GOVERNMENT OF INDIA : THE PATENT OFFICE, 214, LOWER CIRCULAR ROAD, CALCUTTA-17.

Specification No. 96709. Application No. 96709, dated 26th November, 1964.
Complete Specification left on 26th August, 1965. (Application accepted 8th July, 1966.)
Index at acceptance—9B+E+F[XXXII(1)], 70C2[LVIII(5)].

PROVISIONAL SPECIFICATION.

IMPROVEMENTS IN OR RELATING TO THE PRODUCTION OF MISCH METAL.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH , Rafi Marg, New Delhi-1, India, an Indian Registered Body incorporated under the Registration of Societies Act (ACT XXI OF 1860).

This is an invention by VEERARAGHAVA ARAVAMUTHAN, Scientist, KUPPUSWAMY VENUGOPALAN, Scientist, and THALEPARAMPIL PARAMESWARAN MADHAVAN, Senior Scientific Assistant, all of the Central Electrochemical Research Institute, Karaikudi-3, S. Rly., all Indian National.

The following specification describes the nature of this invention.

This invention relates to IMPROVEMENTS IN OR RELATING TO THE PRODUCTION OF MISCH METAL.

Hitherto it has been proposed to electrolyse fused salt mixtures containing cerium chloride with hygroscopic salt mixtures containing calcium chloride and most of the compositions have been kept a trade secret.

This is open to the objection that metal coalescence is not perfect; viscosity of the bath varies; and the compositions are altered; the refractory lining also gets attacked.

The object of this invention is to obviate these disadvantages by

(a) eliminating the use of refractories;
(b) eliminating the use of chlorides like anhydrous calcium chloride which are very hygroscopic in nature;
(c) to prepare the electrolyte composition in a simpler way; and
(d) to melt the electrolyte in the same electrolytic cell set-up.

To these ends, the invention broadly consists in

(1) utilising mixture with high percentages of cerium chloride in admixture with small quantities of barium chloride, potassium chloride and ammonium chloride;
(2) employing an externally heated and vacuum connected vessel wherein the dehydration of the cerium chloride is allowed to take place. In one modification, the external heating in thin layers is effected by employing mild steel rods as a resistor covered with a thin layer of metallurgical or petroleum coke and connecting the resistor to a low voltage, high amperage A.C. (Refer sheet 1 of the accompanying drawings);
(3) the melting of the electrolyte in the cell has been simplified by connecting the vessel itself to one limb of the low voltage, high amperage transformer and the carbon anode itself to the other limb of the A.C.;
(4) No refractory lining is employed for the electrolytic cell. The cell is so shaped that electrolysis takes place between the bottom narrow portions (Refer sheet 2 of the accompanying drawings) of the electrolytic cell and the carbon anode. The cell is simpler in construction and the bottom narrow portions can be repaired whenever necessary.

Thus in every aspect of electrolysis and electrolyte composition novelty has been introduced.

The following typical conditions are given to illustrate the invention :

Example

Raw material consumption for 10 kg of misch metal/day production

70 kilograms $CeCl_3.7H_2O$
1.5 kilograms of ammonium chloride
8 kilograms of potassium chloride
3.5 kilograms of barium chloride $2H_2O$
A.C. requirements=180 kw.hr. for dehydrating 55 kilograms of anhydrous salt.
=52 kw.hr. for melting 55 kgs.
D.C. requirements=11 volts, 900 amperes
For 21 hour run=210 kw. hrs.

R. BHASKAR PAI.

PATENTS OFFICER,

Council of Scientific and Industrial Research.

Dated this 6th day of November, 1964.

COMPLETE SPECIFICATION.

IMPROVEMENTS IN OR RELATING TO THE PRODUCTION OF MISCH METAL.

COUNCIL OF SCIENTIFIC AND INDUSTRIAL RESEARCH , Rafi Marg, New Delhi-1, India, an Indian Registered Body incorporated under the Registration of Societies Act (ACT XXI OF 1860).

This is an invention by VEERARAGHAVA ARAVAMUTHAN, Scientist, KUPPUSWAMY VENUGOPALAN, Scientist, and THALEPERAMPIL PARAMESWARAN MADHAVAN, Senior Scientific Assistant, all of the Central Electrochemical Research Institute, Karaikudi-3, S. Rly, all Indian National.

The following specification particularly describes and ascertains the nature of this invention and the manner in which it is to be performed.

This invention relates to IMPROVEMENTS IN OR RELATING TO THE PRODUCTION OF MISCH METAL.

Hitherto it has been proposed to electrolyse fused salt mixtures containing cerium chloride with hygroscopic salt mixtures containing calcium chloride and most of the compositions have been kept a trade secret.

When proper compositions are not used which is associated with careful preparation and handling of

electrolyte, the metal coalescence is not perfect; viscosity of the bath varies; and the compositions are altered; the refractory lining also gets attacked.

