

[13], where the anode samples were made from raw materials with different sodium content. The data obtained in both cases show an extremely high sensitivity of the anode to oxidation in CO₂ in the presence of sodium, regardless of the content of other metallic impurities. It is believed that the large difference in the catalytic effect of Na₂CO₃ on the reactivity of the anode in CO₂ and in air is due to the different behavior of sodium carbonate at these temperatures during both reactions. In the works [11; 12] Such a behavior of Na₂CO₃ is due to its thermal dissociation.

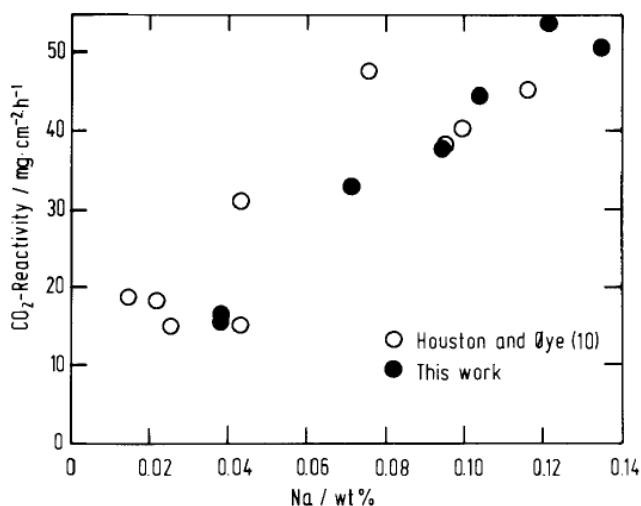
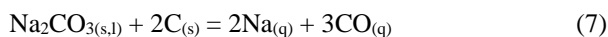
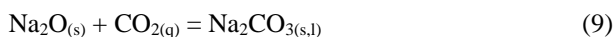
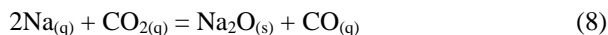


Figure 5: Reactivity of the anode in a current with CO₂ as a function of the sodium content

Due to the high temperature, the following reaction is possible:



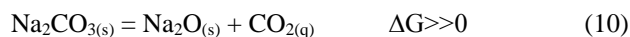
The equilibrium constant for this reaction at 927 °C is $\sim 4 \cdot 10^{-7}$, which can cause a significant increase in the vapor pressure of Na, with decreasing CO pressure. The separation of sodium vapor during the catalytic oxidation of graphite in CO₂ at 1000 °C was indeed noted in [17]. Since sodium vapor has a high reactivity, their formation can lead to the following reactions:



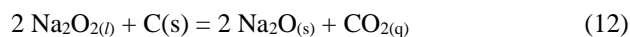
Moreover, at the temperature under consideration, both reactions are thermodynamically possible.

In the case of the oxidation reaction of the anode in air, due to a decrease in temperature <600 °C, the sodium vapor pressure, in accordance with reaction (7), will be insignificant, since the equilibrium constant of reaction (7) is $\approx 4 \cdot 10^{-24}$ at

527 °C, and at this temperature, the dissociation of Na₂CO₃, according to the reaction (4), will be very weak:



Nevertheless, according to [17], the dissociation of sodium carbonate in the presence of carbon and oxygen is enhanced as a result of the following successive reactions:



Which can explain the weak catalytic effect.

The effect of the addition of ZnS with and without the use of Na₂CO₃ is shown in Figure 6.

