

Quality of anode. Overview of Problems and Some Methods of their Solution

Part 1. Coal Foam in an aluminum electrolyzer

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Abstract

The goal of any aluminum plant is to increase electrolyzer productivity and current efficiency, reduce electricity and carbon consumption. These indicators depend significantly on the quality of the anode blocks [1; 2; 3]. In turn, the quality of the anodes is also, to a certain extent, determined by the properties of the anode cinders involved in the recycling of the anodes.

One of the indicators of the anode blocks quality is the amount of carbon foam that is removed during the operation of the anode. Over the past decades, the world's aluminum industry has been paying considerable attention to the study of its negative impact on electrolysis performance. A large amount of carbon foam in the electrolyte leads to operational problems and various technological disturbances that include [4]:

- a slower heating of the new anode, so that more time is needed to equalize the current on the anodes;
- the need to reduce the distance between the anode and the cathode VGH at a given voltage setting due to an uncontrolled increase in the electrical resistance of the electrolyte;
- the increase in the coefficient of dynamic viscosity of the electrolyte;
- the decrease in the rate of dissolution and distribution of alumina, since carbon foam prevents its free distribution in the electrolyte;
- a slower heat removal from melt, which leads to an imbalance in current distribution in the bath;
- the increase of electrical resistance of electrolyte, since carbon foam acts as an insulator;
- the cyclic increase in the temperature of melt, accompanied by an increased consumption of fluoride salts.

The negative effect of the foam is also manifested in the increased consumption of anode carbon, in the increase in the resistance and temperature of the electrolyte, and in a significant decrease in the current yield.

Foam can cause the formation of irregularities on the base of the anode, which in foreign literature are called "mushrooms", "thorns", in the Russian - "cones". The presence of the cone carries the risk of short-circuiting the anode with the cathode directly, or through a layer of liquid aluminum, which leads to temperature fluctuations and a decrease in current efficiency by an average of 1.5%.

The amount of foam formed depends on the electrolyzer design, the conditions of the technology, the quality of the anodes used, the time of problem detection, and many other reasons.

The article provides a brief overview of Russian and foreign studies devoted to the study of the causes of the coal foam appearance in electrolytes of aluminum electrolyzes, as well as proposals aimed at improving the quality of the anode and reducing the yield of coal foam.

Keywords: aluminum electrolyzer, anode, carbon, coal foam, consumption, oxidation, protection, method.

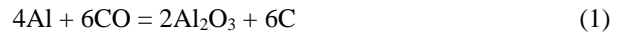
Sources of coal foam in the electrolyte of an aluminum electrolyzer

According to [2], the sources of coal foam in the electrolyte are:

- anode blocks, due to the greater reactivity of coke from the pitch of the binder and its selective oxidation, followed by the shedding of coke-filler particles in the melt;

- wear of the carbonaceous bottom, whose contribution is from 0.5 to 1 kg of carbon particles per 1 ton of aluminum;
- carbonaceous mass, used to protect nipples from oxidation. It gives from 0.5 to 6 kg of carbon per 1 ton of aluminum;
- anode cover material, which contains up to 5% by weight of carbon;
- carbon in secondary alumina, the content of which varies from 0.15 to 0.5% by weight.

millimeters in diameter, comes into the electrolyte. By chemical way, as a result of reactions like:



a fine carbon is obtained in the form of graphite or soot.

The carbon consumption in the interaction with the anode gas [5; 6] is due to the fact that the anode gas penetrates into the pores of the anode under the influence of excess pressure. In this case, in the anode three regions of the oxidation processes can be identified, shown in Fig. 1.

Sources of foam entering the electrolyte can be divided into mechanical and chemical. Due to mechanical destruction of the anodes and carbon lining, the main part of coal in the form of carbon of coarse dispersion, with grains up to several

