЕСКОГО ФРЕЗЕРОВАНИЯ АЛЮМИНИЯ И ЕГО СПЛАВОВ: ИСПОЛЬЗОВАНИЕ ЩЕЛОЧНЫХ АГЕНТОВ

Повышение эффективности технологий химического фрезерования алюминия и его сплавов при упрощении процедуры их осуществления остается актуальной проблемой. Один из эффективных способов ее решения – разработка новых травильных составов. Цель данной работы заключалась в экспериментальном определении оптимальных условий использования солевых добавок (NaNO_3 , NaClO_4) в качестве компонентов щелочных растворов в технологиях химического фрезерования, а также в разработке основ рационального использования отходов в этих технологиях. Показано, что введение солевой добавки в щелочной раствор химического фрезерования увеличивает скорость удаления алюминия. Качество обработанной поверхности улучшается, а количество выделяющегося водорода уменьшается. Предложен оптимальный химический состав травильного раствора. Показано, что из отработанных алюминатных растворов можно получить основную соль алюминия $\text{Al}_2(\text{OH})_5\text{Cl}$, обладающую эффективной коагулирующей способностью. Установлено, что гидроксихлорид алюминия может быть использован в качестве осаждающего реагента при разложении алюминатно-щелочных растворов. Предложена схема процесса химического фрезерования. Она обеспечивает регенерацию компонентов травильного щелочного раствора и способствует рациональному использованию технологических ресурсов.

Ключевые слова: химическое фрезерование, алюминий и его сплавы, щелочной раствор, солевая добавка, скорость растворения.

Problem definition

Alkaline solutions of sodium hydroxide with various additives are mainly used for chemical milling of aluminium and its alloys. For example, in the practice of processing aluminium alloys, an alkaline solution of sodium hydroxide is widely used, containing the following additives (g/dm^3): sodium hydroxide 250–300, aluminium (salt-like compound) 10–15, potassium fluoride 6–8, sodium thiosulfate $\text{Na}_2\text{S}_2\text{O}_3$ 25–30, ammonium peroxydisulfate $(\text{NH}_4)_2\text{S}_2\text{O}_8$ 15–18 [1]. A solution of this composition allows a sufficiently intensive chemical milling of aluminum by reducing the overvoltage of the cathodic process. A significant disadvantage of this solution is that the process is carried out at a sufficiently high temperature of 75–95 °C. In addition, $(\text{NH}_4)_2\text{S}_2\text{O}_8$ and $\text{Na}_2\text{S}_2\text{O}_3$ undergo chemical transformations during etching. This entails the need to adjust the chemical composition of the etching solution to ensure a constant etching rate and the required quality of the treated surface. Such chemical milling procedures are complex and expensive. The problem of improving the efficiency of chemical milling technologies while simplifying the procedure for their implementation remains unresolved. One of the efficient ways to solve this problem is to develop and test new etching compounds.

Related publications

Aluminium alloys have unique structural properties such as low density, acceptable ductility, high thermal and electrical conductivity. However, they are characterized by self-passivation due to the formation of layers of different thickness. These layers consist mainly of Al_2O_3 and $\text{AlO}(\text{OH})$ and have a relatively low chemical activity. Their presence negatively affects the efficiency of chemical milling technologies and forces them to develop new chemical compositions and milling modes. Earlier it was shown that the dissolution of aluminium and its alloys significantly depends on the presence of additives can be changed by adding organic solvents [2-7] or salt-like compounds to the electrolyte solution [8-12]. The use of such composite electrolyte solutions can effectively influence the speed of chemical milling.

Formulation of the purpose of research

The purpose of this work was to experimentally determine the optimal conditions for the use of salt additives (NaNO_3 , NaClO_4) as components of alkaline solutions in chemical milling technologies, as well as to develop the foundations for the rational use of waste in these technologies.

Presentation of the main research material

Improvement of the chemical composition for the implementation of chemical milling technology.

