No. 786,244. PATENTED MAR. 28, 1905.

H. S. BLACKMORE.

PROCESS OF EXTRACTING ALUMINIUM OR OTHER METALS.

APPLICATION FILED JAN. 7, 1905.

Fig. 1.

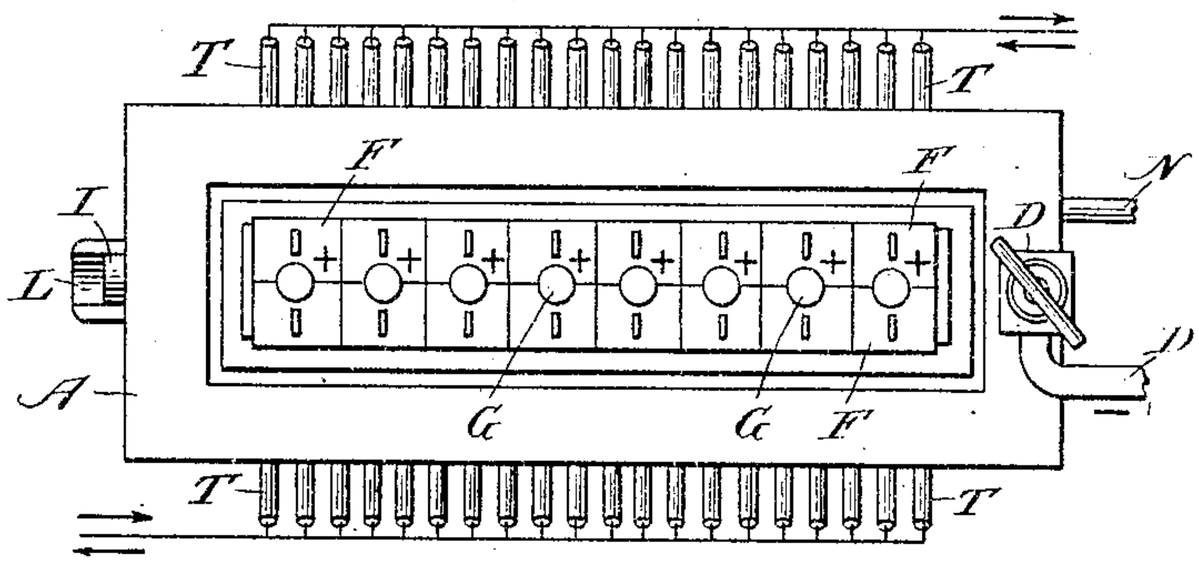


Fig. 2.

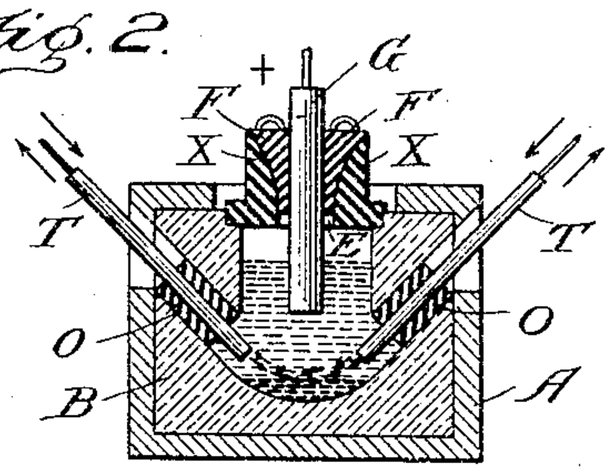
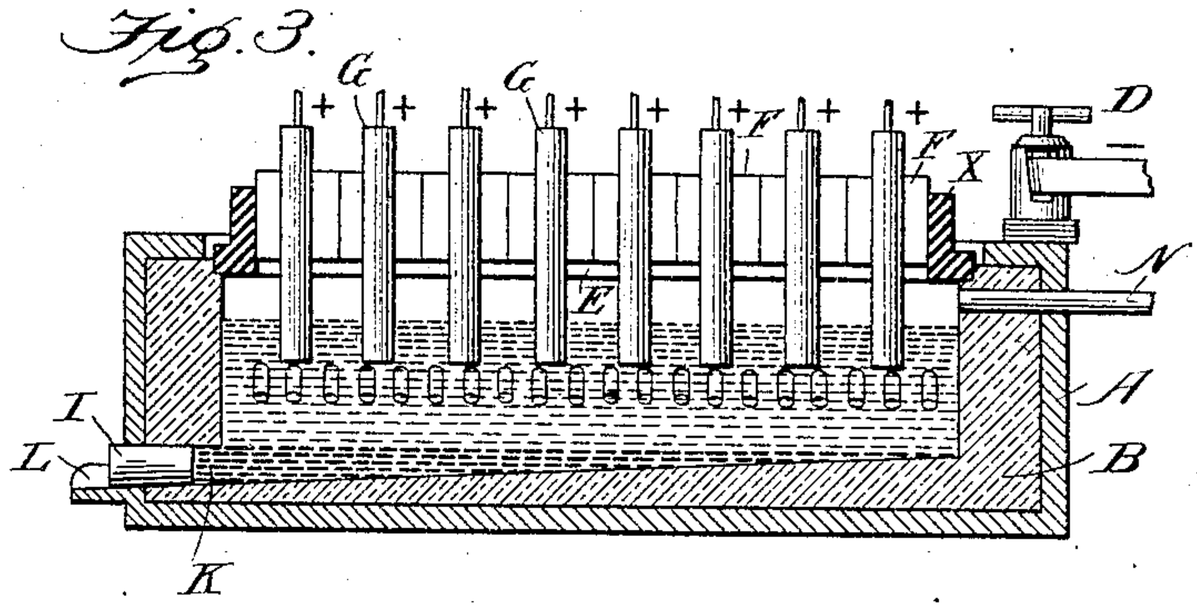