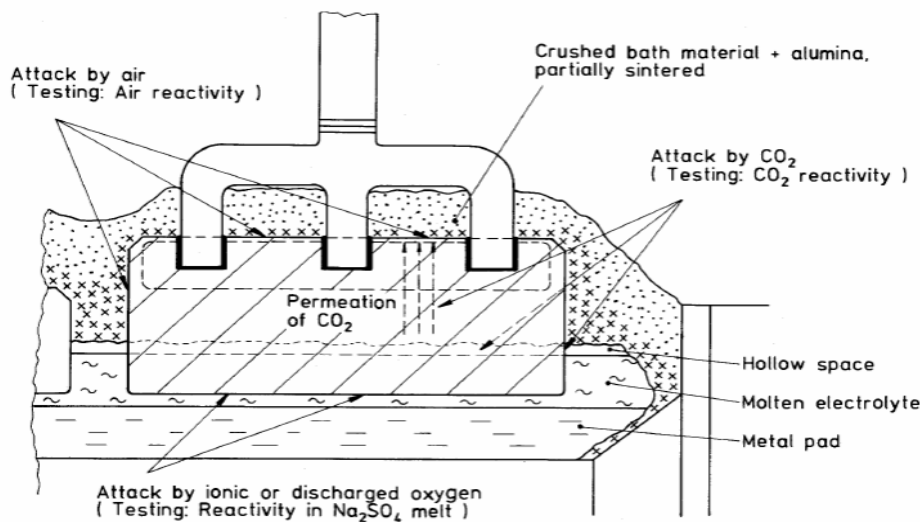


Figure 1: Anode flow mechanism in an aluminum electrolyzer

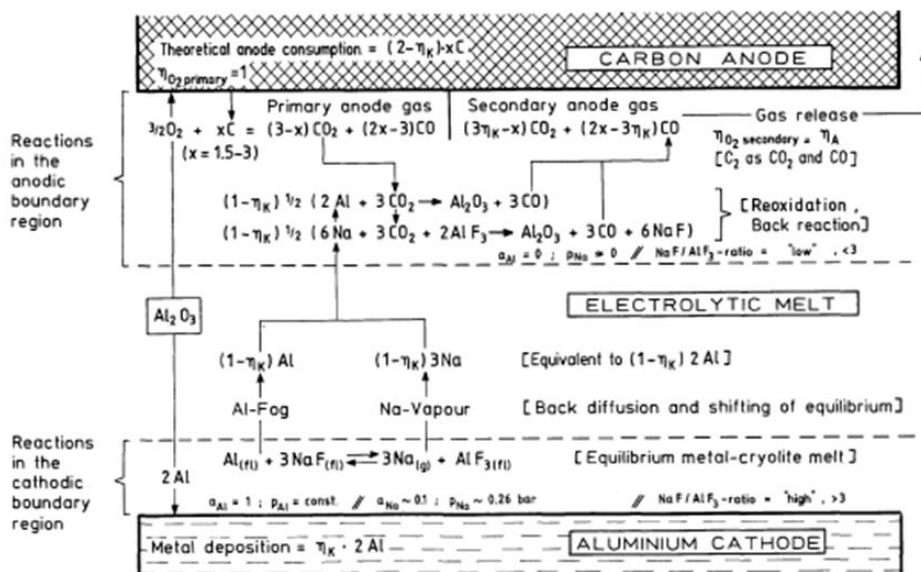
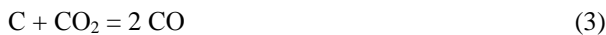


Figure 2: Model illustrating the mechanism of carbon consumption and current yield

One oxidation process occurs at the top of the anode, where CO₂ is reduced to CO. On the surface of the anode lower part, an electrochemical oxidation reaction of the oxygen ion occurs, included in the complex anions, followed by the interaction of oxygen with the anode carbon and the formation of, ultimately, CO₂. Here, it is spent 80 - 85% of consumed carbon. The third process is the oxidation according to the Boudouard reaction of the graphitized carbon material with carbon dioxide, penetrating the pores of the anode block body:



Here, it is spent 15-20% of consumed carbon. Meanwhile, it is difficult to accurately differentiate the amount of carbon that the electrolyte carries to the periphery of the anode, where it rises as a foam and some of its amount is oxidized by air and CO₂. Each of the three mentioned oxidation mechanisms of the anode is accompanied by the formation of fine-dispersed coal foam. Typically, a lot of foam is formed if the matrix of the binder is very reactive and more sensitive to oxidation than the coarse filler material - coke. Faster detachment of coke particles is facilitated by the action on the base of the anode of a turbulent flow of electrolyte, containing gas bubbles, foam and insoluble alumina.

Specific behavior of the anode in oxidizing media - air or CO₂, is determined by the analytical method. For a better understanding of the electrochemical process of anode consumption, a simplified model is proposed, shown in Fig. 2 [5].

There are four stages of formation of anodic gas with different contents of CO₂ and CO [5]. The "primary" gas is formed directly on the anode and originates from the reaction of the liberated oxygen with the anode carbon. Developing in the form of a thin gas layer, the primary anode gas accumulates and separates from the anode in the form of bubbles, and then moves under the anode through the electrolyte layer. As it passes through the electrolyte layer, the primary anode gas reacts with the carbon foam particles, as a result of which CO₂ is reduced to CO by reaction (3) and exits outwards as a "secondary" anode gas (Fig. 2).

Strictly speaking, the electrochemical process is completed by the formation of a secondary anode gas. Prior to the release of the secondary anode gas from under the crust of the electrolyte, it still contains a high content of CO₂, which can react with anode carbon on the surface of the anode or in its pores, as well as with coal foam. As a result of these reactions, the "secondary" anode gas changes to a "tertiary" gas, with a slightly higher CO content. Since the ratio of CO₂ / CO in secondary and tertiary anode gases is insignificant, the differences between them are often not made. The final composition of the anode gas, without taking into account perfluorocarbons, can be defined as a "quaternary" gas. According to the authors of [5], the electrolysis of alumina proceeds with a certain partial pressure of oxygen at the anode. This oxygen determines the theoretical EMF in the

bath, i.e. the sum of the decomposition voltage of alumina and anodic depolarization due to the interaction of oxygen with the anode carbon (Fig. 3). When establishing the thermodynamic equilibrium of the anode carbon, the composition of the anode gas corresponds to the so-called "Boudouard equilibrium" (Fig. 4).

