

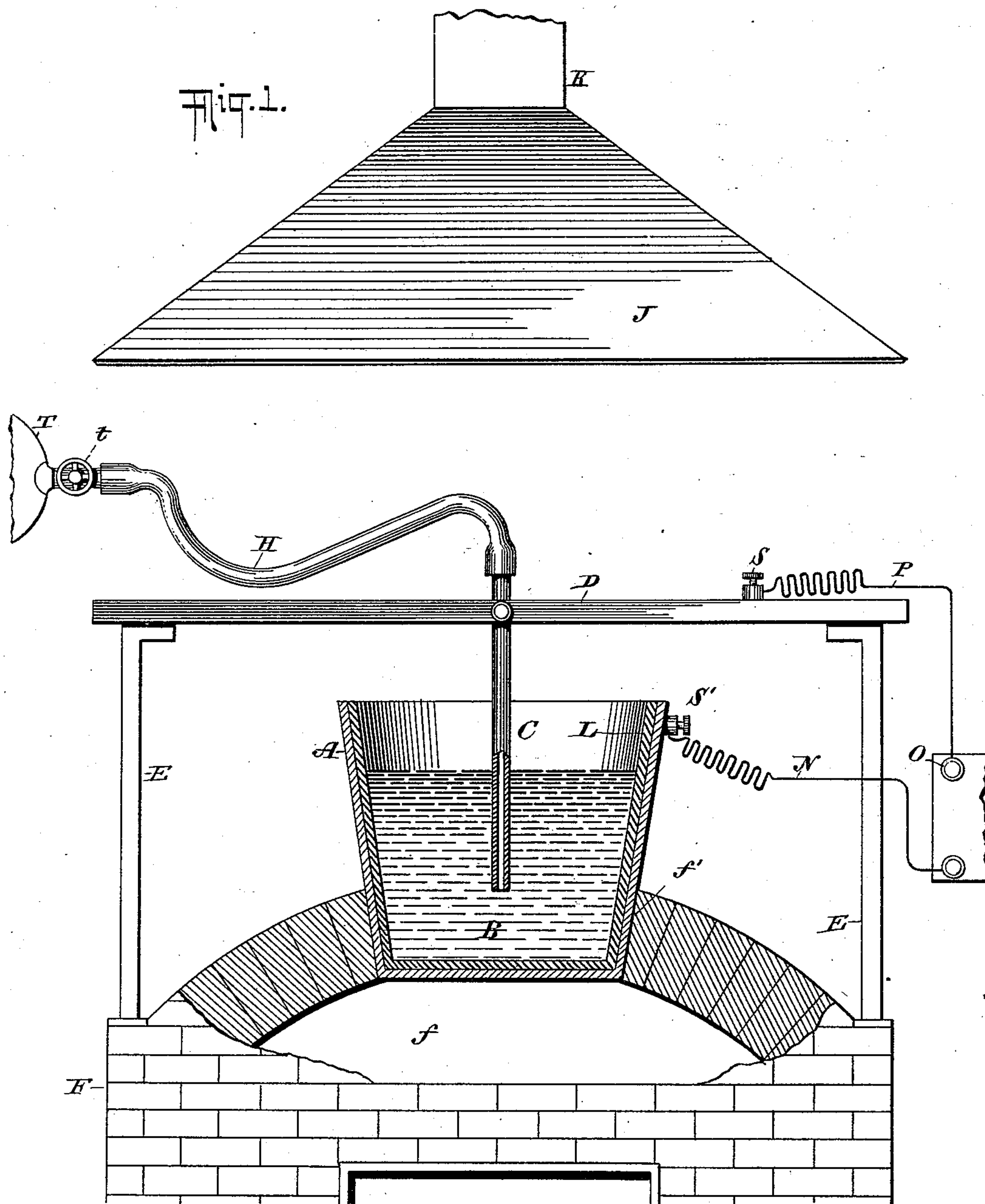
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2 Sheets—Sheet 1.

F. A. GOOCH & L. WALDO.
PROCESS OF REDUCING ALUMINIUM.

No. 527,848.

Patented Oct. 23, 1894.



WITNESSES:

Gustave Dietrich
Joseph A. O'Brien

INVENTORS

Leonard Waldo
Frank A. Gooch
BY
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ATTORNEY.

