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(54) METHOD AND SYSTEM FOR ELECTRICALLY REDUCING A SOLID FEEDSTOCK

VERFAHREN UND SYSTEM ZUR ELEKTROLYTISCHEN REDUKTION EINES FESTEN
ROHSTOFFES

PROCÉDÉ ET SYSTÈME DE RÉDUCTION ÉLECTROLYTIQUE D'UNE CHARGE D'ALIMENTATION
SOLIDE

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EP 2 640 871 B1

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Description

[0001] The invention relates to an electrolysis method using apparatus comprising an electrode module or assembly.

Background

[0002] The present invention concerns a method for the reduction of a solid feedstock comprising a metal compound or compounds, such as a metal oxide, to form a reduced product. As is known from the prior art, electrolytic processes may be used, for example, to reduce metal compounds or semi-metal compounds to metals, semi-metals, or partially-reduced compounds, or to reduce mixtures of metal compounds to form alloys. In order to avoid repetition, the term metal will be used in this document to encompass all such products, such as metals, semi-metals, alloys, intermetallics, and partially-reduced products.

[0003] In recent years there has been great interest in the direct production of metal by reduction of a solid feedstock, for example, a solid metal-oxide feedstock. One such direct reduction process is the Cambridge FFC electro-decomposition process (as described in WO 99/64638). In the FFC process a solid compound, for example a solid metal oxide, is arranged in contact with a cathode in an electrolysis cell comprising a fused salt. A potential is applied between the cathode and an anode of the cell such that the compound is reduced. In the FFC process, the potential that produces the solid compound is lower than a deposition potential for a cation from the fused salt. For example, if the fused salt is calcium chloride, then the cathode potential at which the solid compound is reduced is lower than a deposition potential for depositing metallic calcium from the salt.

[0004] Other reduction processes for reducing feedstock in the form of a cathodically-connected solid metal compound have been proposed, such as the polar process described in WO 03/076690 and the process described in WO 03/048399.

[0005] WO2008/101283 discloses an electrochemical cell for reducing a metal oxide such as titanium oxide in the solid state. The cell includes cathodes that are formed at least in part from the metal oxide that may be removed from the cell and replaced by other cathodes.

[0006] DE4344036 discloses a method for the replacement of carbon anodes in an aluminium electrolysis process.

[0007] Conventional implementations of the FFC process and other electrolytic reduction processes typically involve the production of a feedstock in the form of a preform or precursor, fabricated from a powder of the solid compound to be reduced. This preform is then painstakingly coupled to a cathode to enable the reduction to take place. Once a number of preforms have been coupled to the cathode, then the cathode can be lowered into the molten salt and the preforms can be reduced. It

can be highly labour intensive to produce the preforms and then attach them to the cathode. Although this methodology works well on a laboratory scale, it does not lend itself to the mass productions of metal on an industrial scale.

It is an aim of the invention to provide an electrolysis method that is more suitable for the reduction of a solid feedstock on an industrial scale.

Summary of Invention

[0008] The invention, is defined in the appended claims, and provides a method of electrolytically reducing a solid feedstock as defined in the appended independent claim to which reference should now be made. Preferred or advantageous features of the invention are set out in dependent subclaims.

[0009] Thus, a method of electrolytically reducing a solid feedstock comprises the steps of: positioning an electrode module comprising at least one electrode in a first position for loading of feedstock, loading solid feedstock onto the electrode module, moving the electrode module from the first position and engaging the electrode module with an electrolysis chamber such that the feedstock is in contact with a molten salt within the electrolysis chamber, and applying a voltage to the electrode module such that the solid feedstock is reduced.

Preferably the electrode module comprises at least a cathode and an anode coupleable to a power supply such that a potential can be generated between the anode and the cathode. The electrode module may comprise one or more bipolar electrodes.

Preferably the solid feedstock is loaded such that it contacts a cathode or a cathodic surface of a bipolar electrode.

[0010] The electrode module is transferred from the first position within a transfer module. The transfer module may take the form of a housing defining a chamber into which the electrode module may be raised. Preferably the transfer module is sealable such that the electrode module may be transferred in controlled conditions, for example under an inert atmosphere.

[0011] The electrode module is heated to a predetermined temperature prior to engaging with the electrolysis chamber. In a preferable embodiment the electrolysis chamber contains a molten salt when the electrode module is engaged. If the electrode module is not at an appropriate temperature then thermal distortion or thermal shock of components of the electrode module may occur and may result in failure of components of the electrode module. Thus it is preferable that the electrode module is heated to a temperature close to the temperature of the molten salt. The predetermined temperature may therefore be within the range of about 500°C to 1200°C, depending on the temperature of the molten salt. Particularly preferable temperatures are in the range of 700°C to 1000°C, for example about 800°C or 850°C.

Advantageously, the electrode module may be heated

under an inert atmosphere within the transfer module. The transfer module may comprise heating elements that raise the temperature within the module to a predetermined temperature in order to heat the electrode module. Alternatively, the transfer module may comprise means for allowing a heated gas to be introduced into the transfer module to heat the electrode module.

[0012] The electrode module is transferred from the transfer module to the heating station in order to be heated to the predetermined temperature. The transfer module engages with a heating station and transfer the electrode module into a discrete heating station to allow the electrode module to be heated. The transfer module need not comprise heating elements itself.

[0013] The electrode module is transferred from the loading station within the transfer module, lowered into the heating station, heated to the predetermined temperature, and raised back into the transfer module to be transferred to the electrolysis chamber.

[0014] The electrode module is preferably sealed within the transfer chamber of the transfer module by the closing of a closure. A preferable closure is a gate valve in which the gate is slideable to seal the transfer chamber within the transfer module.

[0015] An opening of the electrolysis chamber, i.e. an opening through which the electrode module may be passed to engage with the electrolysis chamber, is preferably closed by an openable closure. A particularly preferable closure is a gate valve that is openable to allow the electrode module to pass to the electrolysis chamber.

[0016] It may be desirable to remove the electrode module from the electrolysis chamber after electrolysis in order to recover the reduced feedstock. Preferably the removal of the electrode module occurs at or near to the working temperature of the electrolysis chamber and under conditions in which a molten salt contained within the electrolysis chamber is still in the molten state.