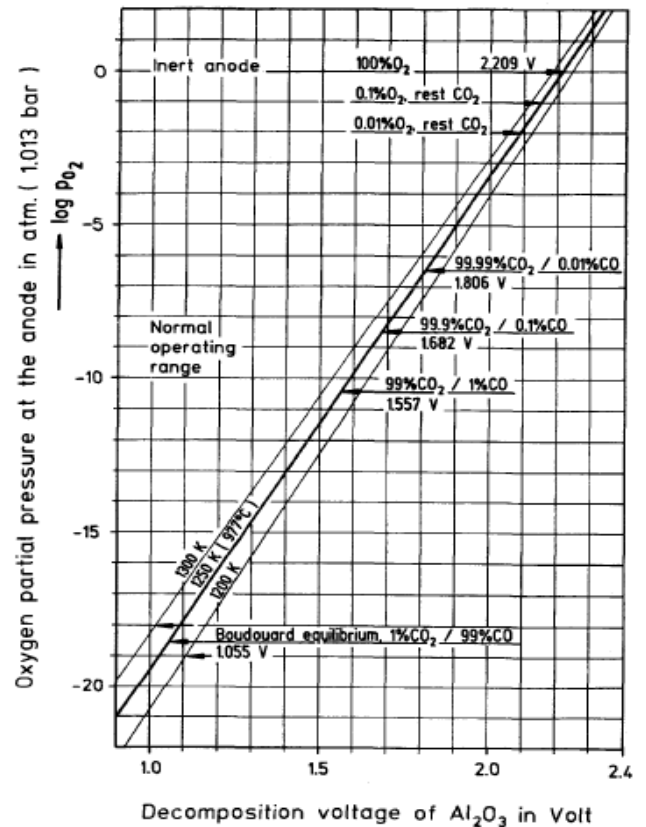


Figure 3: Dependence of the partial pressure of oxygen on the theoretical decomposition voltage of Al₂O₃ at different temperatures and compositions of the primary anode gas

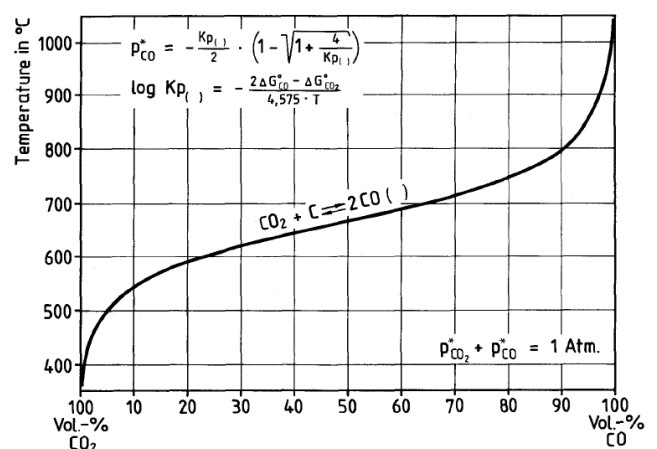


Figure 4: Dependence of the Boudouard equilibrium reaction on the temperature

With an anode gas content of ~ 99% CO and 1% CO₂, the oxygen partial pressure is 10-18 bars, the theoretical EMF is 1.06 V at 977 ° C. For kinetic reasons, due to the slow rate of the anodic reaction, as the current density increases and the reactivity of the anode decreases, the partial pressure of oxygen increases. On fired anodes, under normal electrolysis flow, the oxygen partial pressure is in the range of 10⁻¹⁰ to 10⁻⁸ bar, and the decomposition voltage (reverse EMF) is from 1.6 to 1.7 V with the content in the primary anode gas of CO₂> 99.5%.

In practical operation of the anode, the CO₂ content of the anode gas can be reduced to 50%, which is accompanied by an increase in the decomposition voltage on 0.65 V and an additional power consumption of about 2.1 kWh / kg Al.

When using more reactive Soderberg anodes, the primary anode gas contains a substantial amount of CO. It is proved that higher CO concentrations in the primary anode gas are obtained with a high electrolyte overheat and a sufficiently low current density. Correlation between the content of CO₂ in the primary and secondary anode gas is determined by the formula:

$$\varphi_{CO_2} = \eta(1 + C) - 100 \quad (4)$$

Where: φ_{CO_2} is the volume concentration of CO₂ in the secondary anode gas,% by volume; η - current output,%; C - the specific constant of the anode under normal operating conditions, for burned anodes C = 0.95 - 1.0, for Soderberg anodes C = 0.7-0.9. For 100 the concentration of CO₂ in the primary anode gas is assumed.

The electrochemical consumption of the anode depends on the ratio of CO₂ / CO in the secondary anode gas, as shown in the diagram in Fig. 5.