Our preliminary experiments [3, 11, 12] have shown that when sodium nitrate is introduced into an alkaline solution, the dissolution rate of aluminium and alloys D-16 and V-95 increases. In order to intensify the process of chemical treatment of aluminium and its alloys, we have experimentally determined the optimal conditions for using salt additives (NaNO_3 , NaClO_4) as components of alkaline solutions. Aluminium alloys have the following chemical composition, %: D-16: Al - 93,5÷91,4; Cu - 3,8÷4,9; Mg - 1,2÷1,6; Mn - 0,3÷0,9; Zn - 0,1; T1 - 0,1; Fe - 0,5; Si - 0,5; V-95: Al - 88,3÷89,4; Cu - 1,7÷1,9; Mg - 2,3÷2,5; Mn - 0,4÷0,9; Cr - 0,2÷0,4; Zn - 6,0. A grade Al-00 was used as a pure aluminium. The K_m value characterizes the rate of metal dissolution. It was calculated as the mass of metal dissolved per unit of surface area per unit of time ($\text{kg}/(\text{m}^2 \cdot \text{s})$).

Reducing the concentration of NaOH in the test solution to 180–200 g/dm^3 and increasing the concentration of aluminium to 35–40 g/dm^3 leads to an improvement in the quality of the treated surface. This also allows an alkaline aluminate solution to be used to adjust the etching solution. For the research, a solution of

the composition was used: NaOH (total) = 200,0 g/dm³; Al = 37,1 g/dm³. Both sodium nitrate and sodium perchlorate additives accelerate the ionization of aluminium.

The effect of adding NaNO₃ salt will be considered in more detail. From Fig. 1 it follows that the dissolution rate of the Al-00 sample increases markedly, especially when using perchlorate salt. The duration of exposure to etching salt solutions is also higher than that of an individual alkaline agent NaOH. As the concentration of sodium nitrate increases, the amount of hydrogen released decreases significantly (Fig. 2).

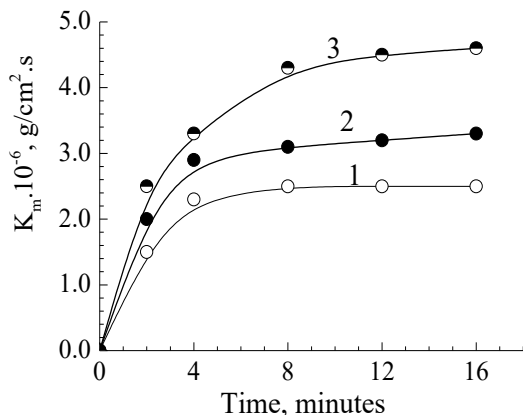


Fig. 1. Time dependence of the dissolution rate of aluminium Al-00. Solutions, g/dm³: 1 – 240 NaOH, 2 – 200 NaOH + 30 NaNO₃, 3 – 200 NaOH + 30 NaClO₄

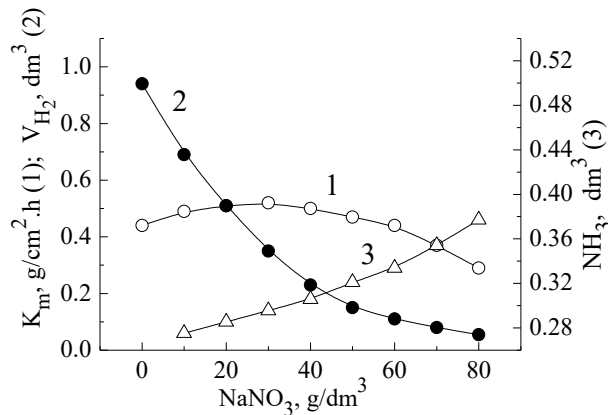


Fig. 2. Influence of NaNO₃ concentration on the dissolution rate of D-16 aluminium alloy (1); the amount of released hydrogen (2), the amount of ammonia (3)

This is due to a natural decrease in the concentration of etching agents. On the contrary, the amount of formed ammonia naturally increases as a product of reduction of nitrate ions (Fig. 2). A similar relationship for the NaNO₃ concentration is more complex and extreme. We found that the effectiveness of nitrate and perchlorate ions depends on the ratio of the concentrations of the studied salts, as well as the duration of etching. During the operation of the etching solution, the effect of the above additives is reduced. This is due to the accumulation of Al(OH)₄⁻ in the solution, which make it difficult to diffuse NO₃⁻ and ClO₄⁻ ions in the near-electrode layer [3, 5]. The data characterizing the process of nitrate ion reduction are presented in Table 1.