[0017] Preferably the electrode module is lifted from the electrolysis chamber into a transfer module.

[0018] It may be advantageous that the electrode module is cooled under an inert atmosphere within the transfer module after being removed from the electrolysis chamber. If the electrode module is at a high temperature, for example 800°C, it is important that the module does not come into contact with oxygen or air until the temperature is reduced sufficiently to avoid auto-ignition of any carbon components of the electrode module or the rapid oxidation of any reduced feedstock located on the electrode module.

[0019] The electrode module may be cooled under an inert atmosphere within the transfer module after being removed from the electrolysis chamber. Thus, the transfer module may comprise cooling means such as water cooling tubes or may comprise means for passing a cooling gas through the transfer module in order to reduce the temperature of the electrode module.

[0020] Alternatively, the electrode module may be transferred within the transfer module to a discrete cool-

ing station to be cooled to a predetermined temperature under an inert atmosphere.

[0021] It is preferable that the cooling station comprises a cooling chamber within which the electrode module may be engaged to effect cooling. The method may therefore comprise the steps of transferring the electrode module to the cooling station within a transfer module, lowering the electrode module into the cooling station, cooling the electrode module to the predetermined temperature, and raising the electrode module back into the transfer module to be transferred away from the cooling station.

[0022] The molten salt remaining on the electrode module will solidify as the electrode module is cooled. Thus, the electrode module, once cooled, will be coated in a film of solidified salt. It may be advantageous, therefore, for the electrode module to be transferred to a washing station for washing salt from the reduced feedstock. The washing station may comprise washing apparatus suitable for directing jets of water towards the electrode module in order to wash salt from the feedstock. The washing station may further comprise means for collecting used water from the washing process.

[0023] It may be advantageous that the electrode module is transferred to a discrete unloading station to facilitate access to the electrode module for unloading the reduced feedstock. It is particularly preferable that the electrode module has removable trays that can be decoupled from the electrode module. Thus, it may be preferable that the solid feedstock is loaded onto removable trays separate from the electrode module and then the removable trays are coupled to the electrode module to load feedstock onto the electrode module. It may also be advantageous that the trays may be removed from the electrode module in order to facilitate unloading of reduced feedstock.

[0024] Preferably the feedstock is loaded such that it contacts a cathode structure of the electrode module for example the surface of a cathode or a cathodic surface of a bipolar electrode. This is essential if the reaction to reduce the feedstock is to occur using the FFC process. Other reduction process may be used, however

[0025] It is particularly preferable that the electrolysis reaction in the method proceeds by electro-deoxidation of the solid feedstock, for example electro-deoxidation by means of the FFC process.

[0026] The method may be applicable for use with any electrode module that is capable of being loaded with feedstock and engaged within an electrolysis chamber for electrolysis of the feedstock. There may be a number of specific embodiments of electrode module that may be preferably used in the method.

[0027] In a preferred embodiment of the invention, it may be advantageous that the electrode module is a removable electrode module for engagement with an electrolysis chamber, the removable electrode module comprising, a first electrode, a second electrode, and a suspension structure comprising a suspension rod cou-

pled, preferably at one end of the rod, to the first electrode, in which the second electrode is suspended by, or supported by, the suspension structure and in which the suspension structure comprises at least one electrically-insulating spacer element for retaining the second electrode in spatial separation from the first electrode.

[0028] In a further preferred embodiment of the invention, it may be advantageous that the electrode module is a removable electrode module for engagement with an electrolysis chamber, the removable electrode module comprising, an anode, and a cathode for supporting a portion of solid feedstock for reduction by electrolysis in a molten salt electrolyte, the feedstock being retained in contact with the cathode.

[0029] In a further preferred embodiment of the invention, it may be advantageous that the electrode module is a removable electrode module for engagement with an electrolysis chamber, the removable electrode module comprising, a first electrode, and a cover, in which, when the removable electrode is engaged with the electrolysis apparatus, the first electrode is located within the electrolysis chamber such that it may be used for electrolysis, and the cover spans an opening of the electrolysis chamber.

[0030] In a further preferred embodiment of the invention, it may be advantageous that the electrode module is a removable electrode module for engagement with an electrolysis chamber, the removable electrode module comprising, a lifting element to enable the module to be lifted, a first electrode coupled to a lower end of a suspension rod, and a resilient means disposed between the lifting element and an upper end of the suspension rod.

[0031] The various aspects and embodiments of the invention described herein are particularly suitable for the production of metal by the reduction of a solid feedstock comprising a solid metal oxide. Pure metals may be formed by reducing a pure metal oxide and alloys and intermetallics may be formed by reducing feedstocks comprising mixed metal oxides or mixtures of pure metal oxides.

[0032] Some reduction processes may only operate when the molten salt or electrolyte used in the process comprises a metallic species (a reactive metal) that forms a more stable oxide than the metallic oxide or compound being reduced. Such information is readily available in the form of thermodynamic data, specifically Gibbs free energy data, and may be conveniently determined from a standard Ellingham diagram or predominance diagram or Gibbs free energy diagram. Thermodynamic data on oxide stability and Ellingham diagrams are available to, and understood by, electrochemists and extractive metallurgists (the skilled person in this case would be well aware of such data and information).

[0033] Thus, a preferred electrolyte for a reduction process may comprise a calcium salt. Calcium forms a more stable oxide than most other metals and may therefore act to facilitate reduction of any metal oxide that is

less stable than calcium oxide. In other cases, salts containing other reactive metals may be used. For example, a reduction process according to any aspect of the invention described herein may be performed using a salt comprising lithium, sodium, potassium, rubidium, caesium, magnesium, calcium, strontium, barium, or yttrium. Chlorides or other salts may be used, including mixture of chlorides or other salts.

