No. 715,625. Patented Dec. 9, 1902.

G. TADDEI.

PROCESS OF OBTAINING METALLIC ALUMINIUM OR OTHER METALS BY ELECTROLYSIS.

(Application filed July 17, 1901.)

(No Model.) 3 Sheets—Sheet 1.

Fig. 2

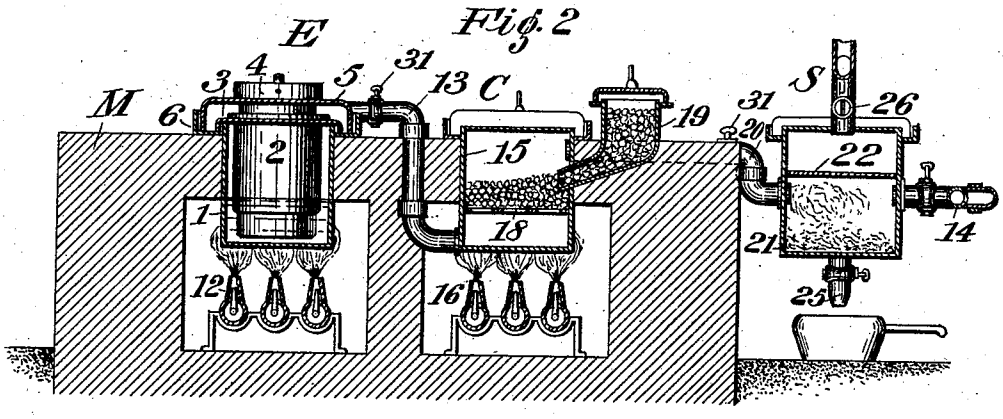
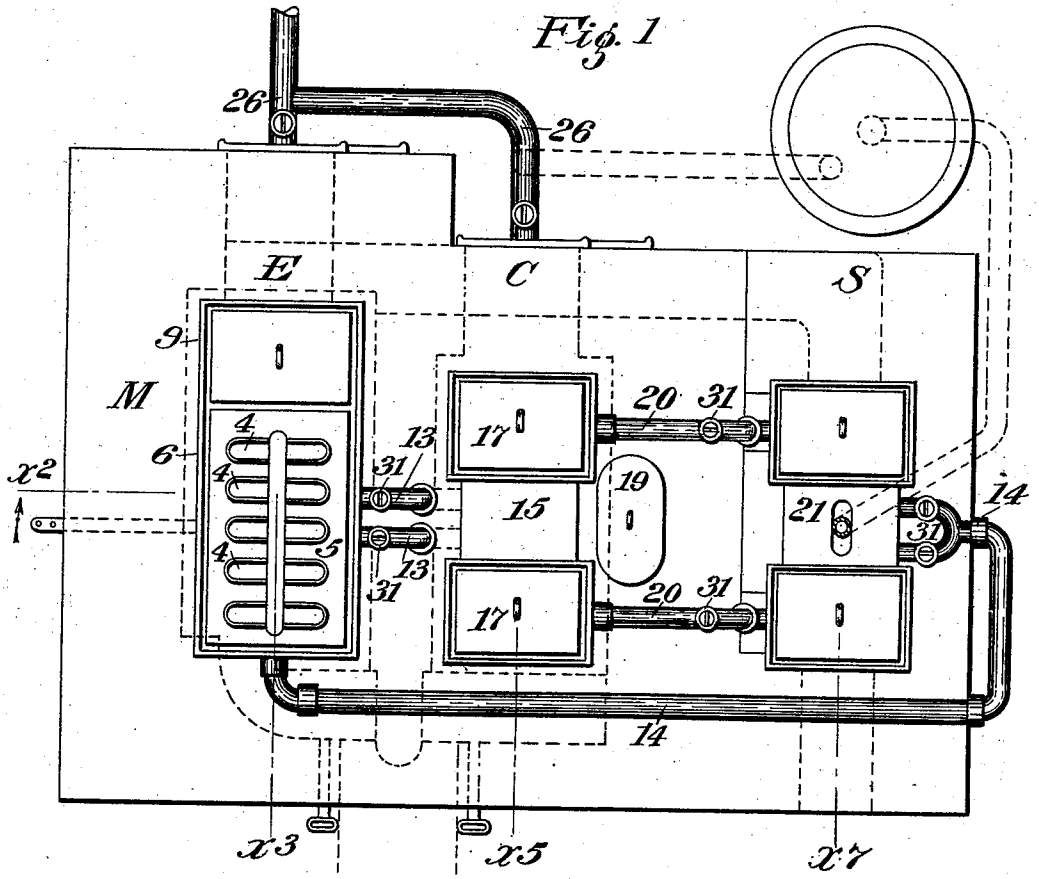