Table 1.

Products of reduction of nitrate-ion during chemical milling of aluminum alloy D-16

The NO ₃ ⁻ content in the solution, g/100 cm ³		The number of nitrate ion reduction products, g/100 cm ³		Correlation NH ₃ /NO ₂ ⁻ in terms of NO ₃ ⁻
Before milling	After milling	NH ₃ in terms of NO ₃ ⁻	NO ₂ ⁻ in terms of NO ₃ ⁻	
0,97	0,69	0,06	0,01	6,00
1,40	1,07	0,11	0,02	5,50
2,90	1,32	0,15	0,04	3,75
4,30	2,45	0,18	0,12	1,50
5,87	3,33	0,34	0,29	1,17

As can be seen from the above data, an increase in the concentration of sodium nitrate in etching solutions leads to the accumulation of an intermediate reduction product – nitrite ion. The completeness of further reduction of the NO₂⁻ ion was controlled using the ratio of the amount of ammonia and nitrite ion in terms of the nitrate ion (Fig. 3). The obtained data indicate that when the NaNO₃ additive contains more than 60 g/dm³, the process of reducing the nitrogen atom to the highest degree of oxidation slows down. The study of the surface roughness of the samples after etching was performed by measuring the radius of depressions (R) on the surface of the samples. The radius of depressions is noticeably reduced in NaOH solutions containing more than 40 g/dm³ NaNO₃. This fact indicates an improvement in the quality of surface treatment.

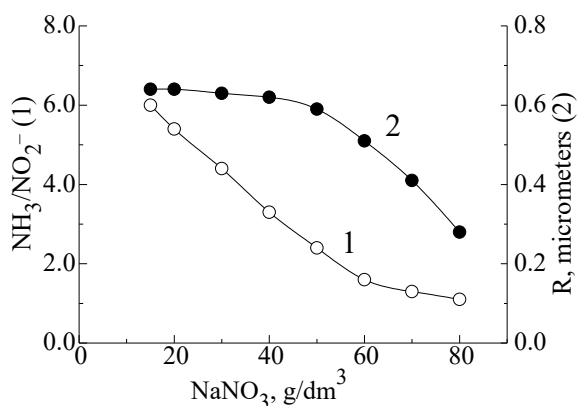


Fig. 3. Dependence of the completeness of the reduction of nitrate ion (1) and the quality of the treated surface (2) on the content of sodium nitrate in etching solutions

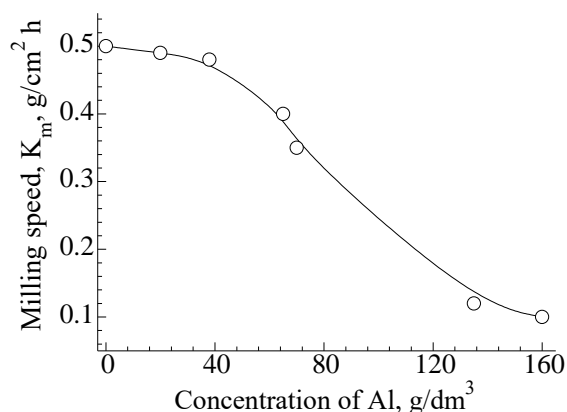


Fig. 4. Dependence of the speed of dissolution of aluminum on the concentration of aluminum in solution. Composition of solution: 200 g/dm³ NaOH and 40 g/dm³ NaNO₃

Based on the obtained results, it can be concluded that when the salts are added to the basic composition of the alkaline etching solution in the amount of 40–50 g/dm³, the metal removal rate increases by 1,2–1,5 times, the surface treatment quality improves, and the amount of hydrogen released during the reaction decreases. These facts indicate that the use of NaNO₃ or NaClO₄ additive for chemical milling of aluminium and its alloys is quite effective and appropriate.

It was found that the amount of free NaOH decreases during etching of aluminium and alloys. However, the etching rate and electrical conductivity of the solution decreases, and the viscosity, on the contrary, increases. If you do not add new portions of NaOH to the etching bath, the milling process slows down. Over time, the solution becomes milky white. There is an increase in the etching rate to values slightly lower than the initial speed. The chemical reaction that occurs can be described as follows: $\text{NaAlO}_2 + 2\text{H}_2\text{O} = \text{Al}(\text{OH})_3 \downarrow + \text{NaOH}$.