The object of this invention is to obviate these disadvantages by following any or combination of procedures suggested below :

(a) eliminating the use of refractories;
(b) eliminating the use of chlorides like anhydrous calcium chloride which are very hygroscopic in nature;
(c) to prepare the electrolyte composition in a simpler way; and
(d) to melt the electrolyte in the same electrolytic cell set-up.

The invented process for the production of misch metal by the electrolysis of fused chloride mixtures is characterised by utilising mixtures with 40-80 per cent. cerium chloride in admixture with 60-20 per cent. of barium chloride or calcium chloride or potassium chloride or mixtures thereof plus 1-3 per cent. ammonium chloride.

The process employs an extrernally heated and vacuum connected stationary or rotating vessel wherein the dehydration of the cerium chloride is allowed to take place, the external heating being preferably effected in thin layers by employing mild steel rods as a resistor covered with a thin layer of metallurgical or petroleum coke and connecting the resistor to a low voltage, high amperage a.c., or by employing any convenient solid, liquid or gaseous fuel heating or resorting to internal heating by combustion gases and the like.

The melting of the electrolyte in the cell has been simplified by connecting the cathode itself to one limb of the low voltage, high amperage transformer and the carbon anode itself to the other limb of the a.c., or employing d.c., itself for melting by a shorting arrangement between anode and cathode.

Either a bottom cathode arrangement could be employed with or without water or air cooling arrangement. Graphite or carbon electrodes could be utilised and under careful conditions Soderberg paste could also be employed.

The following are among the noteworthy novel features :

(1) utilising mixtures with high percentages of cerium chloride in admixture with small quantities of barium chloride, potassium chloride and ammonium chloride;
(2) employing an externally heated and vacuum connected stationary or rotating vessel wherein the dehydration of the cerium chloride is allowed to take place. In one modification, the external heating in thin layers is effected by employing mild steel rods as a resistor covered with a tin layer of metallurgical or petroleum coke and connecting the resistor to a low voltage, high amperage a.c.;
(3) the melting of the electrolyte in the cell has been simplified by connecting the cathode itself to one limb of the low voltage, high amperage transformer and the carbon anode itself to the other limb of the a.c., or employing d.c., itself for melting by a shorting arrangement between anode and cathode;
(4) either a bottom cathode arrangement or a side cathode arrangement could be employed with or without water or air cooling arrangement :
(5) graphite or carbon electrodes could be utilised and under careful conditions Soderberg paste could also be employed.

Thus novelty has been introduced.

The invention will now be described with the help of drawings accompanying the provisional specification where Figure 1 gives the view of an externally heated and vacuum connected stationary vessel wherein the dehydration of the cerium chloride in admixture with alkali and alkaline earth metal chlorides is allowed to take place.

1. Mild steel vessel of suitable dimensions.
2. Fireclay bricks.
3. Mild steel bent rods (electrical resistor).
4. Coke in small lumps.
5. Pipe welded to (1) and connected to partial suction.
6. A channel to seal the vessel with lid.
7. Mild steel lid.
8. Handle for the lid.

Figure II of the drawings accompanying the provisional specification is a view of the electrolytic cell where misch metal is produced.

9. Fireclay brick.
10. Mild steel vessel.
11. Misch metal formed.
12. Insulation.
13. Mild steel cathode lead.
14. Electrolyte.
15. Stand to support the anode.
16. Anode.
17. Insulating sleeve.
18. Mild steel lid for the cell.
19. Ratchet arrangement.
20. Wire rope.
21. Top beam.
22. Pulley arrangement.
23. Bolt and nut arrangement.
24. Supporting point for the anode.
25. Anode contact.
26. Bolt and nut.
27. Flexible anode lead.
28. Mild steel bent connected to cyclone separator.
29. Lid for cyclone separator.
30. Flanges.
31. Bolt and nut.
32. Outlet tube from the cyclone separator.
33. Cyclone separator.
34. Bolt and nut.
35. Flanges.
36. Drop collector.

The apparatus consists of two important parts. One is the dehydrating unit for the preparation of anhydrous rare earth chlorides in admixture with alkali and alkaline earth chlorides. This is represented diagrammatically in Figure I. A mild steel vessel of suitable dimensions (1) is kept on fireclay bricks (2) provided with mild steel bent rods functioning as electrical resistor (3) having a coke bed of suitable thickness (4) above it with a side pipe (5) welded to the body of the iron vessel (1) having a channel (6) to seal the vessel with the lid (7) which lid is provided with a handle (8).