Fig. 1



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Fig. 3

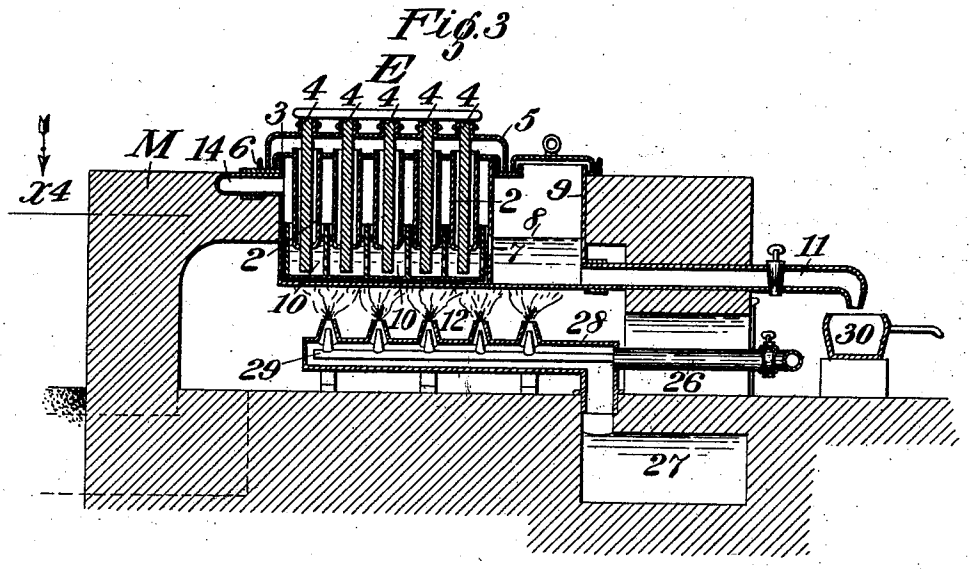
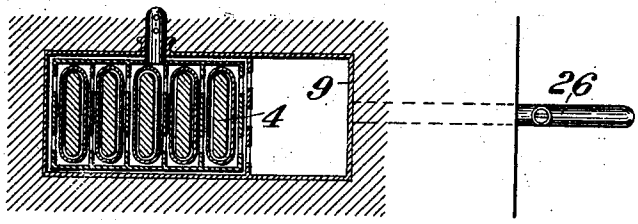


