Sept. 11, 1951 R. D. BLUE 2,567,838

METALLURGICAL APPARATUS

Filed Nov. 16, 1949 2 Sheets-Sheet 1

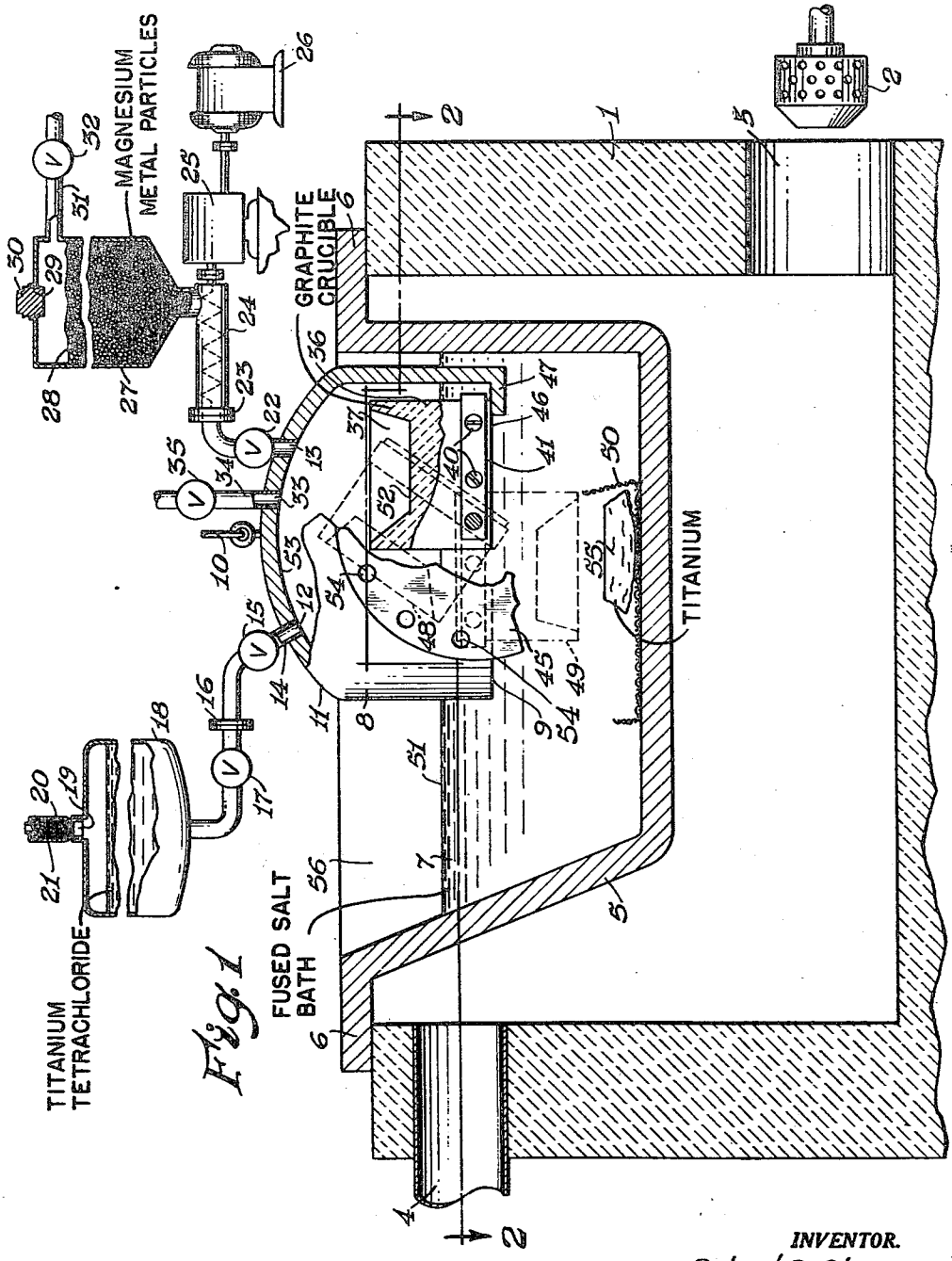


Fig. 1

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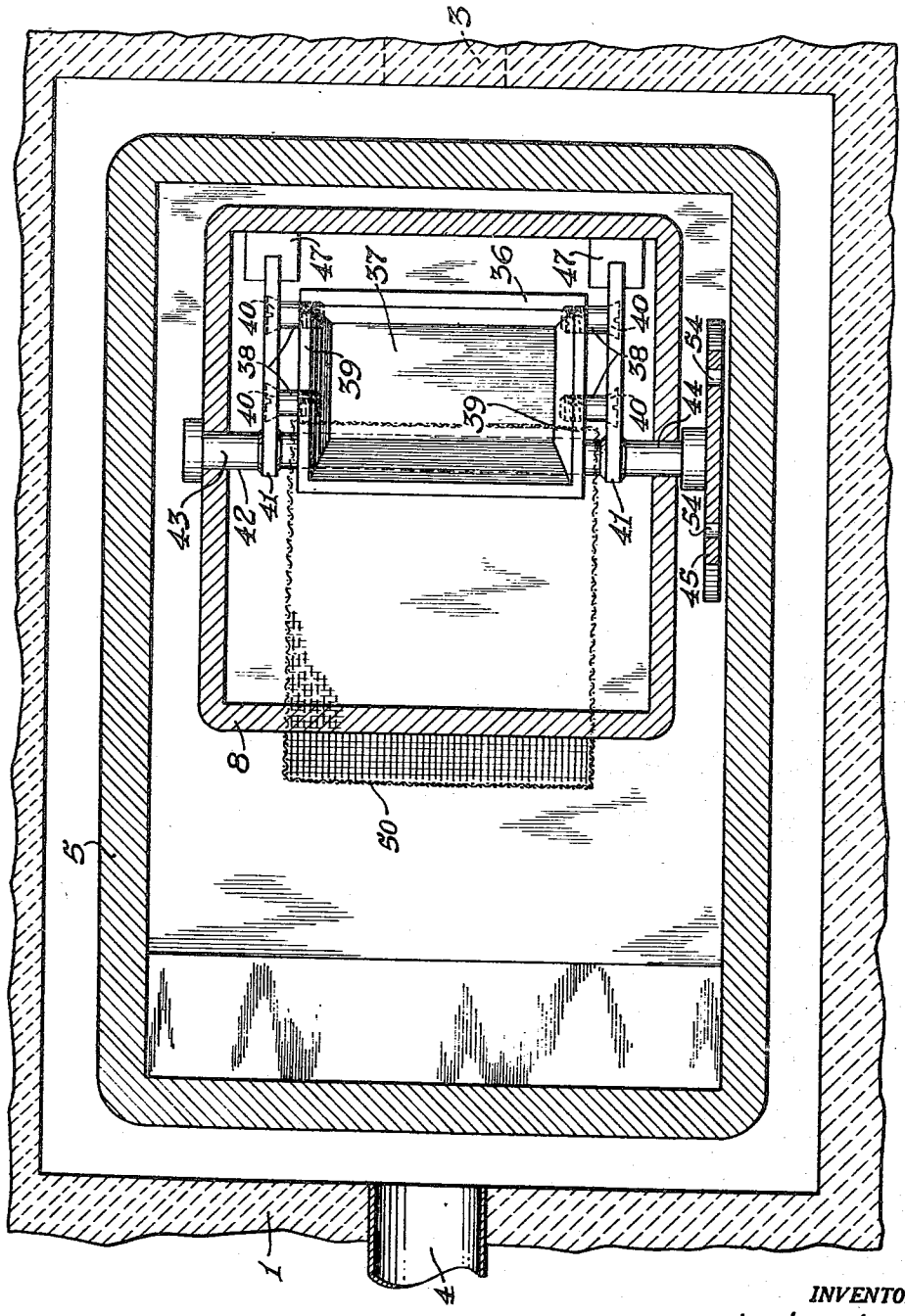


Fig. 2

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METALLURGICAL APPARATUS

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3 Claims. (Cl. 266—24)

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The invention relates to metallurgical apparatus particularly adapted for use in the reduction of titanium tetrachloride to metallic titanium by metallic magnesium.

A particular object of the invention is to provide apparatus in which the reduction of the tetrachloride is effected without contamination from the atmosphere.

A further object is to provide an apparatus which is capable of turning out batches of titanium in a continuous sequence.

In accomplishing these and other objects of the invention, I have provided an improved apparatus, the preferred form of which is illustrated in the accompanying drawings wherein:

Fig. 1 is a side elevation largely in section of the apparatus; and

Fig. 2 is a horizontal section of the apparatus on the line 2—2 of Fig. 1.