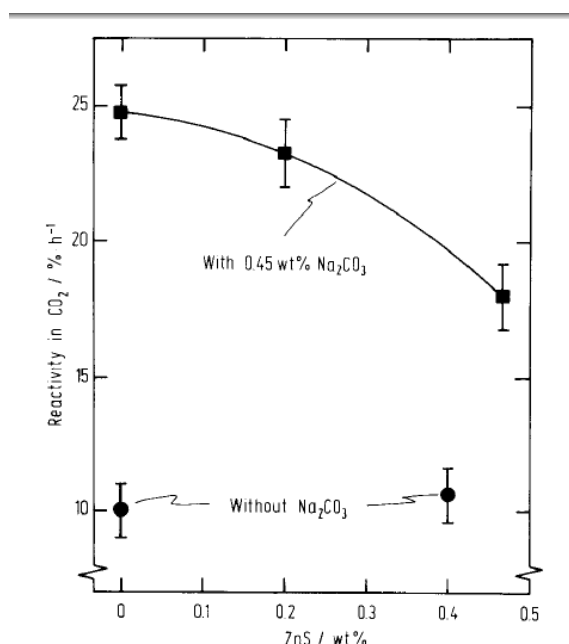
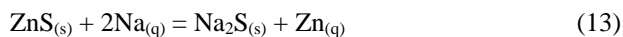


Figure 6: Effect of ZnS on the reactivity of the anode in the current of CO₂ in the absence of Na₂CO₃ and the addition of 0.45% by weight of Na₂CO₃

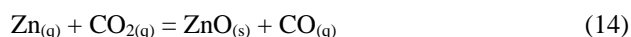
Figure 6 shows that the addition of ZnS only to 0.40% by weight does not cause significant changes in reactivity in the current of CO₂, and the loss of reactivity of carbon is 10-11%, which can be attributed to the experimental error. Addition of 0.45% by weight of Na₂CO₃, as expected, increases the reactivity loss to 25%, in the absence of ZnS.

An interesting feature of the experiment was the fact that the amount of Na₂CO₃ in the samples was maintained at a constant level of 0.45% by weight, while the content of the ZnS additive was increased from 0 to 0.47% by weight, which resulted in a decrease in the reactivity of the anode in CO₂ from 25 to 18%. In other words, the catalytic effect of Na₂CO₃ decreases with increasing S / Na ratio in the sample. The reason for this behavior is probably the presence of additional chemical equilibrium, which can be expressed as follows:



As indicated earlier, the sodium vapor pressure is again formed by reaction (7) due to the dissociation of Na_2CO_3 . However, in the second stage of the catalytic cycle, reaction (8) competes with reaction (13). From the thermodynamic point of view, the Gibbs energy of reaction (13) is $\Delta G = -136.8$ kJ, and the reaction (7) $\Delta G = -67.8$ kJ at 927°C . Therefore, it is reasonable to assume that at least a portion of the sodium vapor is converted to Na_2S , and thus the amount of free sodium for the catalytic cycle decreases.

The vapor of zinc, formed as a result of reaction (13), is usually oxidized to ZnO :



As indicated above, ZnO is not active with respect to oxidation of the anode in CO_2 , and Na_2S is a very stable compound, hardly active as a catalyst for reactivity. Thus, reaction (13) is considered to be an obstacle to the observed catalytic effect produced by Na_2CO_3 .

In the light of the foregoing, it is possible to use a similar interaction of sodium and sulfur in the compositions of industrial anodes. Petroleum coke with a high sulfur content may have a positive effect on reducing the reactivity of the anode in a current of CO_2 by suppressing the catalytic ability of sodium. This may cause disagreement with previously published data on the effect of sulfur on the oxidation of the anode in CO_2 .

The effect of the addition of Al_2O_3 on the physical properties of the anode is shown in Table. 1.

Table I. Some Physical Properties of the Anode Blocks Containing Al_2O_3 as additive

| Al_2O_3 content (wt%) | Apparent Density (g cm^{-3}) | Cumulative Pore Volume ($\text{cm}^3 \text{g}^{-1}$) | Total Poro-sity (%) | Electrical Resistivity ($\mu\Omega\text{m}$) | Thermal Conductivity ($\text{Wm}^{-1}\text{K}^{-1}$) |
|---------------------------------------|---|--|---------------------|--|--|
| 0 | 1.53 | 0.156 | 23.6 | 62.2 ± 1.4 | 3.99 ± 0.03 |
| 1 | 1.50 | - | - | 66.4 ± 1.1 | - |
| 2 | 1.52 | 0.157 | 23.9 | - | 3.94 ± 0.04 |
| 3 | 1.51 | - | - | 60.3 ± 2.0 | - |
| 5 | 1.55 | 0.153 | 23.7 | 65.8 ± 0.6 | 3.80 ± 0.04 |

The apparent density and porosity of the anodes with the addition of Al_2O_3 remain unchanged, as is the pore size distribution, as illustrated in Figure 7 for "pure" anode samples and samples with an additive of 5% by weight of Al_2O_3 .