Fig. 4



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Fig. 6

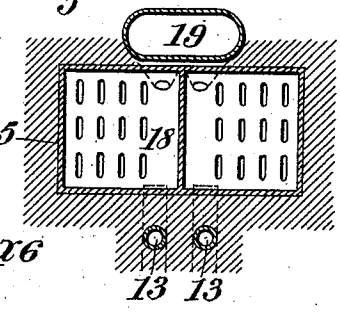


Fig. 5

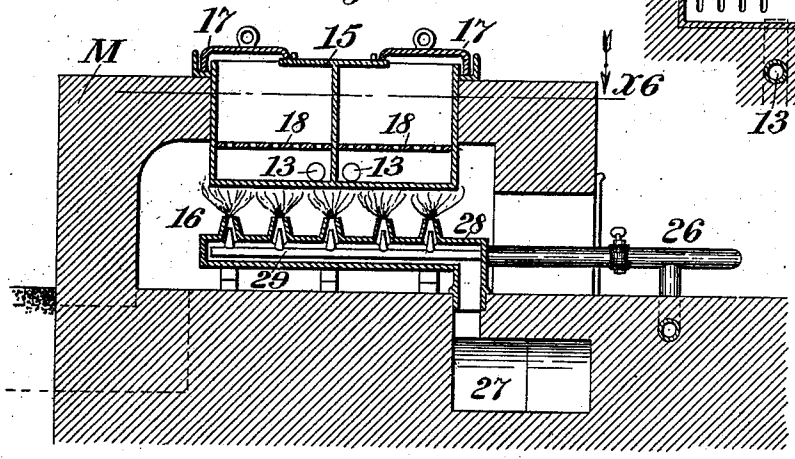


Fig. 7

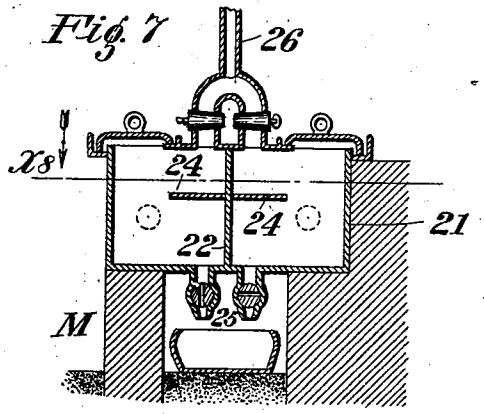
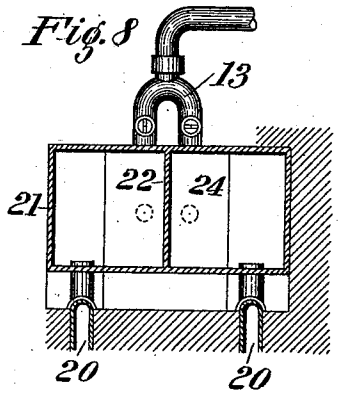


Fig. 8



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UNITED STATES PATENT OFFICE.

GIROLAMO TADDEI, OF TURIN, ITALY, ASSIGNOR TO THE SOCIETA ITALIANA DI APPLICAZIONI ELETTRICHE, OF TURIN, ITALY.

PROCESS OF OBTAINING METALLIC ALUMINIUM OR OTHER METALS BY ELECTROLYSIS.

SPECIFICATION forming part of Letters Patent No. 715,625, dated December 9, 1902.

Application filed July 17, 1901. Serial No. 68,575. (No specimens.)

To all whom it may concern:

Be it known that I, GIROLAMO TADDEI, a subject of the King of Italy, residing in Turin, Italy, have invented a certain new and useful Method of Obtaining Metals from Their Compounds, of which the following is a specification.

This invention relates to a process for obtaining metals from their compounds, and particularly the metal aluminium from its oxid; and the object of the invention is to effect this recovery of the metal with the minimum of energy thermoelectrically considered in relation to industrial conditions and to effect it in an automatic or substantially automatic and continuous sequence of steps or operations.

The process will be readily understood from a description of the operations required for reducing alumina (Al_2O_3) to recover the metal aluminium therefrom. The alumina suitably dehydrated is mixed with tar to form balls or masses in which the carbon and aluminium are in substantially equivalent proportions—viz., six of carbon to 9.1 of aluminium.

The operations or steps of the process are as follows:

1. Fused sodium chlorid (NaCl) is decomposed by electrolysis and its elements in the form of vapor separated. The heat employed will be about 1,000° centigrade, and the current employed will be continuous and have a tension of about 4.5 volts.

2. The chlorin from the step or operation 1 is led to a closed vessel containing the mass of alumina and tar above described. This vessel and its contents are heated up to from 1,300° to 1,500° centigrade, and the chlorin is gradually brought into contact with all parts of the mass therein, thus producing a reaction, from which results aluminium chlorid and carbonic oxid, as expressed in the following:

$$Al_2O_3+3C+3Cl=2AlCl_3+3CO.$$

3. The aluminium chlorid from the reaction of the step 2 will be in the form of vapor, and it passes off, together with the carbonic oxid, to another vessel, where it mingles with the hot sodium vapor from the step 1. This intermingling of the hot vapors produces another reaction, the sodium combining with the chlorin of the aluminium chlorid and setting free the carbonic oxid and the metal aluminium. The reaction is expressed in the following:

$$2AlCl_3+6Na+3CO=6NaCl+2Al+3CO.$$

Thus the metal aluminium is separated, the sodium chlorid is recovered without loss for use again, and the carbonic oxid passes off and may be burned. The operations are continuous, the mixture of alumina and tar producing continuously aluminium and carbonic oxid. The sodium and chlorin separately and in combination move in a closed circuit without waste or being consumed.

In order to make the practical and industrial carrying out of my process the better understood, reference may be had to the accompanying drawings, which illustrate an apparatus that may be employed in the process.

In the drawings, Figure 1 is a general plan of the apparatus, and Fig. 2 is a longitudinal vertical section of the same in the plane indicated by line x^2 in Fig. 1. Fig. 3 is a vertical transverse section at line x^3 in Fig. 1. Fig. 4 is a horizontal section at line x^4 in Fig. 3. Fig. 5 is a vertical transverse section at line x^5 in Fig. 1. Fig. 6 is a horizontal section at line x^6 in Fig. 5. Fig. 7 is a vertical transverse section at line x^7 in Fig. 1, and Fig. 8 is a horizontal section at line x^8 in Fig. 7.