The second part of the equipment is the electrolytic cell where electrolysis is conducted at high temperatures. On to fireclay bricks (9) a mild steel vessel (10) is kept at the bottom of which the misch metal formed (11) collects. This mild steel vessel is insulated (12) and provided with a cathode lead of mild steel (13). (14) represents the space occupied by the electrolyte. (15) is the stand for supporting the anode. (16) is the graphite or carbon anode. This graphite anode is separated from the top lead by insulating sleeve (17). The mild steel vessel (10) is closed by a mild steel lid (18). For raising and lowering the anode a ratchet arrangement (19) is provided. (20) represents the wire rope used in this connection and (21) is the top beam arrangement for supporting the pulley arrangement (22). (23) and (26) represent bolt and nut arrangements from either side with the anode and (24) and (25) from the supporting point and contact point for the anode. (27) is a flexible lead to give anode connection. The gases are drawn off from the mild steel vessel by a mild steel bent (28) which is in connection with a cyclone separator (33). (29) represents the lid for the cyclone separator; (30) represents the flanges ; (31) represents the bolt and nut arrangement and (32) is the outlet from the cyclone separator. (34) and (35) are

bolt and nut and flange arrangement respectively to connect the cyclone separator with the dust catcher (36).

The dehydrating equipment is utilised for the removal of the moisture from the mixed rare earth chlorides in admixture with alkali and alkaline earth metal chlorides with a minimum hydraulitic decomposition. Hence partial vacuum is also maintained.

The electrolytic cell is intended to produce the metal at the bottom of the mild steel vessel functioning as a cathode with the evolution of chlorine gas which escapes from the side tube of the electrolytic cell. The metal is in the molten state and being heavy settles at the bottom. Chlorine gas after a cyclone catcher is conducted away from the cell.

The following typical conditions are given to illustrate the invention :

EXAMPLE

Raw material consumption for 10 kg of misch metal/day production

70 kg $CeCl_3.7H_2O$
1.5 kg of ammonium chloride
8 kg of potassium chloride
3.5 kg of barium chloride $2H_2O$
A.C. requirements=180 kwh for dehydrating 55 kg of anhydrous salt=52 kwh for melting 55 kg
D.C. requirements=11 volts, 900 amperes
For 21 hour run=210 kwh.

We claim :

(1) A process for the production of misch metal by the electrolysis of fused chloride mixture characterised by utilising mixtures with 40-80 per cent. cerium chloride in admixture with 60-20 per cent. of barium chloride or calcium chloride orpotassium chloride or mixture thereof plus 1-3 per cen nium chloride.

(2) A process as claimed in Claim (1), e an externally heated and vacuum connected s or rotating vessel wherein the dehydratior cerium chloride is allowed to take place, the heating being preferably effected in thin employing mild steel rods as a resistor cover thin layer of metallurgical or petroleum connecting the resistor to a low voltage, hig age a.c., or by employing any convenient sol or gaseous fuel heating or resorting to inter ing by combustion gases and thelike.

(3) A process claimed in Claim (1) or (2) the melting of the electrolyte in the cell simplified by connecting the cathode itself to of the low voltage, high amperage transfo the carbon anode itself to the other limb o or employing d.c., itself for melting by a arrangement between anode and cathode.

(4) A process as claimed in any of the p claims wherein either a bottom cathode arr could be employed, with or without water or ing arrangement.

(5) A process as claimed in any of the p claims wherein graphite or carbon electro be utilised and under careful conditions paste could also be employed.

R. BHASKAR

PATENTS OFFIC

Council of Scientific & Industrial

Dated this 24th day of August, 1965.

MGIPC—S-Confdl.—71/78/4 Patent—26-11-74—10 .

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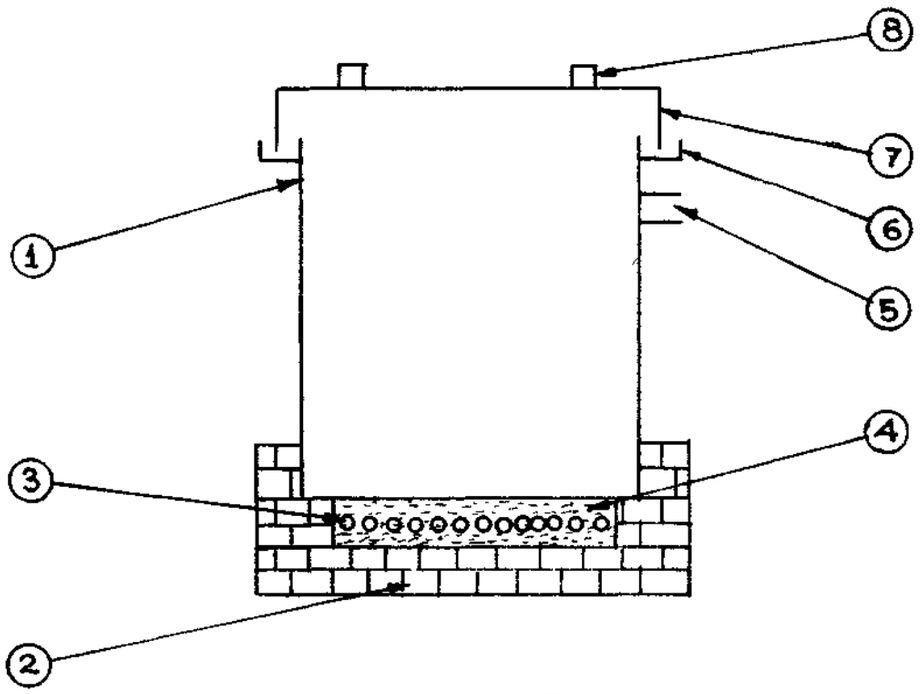

