INERT ANODES: AN UPDATE

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This overview covers the development of inert anodes for the primary aluminium industry in the period 2010 -2013. It continues the review of cermets, including their mechanical and physical properties, their behaviour and their manufacture, especially Cu (NiO-$NiFe_2O_4$) cermets in cryolite melts. However, the overview focuses particularly on the manufacture and behaviour metal anodes, including steels and Ni-Fe alloys. Low-temperature electrolytes are essential to avoid aggravated corrosion; KF-based electrolytes have proved suitable for low-temperature electrolysis. Inert anodes were tested on laboratory and bench scales, and Rusal announces industrial scale tests in the near future.

Introduction

The use of inert anodes instead of carbon eliminates the generation of the greenhouse gas CO_2 when oxygen liberated from the dissociation of alumina reacts with anode carbon. It also eliminates perfluorocarbon (PFC) by-products and other polluting emissions such as PAHs. This update is a continuation of [1].

Cermets

During recent years investigations focussed on the use of nickel ferrite. The technology of spinel $NiFe_2O_4$ powder production was reviewed by Samoylov et al. [2] including mixing of iron oxide powder Fe_2O_3 and nickel oxide powder NiO, pressing as ingot, thermal treatment of pressed forms in air at 1000-1200°C, and subsequent grinding of sintered specimens. Generally cermet production consists in mixing $NiFe_2O_4$, NiO and metal powders (Cu or Ag), then pressing to reach 60% of their theoretical density, and sintering it in argon flow, heating up in for 4 hours to 1350°C and holding at this temperature for 4 hours, and finally cooling down to room temperature. They achieved a density of the specimens between 5.46 to 5.52 g/cm^3.

A two-step sintering process was adopted by Zhang et al. [3] to prepare $NiFe_2O_4$ inert anodes. For the production of $NiFe_2O_4$ spinel, Fe_2O_3 and NiO powders were heated for six hours at 1000°C. After crushing and sifting the oxide they added $NiFe_2O_4$ nanopowder. The as-prepared mixture was then uniaxially compressed and they sintered the nickel ferrite a second time at 1300°C for six hours. The addition of 0 to 40 wt. % $NiFe_2O_4$ nanopowder significantly influenced the density, porosity, bending strength and impact toughness. Inert anodes with 30 wt. % nanopowder had the best compressive properties. With this composition the authors obtained the following results: density 4.86 g/cm^3, porosity 3.5%, bending strength 42.47 MPa, and impact toughness 3.31 J/cm^2.

The effect of the sintering atmosphere was then examined by Zou et al. [4]. 5Cu/(10NiO-$NiFe_2O_4$) cermet anodes were prepared by the cold-pressing sintering method in different atmospheres. Furthermore the phase composition, microstucture and mechanical properties were investigated. The results reveal that 5Cu/(10NiO-$NiFe_2O_4$) cermet materials can be obtained by sintering in a vacuum (oxygen content 0.02 ppm) or in atmospheres of Ar + air with oxygen content 10 ppm, 200 ppm, 2000 ppm and 10000 ppm respectively. The $NiFe_2O_4$ content in the ceramic material increases with the oxygen content in the sintering atmosphere and the Cu in the $NiFe_2O_4$ phase decreases. When the oxygen content in the sintering atmosphere is only 10 ppm the grain size of 5Cu/(10NiO-$NiFe_2O_4$) cermet is 5.43μm, and the bending strength reaches 80.05 MPa at room temperature.

Then Wang and Sun [5] examined the electrochemical behaviour of 5Cu/(10NiO-$NiFe_2O_4$) cermets in low temperature melting electrolytes, alumina-saturated for aluminium electrolysis. Two anodic processes were observed at the cermet anodes: dissolution of copper and evolution of oxygen. The surface activity of anodic films suggests the lack of surface oxide stabilisation. The exchange current density for cermet anodes was 0.19 A/cm^2. The porous surface caused by the depletion of copper during anodic polarization could explain the oxygen evolution blocking on the surface of the cermet anode.