The construction of the apparatus will be readily understood from the drawings and the following description:

For convenience of description the apparatus may be considered as divided into three sections—namely, the electrolyzing-section E, the chlorinating-section C, and the substituting-section S. The several sections of the apparatus will be described in the order given above.

The operative features of the apparatus are set in brickwork or masonry M. The first or electrolyzing section E consists of a vessel 1, into which depend separator-tubes 2, fixed at their upper ends in an inner cover 3. The carbon anodes 4 depend in these tubes 2 from an upper or outer cover 5, elevated above the cover 3. The downwardly-flanged margins of the covers rest in a trough 6 about the vessel

1. At the side of the vessel 1 and connecting therewith by apertures 7 below the level of the electrolyte 8 is a vessel 9 for introducing the electrolyte or adding thereto during the process.

10 represents the iron cathodes, situated between the carbon anodes.

11 is a pipe for drawing off the metallic aluminium, as will be hereinafter explained.

12 is a heater in a chamber beneath the vessel 1 for burning carbonic oxid (CO) mixed with heated air, as will be explained.

From the space between the two covers 3 and 5 two pipes 13 lead to the second or chlorinating section of the apparatus, and a pipe 14 leads from the interior of the vessel 1 to the section S.

The second section of the apparatus (seen in detail in Figs. 5 and 6) consists of a vessel 15, set in the brickwork over the heater 16. This vessel is divided transversely by a partition into two chambers, each provided with a cover 17. Each chamber has a perforated bottom 18, situated some distance above the imperforate bottom, and the pipes 13 enter the space between these bottoms. A hopper 19 supplies material to the two chambers in the vessel 15, and pipes 20 lead from the upper parts of the respective chambers in the vessel 15 to the section S.

The third section S of the apparatus (seen in detail in Figs. 7 and 8) comprises a vessel 21, set in the brickwork and divided transversely by a partition 22 into two chambers provided with covers 23. There is also in each chamber a horizontal partition 24, which extends from the partition 22 part way across the chamber. The pipes 20 from the vessel 15 and the pipe 14 from the vessel 1 (the latter being forked or branched) enter the vessel 21 below the partitions 24. In the bottoms of the chambers of the vessel 21 are evacuating or draining outlets 25, and a pipe 26, having forked connections with the chambers in vessel 21, carries off the gases (CO) from the same to the burners or heaters 12 and 16.

Figs. 3 and 5 show how hot or warm air mixed with carbonic oxid from the vessel 21 is supplied to the burners of sections C and S. The air from flues 27 in the masonry enters a tube-like jacket 28, through which extends a jet-tube 29, connected with the gas-supply pipe 26.

It is proposed with this apparatus to separate metals, and especially the metals of the alkaline earths, from the substances combined chemically with them; and the apparatus may be employed in carrying out other processes—as, for example, the preparation of soda, chlorids, and other similar combinations.

The operation of the apparatus will perhaps be best understood by describing how it is used in the separation of the metal aluminium from alumina, (Al_2O_3,) dehydrated and mixed according to their equivalents with pitch or resin, so as to form balls of from fifteen to eighteen centimeters in diameter in the proportion Al_2O_3+3C. In the vessel 1 of the section E is poured an electrolyte consisting of sodium chlorid, (NaCl,) which is kept in a liquid state by the heater below at a temperature of from 900° to 1,000° centigrade. To the electrolyte will be added in small proportions the chlorids of potassium, lithium, strontium, or calcium in such a manner as to increase the fluidity and render negligible the portion of the electrolyte evaporated. These chlorids remain always in the bath, their action here being simply catalytic. The electrolysis is produced as follows: Chlorin is developed at the anodes 4, passes up to the space between the covers 3 and 5, and from there passes off by the pipes 13 to the bottom of the vessel 15 of section C. The auxiliary vessel 9, as stated before, is the receiver for the electrolyte, and the apertures 7 will always be below the level of the liquid. The flattened separating-tubes 2 are made to dip into the electrolyte, so as to seal their lower ends, say, ten millimeters; but they do not extend to the bottom of the vessel 1. The disposition of these separators 2 is such that as soon as the chlorin and sodium are liberated from the salt and pass to the respective anodes and cathodes they become separated permanently by the tubes 2. The sodium passes off by the pipe 14 to the vessel 21 of the section S. The vessel 15 of the section C is charged with the balls of alumina and tar, which rest on the perforated bottom 18. The chlorin gas entering by the pipes 13 passes up through the mass on the perforated platform or bottom to the space above the mass. By reason of its affinity for aluminium the chlorin decomposes the alumina, forming aluminium chlorid, while the oxygen set free from the alumina combines with the carbon of the tar or rosin to form carbonic oxid, (CO,) the reaction being

