

# UNITED STATES PATENT OFFICE.

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## PROCESS OF REDUCING ALUMINIUM.

SPECIFICATION forming part of Letters Patent No. 631,253, dated August 15, 1899.

Application filed April 29, 1899. Serial No. 714,946. (No specimens.)

*To all whom it may concern:*

Be it known that I, FRANK A. GOOCH, residing at New Haven, in the county of New Haven, State of Connecticut, have invented certain new and useful Improvements in Processes of Reducing Aluminium; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

The invention described herein relates to processes for the reduction of aluminium from its compounds by the electrolysis of a suitable fused mass containing such compounds.

In general terms the invention comprises an improved process for reducing aluminium from its compounds, which consists in forming a bath by fusing together a suitable compound of aluminium and a suitable halogen compound of a metal more electropositive with reference to sulfur than aluminium, adding to the bath a suitable compound of sulfur, together with alumina, and then passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same.

More particularly my invention relates to processes for reducing aluminium from its sulfid by electrolysis.

I am aware that processes have been described for the electrolysis of aluminium sulfid where the sulfid is formed without the bath and then added to the bath or from aluminium oxid suspended in the bath.

My invention is intended to facilitate the formation of a suitable sulfid-bath by forming the aluminium sulfid from the oxid dissolved or combined in the bath. The halogen compounds of the metals more electropositive with reference to sulfur than is aluminium which are to be employed should be such as when fused are capable of dissolving the aluminium sulfid after it is formed in the bath. The compound of aluminium employed should be one which with the other constituents of the bath will unite to form a bath capable of dissolving alumina. In practice I find that the best results are obtainable from baths composed of fluorids of the alkaline metals with the addition of a halogen compound of aluminium. The compound of sul-

fur should be one which is capable of acting upon alumina in the bath, so as to replace by sulfur the oxygen element in the alumina.

For convenience I describe my invention with special reference to the use of carbon disulfid, ( $CS_2$ ), which may be manufactured in any usual manner, as by passing the vapor of sulfur over red-hot charcoal, it being understood, however, that my invention is also applicable to other compounds of sulfur—as, for example, hydrogen sulfid, ( $H_2S$ ). I also select for convenience a bath composed of sodium fluorid and chlorid of aluminium fused, it being understood that I do not confine myself to the bath so constituted, since any bath composed of a suitable halogen compound of the metals hereinbefore mentioned will answer the purpose. I preferably employ the ingredients named in the proportion of three parts of sodium fluorid to two parts of aluminium chlorid. A moderate excess of sodium chlorid does no harm.

In the accompanying drawings, which are hereby made a part of this specification, is shown a form of apparatus suitable to the practice of my invention. I do not, however, limit myself to the apparatus shown, since any other suitable apparatus will answer equally well for the practice of my invention.

Figure 1 represents a sectional diagrammatic view of the apparatus preferably employed by me. Fig. 2 represents a plan view of a portion of the apparatus from above.

Similar letters refer to similar parts throughout the drawings.

In the practice of my invention I prepare a bath by fusing in a suitable tank or crucible sodium fluorid and chlorid of aluminium. The crucible preferably employed by me in the operation of my invention consists of a tank of iron O, provided with sleeves S S' for the insertion of the anodes C' C<sup>2</sup>, and the tank is lined on the bottom and half-way up on the sides with compacted carbon L. The remainder of the tank and the sleeves are lined with compacted alumina I, which serves to keep the anodes from electrical contact with the tank and casing. The anodes C' C<sup>2</sup> are detached carbon electrodes partially immersed in the bath B, connected by copper rods r r' with the metal bar K in any suitable way, as by screw-clamps w w', and perforated

longitudinally. The metal bar K is preferably supported by wooden supports *ff'*. The carbon lining L serves as the cathode. The conductors P N are connected with the positive and negative poles, respectively, of the source of electricity E. Conductor P is also connected with the anodes C' C<sup>2</sup> through bar K and rods *r r'* by a binding-post *m*, and the conductor N is connected in a similar manner with the cathode L by the binding-post *m'*. F is the furnace, having a fire-box *f*. The crucible T is supported over the fire-box by the sides of the furnace. The carbon disulfid or other compound of sulfur is introduced into the bath through the pipes G G', which form a tight joint with the anodes C' C<sup>2</sup>, so that the pipes are continuous with the perforations in the anodes. The gaseous products which escape from the bath pass off through the bell J, which may be made of iron boiler-plate and lined with compacted carbon or other suitable material, the gases passing through the pipe R at the outlet *j*. The bell J is supported by a wooden support T', which is adjustably attached to the metal bar K by screw-clamp *y* or other suitable means. When the bath is in operation, the bell J is lowered below the surface of the bath and the space between the rim of the bell J and the interior surface of the tank T is covered with powdered carbon *p*, sprinkled thickly upon the surface of the bath. Through this space when desired alumina is introduced into the bath B in quantities as required. A moderate excess of alumina does no harm. In the bath described the alumina will be dissolved.