In another experiment Liu et al. [6] examined the phase evolution of 17(Cu-Ni))-(10NiO-$NiFe_2O_4$) inert cermet anodes. A dense $NiFe_2O_4$ layer was observed on the anode surface and thickened with longer electrolysis time. In the newly formed dense ceramic layer, the NiO phase disappeared as a result of being swallowed by the $NiFe_2O_4$ phase, and the metal phase was oxidized during the electrolysis process coupled with a higher dissolution rate of Cu compared with Fe and Ni.

He, Xiao et Zhou [7] tested oxide additions to see whether they could improve the corrosion resistance. They added 1% BaO to xCu(10NiO-$NiFe_2O_4$) cermet (x = 5, 10, 17) inert anodes. They observed significant corrosion of Cu during the experiments: many pores appeared on the surface of the anode, and electrolyte infiltration into the anode during electrolysis. The addition of BaO to the

cermet material proved to be unfavourable to corrosion resistance, possibly because BaO at the grain boundary of the anode accelerated the corrosion of the cermet.

Liu, Zhao and Li [8] observed that the addition of TiO_2 and V_2O_5 to $NiFe_2O_4$ spinel could improve the electric conductivity and corrosion resistance because of the modification of the microstructure of the $NiFe_2O_4$ grain boundary.

Then Du et al. [9] investigated the effect of MnO_2 additions on early stage sintering behaviour and properties of $NiFe_2O_4$ ceramics. The addition of 1.0 wt. % MnO_2 to $NiFe_2O_4$ improves the density. The relative density reaches a maximum of 93.63% and the bending strength is 38.75 MPa.

Zhang et al. [10] studied the effect of addition of ZrO_2 on properties of nickel ferrite cermets. The results show that with 0.5 wt. % ZrO_2 the relative density slightly increases but the corrosion resistance decreases a little, while the bending strength improves remarkably from 55.5 MPa without ZrO_2 addition to 105.26 MPa. The higher the ZrO_2 content, the higher the corrosion rate.

Alloys

In laboratory scale Helle et al. [11] prepared a series of intermetallic compounds with the general formula $(Cu_{65}Ni_{20}Fe_{15})_{100-x}O_x$ by high energy ball milling and sintering, and they evaluated this composition as inert anode material for aluminium electrolysis at 700°C. All compounds had the same composition, but O_2 was added at different amounts during the milling process. All compounds show a γ-phase. However, they obtained the best corrosion resistance in low temperature KF-AlF$_3$ electrolyte when they added 1.4 wt. % of oxygen in the initial alloy

Nickel-iron alloys have been identified as promising inert anode candidates for the Hall-Héroult process. Chapman, Welch and Skyllas-Kazacos [12] tested various compositions, and subjected them to short-term galvanostatic electrolysis in a cryolite alumina bath at 960°C. Prior to electrolysis they oxidized the anodes at 800°C for 48 hours, forming a protective scale in which they identified Fe_2O_3, $Ni_xFe_{3-x}O_4$ and $Ni_xFe_{1-x}O_y$. Anodes having a Ni content of 50-65 wt. % performed best during short-time electrolysis. The preformed oxide scale was efficient in reducing anode wear and fluoridation. In the absence of preformed scale, the anodes suffered appreciable internal corrosion and/or passivation due to metal fluoride formation.

Zhu, He and Wang [13] prepared Fe-30Ni-5NiO alloys as inert anode materials. The metallic alloy with NiO nanoparticle (mean size around 20 nm) anodes were prepared by spark plasma sintering. The authors investigated the oxidation behaviour of these anodes at 800 and 850°C. A continuous Fe_2O_3 film was formed on the anode surface during electrolysis. Although this alloy exhibits good corrosion resistance to fluoride electrolyte

during the electrolysis process, such pre-oxidized anodes remain not suitable for inert anode application.