Fig. 3.



Witnesses

Edwin L. Bradford

H. N. Jenkins

Inventor

Henry Spencer Blackmore,

No. 786,244. Patented March 28, 1905.

UNITED STATES PATENT OFFICE.

HENRY SPENCER BLACKMORE, OF MOUNT VERNON, NEW YORK.

PROCESS OF EXTRACTING ALUMINIUM OR OTHER METALS.

SPECIFICATION forming part of Letters Patent No. 786,244, dated March 28, 1905.

Original application filed September 12, 1903, Serial No. 172,960. Divided and this application filed January 7, 1905. Serial No. 240,022.

To all whom it may concern:

Be it known that I, HENRY SPENCER BLACKMORE, a citizen of the United States, residing at Mount Vernon, in the county of Westchester and State of New York, have invented certain new and useful Improvements in Processes of Extracting Aluminium or other Metals, of which the following is a specification, being a division from Serial No. 172,960, filed September 12, 1903.

The object of my invention is to reduce metals from a compound of the metal desired with fluorin and one or more non-metallic elements other than a halogen by the action of an electric current of sufficient intensity to selectively liberate the lesser electronegative non-metallic element or elements of the compound, simultaneously producing the metal desired either by the direct action of an electric current or with the augmenting reducing action of a substance capable of uniting with one or more of the electronegative non-metallic elements liberated in the presence thereof, by which means the metal is liberated in a pure condition—*i. e.*, not contaminated with foreign metals, whereby the composition from which the metal is reduced is of less specific gravity than the reduced metal and whereby the process may be carried on continuously by the addition of a compound of the same metal with the lesser electronegative non-metallic element or elements selectively liberated from time to time as the first increments are dissociated by the action of the electric current with or without the presence of an augmenting reducing agent capable of uniting with the less electronegative non-metallic elements liberated during action.

My process relates specifically to the reduction of aluminium from a mixture or compound consisting of aluminium combined with fluorin and oxygen by the action of an electric current employing carbon anodes, but is not limited thereto, as it may be applied to the reduction of many other metals without departing from the spirit of my invention.

In prior art it has been proposed to reduce aluminium from its oxid by liquefying or dissolving it in a molten bath consisting of fluorid of aluminium, together with the fluorid of a metal or metals more electropositive than aluminium—such as the fluorid of lithium, potassium, sodium, or calcium, or mixtures or compositions thereof—the object being to produce a solvent bath consisting of aluminium fluorid with a fluorid of a metal more electropositive than aluminium and reducing the aluminium oxid dissolved in said composition by the action of an electric current employing carbon anodes.

The further object of employing the mixture of aluminium fluorid with fluorids of metals more electropositive than aluminium being to provide a bath of sufficiently low specific gravity or lightness that would admit of the aluminium reduced readily settling therein and accumulating below the lighter bath or composition.

The great difficulty encountered in the application of this process has been that great care is required to regulate the electricity employed at such a tension that the aluminium will be reduced without contamination with foreign ingredients—such as lithium, potassium, sodium, calcium, &c., which metals are quite readily thrown down or precipitated from the constituents of the bath into the reduced aluminium, thereby contaminating the same, even to an injurious degree, and it has been found in many cases to be almost impossible to free the contaminated aluminium thus reduced from its associated impurities.