[0034] By selecting an appropriate electrolyte, almost any metal oxide may be capable of reduction using the methods and apparatuses described herein. In particular, oxides of beryllium, boron, magnesium, aluminium, silicon, scandium, titanium, vanadium, chromium, manganese, iron, cobalt, nickel, copper, zinc, germanium, yttrium, zirconium, niobium, molybdenum, hafnium, tantalum, tungsten, and the lanthanides including lanthanum, cerium, praseodymium, neodymium, samarium, and the actinides including actinium, thorium, protactinium, uranium, neptunium and plutonium may be reduced, preferably using a molten salt comprising calcium chloride.

[0035] The skilled person would be capable of selecting an appropriate electrolyte in which to reduce a particular metal oxide, and in the majority of cases an electrolyte comprising calcium chloride will be suitable.

Specific embodiments of the invention

[0036] Specific embodiments of the invention will now be described with reference to the figures in which;

Figure 1 is a perspective-view of a removable electrode module embodying one or more aspects of the invention;

Figure 2 is a side-view of the removable electrode module of Figure 1;

Figure 3 is a plan-view of the removable electrode module of Figure 1;

Figure 4 is a cross-sectional side-view of the removable electrode module of Figure 1 illustrating the structure of the various electrodes and supporting components of the removable electrode module;

Figure 5 is a schematic cross-sectional illustration of an electrolysis apparatus having an electrolysis chamber suitable for receiving the removable electrode module embodiment illustrated in Figure 1;

Figure 6 is a schematic cross-sectional illustration showing the removable electrode module of Figure 1 in engagement with the electrolysis apparatus illustrated in Figure 5;

Figure 7 is a schematic cross-sectional illustration showing the removable electrode module of Figure

1 housed within a transfer module seated on the electrolysis apparatus of Figure 5, in preparation for engaging the electrode module with the electrolysis chamber of the electrolysis apparatus;

Figure 8 is a schematic cross-sectional illustration showing the removable electrode module of Figure 1 after it has been passed from a transfer module and engaged with the electrolysis apparatus of Figure 5;

Figure 9 is a perspective-view of a removable cathode-tray structure suitable for use as a cathode-tray in the removable electrode module of Figure 1;

Figure 10 is a plan-view of the cathode-tray structure of Figure 9;

Figure 11 is a side-view of the cathode-tray structure of Figure 9;

Figure 12 is a cross-sectional illustration of a second embodiment of a removable electrode module according to one or more aspects of the invention;

Figure 13 is a cross-sectional illustration of a third embodiment of a removable electrode module according to one or more aspects of the invention.

Figure 14 is a schematic cross-sectional illustration of an alternative method of coupling a removable electrode module according to an embodiment of the invention to a lifting means;

Figure 15 is a schematic illustration of an electrode module situated at a loading station with a transfer module located above the electrode module;

Figure 16 is a schematic illustration of an electrode module held within a transfer module above a heating station;

Figure 17 is a schematic illustration showing a transfer module engaged with a heating station;

Figure 18 is a schematic illustration of an electrode module held within a transfer module above a cooling station;

Figure 19 is a schematic illustration showing a transfer module engaged with a cooling station; and

Figure 20 is a schematic illustration of an electrode module undergoing washing at a washing station.

[0037] A removable electrode module according to a first embodiment of the invention will now be described with reference to Figures 1 to 4. The electrode module

10 comprises a terminal anode 20, a terminal cathode 30, and seven bipolar electrodes 40, 41, 42, 43, 44, 45, 46 distributed in spatial separation from each other above the terminal cathode 30 and below the terminal anode 20. The terminal cathode 30, the terminal anode 20, and each of the intermediate bipolar electrodes 40, 41, 42, 43, 44, 45, 46, are substantially circular in shape and have a diameter of about 550 mm.

[0038] The terminal cathode 30 has a composite structure consisting of a lower portion and an upper portion. The lower portion is a substantially cathode base element 30a formed from a disc of grade 310 stainless steel having a diameter of 550 mm and a thickness of 60 mm. The upper portion is provided by a removable tray-assembly 30b seated on an upper surface of the base element 30a. The removable tray-assembly 30b is illustrated in Figures 9, 10 and 11 and will be described in more detail below. A central hole having a diameter of about 130 mm is defined through the central portion of the assembled tray-assembly 30b.

[0039] Each of the seven bipolar electrodes 40, 41, 42, 43, 44, 45, 46, has a composite structure comprising a lower portion 40a, 41a, 42a, 43a, 44a, 45a, 46a and an upper, or tray-assembly, portion 40b, 41b, 42b, 43b, 44b, 45b, 46b. The upper, tray-assembly, portions of each of the bipolar electrodes are identical to the upper, tray-assembly, portion 30b of the terminal cathode 30.

[0040] The lower portions 40a, 41a, 42a, 43a, 44a, 45a, 46a of each of the bipolar electrodes are formed from discs of carbon, for example graphite, having a diameter of 550 mm and a thickness of 60 mm. A hole having a diameter of about 130 mm is defined through the central portion of each of the bipolar electrodes 40, 41, 42, 43, 44, 45, 46.

[0041] On a lower surface of each bipolar electrode a plurality of channels 50 of approximately 10 mm in width are defined in order to aid the channelling of gas evolved on the lower surface of each bipolar electrode to the outer circumference of each bipolar electrode.

[0042] A first bipolar electrode 40 is supported directly above the terminal cathode 30 by a first electrically-insulating spacer element 60. The first electrically-insulating spacer element 60 is a tubular spacer formed from alumina. The first electrically-insulating spacer element may alternatively be formed from other electrically-insulating ceramic materials such as yttria or boron nitride.

[0043] In some embodiments the first electrically-insulating spacer element 60 is seated directly on the cathode base element 30a. In other embodiments, a ceramic insert 70, formed from a ceramic material that will not reduce under the cell operating conditions, is disposed between the terminal cathode base element 30a and the first electrically-insulating spacer element 60.

[0044] A lower surface of the lower portion 40a of the first bipolar electrode 40 is seated on the first electrically-insulating spacer element 60 such that the first bipolar electrode 40 is supported, through the first electrically-insulating spacer element 60, by the terminal cathode

base element 30a.