The anodic behaviour of surface-oxidized Ni-Fe-Co alloys was investigated over short periods of aluminium electrolysis. Singleton, Welch and Skyllas-Kazacos [14] found that additions of 10 wt. % Co significantly improve the anodic wear resistance due to the suppression of Fe_xO formation. In general, the protective ability of the preformed oxide scale was greatly affected by the level of porosity and surface adhesion. In electrolytes containing less than 4 wt. % Al_2O_3, they observed catastrophic failure of the anodes due to an overpolarization at the reaction interface.

Inert anode compositions containing Ni-Fe-Cu were investigated several times [15-17]. Helle et al. [15] prepared $Cu_xNi_{85-x}Fe_{15}$ ($0 \leq x \leq 85$ wt. %) alloys by mechanically alloying. A stable anode for aluminium electrolysis in low temperature KF-AlF$_3$ electrolyte was obtained for $65 \leq x \leq 85$. However, a substantial increase of the Cu contamination in produced aluminium was observed for x>70. Goupil et al. [16] evaluated cold spray deposition for the production of inert anodes for the aluminium industry. $Cu_{65}Ni_{20}Fe_{15}$ (in wt %) alloy was prepared from the respective metal powders. In a first step they showed that they could control the particle size of the milled powder with the addition of stearic acid to the initial powder mixture. Then they optimized the cold spray parameters (pressure and carrier N_2 gas) and produced thick (1100μm), dense (porosity 1-2%) and adherent (adhesion strength of 15 MPa) coatings of $Cu_{65}Ni_{20}Fe_{15}$ on nickel aluminium bronze alloy. This permits envisioning the fabrication of large area electrodes, as required for pilot scale aluminium electrolysis tests.

Simakov and co-workers [17] reported laboratory electrolysis tests lasting about 270 hours at Rusal's Engineering & Technology Centre at Krasnoyarsk. Tests showed that the consumption of inert anodes is less than 2.5 cm/y and the aluminium purity is better than 99.2%. The near future will see the development inert anode tests for more than 2 kA, and then the development of a cell that can operate at a current of more than 100 kA. The anode metal structure contains two phases due to the separation of a liquid solution into two solid solutions; a solid solution of copper in iron and a solid solution of iron in copper. Nickel is dissolved in both phases.

Nguyen [18] optimized an alloy composition containing 64-66% Ni, 25-27% Fe, 7-9 % Mn, 0-0.7% Cu and 0.4-0.6% Si, where the weight ratio Ni/Fe is preferably 2.3-2.6, the weight ratio Ni/Ni + Cu is greater than 0.98, the weight ratio Cu/Ni is less than 0.01 and the weight ratio Mn/Ni is from 0.09 – 0.15. The alloy surface can include $NiFe_2O_4$ produced by pre-oxidation of the alloy.

The influence of yttrium addition (5 wt. %) on the corrosion resistance of the mechanically alloyed $Cu_{65}Ni_{20}Fe_{15}$ [16] anode for aluminium electrolysis in low temperature KF-AlF$_3$ electrolyte was investigated by Ouvarov-Bancalero et al. [19]. Y atoms do not dissolve in

the Cu(Ni, Fe) phase by milling and they induce the formation of Y-Ni precipitates during subsequent powder consolidation procedure. These inclusions improve the alloy corrosion resistance by slowing down the outward diffusion of Cu in Cu oxides. However, the purity of the produced aluminium still seems to be limited.