$$Al_2O_3+3C+3Cl=2AlCl_3+3CO.$$

The aluminium chlorid mixed with carbonic oxid flows by the pipes 20 to the vessel 21 of the section S. These pipes, as well as those before mentioned, should be well protected against radiation of heat in order to maintain the substances passing through them in a state of vapor. The vapor of aluminium chlorid mixed with the carbonic-oxid gas on entering the vessel 21 mixes thoroughly with the hot sodium vapor from the vessel 1 and a double reaction takes place. The chlorid is decomposed and the carbonic oxid separated, as expressed in the following formula:

$$2AlCl_3+6Na+3CO=6NaCl+2Al+3CO.$$

The sodium chlorid thus obtained and the aluminium will be found mixed.

To separate the aluminium from the sodium chlorid with the aid of this apparatus, so as to produce the metal in the form of bars or bricks, the mixture of the metal and the chlorid is put in the auxiliary vessel 9 of the section E. The sodium chlorid is at once taken

up by the electrolyte and the heavier aluminium in a molten state falls to the bottom and may be drawn off at the pipe 11, as before explained. Fig. 3 shows a ladle 30 to receive the molten metal and convey it to the molds.

The purpose of dividing the vessels 15 and 21 of the respective sections C and S into two parts or chambers, as shown, is to enable one chamber to be emptied and cleaned while the other is in operation, thus avoiding interruption of the process. Suitable stop-cocks 31 are provided in the several pipes to enable this to be effected.

Only one section E or electrolytic section is herein shown; but it will be obvious to any one skilled in the art that several of these electrolyzers may be employed in connection with a single large chlorinating apparatus C and a single large substitution apparatus S.

It may be stated as to the electrolyzing apparatus that spaces occupied by the electrolyte between the anodes and cathodes may be about one and one-half centimeters, and the superficial area of the anodes may equal that of the cathodes. There may be for each dynamic horse-power one hundred square centimeters of area of the electrodes. In these measurements the entire exposed faces of the electrodes are counted. These superficial areas extend to one hundred and fifty square centimeters for kilowatt; but this can be reduced about one-fourth. A good proportion for the capacity of the electrolytic bath or containing vessel will be about one-half liter per horse-power or three-fourths liter per kilowatt. The thickness of the carbon anode may be 1.9 centimeters and that of the iron cathode 0.1 centimeter. The form of the several receptacles or vessels will vary with the dimensions and output of the plant.

This process is applicable to the separation of any metal from its compound where its chlorid is less exothemic than sodium. The anhydrous chlorid is first formed, even though the mineral treated may be sulfurous, and sodium is then substituted for the base of the chlorid, thus setting the metal free.

I do not herein claim the apparatus shown and described, as this invention is limited to the process; but I have described and claimed this apparatus in another application, Serial No. 85,772, filed December 13, 1901.

Having thus described my invention, I claim—

1. The herein-described method of obtaining a metal from its compound, which consists in separating sodium from its chlorid, while at a high temperature, by electrolysis, then decomposing the oxid of the metal treated while hot, with the chlorin from the first step, whereby a chlorid of the metal is formed, and then decomposing the said chlorid by the hot sodium vapor from the first step, whereby sodium chlorid is formed and the metal treated set free, said operation being continuous, substantially as set forth.

2. The herein-described method of obtaining the metal aluminium from its oxid, which consists in first decomposing sodium chlorid at a high temperature and separating its elements, then passing the hot chlorin from the first step through alumina mixed with a carbonaceous substance, thereby producing, by reaction, carbonic oxid and aluminium chlorid, and then mixing with the hot, vaporous aluminium chlorid the hot, vaporous sodium from the first step, thereby producing, by reaction, sodium chlorid and aluminium in metallic form, substantially as set forth.

In witness whereof I have hereunto signed my name, this 26th day of June, 1901, in the presence of two subscribing witnesses.

GIROLAMO TADDEI.

Witnesses:
SECONDO TORLA,
VITTORIO GARNIER.