It has also been proposed to fuse aluminium oxid *per se* by the action of an electric current and to maintain the current of sufficient volume and intensity above that required for fusion of the oxid to produce an electrolytic dissociation, employing carbon anodes, whereby the oxygen of the oxid unites with the carbon, liberating aluminium. It is found, however, in this process that the temperature at which the aluminium oxid *per se* fuses and becomes electrolytically dissociated is so high and the aluminium liberated is so light that it either becomes volatilized by the heat or dissipated by the escaping gaseous products of reduction,

no metallic aluminium *per se* being obtained in practical commercial quantities by this process for the reasons stated.

In order to obviate the loss of metal by volatilization or dissipation in this process, metallic copper or other metal has been employed as cathode which absorbs the liberated aluminium, producing alloys thereof; but it has been found impractical to use this process for the production of unalloyed aluminium.

It is well known that many metals and compounds thereof will assume a fused or molten condition when mixed or associated with each other at a lower temperature than is possible to fuse, melt, or liquefy either of the constituents individually by the action of heat, and practically applying this fact and condition I have ascertained one can mix together compounds of a metal containing fluorin, such as aluminium fluorid, and one or more non-metallic elements other than a halogen, such as aluminium oxid, and expose this mixture or composition consisting of aluminium combined with oxygen and fluorin to the action of an electric current whereby the fusion of the composition and the electric dissociation of the oxid content is accomplished at a temperature and under conditions whereby the aluminium reduced is not volatilized or dissipated, but will accumulate and settle beneath the associated ingredients for the reason that the compounds of aluminium and oxygen and aluminium and fluorin in the fused condition are of less specific gravity than the aluminium separated and which composition being free from other metals than aluminium avoids the production of aluminium contaminated with other metals, yielding a product of practically pure and uncontaminated form, the electrolytical dissociation being maintained at substantially the same temperature and with the same expenditure of electric current by adding to the composition more aluminium oxid from time to time to compensate for the amount electrically dissociated, thus maintaining the composition in a practically uniform condition, the dissociation being preferably carried on with the employment of an anode capable of uniting with oxygen, such as carbon.

In carrying out my invention for the production of aluminium, I employ a substance consisting of aluminium combined with oxygen and fluorin, such as a substance produced by the mixture of aluminium oxid with aluminium fluorid in proportion of about one of the former to two of the latter by weight. I then heat this substance until it assumes a fused or molten condition and pass a current of electricity therethrough of sufficient volume and intensity to electrically dissociate or reduce the oxygen content of the substance from the metal aluminium, while employing a carbon anode, which combines with the oxygen thus liberated, evolving carbonic oxid. As the process of electrolytic dissociation progresses the aluminium liberated settles and accumulates below the aluminium compounds present and may be withdrawn from time to time as desired, while the aluminium-containing compound or substance subjected to the action of the electric current is supplied with aluminium oxid from time to time as the aluminium and oxygen content becomes dissociated or reduced, thus avoiding the depletion of the substance of the aluminium and oxygen content thereof. In this manner the process can be carried on continuously, aluminium oxid being supplied from time to time and the metal withdrawn as desired, while at the same time the expenditure of electrical energy is maintained at practically a uniform ratio and the reduction maintained at a temperature of about 1,800° Fahrenheit, which avoids the loss of the aluminium reduced by volatilization or dissipation and produces a yield of metal practically free from injurious metallic contamination.

It should be noted that my process is carried on with the employment of a substance containing practically only the metal desired in combination with flourin and one or more non-metallic elements other than a halogen, the association or combination of which non-metallic elements, either as a whole or mixture of separate compounds of the same metal, is so united or blended as to facilitate reduction in the presence of reducing agents or electrical action at lower temperatures and with better results than can be obtained with the employment of binary compounds of the metal with the non-metallic elements individually.

I do not desire to confine myself to the reduction of aluminium, but reserve the right to employ my process in connection with the reduction of any or all metals to which it may be applicable without departing from the spirit of my invention, which consists in exposing a compound or composition of the metal desired with fluorin and one or more non-metallic elements other than a halogen so associated or blended that the reduction or fusion temperature is reduced below the point at which either may be accomplished individually and whereby a continuous reduction may be carried on by the action of an electric current of sufficient volume and intensity to reduce or separate the metal from the less electronegative elements of the composition, which is replenished from time to time by the addition to the same of fresh increments of the substance reduced as required.