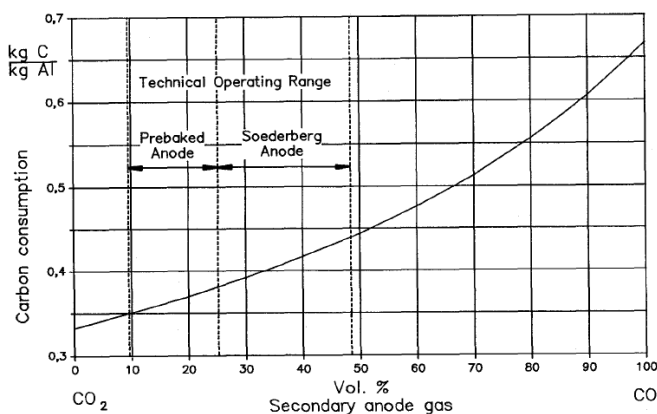


Figure 5: Dependence of the specific consumption of the anode on the ratio of CO₂ / CO in the secondary anode gas

The dominant part of electrolysis occurs at the anode bottom [7-8]. The contribution of the side surfaces of the anode is also significant. The current density on them is estimated on the average as 1/3 of the current density at the base of the

anode. Although there is a lower current density in this zone, it is subjected to intense exposure to bubbles of anodic gas rising near the vertical surfaces of the anode. Most of the shedding occurs on these edges, due to the reaction (3) of the interaction of anode gases with carbon. Laboratory studies of the consumption rate of anodes of various quality show [9] that a low current density promotes the development of surface roughness of the anode due to exfoliation, increasing its consumption.

With regard to the foam formation on the sides of the anode, there is still no final clarity, it is formed in the contact zone with the electrolyte or in the upper hot zone in contact with air.

Oxidation of the anode surface by CO₂ or air is significantly catalyzed by various metals (for example, V, Na, Ca), while the effect of these catalysts on the anode bottom is negligible.

The effect of sodium on the anode consumption (an approximate subtitle)

It is well known that the cinders of long-running anodes is heavily contaminated with sodium, and for this reason it is not advisable to involve them in processing, since one such cinder can contaminate up to 30 new anodes at the stage of their production.

One of the sources of anode contamination with sodium is the anode cinders involved in the production of carbon materials, into which sodium comes from the melt. The research results of the penetration rate of sodium from the melt of an aluminum electrolyzer to the recovered cinder, presented in [9], show that the use of porous cinders (due to insufficient oxidation of the anode) and poorly purified from coal foam impairs the quality of the produced anodes and thus creates a vicious circle, shown in Fig. 6, which leads to extreme accumulation of foam in the bath and disruption of the electrolysis process.



Figure 6: A vicious circle created by coal foam

The study [10] considers two penetration scenarios:

- closing of the de-energized anode with cathode aluminum;
- interruption of current in the electrolyzer.

The investigations were carried out on 24 electrolyzers with a current strength of 80 kA, each of which is equipped with 24 anodes measuring 1100x520x520 mm. Closure of the anode with the cathode during the planned removal of the cinder was carried out by lowering it after switching off from the busbar on 8 cm down to contact with the metal (Fig. 7). At the same time, the top of the cinder was in contact with the electrolyte.

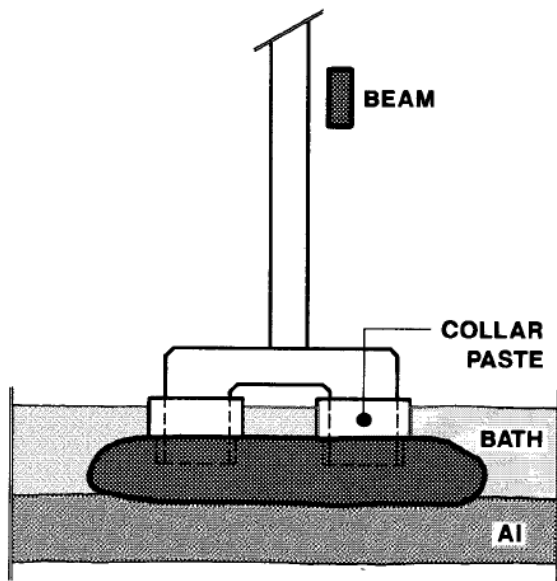


Figure 7: Anode cake after 30 days of anode operation and immersion in liquid aluminum by 2 cm

In various tests, the contact time of the anode with the metal and electrolyte was: 0, 1, 2, 4, 8 and 16 minutes, after which the cinder was removed from the bath and samples from its ends were taken for further analysis.

The current supply interruption was performed as follows. During the last week of testing, half of the cells (12 of 24) were decommissioned at a rate of 2 baths per day. The remaining 12 baths were decommissioned on the last day of production. Immediately after the current was turned off, the anode cinders were extracted from the bath. At the same time from the second batch of electrolyzers were confiscated for 4 cinders, exploited for 10, 20 and 30 days. In the future, these 12 cinders were tested. Due to the constant removal of cinders from the electrolyzers of the second batch, with an average interval of 20 minutes, the total time of the bath in the de-energized state was about 20 minutes.

For testing from each cinder, according to the scheme shown in Fig. 8, 10 cores with a diameter of 50 mm each and a length equal to the height of the cinder were drilled out.

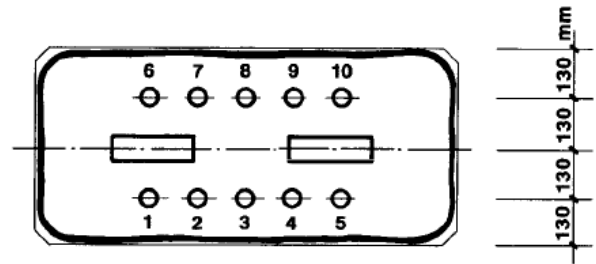


Figure 8: Points for drilling cores from the anode cinder

Even cores on both sides were cut to 20 mm and then, by X-ray fluorescence, they determined the impurity content. Odd cores were used to measure the compressive strength, apparent density, density in xylene, elemental composition.