[0045] The second bipolar electrode 41 is supported directly above the first bipolar electrode 40 by means of a second electrically-insulating spacer element 61. The second electrically-insulating spacer element 61 is a tubular alumina element that is substantially identical to the first electrically-insulating spacer element 60. The second electrically-insulating spacer element is seated on an upper surface of the lower portion 40a of the first bipolar electrode 40. A lower surface of the lower portion 41a of the second bipolar electrode is, in turn, seated on the second electrically-insulating spacer element such that the second bipolar electrode 41 is supported, by means of the second electrically-insulating spacer element 61, by the first bipolar electrode.

[0046] This support structure is repeated for each of the bipolar electrodes. Thus, a third bipolar electrode 42 is supported by the second bipolar electrode 41 by means of a third electrically-insulating spacer element 62. A fourth bipolar electrode 43 is supported by the third bipolar electrode 42 by means of a fourth electrically-insulating spacer element 63. A fifth bipolar electrode 44 is supported by the fourth bipolar electrode 43 by means of a fifth electrically-insulating spacer element 64. A sixth bipolar electrode 45 is supported by the fifth bipolar electrode 44 by means of a sixth electrically-insulating spacer element 65. A seventh bipolar electrode 46 is supported by the sixth bipolar electrode 45 by means of seventh electrically-insulating spacer element 66.

[0047] The terminal anode 20 is formed from a disc of graphite having a diameter of 550 mm and a thickness of 60 mm. Channels are defined on the lower surface of the anode the same way as defined above in relation to the bipolar electrodes. One purpose of these channels is to assist the removal of gas evolved at the lower surface of the terminal anode 20. A hole is defined through a central portion of the terminal anode 20 having a diameter of about 130 mm. The terminal anode is supported directly above the seventh bipolar electrode 46 by means of an eighth electrically-insulating spacer element 67.

[0048] The removable electrode module 10 further comprises an insulating ceramic cover 100 disposed directly above the terminal anode 20. The cover 100 is formed from alumina, although any thermally-insulating ceramic material could be used, and is designed to cover an electrolysis chamber of an electrolysis apparatus during an electrolysis reaction. The cover 100 is supported by an upper surface of the terminal anode 20 by means of a ninth electrically-insulating supporting element 68. The ninth electrically-insulating support 68 is similar to the electrically-insulating support elements previously described, but has greater length.

[0049] A central hole is defined through the cover 100. Thus, a hole or cavity is defined that extends downwardly through the removable electrode module from an upper surface 101 of the cover 100 through the tubular electrically-insulating spacer 68, through the centre of the anode, and through each of the bipolar electrodes and their

associated spacer elements. A suspension rod 110 extends through this hole or cavity and is coupled to the cathode base element 30a of the terminal cathode 30 by means of a thread that engages with a threaded hole defined in the cathode base element 30a. The suspension rod 110 does not contact any other electrode or spacing element. At the point that the suspension rod 110 passes through the central hole defined through the cover 100, a seal is formed by means of a graphite gland packing, for example braided graphite rope or other similar gland packing materials 120.

[0050] At its upper portion, the suspension rod 110 is coupled to a j-slot type connector 130. A j-slot connector is a bayonet connector that is well known for coupling sections of pipe in the oil industry. The coupling between the suspension rod and the j-slot connector is achieved by means of washers and nuts 111.

[0051] The suspension rod 110 may be used to lift the entire removable electrode module 10, for example when raising or lowering the electrode module. In use, the suspension rod may need to function at high temperatures. Therefore, the rod 110 and associated nuts and washers 111 that couple the rod 110 to the j-slot connector 130 are formed from a high nickel alloy suitable for operation at high temperatures.

[0052] The anode 20 is coupled to two graphite risers 21, 22 to enable an electrical connection to be made between a power supply (not shown) and the terminal anode 20. The graphite risers 21, 22 are coupled to the terminal anode 20 by means of graphite studs 23, 24. The graphite risers 21, 22 extend vertically above the terminal anode 20 through holes defined in the cover 100, such that an electrical connection can be made with an uppermost portion of the risers when the removable electrode module is located in engagement with an electrolysis chamber of an electrolysis apparatus. A gap between the risers 21, 22 and the associated holes defined through the cover 100 for the risers to pass through is sealed by means of braided graphite rope or other similar gland packing materials 25.

[0053] The removable electrode module 10 is designed to have three loading or support conditions.

[0054] In the first of these three conditions, the removable electrode module is seated on a lower surface of the cathode base element 30a. In this condition the weight of all of the bipolar elements, the anode, and the cover are transferred through the cathode base element 30a and the suspension rod 110 is not in tension.

[0055] In a second loading condition, the j-slot connector 130 is coupled to a lifting mechanism, and the entire weight of the module is supported through the suspension rod 110, which is coupled to the cathode base element 30a.

[0056] In a third loading condition, the removable electrode module 10 may be supported at multiple points on a lower surface 102 of the cover 100. In this condition the weight of the module is supported by the cover 100 and transferred through the suspension rod 110, which

is coupled to the cathode base element 30a.

[0057] Thus, the module may be free-standing on its cathode base element 30a, it may be suspended by the j-slot coupling 130 at an upper end of the suspension rod 110, or it may be suspended by the underside 102 of the cover 100.

[0058] The suspension rod 110 is coated or clad with an electrically-insulating material 115 throughout its length from the point of coupling to the cathode base element 30a to the point of sealing with the braided graphite rope 120 as the suspension rod 110 passes through the cover 100. This electrically-insulating material is an alumina coating 115, but may be any high temperature electrically-insulating material. For example, the coating 115 may be boron nitride. The coating may be applied by any known method, for example by dip coating or by spray coating.

[0059] The removable tray-assembly that forms part of the terminal cathode 30 and each of the seven bipolar electrodes 40, 41, 42, 43, 44, 45, 46 is illustrated in Figures 9, 10 and 11. The tray assembly 30b, 40b, 41b, 42b, 43b, 44b, 45b, 46b, is formed of two couplable portions 151, 152. When coupled together, the entire tray-assembly is substantially circular and has a diameter of about 542 mm at room temperature. The tray-assembly is metallic and so the diameter may increase to about 550 mm at the working temperature of the removable electrode module (usually between about 500°C and 1200°C when used in an electrolysis reaction in a molten salt) due to thermal expansion.