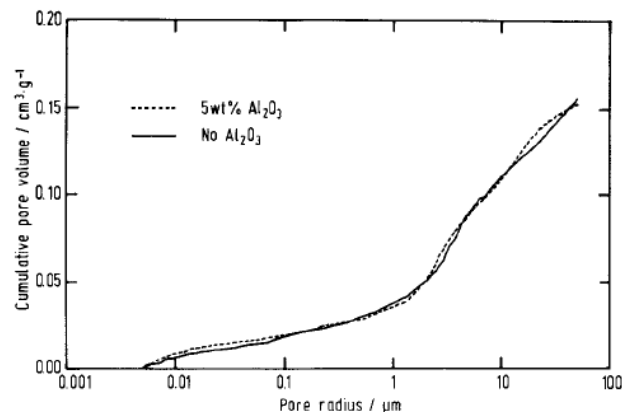


Figure 7: Typical pore spectra of anode samples without the addition of Al_2O_3 and with the addition of 5% by weight of Al_2O_3

The correlation between the resistivity of the anode and the content of Al_2O_3 is not observed, and the thermal conductivity decreases almost in proportion to the increase in the Al_2O_3 addition.

Reactivity of anode samples with an additive of 5% by weight of Al_2O_3 is shown in Figure 8.

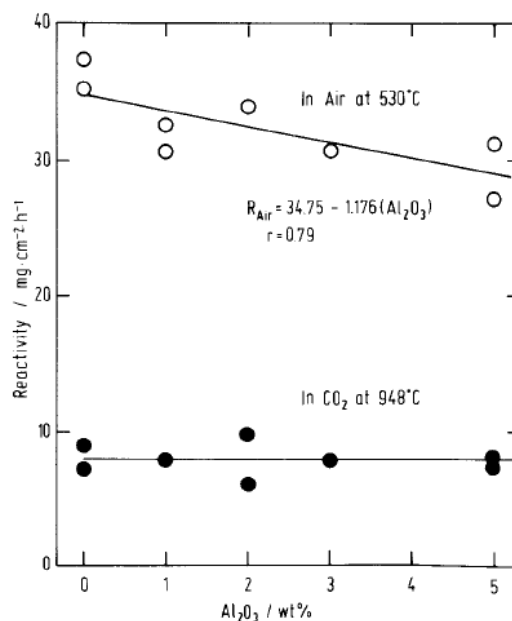


Figure 8: Effect of Al_2O_3 on the reactivity of the anode in air and CO_2

While the reactivity in air linearly decreases with increasing Al_2O_3 content, the reactivity in the current of CO_2 remains at the same level, at any content of aluminum oxide. This different behavior of the anode samples in air and in CO_2 is believed to be due to the temperature difference at which these reactions were studied.

From the technological point of view, the reduction in oxidizability in air is very useful, since the consumption of the anode decreases. Another useful aspect is a decrease in thermal conductivity with the addition of Al_2O_3 , which will reduce the temperature of the anode in the industrial cell. According to [13], a decrease in thermal conductivity of $0.1 \text{ W}^{-1}\text{K}^{-1}$ reduces the anode surface temperature by 2.75°C . Even such a small decrease in temperature in the upper part of the anode will lead to significant savings in the carbon material, since its reactivity in air depends strongly on temperature. This combined effect makes the addition of Al_2O_3 to industrial anodes attractive.

The effect of the addition of Al_2O_3 on the anode consumption

The effect of the addition of Al_2O_3 on the anode consumption in the laboratory cell is shown in Figure 9.