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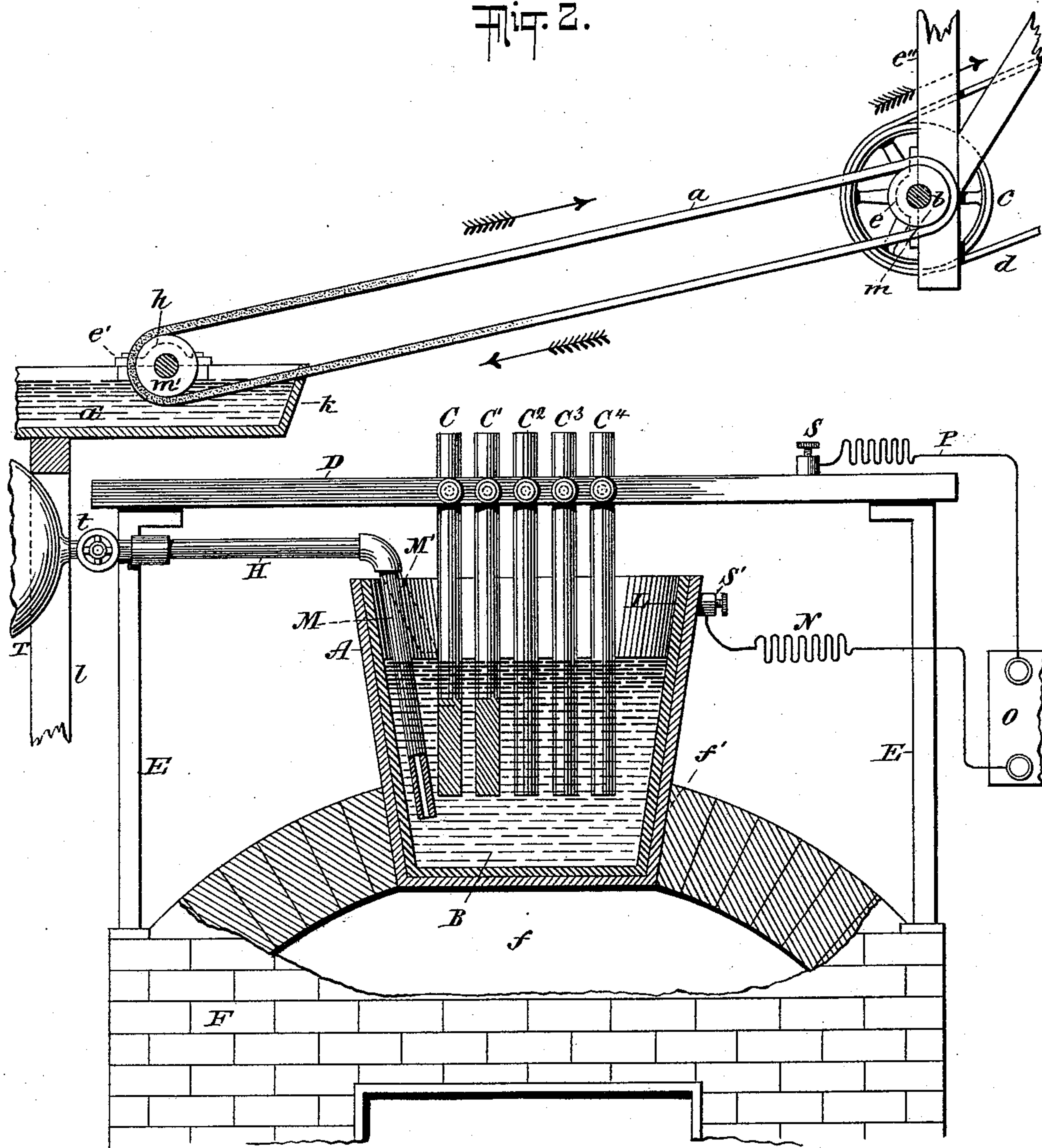
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Fig. 2.



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UNITED STATES PATENT OFFICE.

FRANK A. GOOCH, OF NEW HAVEN, AND LEONARD WALDO, OF BRIDGEPORT, CONNECTICUT; SAID WALDO ASSIGNOR TO THE WALDO FOUNDRY, OF NEW JERSEY.

PROCESS OF REDUCING ALUMINIUM.

SPECIFICATION forming part of Letters Patent No. 527,848, dated October 23, 1894.

Original application filed September 6, 1893, Serial No. 484,905. Divided and this application filed April 10, 1894. Serial No. 507,035. (No specimens.)