[0060] A base 153, 156 of each of the tray-assembly portions 151, 152 is formed from a mesh suitable for supporting a solid feedstock. Around the circumference of the assembled tray-assembly a circumferential lip is raised extending about 30 mm above the level of the mesh 153, 156. A plurality of downwardly extending feet 155 extend downwards from the circumferential lip 154 by a distance of about 10 mm below the level of the mesh 153, 156.

[0061] The entire tray-assembly may be seated on an upper surface of an associated electrode portion to form an electrode of the electrode module. For example, a tray assembly 30b may be seated on an upper surface of the terminal cathode base plate 30a to form a terminal cathode 30, or a tray assembly 40b, 41b, 42b, 43b, 44b, 45b, 46b may be seated on an upper surface of the lower portion of a bipolar electrode 40a, 41a, 42a, 43a, 44a, 45a, or 46a to form a bipolar electrode. Electrical contact is made between the tray-assembly and its associated electrode portion through the downwardly extending feet 155.

[0062] When a removable electrode module comprising the removable tray-assemblies 30b, 40b, 41b, 42b, 43b, 44b, 45b, 46b is located in an electrolysis chamber containing a molten salt, molten salt is able to flow into a gap created between the upper surface of an electrode portion on which the tray assembly is seated and the mesh base 153, 156. The molten salt is therefore able

to flow upwardly through the mesh base 153, 156 of the tray-assembly and, therefore, over any solid feedstock supported on the base 153, 156.

[0063] The tray-assembly is formed having a central hole for surrounding an electrically-insulating spacer element, for example the electrically-insulating spacer element 60 that supports the first bipolar electrode 40.

[0064] The tray-assembly is formed in two couplable portions, i.e. the first portion 151 and the second portion 152, each portion being substantially semicircular. The two portions 151, 152 are coupleable by means of a stud and slot arrangement. Studs 160 extend from a mating surface or mating edge 162 of the second portion and slots 161 for receiving the studs 160 are defined in a corresponding mating surface 163 of the first portion 151.

[0065] In use, each half or each portion 151, 152 of the tray-assembly may be separately removed from the removable electrode module 10 in order to load feedstock or unload reduced product.

[0066] The removable tray-assemblies form the uppermost portion of the terminal cathode and each of the bipolar electrodes. These portions of the respective electrodes become cathodic when the removable electrode module is used for electrolysis.

[0067] The removable tray-assemblies 30b, 40b, 41b, 42b, 43b, 44b, 45b, 46b are manufactured from 310-grade stainless steel. The removable tray-assemblies may be made from many other materials, and the choice of material may depend on the nature of the feedstock to be reduced. For example, it may be desirable to use a tray-assembly formed from a metal that will not contaminate the reduced product. For example, it may be desirable to form the cathode tray assembly from tantalum, or tantalum coated metal, where the removable electrode module is to be used for the reduction of a tantalum oxide to tantalum metal.

[0068] A removable electrode module according to the first specific embodiment described above may be of particular advantage when used for the reduction of a solid feedstock in a molten salt electrolyte. The removable tray-assemblies allow a solid feedstock to be conveniently loaded onto each separate removable tray-assembly portion 151, 152 and loaded into the removable electrode module by seating the loaded tray-assembly portions in an appropriate position in the electrode module.

[0069] At room temperature, the removable electrode module 10 has a total height from the lower surface of the cathode base plate 30a to the lower surface of the cover 100 of 1645 mm. The height from the lower surface of the cathode base plate 30a to the top of the j-slot connector 130 is 2097 mm. As stated above, the diameter of the electrodes 30, 40-46 is 550 mm. The maximum diameter of the cover 100 is 830 mm. Some of these dimensions will be subject to change as the temperature varies. In particular, the height values may be increased by 5 to 10 mm at the working temperature of the electrode module.

[0070] The removable electrode module 10 according

to the first embodiment of the invention described above may be advantageously used with any electrolysis apparatus having an electrolysis chamber suitable for receiving the module 10 in engagement. A schematic illustration of such an electrolysis apparatus 200 is provided by Figure 5.

[0071] The electrolysis apparatus 200 comprises a housing 210 containing an electrolysis chamber 220 defined within a graphite crucible 230, an upper rim 231 of the graphite crucible 230 defining an opening into the electrolysis chamber 220. An upper surface of the rim 231 is coated with a 15 mm thick section of a resilient graphite material for sealing the rim 231 against an underside of the cover 100 of the removable electrode module 10. The sealing material seated on the upper rim 231 is a braided graphite gland packing material that may be deformed and regain its shape.

[0072] The housing 210 furthermore contains furnace heating elements 240 for maintaining the temperature of the graphite crucible 230, a molten salt inlet 250 and a molten salt outlet 260 for allowing a flow of molten salt through the electrolysis chamber 220. A gas vent line 270 is provided towards an upper portion of the electrolysis chamber 220 to allow the escape of gases evolved during any electrolysis reaction taking place within the electrolysis chamber. A DC supply cathode bus bar 280 is coupled to the graphite crucible 230 and enables the entire graphite crucible 230 to directly couple the graphite crucible to a power supply.

[0073] The graphite crucible 230 is lined with an alumina liner 290. The alumina liner 290 provides an electrical insulation between side-walls of the graphite crucible 230 and any removable electrode module 10 engaged within the electrolysis chamber 220. Although made from alumina, the liner may be made from any suitable electrically insulating ceramic material that is substantially inert under the processing conditions within the electrolysis chamber 220.

[0074] An upper portion of the electrolysis apparatus comprises a gate-valve type closure 300 that enables external access to be provided to the electrolysis chamber 220. The gate-valve closure 300 comprises a gate 310 formed from a thermal barrier material, for example a ceramic material. An actuation device 320 allows the gate 310 to slide back-and-forth to open and close the gate valve 300, thereby allowing access to the electrolysis chamber 220 within the electrolysis apparatus 200.