Samoylov et al. [20] examined specimens at Rusal's Engineering & Technology Centre produced by auto-pyrolysis. These alloys are: Ni-6Al-10Cu-11Fe-X alloy. X is a small dopant (1-3%). During electrolysis tests they highlighted an augmentation if the mass due to high residual porosity of 28-35%, to the presence of poor-adhesive oxide layers, and to some swelling of the lower part of the anode which was immersed in the electrolyte. This kind of anode cannot be used in aluminium production. But another homogenous metallic anode performed better: the composition of Ni-6Al-10Cu-3Zn alloy, produced by hot pressing did not show swelling or mass growth during the electrolysis test. Homogenous as well as gradient metal-ceramic anodes, produced by hot pressing, provided a dense cermet material with high concentration of oxide phases. As-prepared anodes have presented better stability during electrolysis tests. Metal-ceramic samples made by hot pressing showed the best resistance to cryolite-alumina melt during electrolysis. This was linked to the low residual porosity remaining in the operative material at high $NiFe_2O_4$ and NiO concentration.

Tkacheva et al. [21] examined in laboratory scale the operating parameters of aluminium electrolysis in KF-AlF_3-based electrolyte with an aluminium bronze anode and a wettable cathode. After each electrolysis test, the aluminium bronze anode was covered by a thin, flaky oxide layer. No correlation was found between the anode layer and the electrolyte composition, nor between the anode layer thickness and the operating temperature The thickness of the anode layer increased during the first 50 hours of electrolysis and then stabilized at 0.6-0.7 mm. Aluminium bronze anodes covered by a protective oxide layer can be effectively used as inert anodes during long-time testing. In continuation of these results Hryn et al. [22] reported about aluminium electrolysis testing in a 1000 A cell at 750°C. The cell was fitted with vertically oriented aluminium bronze anodes and wettable cathodes. The electrodes were immersed in a KF-AlF_3-electrolyte with a cryolite ratio of 1.3. Alumina was fed automatically to the cell to maintain a dissolved alumina concentration at 5 wt.%. Oxygen gas evolved from the cell and was measured. During the 24 hour electrolysis test, the anodes were protected by a dense oxide layer.

Chemical analysis

Ren and Sha [23] measured the concentration of fluoride ions in low-temperature aluminium electrolyte using the fluoride ion-selective electrode method. Based on the formula of molecular proportion they compared the actual and the calculated molecular proportion in low-temperature aluminium electrolyte. They found that the correction coefficient of molecular proportion was $\lambda = 0.9541$. This method has been used to measure molecular proportion in KF-AlF_3-Al_2O_3 and KF-AlF_3-Al_2O_3-NaF low temperature electrolytes. The results were consistent with those obtained by chemical analysis. This proves that the ion-selective electrode method can be used to measure molecular proportion of KF-AlF_3-Al_2O_3-X low temperature electrolyte systems.

Other processes

Nickel-based hydrogen diffusion anode

Namboothiri et al. [24] conducted laboratory scale electrolysis experiments to investigate the electrowinning of aluminium using a hydrogen diffusion anode. A potassium-based electrolyte (KF-AlF_3-Al_2O_3), porous nickel anode and molybdenum disk cathode were used in the experiments at 750°C. Hydrogen was supplied to the anode/electrolyte interface through the porous anode. They observed a measurable depolarisation of the anode potential and also an anode reaction of hydrogen and oxygen ions in the bath to form steam detected at the electrolysis exit gas pipe. Aluminium was found on the spent cathode. The experiments suggest that the rate limiter for hydrogen oxidation was the availability of surface hydrogen at the anode/electrolyte interface. The anode surface was corroded during electrolysis.

Mushy electrolysis

Beck [25] reported a new process which has been under development since 1990 that promises 20% lower capital cost and 20% lower operating cost and no CO_2 or fluorocarbon emissions. A new cell design, new anode and cathode materials, new electrolyte and new operating conditions are based on experience over the past 60 years. Multiple vertical anodes and cathodes are possible resulting in smaller cells than the conventional Hall-Héroult cells. Electrical connectors for the cathode must be protected from oxidation. Solid alumina parts require that they do not dissolve in the electrolyte. The bottom of the cell is the anode to produce oxygen bubbles that prevent alumina particles from settling The liner is a CuNiFe-alloy in a thermally insulated enclosure. The cathode is made of a fine porous layer of TiB_2 deposited by chemical vapour deposition on porous alumina filter plates. The electrolyte consists of eutectic NaF:KF:AlF_3, with a melting point of somewhat above 600°C. However, to continue this work further funding and facilities are needed to scale up the process.