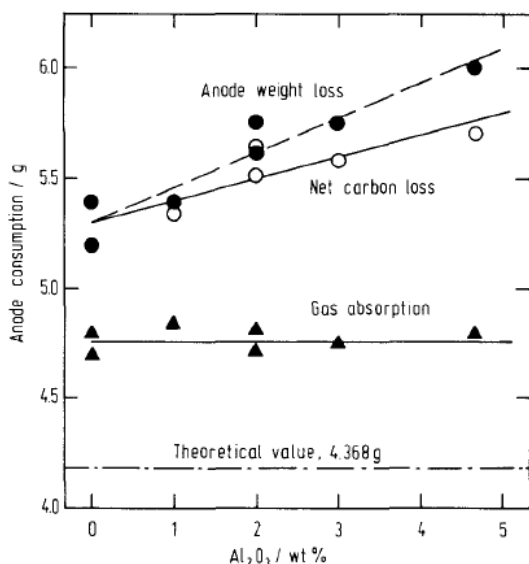


Figure 9: Effect of Al_2O_3 on the anode consumption after electrolysis at 980°C and 13 A, for 3 hours. The anode consumption is determined by the loss of its weight minus the Al_2O_3 dissolved in the melt

The theoretical anode consumption corresponds to its 100% oxidation in CO_2 . It is seen that with an increase in the Al_2O_3 content, the anode is consumed at a faster rate. Nevertheless, some of these losses are caused by the dissolution of Al_2O_3 in the electrolyte.

Anode weight loss correction for the corresponding Al_2O_3 content gives a net carbon consumption, which also increases with the addition of alumina. These data are presented in Figure 10. Oxidability of the anode does not cause an increase in the anode flow rate and this agrees with the results of the tests for the determination of the reactivity in the CO_2 current.

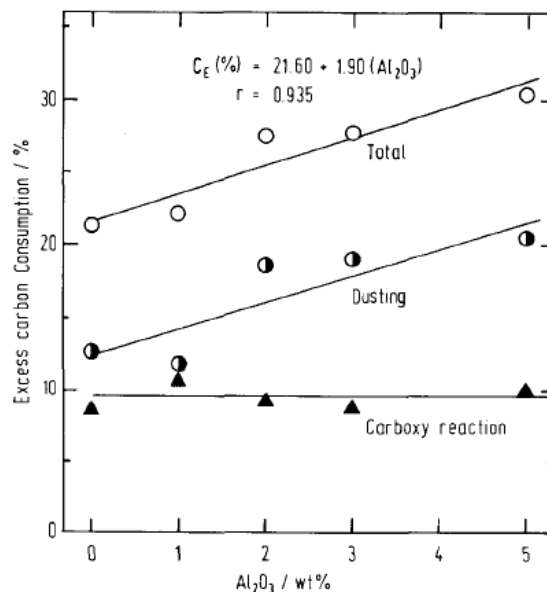


Figure 10: Dependence of excess carbon consumption on the content in the anode of Al_2O_3

To a higher consumption of carbon material leads the formation and removal of coal foam. In accordance with Figure 10, the most optimal is the addition of Al_2O_3 in an amount of 1.9% by weight.

Summarizing the results obtained, the authors of [14] draw the following conclusions:

- Na_2CO_3 acts as a strong catalyst for reactivity, while its effect on the rate of carbon oxidation in air is very modest;
- ZnS is not active for reactivity, nevertheless it has the ability to significantly reduce the catalytic ability of sodium, especially in the presence of Na_2CO_3 ;
- Al_2O_3 does not affect the reactivity in CO_2 , but reduces the reactivity in air.

It was established in [15] that V and Ni are catalysts for oxidation of the anode in air. The same is true for Na and Fe if we consider the reactivity of the anode in CO_2 . However, it is often difficult to separate the effect of different elements due to the joint presence of other impurities. An example of this is the presence of sulfur, about which contradictory results were reported in the work.