Referring to the drawing in detail, there is shown a furnace setting 1 heated by burner 2, the heat from which enters the setting through opening 3. The setting is vented through vent 4. A melting pot 5, which may be made of steel, is supported in the furnace setting 1 by the rim 6. The pot is adapted to contain a fused salt bath 7. A dome-shaped open end vessel or hood 8 has its open end 9 depending in the salt bath 7 so that the open end 9 is submerged in the fused salt which thereby forms a gas-tight seal over the open end. The hood 8 is suspended in the pot 5 on cable 10 as from a crane, not shown, which is capable of raising or lowering the hood.

The top 11 of the hood is provided with openings 12 and 13. Opening 12 is connected by pipe 14 through metering valve 15 and pipe union 16 to a titanium tetrachloride supply tank 18 having a shut off valve 17 and a filling opening 19 closed with a drying tube 20 containing a desiccant 21 whereby air entering the tank through the tube is dried. Opening 13 is connected through valve 22 and union 23 to the screw feeder 24 for metering the introduction of magnesium into the hood. The screw feeder 24 is driven through reducing gear 25 by motor 26. The screw feeder is provided with a supply hopper 27 for holding a supply of magnesium metal 28 in particle form. The hopper is provided with a filling opening 29 which may be closed by a removable plug 30. One end of pipe 31, communicates with the interior of hopper 27, the other end is connected through valve 32 to an inert gas supply, not shown. An opening 33 is provided in the top of the hood connected by pipe 34 through valve 35 to a source of inert gas, not shown, or vented to the atmosphere.

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Disposed under hood 8 beneath the point of introduction of the magnesium, i. e. under opening 13, is the reaction crucible 36. This is formed of graphite and comprises essentially a rectangular block, the top of which is dished to form the receptacle 37. As shown, the crucible 36 is supported by threaded bolts 38 which are screw-threadedly attached to the ends 39 of the crucible. The heads 40 of the bolts engage the side rails 41 which are attached to the cross shaft 42 passing through the bottom of the crucible. The ends 43 and 44 of the shaft are journalled in the bottom 9 of the hood 8. Attached to the end 44 of shaft 42 is a wheel 45 by means of which shaft 42 may be turned 180° so as to invert the crucible from the position shown in the full line in Fig. 1, with the bottom resting on the brackets 47 attached to hood 8, to the positions 48 and 49 shown by the dotted lines.

A basket 50, as of wire mesh, is disposed in the salt bath below the inverted position 49 of the crucible 36.

In operation, the pot is charged with a fusible anhydrous metal chloride of an alkali or alkaline earth metal or mixture of such salts, for example, the salts magnesium chloride, calcium chloride, sodium chloride may be used. A particularly suitable salt bath is made from $MgCl_2$ 15 to 25 per cent, $CaCl_2$ 20 to 25 per cent, CaF_2 about 1 per cent, the balance being NaCl. Sufficient salt charge is used to form a bath covering the lower end 9 of the hood 8, as indicated in Fig. 1, as by filling the pot to the level 51 which is below the bottom 52 of the receptacle 37 in its upright position.

The salt bath is heated to a temperature sufficient to maintain the salt charge in the molten state. The air under the hood is displaced as by flushing with inert gas. However, it is more satisfactory to disconnect the pipe unions 16 and 23 and lower the hood into the bath while valves 15 and 22 are closed and valve 35 opened and then lower the hood until the underside 53 of the top touches the top 51 of the salt bath, thereby allowing the air to escape. Thereafter inert gas is admitted through valve 35 and the hood 8 raised on cable 10 up to the position shown in Fig. 1, thereby filling the hood 8 with inert gas, and unions 16 and 23 are reconnected. Fused salt picked up in the dished portion 37 of the crucible as a result of lowering the hood into the salt bath is emptied out by turning the crucible into the position shown at 49 with the aid of the wheel 45. Wheel 45 may be rotated by pulling on a hook (not shown) inserted into the holes 54 of the wheel. After emptying out

fused salt, the crucible is returned to the position 36.