The products of this reaction are the release of sodium hydroxide, which is necessary for further etching, as well as the formation of aluminium hydroxide. Therefore, it is important to investigate the effect of the amount of aluminium present in the solution on the speed of chemical milling. It was found (Fig. 4) that the NaOH solution, when it used continuously, "absorbs" aluminium until the volume of free sodium hydroxide is reduced to about 25 % of the original volume. After that, etching continues with free sodium hydroxide, remaining at approximately the same level with a frequency that depends on the temperature. Aluminium hydroxide in this case crystallizes at the bottom and sides of the etching bath to form a solid precipitate that is difficult to remove. This stage can be described by the reaction of aluminium hydroxide dehydrogenation to form aluminium oxide: $2\text{Al}(\text{OH})_3 \rightarrow \text{Al}_2\text{O}_3 + 3\text{H}_2\text{O}$. The explanation of this transformation is shown in Fig. 5.

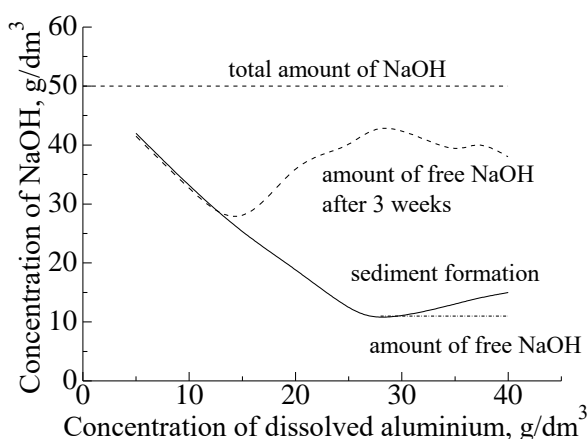


Fig. 5. Influence of dissolved aluminum concentration on the amount of free sodium hydroxide

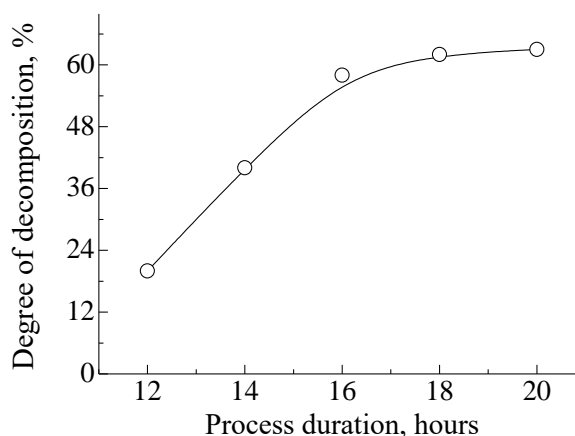


Fig. 6. The effect of the duration of decomposition on the degree of decomposition of the solution

When the concentration of dissolved aluminium exceeds 15 g/l, free NaOH is regenerated up to 80 % relative to the original value. With prolonged use of the etching solution, this value ranges from 20 to 30 %, and in the case of downtime, it increases to 50 %. A similar ratio is also observed for higher concentrations of sodium hydroxide. Note that the etching rate decreases significantly when the content of dissolved aluminium

increases (see Fig. 4). In practice, this means that to maintain a constant etching rate, the content of free sodium hydroxide should be increased. However, a negative consequence of this is large volumes of waste process solutions containing 50–60 % of unused sodium hydroxide and 80 % of dissolved aluminium. Therefore, the disposal and rational use of waste solutions becomes an urgent task.