The research results of the degree of sodium penetration into the cinder when it comes into contact with the cathodic metal and electrolyte are shown in Fig. 9, from which it follows that in just 4 minutes of contact the average concentration of sodium in the cinder reached almost 1%.

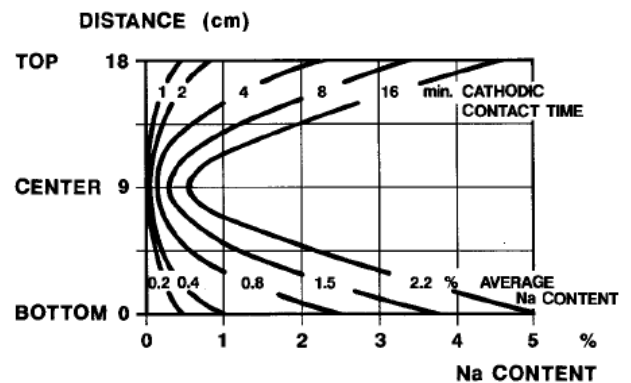


Figure 9: Profiles of sodium concentration at the ends of the anode cinder after its contact with the cathode metal

In this case, in accordance with Fig. 10, the sodium content in the central zone of the cinder is not as high as at its bottom and at the top. Even after 16 minutes of contact, the central zone was not saturated, while the sodium concentration on the peripheral surfaces of the anode significantly exceeded 2%. Also, from Fig. 10 it can be seen that the concentration of sodium on the bottom of the cinder is not much higher than on its surface, and this despite the fact that the bottom is about 2 cm immersed in the metal, while its upper part was completely surrounded by an electrolyte. The average penetration rate of sodium into the peripheral surfaces of the cinder was 0.2% per minute, and the maximum volume concentration of sodium approached 5% vol.

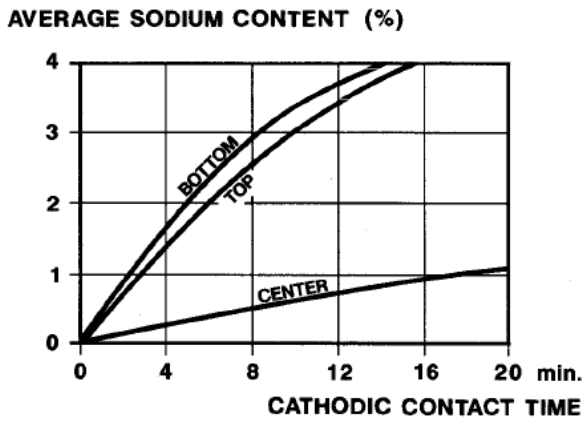


Figure 10: The sodium content in the lower and upper sides, as well as in the center of the anode cinders after their contact with the cathode metal

Table 1: Butt core properties with 16 minutes cathodic contact compared to the butts without cathodic contact (0 minute)

PROPERTIES	UNIT	0 min.	16 min.
Apparent Density	kg/dm ³	1.59	1.69
Compressive Strength	MPa	25	38
Na	%	0.02	2.2
Al	%	0.01	1.1
F	%	0.01	3.0

As shown in Fig. 12, the effect of sodium contamination on the reactivity of the anode cinder is very significant. When heated at a rate of 10°C / min for the first 4 minutes, the reactivity in CO₂ sharply increases, then, after the next 4 minutes, decreases, as ash, formed during the combustion of carbon, protects the surface of the cinder from further oxidation.

When the current is interrupted, the average sodium content on the surfaces of the cinder is much lower than in the cinder which came into contact with the cathode metal. The anode cinders that lasted 10 days had no traces of sodium at all, and only half of the anode cinders that lasted 20 days contained an insignificant amount of it. At the same time, a relatively large content of sodium was found in all the anode cinders that lasted 30 days. As shown in Fig. 11, the profiles of sodium content in the cinder coincide with the profiles obtained as a result of its contact with the anodic metal.

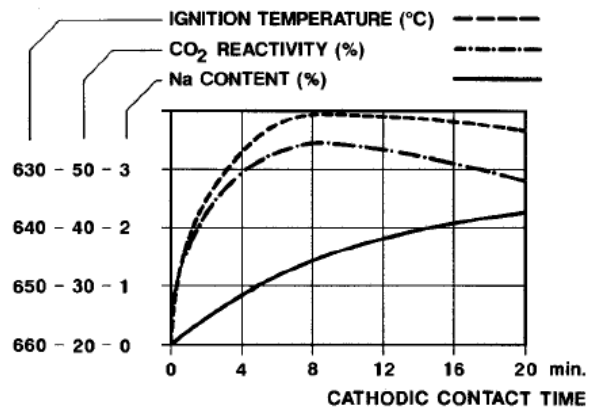


Figure 12: Reactivity of the anode as a function of temperature, CO₂ content in the anode gas, sodium in the anode, and contact time with the cathode metal