With the crucible empty and in the upright position, as shown at 36, and having an inert atmosphere in the hood 8, magnesium in solid particle form is introduced into the receptacle 37 by opening valve 22 and running motor 26 so as to feed particles of solid magnesium into the receptacle 37 from supply hopper 27. Inert gas may be admitted through valve 32 to take the place of the magnesium as it is run out of the supply hopper 27. Valve 17 is opened and also valve 15, the latter cautiously, so as to regulate the rate of addition of titanium tetrachloride. The titanium tetrachloride vaporizes upon entering the hood and tends to displace inert gas which may be vented through valve 35. The vapor reacts with the magnesium to form solid titanium metal and anhydrous magnesium chloride which are received by the receptacle 37. The titanium tetrachloride may be introduced into the hood 8 as fast as it will react with the magnesium and preferably in about 90 to 95 per cent of the stoichiometrical proportions as shown by the following equation: $2Mg+TiCl_4=Ti+2MgCl_2$, thereby maintaining an excess of magnesium during the reaction. The rate of $TiCl_4$ addition may be gauged from changes in level of the fused salt bath which serves as a manometer, the level of the salt bath rising outside of the hood when excess pressure is produced in the hood, and falling when subatmospheric pressures obtain.

When the dished portion 37 of the crucible becomes filled with produced titanium (the excess magnesium chloride being liquid overflows into the salt bath), the introduction of the $TiCl_4$ and magnesium metal is stopped and the crucible is inverted by rotating it about 180° with the aid of the wheel 45 into the position 49 whereupon the titanium metal, collected in the receptacle 37 as a result of the foregoing reaction, is dumped into the basket 50, as indicated at 55. By employing graphite as the crucible material, I have found that the titanium metal does not stick to it and is not significantly contaminated with carbon. The titanium product 55 is then removed from the salt bath by withdrawing the basket 50 with a suitable hook through the opening 56.

After emptying the basket of its titanium, the basket is returned to the position 50 and the crucible is rotated back into the position 36 as shown in Fig. 1. The reaction then may be repeated with additional charges of $TiCl_4$ and metallic magnesium without the need for again displacing air from the hood with inert gas and without atmospheric contamination of the produced titanium, thereby turning out batches of titanium in a continuous sequence. The salt bath level which rises as produced magnesium chloride accumulates is restored by bailing out the excess from time to time.

I claim:

1. In an apparatus in which to conduct the reducing reaction of metallic magnesium upon titanium tetrachloride vapor at elevated temperature to produce metallic titanium the combination of a vessel adapted to contain titanium tetrachloride vapor at elevated temperature, a

first metering means associated with the vessel adapted to introduce solid magnesium into the vessel at a controllable rate, a second metering means associated with the vessel adapted to regulate the rate of introduction of liquid titanium tetrachloride into the vessel, a graphite crucible disposed in the vessel beneath the point of introduction of the magnesium adapted to collect the metallic titanium reduced by the magnesium, and turning means connected with the crucible adapted to invert the crucible.

2. In an apparatus in which to conduct the reducing reaction of metallic magnesium upon titanium tetrachloride vapor at elevated temperature to produce metallic titanium the combination of an open top melting pot adapted to hold a body of a fused saline melt, a dome-like hood suspended in the melting pot with the open end of the hood depending downwardly into the pot so as to be sealed by a body of a fused saline melt in the pot, a first metering means associated with the hood adapted to introduce solid magnesium into the hood at a controllable rate, a second metering means associated with the hood adapted to regulate the rate of introduction of liquid titanium tetrachloride into the hood, a graphite crucible disposed under the hood with its inside bottom below the top of the melting pot and above the bottom of the hood, said crucible having an open top, turning means outside the hood connected with the said crucible adapted to invert the crucible while under the hood so as to dump out the contents of the crucible.

3. In an apparatus in which to conduct the reducing reaction of metallic magnesium upon titainium tetrachloride vapor at elevated temperature to produce metallic titanium the combination of a furnace setting; an open top melting pot adapted to hold a body of a fused saline melt; a dome-like hood suspended in the pot with the open end depending downwardly into the melting pot so as to be sealed by a body of a fused saline melt in the pot; a first metering means associated with the hood adapted to introduce solid magnesium into the hood at a controllable rate; a second metering means associated with the hood adapted to regulate the rate of introduction of titanium tetrachloride into the hood; a graphite crucible having an open top, said crucible being disposed under the hood with its inside bottom below the top of the melting pot and above the bottom of the hood; turning means outside the hood connected with the crucible adapted to invert the crucible while under the hood so as to dump out the contents of the crucible.

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