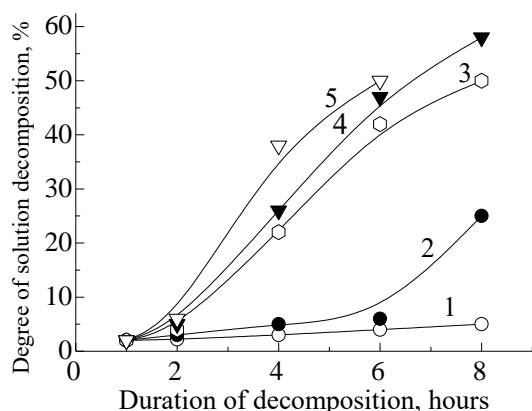


Fig. 7. Kinetic curves of alkaline aluminate solution decomposition using $\text{Al}_2(\text{OH})_5\text{Cl}$ as a precipitating reagent. Temperature, °C: 1 – 20; 2 – 25; 3 – 30; 4 – 35; 5 – 40

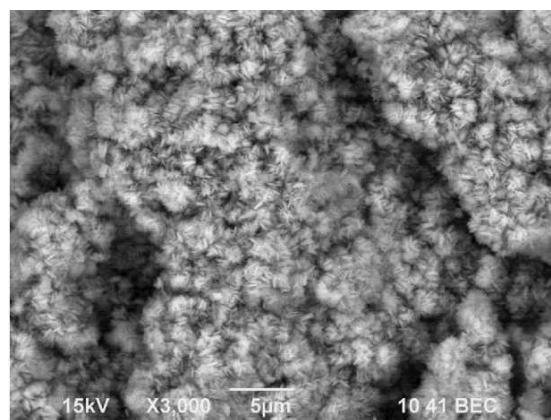


Fig. 8. Electronic microphotography of aluminum hydroxide obtained using a precipitator reagent

Rational use of waste in chemical milling technologies of aluminium. The current direction is the development of processes that allow using used etching solutions as raw materials for obtaining valuable products. Methods of regeneration of alkaline solutions of aluminium etching are intended to prevent the flow of industrial waste into the environment [2, 3, 8, 9]. Therefore, further research was directed to the processing of spent alkaline solutions that are used for chemical milling of aluminium and its alloys. We studied the processes of decomposition of aluminate solutions with subsequent use of the precipitate to obtain basic aluminium salts, since they have high coagulating properties in a wide range of acidity values.

In order to study the conditions under which the decomposition of alkaline-aluminate solutions is possible in the presence of aluminium hydroxychloride $\text{Al}_2(\text{OH})_5\text{Cl}$ as a precipitator-reagent, we investigated the influence of NaOH concentration, $\text{NaAlO}_2/\text{Al}_2\text{O}_3$ ratio, temperature and mixing mode. We proceeded as follows when simulating the conditions for obtaining of hydroxychloride. A 0,5 mol/L hydrochloric acid (with a density of 1,19 g/cm³) was added to a certain amount of an aluminate solution containing 280 g of NaOH and 80 g of Al in 1 cm³. The freshly precipitated $\text{Al}(\text{OH})_3$ was filtered off and thoroughly washed with hot water. Particular attention was paid to the fact that the time for washing the precipitate of aluminium hydroxide from NaCl was no more than 15–20 minutes, since the aging of the precipitate impairs the technological properties of the product. Then $\text{Al}(\text{OH})_3$ was dissolved in 1 dm³ of 0,5 mol/L HCl in a flask with a reflux condenser and constant stirring at a temperature of 90–95 °C. Aluminate alkaline solutions were analyzed for NaOH and Al content such as [5]. The content of Al^{3+} and Cl^- ions was determined in the obtained aluminium hydroxychloride. According to result of chemical analysis, it was found that the ratio of molar concentrations $\text{Cl}:\text{Al}^{3+}$ is equal to 0,6. This corresponds to the chemical composition of the main salt of aluminium $\text{Al}_2(\text{OH})_5\text{Cl}$. The formation of this salt can be expressed by the equation: $2\text{Al}(\text{OH})_3 + \text{HCl} \rightarrow \text{Al}_2(\text{OH})_5\text{Cl} + \text{H}_2\text{O}$.

Experimental data on the effect of the process duration on the degree of decomposition of the spent alkaline-aluminate solution at a temperature of 25 °C without mixing are shown in Fig. 6. Decomposition was performed at $\text{NaAlO}_2/\text{Al}_2\text{O}_3 = 1,63$ with the addition of 6,5 g/dm³ $\text{Al}_2(\text{OH})_5\text{Cl}$. From the above data, it can be seen that at first there is a long period of induction. Intensive precipitation of aluminium hydroxide begins after 10 hours, and after another 4 hours 40 % Al_2O_3 is released from the solution. The dependence of the degree of decomposition of the aluminate solution on time at different temperatures is shown in Fig. 7; $\text{Al}_2(\text{OH})_5\text{Cl}$ as a precipitator reagent was used in each experiment in an amount of 6,5 g/dm³.