In extracting the aluminium or other metal in accordance with my process as aforestated I fuse the composition by the action of an alternating current and dissociate or reduce the metal by the simultaneous action of a direct

current. In this manner the constituents may be maintained in a molten condition without interrupting the action of the direct current or deteriorating the value of the same as an electrolytic agent, whereby the whole of the direct current may be expended in yielding metal instead of being utilized to a considerable extent to maintain fusion, as employed in processes hitherto.

The apparatus which I prefer to employ in carrying out my process is illustrated in the accompanying drawings, in which—

Figure 1 is a plan or top view. Fig. 2 is a transverse vertical section, and Fig. 3 is a longitudinal vertical section.

In referring to the drawings, the letter A designates a box or receptacle, preferably of cast-iron, lined with a conductive substance, such as carbon B, into which project the electrodes T T, which pass through the insulators O O.

The material is introduced into the apparatus through the openings E, which are closed by the insulating covers or plugs F passing into the insulating-cover X, through which pass the electrodes G.

In the operation of the process I place in the apparatus a quantity of the substance containing the metal desired combined with fluorin and one or more non-metallic elements other than a halogen, such as aluminium with oxygen and fluorin, and fuse the same by passing an alternating current therethrough between the electrodes T T, which heating means is independent of the electrodes G employed for electrolytic purposes. I then add from time to time quantities of the composition until the interior of the apparatus has been sufficiently filled with fused material. When the substance is fused, a direct or electrolytic current is passed through the mass between the electrodes G, which constitute the anode and which consists, preferably, of carbon, and the carbon lining B, which constitutes the cathode of the electrolytic circuit, the tension and intensity of the current being approximately three volts and eighty-five amperes per square inch of anode-surface exposed with the temperature maintained by independent means at approximately 1,800° Fahrenheit, at which time the oxygen of the substance containing aluminium, oxygen, and fluorin is liberated at the anode, combining with the carbon thereof, evolving carbonic-oxid gas, while metallic aluminium accumulates in the bottom of the apparatus and is withdrawn from time to time through the taphole K and trough L by removing the taphole plug I. The presence of carbon as anode augments the reduction by facilitating reduction and generating heat by union of the oxygen content therewith, thus economizing both the electrolyzing and heating currents of the electricity employed. If the electrode G consists of a material which is not acted on by the electronegative constituent evolved at the said electrode, this augmenting action is not available. As the oxygen and aluminium are separated by electrolytic action of the direct current of such selective tension as not to affect the fluorin content, the material may be replenished from time to time by adding further increments of aluminium and oxygen in the form of aluminium oxid.

The current for heating purposes is applied independently of the electrolytic electrodes G and is passed through the substances contained in the reduction-receptacle between the electrodes T T, fusing the substances and maintaining them in a state of fusion while the electrolytic or direct current is applied through the substances between the electrodes G and the carbon lining B, whereupon the electrolytic efficiency of the direct or electrolytic current may be utilized without loss. The gaseous by-products escape through the conduit N.

It can be seen that by this method of procedure the substances to be decomposed or electrolyzed are fused and maintained in a fused condition with the heat evolved by the resistance to the passage of the alternating or non-metal-yielding current through a fusible or fused conductor in such a manner as to heat the ingredients independent of the electrolyzing or direct current and of the electrodes through which the said current is applied.

I am aware that aluminium has been reduced from its oxid by mixing it with a mixture of aluminium halogen salts, such as fluorid and chlorid, the mixed halogen salts of which were intended to produce a highly-resistant bath within the aluminium oxid to be reduced by electrolysis. It is found, however, that when halogen compounds of a metal with halogens other than fluorin are employed associated or in combination with the fluorid in electrolytic processes the halogen salts other than fluorid—such as chlorid, iodid, or bromid—are so easily volatilized, dissociated, and dissipated by the action of heat as to greatly retard electrolytic action by occasioning loss of heat from the bath by volatilization, increasing the electrical resistance of the fused ingredients and retarding the solubility or liquefaction of aluminium oxid when it is desired to reduce aluminium therefrom. It has therefore been found detrimental and impracticable from an economical and commercial standpoint to employ compounds or compositions of haloids other than fluorin in the mass in which the metal is being reduced by electrolysis.

Having now described my invention, what I claim as new, and desire to secure by Letters Patent, is—

1. The process of reducing metal which consists in exposing a fused substance containing only one metal combined with one halogen, fluorin, and one or more non-metallic elements other than a halogen, to the action of an electric current of a voltage below that necessary to liberate fluorin therefrom, but capable of liberating one or more non-metallic elements.