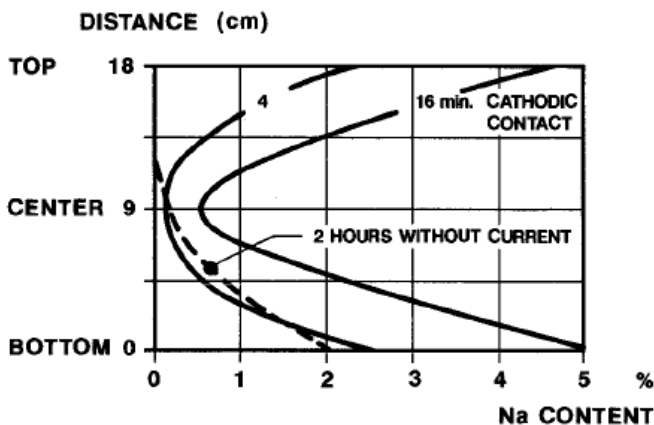


Figure 11: Profile of sodium concentration in anode cinder removed from the electrolyzer after 4 and 16 minutes and 2 hours after the electrolyzer is disconnected from the power supply

To assess the effect of adding crushed cinders on the quality of produced carbon products, anodes of four different compositions were prepared and tested, the formulas of which are given in Table. 2.

Table 2: Bench scale recipes: butts addition characteristics

RECIPE	Total Butts	8 - 1 mm	1 - 0.5 mm	0.5 - 0.1 mm	Ball mill feed
①	20 %	14.0 %	3.0 %	3.0 %	-
②	20 %	11.2 %	2.4 %	2.4 %	4 %
③	17 %	14.0 %	3.0 %	-	-
④	14 %	14.0 %	-	-	-

When impregnating the cinder with the components of the electrolyte, the apparent density and compressive strength significantly increase, as shown in Table. 1.

As shown in Fig. 13 and 14 cinders with a high sodium content significantly increase the reactivity of the anodes in air and in CO₂ - the deterioration of the anode's resistance to oxidation in air was due to the addition of cinder which only contacted the metal for two minutes. The change in reactivity in CO₂ is much lower than in air (Figure 14).

AIR REACTIVITY RESIDUE (%)

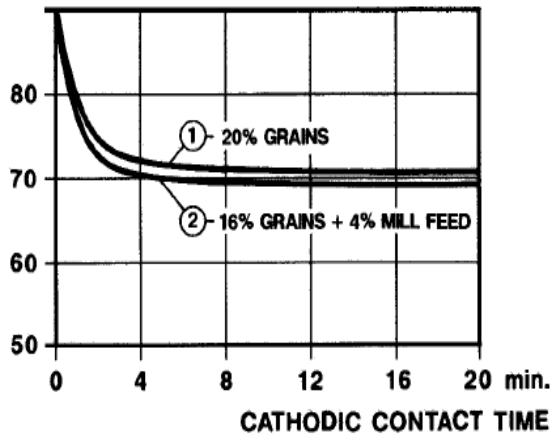


Figure 13: Dependence of the reactivity of the anode on air on the contact time of the anode cinder with the cathode metal

CO₂ REACTIVITY RESIDUE (%)

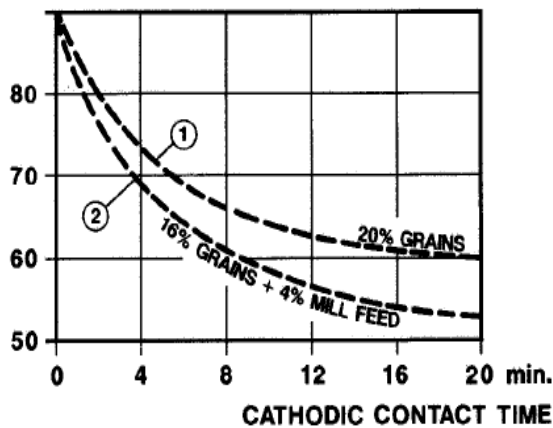


Figure 14: Dependence of the reactivity of the anode in CO₂ on the contact time of the anode cinder with the cathode metal

Removal from the anode of fine fractions of crushed cinder (formulations 3 and 4) increases the reactivity of the anode in CO₂ and in air (Figures 15, 16), which proves the inexpediency of involving sodium-impregnated cinders into the recycling of anodes.