When the carbon disulfid is introduced, it acts upon the dissolved alumina to convert that oxid to aluminium sulfid, while carbon oxysulfid or carbon monoxid is formed at the same time. Then an electric current of suitably low voltage (which will vary according to the constitution of the bath and the size of the electrodes) is passed through the bath, thereby electrolyzing the aluminium sulfid. Aluminium will be deposited at the cathode and may be removed by ladle or siphon or other convenient means, while sulfur is liberated at the anode and passes off through the pipe J and outlet *j*. By supplying alumina to the bath and maintaining the current of carbon disulfid the production of aluminium sulfid will go on continuously and if properly regulated will keep pace with the electrolytic action of the electric current.

One great advantage in the process thus described is the low voltage of the current required to reduce the aluminium, being about nine-tenths of a volt. I therefore employ a current of just sufficient voltage to overcome the resistance of the bath and other resistances under the existing conditions of the process, together with the added voltage of nine-tenths required to electrolyze aluminium sulfid. Such a current would of course have no effect in electrolyzing alumina dissolved in the bath. I have

found in practice that similar results are obtained when instead of introducing carbon disulfid directly into the bath sulfur is introduced into a bath covered with charcoal. In such a case aluminium sulfid is formed and aluminium is precipitated when the current is passed. Preferably, however, the crude carbon disulfid, either as it goes from the generator or after intermediate condensation, is passed through the crucible T and the carbon disulfid and sulfur which escape from the crucible through the pipe J are condensed and again sent through the generator and into the crucible. From the carbon oxysulfid which escapes from the condenser the sulfur may be recovered in any known manner or the carbon oxysulfid may be burned to form sulfur dioxide and carbon dioxide, the former of which being a by-product of value may be absorbed by suitable reagents, while the carbon dioxide will ordinarily be permitted to escape.

I have described a form of my process in which the bath is composed of a halogen compound of an alkaline metal and of a halogen compound of aluminium, which bath is capable when fused of dissolving alumina. I find also that excellent results are obtained from the use of baths composed of the fluoride of aluminium and fluorids of alkaline metals, particularly the fluorid of sodium. As is well known, when alumina is introduced into the last-named bath it dissolves. When the compound of sulfur is introduced into such a bath, the sulfid of aluminium is rapidly formed in solution, and on passing a current of the voltage above specified electrolysis takes place and aluminium is produced at the cathode.

Having described my invention, what I claim, and desire to procure by Letters Patent, is—

1. As an improvement in the art of manufacturing aluminium, the herein-described process which consists in forming a bath by fusing together a suitable compound of aluminium and a suitable halogen compound of a metal more electropositive with reference to sulfur than is aluminium, adding to the bath a suitable compound of sulfur together with alumina, and then passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same, substantially as and for the purposes set forth.

2. As an improvement in the art of manufacturing aluminium, the herein-described process which consists in forming a bath by fusing together a halogen compound of aluminium and a suitable halogen compound of a metal more electropositive than aluminium with reference to sulfur, adding to the bath a suitable compound of sulfur, together with alumina, and then passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same, substantially as and for the purposes set forth.

3. As an improvement in the art of manufacturing aluminium, the herein-described

process which consists in forming a bath by fusing together a halogen compound of aluminium and suitable halogen compounds of metals which are more electropositive than aluminium with reference to sulfur, adding to the bath in suitable quantity carbon disulfid, together with alumina, and then passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same, substantially as and for the purposes set forth.

4. As an improvement in the art of manufacturing aluminium, the herein-described process, which consists in forming a bath by fusing together the fluorids of aluminium and of metals more electropositive than aluminium with reference to sulfur, adding to the bath in suitable quantity carbon disulfid, together with alumina, and then passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same, substantially as and for the purposes set forth.

5. As an improvement in the art of manu-

facturing aluminium, the herein-described process, which consists in forming a bath by fusing together fluorids of aluminium, and of an alkaline metal, adding to the bath in suitable quantity carbon disulfid, together with alumina, and then passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same, substantially as and for the purposes set forth.

6. As an improvement in the art of manufacturing aluminium, the herein-described process which consists in forming a bath by fusing together the fluorid of aluminium and the fluorid of sodium, adding to the bath in suitable quantity carbon disulfid and alumina, and then passing an electric current through the fused mass, thereby electrolyzing the same, substantially as and for the purposes set forth.

FRANK A. GOOCH.

Witnesses:

SARAH E. GOOCH,

PHILIP E. BROWNING.