2. The process of reducing metals which consists in exposing a fused substance consisting of one metal combined with one halogen, fluorin, and one or more non-metallic elements other than a halogen, to the action of an electric current of a voltage below that necessary to liberate fluorin but of sufficient intensity to selectively liberate the less electronegative non-metallic element or elements.

3. The process of reducing metal which consists in exposing a fused substance consisting of one metal combined with one halogen, fluorin, and one or more non-metallic elements other than a halogen, to the action of an electric current in the presence of a substance capable of uniting with a non-metallic constituent of the compound while subjecting the material to the action of an electric current of a voltage below that necessary to liberate fluorin, but capable of liberating the less electronegative non-metallic element or elements in the presence of the combining substance.

4. The process of reducing metal which consists in mixing two or more compounds composed of a single metal combined with one halogen, fluorin, and different non-metallic elements other than halogens, and exposing the substance consisting of the metal combined with fluorin and one or more non-metallic elements other than a halogen in a fused state, to the action of an electric current of a voltage below that necessary to liberate fluorin, but of sufficient intensity to liberate one of the non-metallic elements.

5. The process of reducing metal which consists in mixing two or more binary compounds composed of a single metal combined with one halogen, fluorin, and different non-metallic elements other than halogens, and exposing the substance consisting of the metal combined with fluorin and one or more non-metallic elements other than halogens, in a fused state, to the action of an electric current of a voltage below that necessary to liberate fluorin, but of sufficient intensity to liberate one of the non-metallic elements.

6. The process of reducing metal which consists in exposing a fused substance consisting of one metal united with one halogen, fluorin, and one or more non-metallic elements other than halogens, to the action of an electric current of a voltage below that necessary to liberate fluorin, but of sufficient intensity to liberate a portion of the metal and one non-metallic element, and supplying to the undecomposed composition, portions of a compound of the same metal with a non-metallic element similar to that previously liberated from time to time while maintaining the electric current and withdrawing the reduced metal at intervals, as desired.

7. The process of reducing aluminium which consists in exposing a fused substance consisting of one metal, aluminium, united with one halogen, fluorin, and one or more non-metallic elements other than a halogen, to the action of an electric current of a voltage below that necessary to liberate fluorin, but of sufficient intensity to liberate the less electronegative non-metallic element or elements.

8. The process of reducing aluminium which consists in exposing a substance consisting of one metal, aluminium, combined with oxygen and one halogen, fluorin, to the action of an electric current of a voltage below that necessary to liberate fluorin, but of such tension as to induce the selective liberation of oxygen.

9. The process of reducing aluminium which consists in exposing a fused substance consisting of one metal, aluminium, combined with oxygen and one halogen, fluorin, to the action of an electric current of a voltage below that necessary to liberate fluorin, but capable of liberating oxygen in the presence of carbon.

10. The process of producing aluminium and carbon oxid which consists in exposing a fused substance consisting of one metal, aluminium, combined with one halogen, fluorin, and one or more non-metallic elements other than a halogen, one of which is oxygen, to the action of an electric current of a voltage below that necessary to liberate fluorin, but of sufficient intensity, while employing a carbon anode, to liberate oxygen in the presence of the carbon whereby the oxygen unites with the carbon of the anode, evolving carbon oxid and liberating aluminium.

11. The process of reducing aluminium which consists in exposing a fused mixture of aluminium fluorid and aluminium oxid to the action of an electric current of a voltage below that necessary to liberate fluorin, but of sufficient intensity to liberate oxygen from the oxid in the presence of a combining agent with the simultaneous production of aluminium and supplying aluminium oxid to the composition from time to time while maintaining the electric current and withdrawing the aluminium reduced at intervals, as desired.

12. The process of reducing aluminium which consists in exposing a fused mixture of aluminium fluorid and aluminium oxid to the action of an electric current of a voltage below that necessary to liberate fluorin, but of sufficient intensity to liberate the oxygen from the oxid in the presence of carbon with the simultaneous production of aluminium and supplying aluminium oxid to the mixture from

time to time while maintaining the electric current and withdrawing the aluminium reduced at intervals, as desired.

13. The process of reducing aluminium which consists in exposing fused compounds of one halogen, fluorin, and different non-metallic elements other than halogens with aluminium only, to the action of an electric current of a voltage below that necessary to liberate fluorin, but of sufficient intensity to liberate one of the electronegative non-metallic elements from the composition with the simultaneous separation of aluminium.

In testimony whereof I affix my signature in presence of two witnesses.

HENRY SPENCER BLACKMORE.

Witnesses:
C. C. WRIGHT,
C. M. FORREST.