[0075] Figure 6 illustrates a removable electrode module, according to the first embodiment described above in relation to Figures 1 to 4, engaged with an electrolysis apparatus of the type illustrated in Figure 5.

[0076] A lower internal surface of the graphite crucible 230 is raised forming a pedestal 232. When engaged with the electrolysis chamber 220, the removable electrode module 10 is seated on this raised pedestal 232 within the graphite crucible 230. Thus, the lower surface of the terminal cathode 30 of the removable electrode module is in physical and electrical contact with an inter-

nal surface of the graphite crucible 230.

[0077] The bipolar electrodes 40-46 and the anode 20 of the removable electrode module 10 are situated within a portion of the electrolysis chamber that is electrically-insulated from the side-wall of the crucible 230 by the ceramic liner 290. A lower surface 102 of the cover 100 of the removable electrode module 10 makes contact with the upper rim 231 of the graphite crucible 230. As the cover comes into contact with the rim 231 the flexible graphite sealing material seated on the upper rim deforms to enable a seal to be made. It is noted that the graphite sealing material could alternatively or additionally be located on the lower surface 102 of the cover 100.

[0078] In use, the temperature within the electrolysis chamber may vary considerably. Thus, the dimensions of some components of the removable electrode module, for example the suspension rod 110, may change by several millimetres. The resilient material seated on the upper rim of the graphite crucible 230 preferably has sufficient resilience and deformability to accommodate any such thermal distortion and maintain a viable seal with the underside 102 of the cover 100.

[0079] The anode risers 21, 22 of the removable electrode module extend upwardly through the cover 100. Electrical contact may be made with these risers by actuable DC anode bus bars 250, which may be actuated to contact the anode risers and thus provide an electrical connection between the anode and the power supply.

[0080] In use, the electrolysis chamber 220 is filled with a molten salt and a removable electrode module loaded with a reduceable feedstock is engaged with the electrolysis chamber. The anode bus bars are actuated to contact the anode risers 21, 22 and a potential is applied between the anode 20 (by way of the anode risers and the actuable anodic bus bars 250) and the terminal cathode 30 (by way of the graphite crucible 230 and the cathodic DC bus bar 280). The potential applied is sufficient to reduce the feedstock. The required potential may vary dependent upon the type of feedstock and the composition of the molten salt.

[0081] In many situations, in particular for the reduction of a solid feedstock in a molten salt electrolyte, it may be advantageous to be able to engage a removable electrode module with an electrolysis chamber of an electrolysis apparatus that is at or near to its working temperature. For many molten salt electrolytes this means that the electrolysis chamber contains a molten salt at a temperature of between 500°C and 1200°C. If a removable electrode module at room temperature was to be inserted into an electrolysis chamber containing a molten salt at a temperature of, for example, 1000°C, then the components of the removable electrode module would be likely to undergo severe and rapid thermal distortion. In particular, the ceramic components of the removable electrode module may undergo severe thermal shock and, thus, fail. As a complication, if the removable electrode module as described above in relation to the first embodiment of a removable electrode module were pre-heated to a tem-

perature of 1000°C in air, the graphite components of the removable electrode module would combust.

[0082] It may be particularly desirable to be able to remove a removable electrode module from an electrolysis chamber of an electrolysis apparatus immediately after electrolysis has taken place and without waiting for the electrolysis chamber to cool. Care would need to be taken to ensure that oxygen containing atmosphere such as air did not come into contact with the removable electrode module at high temperatures. Failure to safeguard against this could result in the graphite components of the electrode module combusting, reduced metallic product located within the removable electrode module combusting or oxidising and severe thermal deformations and failures occurring due to rapid cooling of the module.

[0083] In order to allow the removable electrode module to be engaged with the electrolysis chamber of the electrolysis apparatus at temperature near to working temperature, and in order to allow the removable electrode module to be disengaged from the electrolysis chamber at a temperature close to working temperature, it is desirable that the removable electrode module can be withdrawn into a transfer module before being transferred or transported to the electrolysis apparatus. A transfer module may include heating and/or cooling elements. A transfer module may simply be a shroud within which an inert atmosphere can be maintained that insulates a preheated electrode module prior to loading into the electrolysis chamber or insulates an electrode module recently disengaged from an electrolysis chamber prior to being transported to a separate location for a controlled cooling.

[0084] Figure 7 illustrates a removable electrode module as described above in relation to Figures 1 to 4 located within an embodiment of a removable transfer module 400. The removable transfer module 400 comprises a housing 410 formed from 310-grade stainless steel and lined with a refractory lining. The refractory lining may be a ceramic brick lining or any other suitable material, such as fibreboard, that thermally insulates the interior of the transfer module. The interior of the transfer module comprises a transfer cavity 420 within which a removable electrode module 10 may be located.

[0085] A transfer module may comprise a means for coupling to the j-slot connector at the top of the removable transfer module and means for withdrawing the removable transfer module into the transfer chamber 420. For example, the transfer module 400 may comprise a winch for lifting the removable electrode module.

[0086] An upper portion of the transfer module 400 comprises means for lifting the transfer module such as a hook or hooks 430. Such lifting means enable the entire transfer module to be lifted and moved to and from an electrolysis apparatus 200.

[0087] A lower portion of the transfer module 400 is closed by a gate-valve 440. This gate-valve comprises a thermally resistant gate 450 that is actuable to open and close an opening into the transfer module chamber

420. The transfer module, including the gate-valve, may conveniently be seated atop the gate-valve of an electrolysis apparatus 200, as described above in relation to Figure 5. By opening the gate-valves associated with both the transfer module 440 and the electrolysis apparatus 200, access can be provided to the opening of the electrolysis chamber 220. The removable electrode module 10 can then be lowered from the transfer chamber 420, through the openings of both the gate-valve associated with the transfer module and the gate-valve associated with the electrolysis apparatus, to enable the electrode module to be located within the electrolysis chamber 220. The respective gate-valves can then be closed, as illustrated in Figure 8, and the transfer module 400 may then be removed.