Additives AlF_3 , B_2O_3 , SiO_2 and phosphorus-containing compounds were tested as inhibitors of anode oxidation [9]. However, only a few published data about the effect of Al_2O_3 on the reactivity and anode carbon consumption. It was reported in [16] that Al_2O_3 catalyses the oxidation of graphite in air. A significant increase in the consumption of Soderberg test anodes containing Al_2O_3 as an additive was reported in [17]. In the works [18; 19] as an alternative to the traditional, composite anodes containing from 20 to 85% by weight of Al_2O_3 were proposed, while in [13] there is a minimum

oxidation of such an anode by air, apparently due to a decrease in the fraction of carbon in it.

Proposed measures to reduce the consumption of pure carbon and the yield of coal foam.

According to [20], the surface area available for reaction (7) depends on the properties of the pitch and the depth of its penetration into the pores of the coke-filler. When coke is impregnated, the maximum capillary rise height h_{max} is determined by the surface tension (σ) of the liquid phase, the wetting contact angle ($\cos \theta$) and the density ρ_l and can be determined from the expression:

$$h_{max} = \frac{2\sigma_l \cos\theta}{r\rho_l g} \quad (15)$$

According to [21], the Maximum lifting height of the pitch along the capillaries depends on its softening temperature and reaches 7 mm. The most rapid lifting height of the pitch in the capillaries varies in the temperature range 150 - 175 ° C. A further increase in temperature to 200 ° C does not lead to a noticeable change in the lifting height of the pitches in the capillaries.

Increase lifting height of the pitch in coke capillaries, and thus reduce the consumption of the binder is proposed through the use of a sound capillary effect, when ultrasound causes an abnormally deep penetration of liquid into capillaries and narrow slits. In this case, the height of the lifting and the depth of penetration significantly exceed the corresponding values, caused only by the surface tension of the liquid pitch [22]. The use of a sound-capillary effect will ensure an improvement in the quality of the anode mass with a lower pitch consumption, which will have a favorable effect on the environmental performance of both the anode production and aluminum production in general.

Another way to increase the degree of impregnation by pitch is to impart a multi-polar electrical charge of coke dust and pitch using a high-voltage DC charging unit that provides a voltage on the electrodes from 24 to 50 thousand volts. In this case, the pitch is given a negative charge, the coke dust is positive [23].

Laboratory studies have shown that charging the components of the anode mass increases the rate of wetting and impregnation of dust by the pitch in 2-3 times, as evidenced by the shorter time interval during which the moistened coke was immersed in the pitch melt (Figures 11-16) [24].