CO₂ REACTIVITY RESIDUE (%)

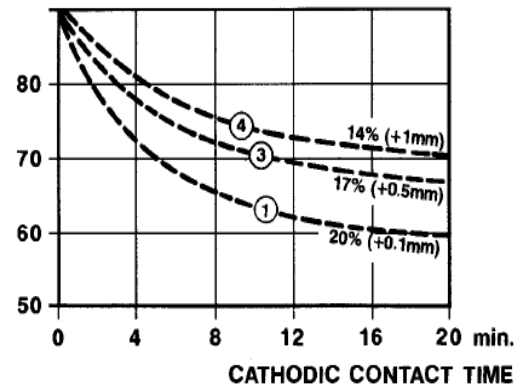


Figure 15: Dependence of the reactivity of anodes in CO₂ on the granulometric composition of crushed cinders involved in the recycling of anodes

AIR REACTIVITY RESIDUE (%)

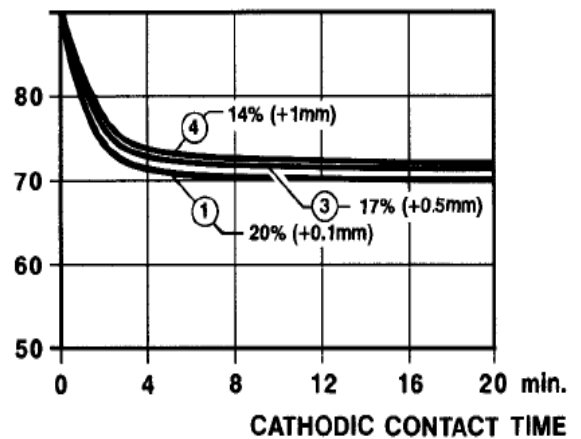


Figure 16: Dependence of the reactivity of anodes in air on the granulometric composition of crushed cinders involved in the recycling of anodes

The rate of impregnation of a porous body depends on the boundary angle between the solid and the liquid. For amorphous carbon, for example, an anode moistened by electrolyte of bath without applying electrical potentials (without electrolysis), the value of this angle is sufficiently small. All the values of the contact angles at the usual temperature and composition of electrolyte for electrolysis are given in the literature, [9, 10, 11, 12], significantly exceed 90° (180° = no wetting and 0° = total wetting). This explains the low impregnation rate observed in the laboratory, when the carbon was placed in a crucible with a cryolite melt. As shown in Fig. 17 the angle of wetting with positively charged carbon is slightly lower than in conditions without electrolysis.

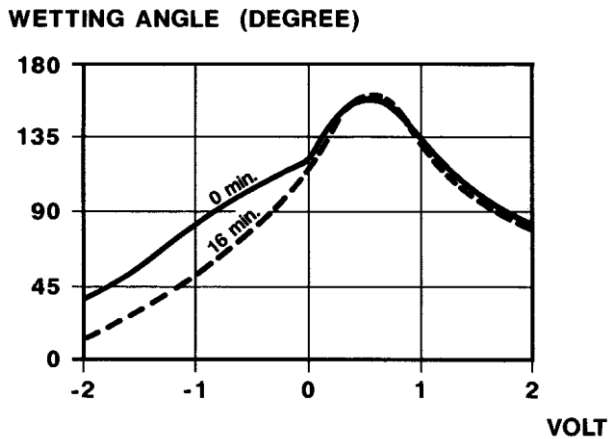


Figure 17: Marginal angle of graphite wetting by cryolite melt, depending on the potential of the reference graphite electrode. It is taken from [13, 14]

Nevertheless, penetration of the electrolyte into the anodes under normal conditions of electrolysis (- 2 volts), can be neglected. This is partly due to the rate of anode consumption (1 - 2 cm / day) - the anode is consumed by the same amount as impregnated with an electrolyte.

Conversely, when the electrode is negatively charged, its electrolyte wetting becomes quite noticeable and even increases with the time of electrolysis. This is a sign of changes in the chemical composition at the interface carbon(anode) - electrolyte during electrolysis.

It is believed that sodium is formed in the negatively charged part of the anode (in the case of contact with the cathode metal) and rapidly diffuses into carbon due to rapid intercalation of Na-C. Because of the intercalation, sodium at the carbon-electrolyte interface interacts with cryolite with forming of sodium fluoride, which enhances the penetration of the electrolyte into the carbon of the anode [15, 16].

That is why, impregnation with the electrolyte occurs quickly when the carbon anodes are in contact with a negatively charged metal. The concentration of sodium in the upper part of the cinders is less than in the lower part, which was in contact with the metal. Thus, the main driving force for impregnation is the hydrostatic pressure of the electrolyte.

In the event of current interruption, the reverse electrolysis process may occur if oxygen ions are available at the anodes (in this case the electrolyzer will work as a galvanic cell), but this is clearly not the case.

The mechanism of impregnation of anodes with electrolyte is not sufficiently studied, there are no reasons why only old anodes were impregnated with electrolyte. Data on the sodium content indicate a small effect on the impregnation of capillary forces, because the level of impregnation of the anode with electrolyte was not higher than the level of immersion of the anode in the electrolyte. This fact can be

explained by the fact that the capillary forces are inversely proportional to the pore size, and as can be seen from Fig. 18, as the electrolyte does not penetrate the micropores of carbon.