To all whom it may concern:

Be it known that we, FRANK A. GOOCH, residing at New Haven, in the county of New Haven, and LEONARD WALDO, residing at Bridgeport, in the county of Fairfield, State of Connecticut, have invented certain new and useful Improvements in Processes of Reducing Aluminium; and we 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.

This is a divisional application of the application filed by us September 6, 1893, Serial No. 484,905.

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

In an application filed by us September 1, 1893, Serial No. 484,561, we have described and claimed an improved process for reducing aluminium from its compounds, which in general terms consists in passing an electric current through a suitable fused bath containing a suitable compound of aluminium, thereby electrolyzing the bath, and in supplying hydrogen to the bath.

The invention herein described consists, in general terms, in passing an electric current through a suitable fused bath containing a suitable halogen compound of aluminium, thereby electrolyzing the bath, and in supplying to the bath a suitable gaseous compound of hydrogen.

In practice we find that the halogen compounds of aluminium, which are most suitable and best adapted to be used in our process, are the chloride of aluminium and the fluoride of aluminium.

For convenience, we describe our invention with special reference to the reduction of aluminium from its chloride, it being understood, however, that our invention is also applicable to other compounds of aluminium.

Hydrogen in its free state is comparatively expensive to produce. For this reason, we

have found it desirable to use some one of the gaseous compounds of hydrogen, many of which are easily and cheaply produced, and answer the purpose to be attained as well as the free hydrogen. We preferably use only such gaseous compounds as are rich in hydrogen and as permit their hydrogen constituent readily to unite with the halogen elements set free by the electrolysis of the bath. It is also best not to use any hydrogen compound which, when the hydrogen unites with the halogens will add any deleterious element to the bath. There are many gaseous compounds of hydrogen suitable for the purpose intended, of which the so-called "water-gas" of commerce, and the vapor of water, are two of the best known and most easily obtainable.

While our invention is applicable to every one of a large class of gaseous compounds, we shall, for convenience, describe it with reference to vapor of water only, selecting steam as the most convenient form thereof, it being understood that our description is in substance applicable to the use of all other suitable gaseous compounds of hydrogen.

In the accompanying drawings, which are hereby made a part of this specification, is shown one form of apparatus suitable to the practice of our invention. We do not, however, limit ourselves to the apparatus shown, since any other suitable apparatus would answer equally well for the practice of our invention.

Figure 1 represents a sectional diagrammatic view of the apparatus employed by us. Fig. 2 represents a sectional view of a modified form of apparatus employed by us.

Similar letters refer to similar parts throughout the drawings.

F is a furnace having a fire box *f*, and opening *f'* in the top of the furnace, into which is set a tapering crucible A, the same being supported by the sides of the opening *f'*.

The construction of the crucible and the arrangement of the electrodes and conductors are fully described hereinafter.

T is a tank from which the gaseous com-

pound of hydrogen is supplied through the tube H, the supply being regulated by a cock *t*.

s s' are binding posts for the conductors
5 N P.

B is the bath contained in the crucible A, through which the electric current is passed.

The construction and arrangement of other parts of the apparatus employed are herein-
10 after fully described.

In the practice of our invention, so far as it relates to the reduction of aluminium from its chloride, we preferably prepare a bath by fusing in a suitable tank or crucible
15 A in suitable proportions, the chloride of aluminium and the chloride of an alkaline metal (preferably sodium) or of an alkaline earth metal, with the fluoride of aluminium and the fluoride of sodium. A convenient way to
20 form a suitable bath is to combine the chloride of aluminium and the chloride of magnesium with the mineral cryolite. We find it of advantage to add to the combination just named a certain proportion of chloride of sodium,
25 which has the effect of making the bath more liquid. These ingredients are preferably mingled together in the following proportions: cryolite, fifty-five parts; chloride of aluminium, twelve parts; chloride of magne-
30 sium, five parts; chloride of sodium, twenty-eight parts; but the proportions above given are variable serviceably within considerable limits, without materially affecting the operation or function of the bath, as in fact any
35 proportions which may be found suitable may be employed.

The reason for employing cryolite or the fluorides of aluminium and sodium in the bath, is the well known fact that when they
40 are present and reduction by the action of the electric current takes place, the aluminium melts and flows to globules, while, when the bath contains only the chlorides named, the aluminium is obtained in the form of a
45 powder, which has to be further treated before it can be made commercially available.

The crucible A preferably employed by us in the operation of our invention, consists of a tank of iron lined with compacted carbon.
50 The carbon lining L of this tank serves as the cathode, the iron of the tank being connected by the conductor N with the negative pole of a dynamo-electric machine or other suitable source of electricity O.