Cell design

While Lamaze [26] proposes a device and method for connecting inert anodes for the production of aluminium,

Galasiu and Galasiu [27] propose an electrolysis cell consisting of inert anodes and wettable cathodes both consisting of perforated plates, through the holes of which oxygen is released at the anodes while aluminium drops through the holes of the cathode. Short interpolar distance of about 1 cm should be appropriate, as the oxygen gas bubbles are small and escape along the anode sides. Only $KF-AlF_3$-based electrolytes with a low operating temperature of about 750°C can be used.

State-of-the-art

Cermet inert anodes

In 2012, Li and co-workers [28] presented results of a pilot test in a 20 kA cell in order to investigate the corrosion of cermet inert anodes. TiB_2/C wettable cathodes and low temperature electrolytes were used in this experiment. Based on 300 cup-shaped cermet inert anodes, 8 TiB_2/C cathode blocks, and on low temperature electrolyte, the test was carried out during 100 days. $NiFe_2O_4$ was taken as the ceramic phase and Cu-Ni alloy was used as an optimized metal phase in the inert anode. The relative density of the cermet was 96-97%, the electrical resistivity 60 μΩm, corrosion resistance ≤ 2 cm/y.

The test was terminated due to several factors: Irregular anode current distribution led to some anode current overload, and it dropped off at the beginning; The anode current rod was so fragile that the damage was focused at the junction between the electric conductor and the anode; The impurity content in the aluminium exceeded the standard due to anodic corrosion dissolution, and to the dissolution of the broken anode in the molten aluminium.

Future development should focus on further improvement of: corrosion resistance; thermal shock resistance and conductive consistency of inert anode; the connection of the electric conductor rod with the anode to solve fall off anode problem; cathode and anode materials to maintain the compatible electric current density, anode shape and structure of electrolysis cell; augmentation of Al_2O_3 solubility in low temperature electrolyte using pilot scale tests.

Metal inert anodes

As can be seen from [17,20] UC Rusal is actively developing inert anodes based on a Ni-Fe alloy. In 2011[29, 30] the Engineering & Technology Centre in Krasnoyarsk developed engineering solutions like pot design, control system etc. During the development of the cell they proposed and tested a new concept of the cathode assembly. This pilot cell with amperage of 1-5 kA is a prototype for a future cell with amperage of 100 kA. Tests are planned in 2015. The Rusal website [31] shows that from 2017 onwards Rusal may start shifting smelting capacity to inert anode technology, starting at Krasnoyarsk Smelter (KrAZ). The new generation pots produce 1 tonne of oxygen for every 900 kg of aluminium produced. Scaled up to KrAZ this figure will reach 900,000 tpy of oxygen. The burning speed of an inert anode is 300 to 400 times slower than that of a traditional carbon anode, and it wears down only 1-2 cm/y, compared to 1-2 cm /d with the carbon anode. A plan of the new pot room layout can be seen in [32].

The project joined the Skolkovo foundation in June 2011. Planned co-financing of the inert anode aluminium production research from the foundation amounts to US$25 million till 2015. To date US$ 4.3 million has already been spent by Skolkovo.

Conclusion

During recent years research focussed on the development of $NiFe_2O_4$-based cermets and NiFe-based alloys as inert anode materials for aluminium production. While Chinese scientists chose the cermet way, western laboratories and Rusal chose the metal alloy solution. After developing a new cell design Rusal is convinced it can improve NiFe-alloy manufacture up to 2015, and then equip the first industrial electrolysis cell of 100 kA with alloyed anodes, which should be in operation by 2017. The future will show whether aluminium electrolysis can function industrially when replacing carbon anodes with inert anodes.

Acknowledgement

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