The advantages of using $\text{Al}_2(\text{OH})_5\text{Cl}$ as a precipitator reagent for the decomposition of alkaline-aluminate solutions should also include the fact that the resulting aluminium hydroxide has a developed surface (see Fig. 8); it is easily soluble in acids and is an important product for obtaining cheap coagulants.

The results of studying the amount of active $\text{Al}(\text{OH})_3$ priming during decomposition of an alkaline-aluminate solution are shown in Fig. 9. The process was carried out without stirring at 25 °C. These data suggest a high activity of aluminium hydroxide obtained using $\text{Al}_2(\text{OH})_5\text{Cl}$ without heating. As can be seen from the Fig. 9, the decomposition rate of 60 % is reached after 18 hours.

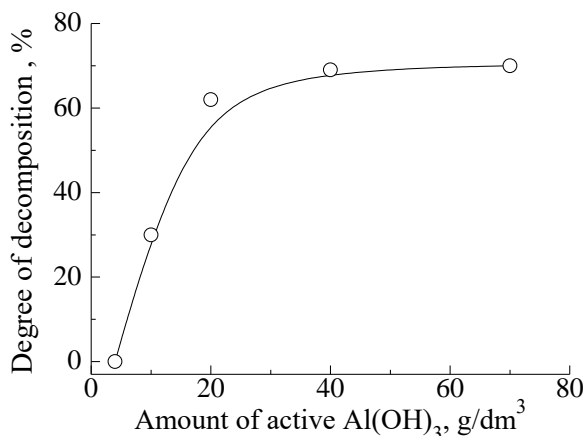


Fig. 9. Influence of the amount of active aluminum hydroxide on the degree of decomposition of alkaline-aluminate solution

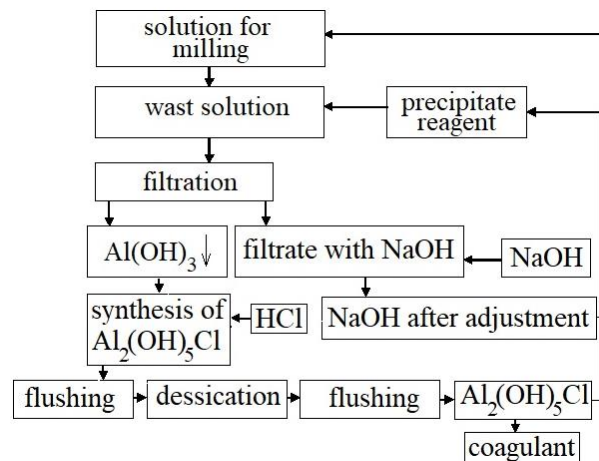


Fig. 10. Scheme of regeneration of solutions for chemical milling of aluminum and its alloys

The obtained results are the basis for the development of a new environmentally friendly technology for chemical milling of aluminium and its alloys. A schematic diagram of the technological cycle is shown in Fig. 10. This development contributes to the rational use of technological resources.

Conclusions

It is shown that the introduction of a salt additive NaNO_3 or NaClO_4 to an alkaline solution of chemical milling increases the rate of aluminium removal by 1,2–1,5 times. In this case, the quality of the treated surface is improved, and the amount of evolved hydrogen decreases. The optimal chemical composition of the etching solution for dimensional processing of aluminium and its alloys contains NaOH – 180–200, Al – 30–40, NaNO_3 – 40–50 g/dm^3 . It is shown that the spent aluminate solutions can be used to obtain the basic aluminium salt $\text{Al}_2(\text{OH})_5\text{Cl}$, which has a high coagulating ability.

It has been determined that aluminium hydroxychloride can be used as a precipitating reagent for the decomposition of alkali-aluminate solutions. The technological scheme of the chemical milling process is proposed. It provides for the regeneration of the components of the etching alkaline solution and contributes to the rational use of technological resources.

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