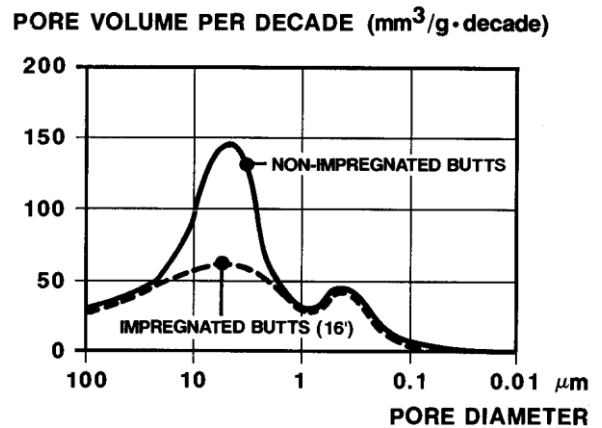


Figure 18: Distribution of pores in the volume of crushed cinders materials (1-1.4 mm) showing the impregnation limit of the electrolyte (pore size ~ 2 nm)

Due to limited penetration, the maximum electrolyte content in the impregnated cinders is about 15% by weight, although the total porosity of the anode is 25%.

Impregnation of carbon anodes can occur, with the following problems [17]:

- too small MPR (pinch effect);
- pieces of carbon under the anode;
- burnout of the nipple;
- the cones presence or the anode are mounted so that it touch/is immersed in metal;
- the presence of current interruption;

To avoid impregnation, care should be taken when extinguishing anode effects by moving the anode array (twitching [18] or tilting [19]) especially when the number of anode effects is higher than normal. Avoid contact between the anode and the metal when replacing the anodes. A neat punching of the crust helps to avoid this bad practice.

The noticeable effect of impregnated cinders on the reactivity of the anode is enhanced, because Sodium fluoride, penetrates predominantly into the anode body as suggested by Grjotheim with co-authors [14] and is confirmed by the data of Table 1. In the case under consideration, in the electrolysis, the relative Na content (35%) is significantly higher than in the initial acid electrolyte (BR = 1.2), where Na content was 25% The alkalization of the electrolyte on one side, and the uniform distribution of ground cinders grains throughout the anode body on the other hand, explains the strong catalytic activity of sodium that was observed when impregnated cinders was used.

It should be emphasized that of the two calcined anodes with the same average sodium content and reactivity, the anode contaminated with ground electrolyte (with millimeter size of inclusions), obtained because of poor cleaning of cinders, is much better than the anode produced with the addition of impregnated cinders.

Interestingly, in the case of a serious impregnation problem, any improvements made on the electrolysis side or mill for the use of cinders, primarily improve the reactivity of the anodes current in the CO₂, and then the reactivity of the anodes in air. This is in good agreement with the observation made from full-size anodes obtained from the same raw material, where positive trends were first observed for the remainder of the reactivity study in the CO₂ current.

As conclusions, we note that the strong influence of cinders impregnated with electrolyte cannot be sufficiently compensated by changes in the processing of cinders before their use, for the production of anodes. It is shown that the contact of cinders with liquid aluminum, quickly leads to unacceptable contamination of the anode with sodium. The interruption of the current in the bath is a potential risk of impregnating the anode with electrolyte and further studies are needed to fully quantify the impregnation phenomenon under these conditions.

If the impregnation capability is a function of the anode cycle, as suggested in this preliminary study, then optimization of the anode work cycle, discussed in [20-32], can affect it.

Obviously, sorting of impregnated cinders is also important as an effective cleaning of cinders. But the main recommendation is to prevent impregnation of cinders by electrolyte during electrolysis.

At the same time, in papers [10; 11] It is recognized that the involvement in the processing of dense, well-cleaned from coal foam cinders significantly improves the quality of the anodes produced. At present, the average sodium content in the anode cinders is in the range of 0.05 to 0.15% by weight. This is due to the introduction of advanced technologies for cleaning cinders, using shot blasting machines or by using the impact of falling balls [12]. Thus, the knowledge of the reasons for the penetration of sodium into the anode cinder is of paramount importance in order to avoid problems in the further operation of the electrolyzer.

In recent years, there has been considerable progress in understanding the reactions that contribute to the general consumption of the anode, and how they relate to the operating parameters and bath designs [13; 14; 15; 16]. When developing various models for the quantitative evaluation of the oxidation reaction, it became apparent that the quality of coke is the most important factor determining the anode consumption [17]. Quantification of foam formation varies considerably and, as a rule, is based on balance data. With an increase of the sodium content in the anode blocks, their

reactivity in the current of CO₂ and in air increases, and as a result, the yield of coal foam increases.

Behavior of coal foam in the electrolyte of aluminum electrolyzers

The behavior of coal particles in the electrolyte has been investigated by many authors. According to [18], the resistivity of the electrolyte increases with the presence of foam, and the smaller the particle size of the foam, the stronger the resistance increases. The reason for this is a decrease in the live section of the electrolyte, since in the electric field of the electrolyzer the carbon particles act as dielectrics that increase the electrical resistivity of the electrolyte.

When the electrolyzer operates with a reduced power consumption and a reduced MWR, an increase in the number of unevenness at the anode bottom is observed. The installation of new anodes with increased geometric parameters causes a significant energy imbalance, accompanied by solidification of the electrolyte at the bottom of the anode and significant fluctuations in its chemical composition, and when operating large anodes, the melting rate of the solidified electrolyte is reduced and the tendency to create unevenness on the bottom is more pronounced. Increases the sensitivity of the anode to the adherence of coal foam, which destabilizes the situation. In this case, there is a need to raise the voltage on the electrolyzer before installing the anode to increase the electrolyte overheating and then reduce it [19].

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