[0088] The first embodiment of a removable transfer module, as described above and illustrated in Figures 1 to 4, comprised eight effective working electrodes on which solid feedstock could be reduced (i.e. the upper portion of the terminal cathode 30 and the upper portions of each of the bipolar electrodes 40-46). For some reactions it may be desired to reduce a lower volume of a solid feedstock. For such purposes, it may be desirable that a removable electrode module has a lower area of cathodic-electrode surface. A second embodiment of a removable electrode module according to one or more aspects of the invention is illustrated by Figure 12.

[0089] The overall dimensions of the removable electrode module as illustrated in Figure 12 are the same as the removable electrode module illustrated in Figures 1 to 4 and, thus, this second embodiment of a removable electrode module may be used in conjunction with the same electrolysis apparatus as the first embodiment. However, the removable electrode module of the second embodiment of the invention 1200 comprises a terminal cathode 1230 and a terminal anode 1220, with only a single bipolar electrode 1240 disposed between the terminal anode 1220 and the terminal cathode 1230. The terminal anode terminal cathode and the bipolar electrode are identical in construction to the equivalent structures described above in relation to the first embodiment of the invention. As there are fewer bipolar electrodes disposed between the terminal anode 1220 and the terminal cathode 1230, the graphite electrode risers 1221 and 1222 are substantially longer than those described above in relation to the first aspect of the invention. If needed, several sections of graphite risers may be joined by internal threaded studs 1226. The cover 1201 is supported directly above the upper surface of the anode 1220 by means of a plurality of electrically insulating ceramic spacers 1268.

[0090] Apart from these specific adaptations required to ensure the external dimensions of this removable electrode module are the same as the dimensions of the module of the first embodiment of the invention, all other elements of the removable electrode module according to the second embodiment of the invention are the same as described above.

[0091] According to certain aspects of the invention, it is not essential that a removable electrode module comprises a bipolar electrode. Figure 13 illustrates a third specific embodiment of a removable electrode module according to one or more aspects of the invention. This third embodiment comprises a terminal anode 1320 and a terminal cathode 1330, but does not comprise a bipolar electrode. The terminal cathode 1330 and the terminal anode 1320 are constructed in the same way as the terminal anode 20 and the terminal cathode 30 described above in relation to the first embodiment of the invention. The external dimensions of the removable electrode module 1300 of the third embodiment are the same as the dimensions of the first and second embodiments of a removable electrode module. All other details of the third embodiment of a removable electrode module as illustrated in Figure 13 are as described above in relation to the first embodiment or the second embodiment of the removable electrode module.

[0092] In the embodiments described above a suspension rod 110 is coupled to a j-slot connector 130 by clamping an end of the rod 110 to the connector 130 by means of washers and bolts 111. Any tolerance needed to form a seal between an underside of the cover 100 and a rim 231 of a crucible 230 forming an opening into an electrolysis chamber 220 is achieved by the use of a resilient sealing material on the rim. Figure 14 illustrates an alternative coupling that may be used in an embodiment of a removable electrode module. For ease of reference, components that are identical to those present in the first embodiment described above have been given the same reference numerals.

[0093] In the alternative embodiment illustrated in Figure 14 a suspension rod 110 of the electrode module is coupled to a j-slot connector 130 by means of a flange 1410 which transfers load through a set of Belleville springs 1400 and on to the j-slot connector. The flange 1410 is secured against the spring 1400 by means of nuts 1420.

[0094] When the module is lifted, the weight of the module is transferred through the suspension rod 110 and compresses the spring 1400. The spring urges upwards against a lower surface of the flange 1410. The spring 1400 may be any suitable spring means. For example, the spring may comprise a helical spring.

[0095] Coupling an electrode module to a lifting means such as a j-slot connector with a resilient spring disposed between may provide advantages in use. For example, as the electrode module is lowered into an electrolysis chamber as described above, contact is made between a rim surrounding the opening of the chamber and a lower surface 102 of the cover 100 in order to form a seal. In the embodiments described above, the base plate 30a of the module must be seated in physical contact with the internal wall of the crucible in order to provide a cathodic connection. The use of a resilient means such as a Belleville spring 1400 disposed between the lifting means and the suspension rod may allow additional trav-

el of the electrode module after a seal has been formed by the cover 100. Furthermore, such a resilient means may advantageously accommodate dimensional changes in the suspension rod caused by thermal fluctuations.

[0096] An embodiment of a removable electrode module that includes a resilient means disposed between a suspension rod or rods supporting the electrodes and a lifting means may be employed as an alternative to using a resilient sealing material surrounding the opening of an electrolysis chamber or in addition to it.

[0097] The following description of a method for electrolytically reducing a solid feedstock according to a specific embodiment of the invention uses a removable electrode module 10 as described above. The processing method can be broken down into a number of steps.

[0098] Step 1 - The removable electrode module 10 comprises eight layers of cathodic electrodes (i.e. one cathode and seven bipolar electrodes that act as both anode and cathode) and an anode. An upper surface of each of the working electrodes comprises a removable tray-assembly itself formed of two coupleable parts 151, 152. As a first step in the process a feedstock consisting of a plurality of solid oxide preforms is loaded onto a surface of each of the removable tray-assembly elements 151, 152.

[0099] Step 2 - The loaded tray-assemblies are transferred to a removable electrode module located at an electrode module loading station. The loaded tray-assemblies are seated on the removable electrode module 10 and each pair of tray-assembly portions 151, 152 forms a cathodic portion of a cathodic electrode (for example electrodes referenced 30, 40, 41, 42, 43, 44, 45, 46).

[0100] Step 3 - The loaded removable electrode module 10 is raised up into a transfer chamber 420 of a transfer module 400. In order to raise the removable electrode module 10 into the transfer chamber 420, the transfer module 400 is positioned vertically above the working station. A gate valve 440 of the transfer module 400 is actuated causing a gate 450 to slide open and allow access to the transfer chamber 420. A bayonet coupling (not shown) is lowered by a winch 465 located on the transfer module 400. The bayonet fitting couples to a j-slot connector 130 on the removable electrode module 10, thereby allowing the removable electrode module 10 to be raised into the transfer module chamber 420.