55 The anode C is a detached carbon electrode partially immersed in the bath B and connected in the following manner with the source of electricity. We preferably suspend the anode C from an iron bar D raised above the crucible and resting on wooden supports
60 E E. The bar D is connected by the conductor P to the positive pole of the source of electricity. If desired the cathode may also be formed of a detached carbon, suspended in
65 a similar way to that just described for the anode, or otherwise supported, and partially immersed in the bath B.

If desired, a number of separate carbons may be used to form the electrodes, as shown in Fig. 2, where C, C', C², C³, and C⁴, all sus-
70 pended from the bar D, collectively form the anode.

The relative superficial areas of the electrodes in any particular case are adjusted so as to secure the greatest economy in action.
75 In place of a carbon anode, an anode of any other suitable material, as of platinum, may be used. The bath may be fused and the fusion may be maintained by the application of heat from any suitable source, as from a
80 furnace F. The temperature required to fuse the baths suggested above is that of a dull red heat. We may also fuse the ingredients of the bath in a separate apparatus, and introduce the same in a molten condi-
85 tion into the tank at the beginning of the process, the fusion being thereafter maintained in any suitable way. When the electric current is passed through the bath and electrolysis takes place, aluminium is de-
90 posited at the cathode, and the halogens, (chiefly chlorine) are liberated at the anode. It is well known that these gases, in a free state, are highly noxious to health. On this
95 account, and for the further reason that they attack and quickly destroy the positive electrode in the apparatus used in any process similar to the one described, their escape in
100 a free state would render such process commercially useless. To obviate these difficulties, and to accomplish other desirable results, we supply to the bath the vapor of water, preferably in the form of steam, which
105 we preferably deliver at or beneath the surface of the fused mass in a substantially continuous manner and in near proximity to, or through the anode. In the latter case, a longi-
110 tudinally perforated carbon rod C is preferably employed for the anode, and the steam is passed through the same as through a pipe, the steam being supplied through a tube H
115 connected with the upper end of the anode C, which may project above the bar D. The steam may also, if preferred, be conducted through a separate pipe, as the pipe M in
120 Fig. 2. Where the steam is supplied at the surface of the bath, it may be passed through pipe M' represented in Fig. 2 by dotted lines, in which case pipe M would be dispensed
125 with. Where we use steam in our process, we may dispense with the conduction pipe altogether and spray water on the surface of the bath. The water will instantly assume the spheroidal condition and quickly vaporize. We may also produce the steam by
130 dropping minute fragments of ice on the surface of the bath.

We do not limit our invention to any particular mode of supplying the steam or other gaseous compound of hydrogen to the bath,
130 since any suitable mode may be employed without departing from the essential nature of our invention.

The constituents of the steam or other gas-

eous compound of hydrogen used being dissociated in or at the surface of the bath during action, the hydrogen required for our process is supplied in a substantially continuous manner.

When the halogen compounds are liberated by the electrolysis of the bath, they combine instantly with the hydrogen constituent of the steam or other gaseous compound of hydrogen used, and escape at the anode in the form of hydrochloric acid or hydrofluoric acid (chiefly the former). These gases, in the quantities evolved by our process, are easily controlled and disposed of without injury to apparatus or workmen, by appropriate means external to the bath, as by the use of a flue K, having a strong draft and provided with a hood J projecting over the crucible A; (see Fig. 1;) or cloths or other absorbent media saturated with water may be suspended over the crucible, the escaping gases being taken up by the water, whose power of acting as a solvent for hydrochloric and hydrofluoric acid is well known. Such an arrangement is shown in Fig. 2, where *a* represents an endless band or apron of cloth or other suitable fabric, passing over drums *m m'* attached to shafts *b, h*. The shaft *b* revolves in journal boxes *e* and is supported in any suitable manner, as by a strip *e''* suspended from the ceiling. The shaft *h* revolves in boxes *e'* formed on the edges of the trough *k*. The shafts and band or apron *a* are kept in revolution in the direction indicated by the arrows by power from any suitable source transmitted by the belt *d*, passing over the pulley *c*, which is rigidly attached to the shaft *b*. The shaft *b* is hung somewhat higher than the shaft *h*. The drum *m'* on the shaft *h* runs close to or under the surface of water *w*, contained in a shallow pan or trough *k*, having suitable support *l*. The band or apron *a* being inclined passes through the water *w* at the lower side of the drum *m'* and is thus kept saturated with water. Thus saturated it passes continuously over the crucible.