Fig. I.

R B Pai

(R.B. Pai)
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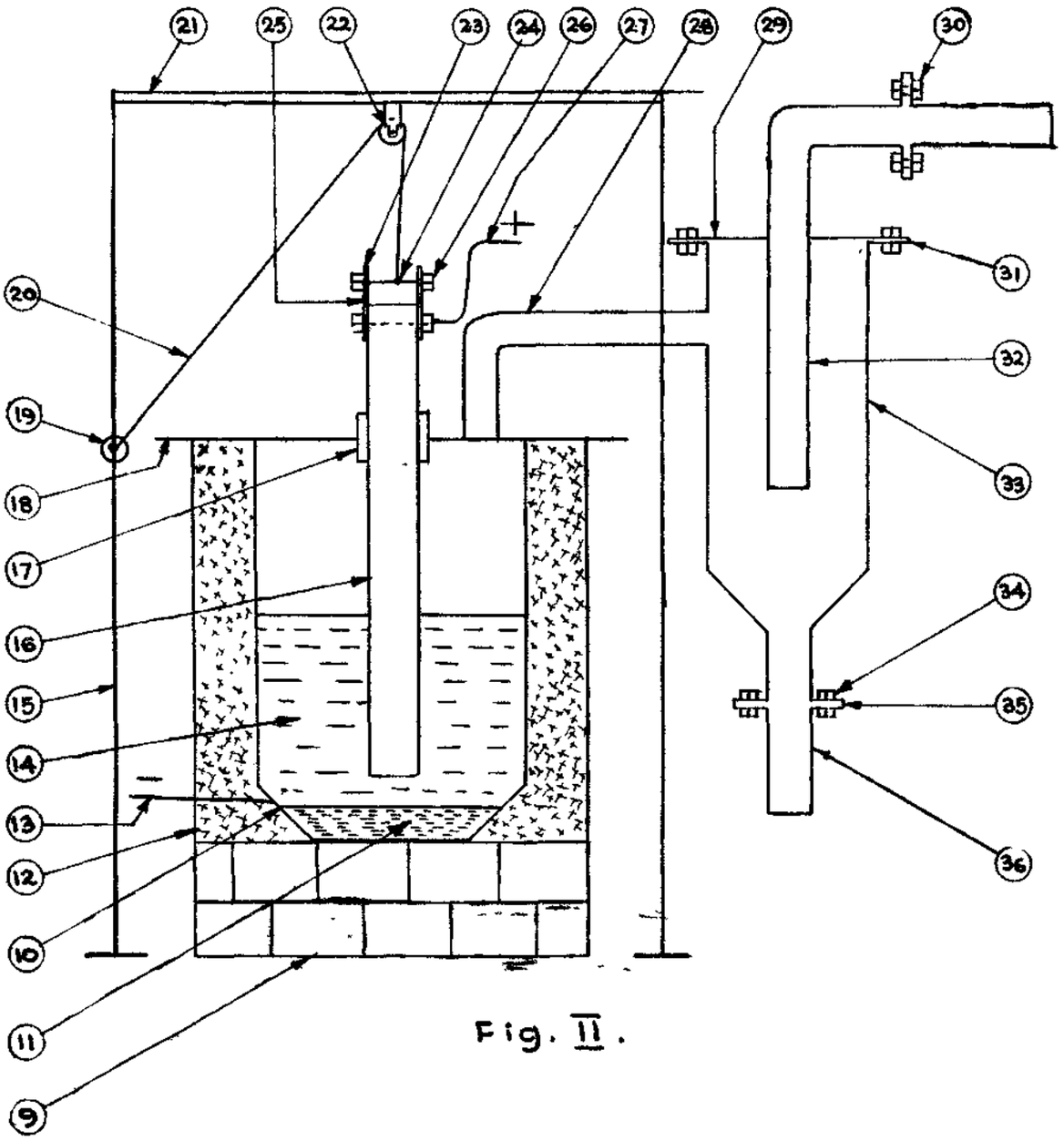

Fig. II.

R B Pal
(R.B. Pal)
Patents officer
C.S.I.R.

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