Figure 11: Without giving charge to AM components, the initial state



Figure 12: With the charged the AM components, the initial state



Figure 13: Without charging the AM components after 50 seconds

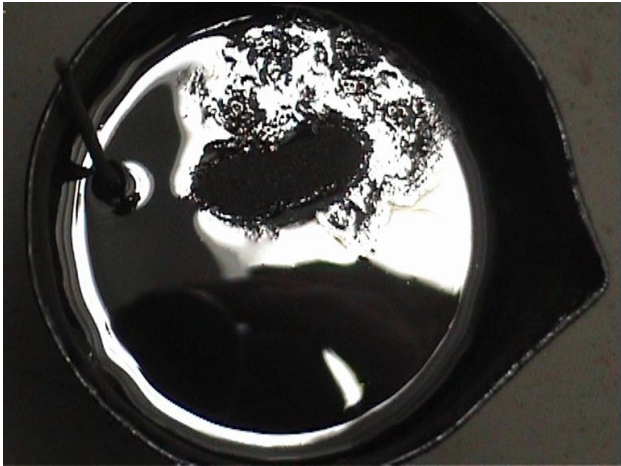


Figure 14: With charged the AM components in 50 seconds



Figure 15: Without charging the AM components after 5 minutes

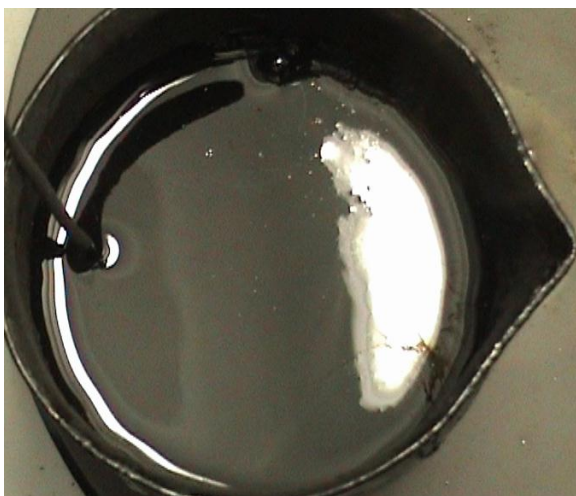


Figure 16: With charged the AM components in 3 minutes

The technology of charging components of pitch and coke with multi-polar charges can be easily adapted to the existing technological process of obtaining anode mass. In this case, a high-voltage dust charging installation is located between the coke dust preheater and the mixer, pitch - between the pitch meter and the mixer (Figure 17).

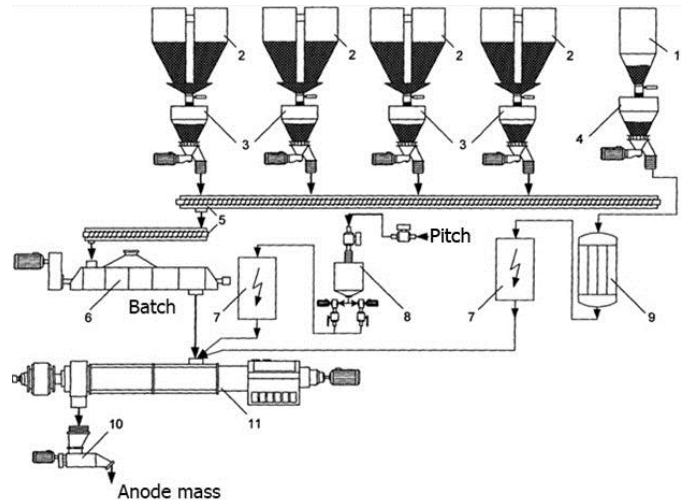


Figure 17: Technological scheme for obtaining anode mass with a high-voltage charging installation: 1,2 – bunkers of dust and coke bunches; 3,4 - dosing equipment for dust and coke bunches; 5 - auger conveyor; 6 - charge heater; 7 - high-voltage charging installations of coke dust and pitch; 8 - pitch dispenser; 9 - dust preheater; 10 - extruder; 11 - mixer

The algorithm for obtaining anode mass according to the proposed technological scheme is the following. The components of coke batch - bunches of different fractional composition from bunkers 2 by metering devices 3 and transport screws 5 are fed to preheater 6 where they are heated to a temperature of 200-220 ° C. Further, the heated components of the coke batch are fed into the mixer 11. Coke dust before entering the mixer 11 by the dispenser 4 from the bunker 1 is fed into the dust preheater 9 where it is also heated to a temperature of 200-220 ° C. Pitch, preheated to a temperature of 200-2400C, is fed into the dispenser 8. Further heated coke dust and pitch enter high-voltage charging units 7, where they acquire electrical charges for 1-3 seconds, pitch-negative, and coke dust-positive. At the same time, the voltage at the electrodes of high-voltage charging stations is in the range of 24000-50000 V DC. Charged in this way, pitch and coke dust also enter mixer 11, where they are mixed for 3-4 minutes with each other and with coke batch. From the mixer 11, the finished anode mass is fed into the extruder 10 to form briquettes. In general, the process of anode preparation takes 4-5 minutes, which is 2 times lower than with "traditional" technology [25-33].

The presented technology allows essentially to raise productivity of the manufacture equipment of an anode mass at the expense of time reduction of mixing more than in 2

times. Reducing the mixing time of components reduces the specific electricity consumption for anode production by 20-25 kW / t, taking into account the energy consumption of the high-voltage charging unit. An increase in the degree of impregnation of coke dust with pitch reduces the anode fall-off under the conditions of the active electrolyzer and reduces the yield of coal foam by 5-7 kg / ton of Al. Reducing the yield of coal foam reduces the time of the electrolyzer in the depressurized state by an average of 0.2-0.25 hours during the day, which reduces the specific fluoride emissions by 0.1-0.2 kg / ton Al.