We preferably supply the steam or other form of water vapor to the bath only in an amount sufficient to furnish the hydrogen for the combination with the halogen elements, or slightly in excess of such amount.

In the practical operation of our invention where we use the suggested bath composed of the chloride of aluminium and the chloride of magnesium, and the fluorides of aluminium and sodium, with or without the chloride of sodium, we have found that we obtain at the anode hydrochloric acid almost exclusively, with very little trace of fluorine. This shows that the aluminium deposited at the cathode is derived chiefly from the chloride of aluminium in the bath; and we have found that, by the addition to the bath from time to time of chloride of aluminium (Al_2Cl_6) in sufficient quantities, the bath will be replenished and the process will be a substantially continuous one during a considerable period of

time. An excess of the chloride of aluminium produces no injurious effect, as it is gradually utilized in the subsequent operation of the process. We have spoken of fluorine being set free, though we are aware that it is considered very difficult to isolate this element. We, however, use the term "fluorine" merely for convenience, meaning thereby to include not only free fluorine, if any be liberated, but also any compound thereof which might be set free at the anode if hydrogen either free or in combination were not used in the process.

As before stated, the aluminium is deposited at the cathode, where it melts forming globules, which (when the suggested bath whose proportions are hereinbefore stated is employed) gather at the bottom of the bath, the aluminium having a higher specific gravity than the fused bath. The aluminium may then be removed in any suitable or convenient manner, as for example by means of a ladle lined and covered with carbon, or by drawing it off through a suitable opening at the bottom. In case a bath should be used whose specific gravity is higher than that of aluminium, the aluminium would rise to the top, where it could be easily removed.

In the process herein described, we use an electric current of suitably low electro-motive force, usually of from four to ten volts, but the voltage of the current required will depend on the size and form of the electrodes as well as on the composition of the bath, it being only necessary to have a voltage sufficient to decompose electrolytically the compound of aluminium in the bath under all the existing conditions of resistance and of chemical constitution. The electric current which we employ in our process, and which is herein referred to as being passed through the bath, is employed solely for purposes of electrolysis. The gaseous compound of hydrogen may in the first instance be supplied to the bath before or after the electric current is passed through the same, or simultaneously therewith, the order of the operations being unimportant.

Where the gaseous compound of hydrogen employed contains oxygen, as in the case of steam, some oxidation of the anode is likely to take place, if the material of the anode is oxidizable; but, unless the material of the anode is very easily oxidizable, the waste of the anode will be usually much less than would be the case if the anode were exposed to the action of free halogens. It being possible to form a number of different baths to any of which our invention would be applicable, we do not limit our invention to the use of any particular bath which has been described herein.

In the process herein described, we do not employ gaseous compounds of hydrogen as agents in accomplishing the reduction of the metal from the aluminium compounds used, nor do we here claim any process in which

such gaseous compounds are so employed. The objects of supplying a gaseous compound of hydrogen to the bath in our improved process herein described are, as above stated, 5 largely to protect the anodes from the corrosive action of the halogens set free by the electrolysis of the bath, and to convert such free halogens into a more manageable form.

Having described our invention, what we 10 claim, and desire to secure 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 15 fusing the chloride of aluminium and the chloride of an alkaline earth metal with the fluoride of aluminium and the fluoride of sodium, passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same, and in supplying to the 20 bath a suitable gaseous compound of hydrogen, 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 25 fusing the chloride of aluminium and the chloride of an alkaline earth metal with the fluoride of aluminium and the fluoride of sodium, passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same, and in supplying steam to the bath, substantially as and for the purposes set forth. 30

3. As an improvement in the art of manufacturing aluminium, the herein described process, which consists in forming a bath by 35 fusing together the chlorides of aluminium

and magnesium with the fluorides of aluminium and sodium, passing an electric current 40 of suitably low voltage through the fused mass, thereby electrolyzing the same, and in supplying steam to the bath, 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 45 fusing together the chloride of aluminium, the chloride of an alkaline earth metal and the chloride of sodium with the fluorides of aluminium and sodium, passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same, and in supplying steam to the bath, substantially as and for the purposes set forth. 50

5. As an improvement in the art of manufacturing aluminium, the herein described process which consists in forming a bath by 55 fusing together the chlorides of aluminium, magnesium and sodium, and the fluorides of aluminium and sodium, passing an electric current of suitably low voltage through the fused mass, thereby electrolyzing the same, and in supplying steam to the bath, substantially as and for the purposes set forth. 60

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