According to [25], oxidation of the anode occurs over all its surfaces, as shown in Figure 18

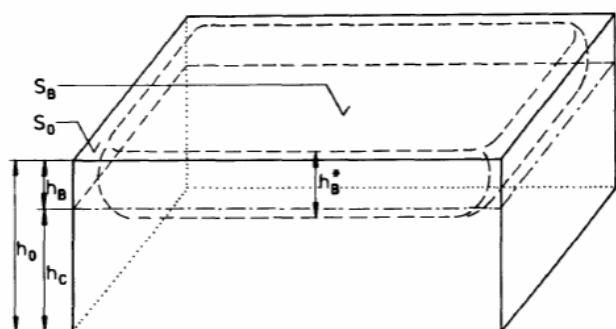


Figure 18: Dimensions and contour of the anode and anode cinder

To reduce the oxidation of the side surface of the anode is proposed by means of a device on its side, facing the flange sheet of the cathode device of the electrolyzer of the notch-shelf, as shown in Figure 19 [26].

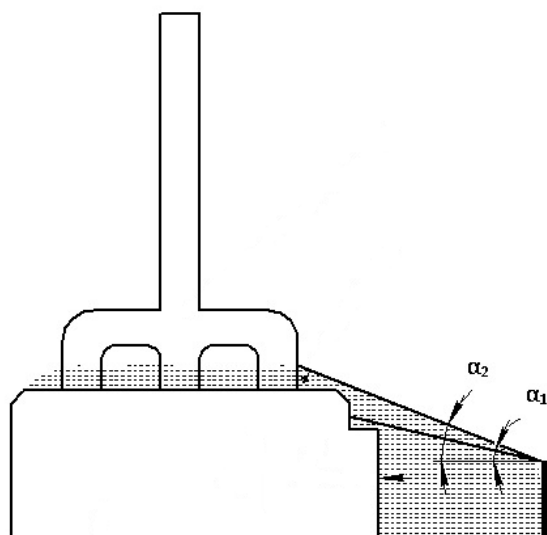


Figure 19: Annealed anode with a notch on the side surface

With this anode configuration, the loading of the covering material is carried out in two stages. The first layer of the

covering material is loaded onto the notch-shelf, forming an angle α_1 between the upper boundary of the covering material and a horizontal plane within the range of $37 - 42^\circ$, which corresponds to its angle of repose. Further; after sintering of the covering material and formation of a firm crust on its surface, a second layer of covering material is loaded onto it, forming an angle α_2 between the upper boundary of the covering material and a horizontal plane in the range $55-60^\circ$. Thus, the efficiency of protecting the front side of the baked anode from air oxidation is increased, its mass is reduced, and also the anode cinder, involved in the recycling of the anodes.

Increase the stability of the upper surface of the anode to oxidation by air is proposed by changing the algorithm for loading the cover material onto it. Initially, a layer of cryolite-alumina batch with a thickness of 5-10 cm is loaded onto the anode surface. Then, after 1-1.5 hours, an alumina layer 3-5 cm thick is loaded onto the crust of the sintered cryolite-alumina batch [27, 34-37]. When loading the cryolite-alumina batch on the surface of the anode massif, the alumina entering it is impregnated with electrolyte vapors, and the fluoride salts begin to melt when heated to 700°C , thus forming a strong crust with low gas permeability, from 0.02 to 0.04 NPM. However, the thermal conductivity of the crust can reach $1.5-1.6\text{ W / m} \cdot \text{K}$, which is accompanied with high losses of the heat by the electrolyzer. A decrease in these losses is ensured by an alumina layer whose thermal conductivity is $\sim 0.14\text{ W / m} \cdot \text{K}$.

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