[0101] Step 4 - The entire transfer module 400 containing the removable electrode module 10 may be lifted and moved by means of a plurality of hooks 430. At Step 4 the entire transfer module, containing the electrode module, is moved to a position vertically above a heating station. This is illustrated in Figure 16. The transfer module 400 is coupled to the heating station 500 with the removable electrode module 10 positioned directly above a heating chamber 510. The heating station comprises a housing 501 containing a heating chamber 510 surrounded by a plurality of heating elements 520.

[0102] Step 6 - The electrode module is lowered, by

means of the winch 465 until a lower surface 102 of the cover 100 is seated on a rim 502 of the heating chamber 510. The bayonet coupling is removed from the j-slot connector 130 and the gate 450 is closed. The removable electrode module 10 is now engaged with the heating station and the entire weight of the module is supported through the lower surface of the cover 100. This arrangement is illustrated in Figure 17.

[0103] Step 7 - The electrode module 10 is heated within the heating chamber 510 of the heating station 500 to a predetermined temperature. For electrolysis in a molten salt this predetermined temperature is likely to be somewhere between 500°C and 1200°C. For example, the temperature may be raised to 700°C or 800°C. The rate of heating the module is controlled so that the ceramic components of the electrode module do not undergo thermal shock. Thus the heating may occur at a rate of between 1 °C per minute to 10 or 20°C per minute. For example, heating may occur at a rate of about 5°C per minute. The heating occurs under an inert atmosphere, for example an argon atmosphere or a nitrogen atmosphere.

[0104] Step 8 - Once the electrode module has been heated to the predetermined temperature the electrode module 10 is once more raised into the transfer chamber 420 of the transfer module 400.

[0105] Step 9 - The gate valve is closed, thereby sealing the transfer chamber 420. An inert atmosphere, for example an atmosphere argon or nitrogen, is maintained within the transfer module.

[0106] Because the walls of the transfer module are insulated, the rate of heat loss is low. Thus, once the electrode module 10 has been heated to a predetermined temperature and is sealed within the transfer module 400 the temperature of the module 10 reduces slowly.

[0107] Step 10 - The transfer module 400 containing the electrode module 10 at a predetermined temperature is moved to a position directly above an electrolysis chamber. Access to the electrolysis chamber 220 is restricted by the presence of a gate valve 300 seated above the electrolysis chamber. This is illustrated in Figure 7.

[0108] Step 11 - The transfer module 400 is coupled to an electrolysis apparatus 200 containing the electrolysis chamber 220. The gate valve 440 associated with the transfer module is aligned with the gate valve 300 associated with the electrolysis apparatus such that, when both gate valves are opened the electrode module 10 is able to gain access to the electrolysis chamber 220.

[0109] Step 12 - The gate valve 440 on the transfer module is opened.

[0110] Step 13 - The gate valve on the electrolysis apparatus 200 is opened.

[0111] Step 14 - The electrode module 10 is lowered into engagement with the electrolysis chamber. The electrolysis chamber contains a molten salt at, or close to, its desired working temperature. The preheated electrode module 10 is also at, or near, to the working temperature. This working temperature may, for example, be about

800°C. The electrode module is seated within the electrolysis chamber 220 such that a lower surface of the terminal cathode 30 of the electrolysis module 10 makes physical contact with the graphite crucible 230 defining the electrolysis chamber 220. This has been described above and illustrated in Figure 8.

[0112] Step 15 - Anode bus bars 250 are moved into contact with anode risers 21, 22, of the removable electrode module 10.

[0113] A potential is applied between the anode 20 and the cathode 30 of the electrode module 10. This potential is sufficient to reduce the feedstock that is in contact with the cathode and cathodic surfaces of each of the bipolar electrodes. The potential required to reduce the feedstock will depend on the nature of the system. Thus, for reduction of the titanium oxide feedstock in a calcium chloride based molten salt, the potential voltage at each cathodic surface of the electrode module 10 may be between 2 and 3 Volts.

[0114] The potential is applied for a sufficient period of time to reduce the solid feedstock.

[0115] After electrolysis to reduce the feedstock there may be a number of further processing steps in order to recover a reduced product from the electrode module 10. Thus, the following further steps are used in a specific embodiment of the invention in order to recover a reduced feedstock after electrolysis.

[0116] Step 17 - The electrolysis cell current is switched off and, thus, the voltage at the surface of each electrode is removed.

[0117] Step 18 - The anode bus bars are withdrawn from contact with the graphite anode risers 21, 22.

[0118] Step 19 - The electrode module is raised out of the molten salt and into the transfer module 400. The electrode module is raised slowly in order to allow time for molten salt to drip free from the electrode module 10.

[0119] Step 20 - The gate valve 300 on the electrolysis apparatus 200 is closed.

[0120] Step 21 - the gate valve 440 on the transfer module is closed.

[0121] Step 22 - The transfer module containing the electrode module 10 is then disengaged from the electrolysis apparatus 200 and moved to a position immediately above a cooling station 600. This is illustrated in Figure 18.

[0122] Step 23 - The transfer module 400 is coupled to the cooling station 600.

[0123] Step 24 - The gate valve 440 is opened to allow access for the removable electrode module 10 into a cooling chamber 610.

[0124] Step 25 - The electrode module 10 is lowered into the cooling chamber until a lower surface 102 of the cover 100 rests on an upper rim of the cooling chamber 610. The cover 100 effectively forms a seal to the cooling chamber 610. This is illustrated in Figure 19.

[0125] Step 26 - The cooling chamber comprises walls 620 of a high thermal conductivity material, for example a metallic material such as a stainless steel. The walls

contain a water jacket 625, through which a constant supply of cold water is provided by means of an inlet 630 and outlet 631. The water jacket allows heat to be removed efficiently from the thermal module 10. The rate of cooling may be controlled by varying the rate at which water is passed through the cooling jacket 625.

[0126] Step 27 - Once the electrode module 10 has been cooled to a predetermined temperature it is raised into the transfer module 400.

[0127] Step 28 - The cooled electrode module 10 within the transfer module 400 is moved to a position directly above a wash station.

[0128] Step 29 - The cooled electrode module is lowered into the wash station and seated on its cathode base plate 30a.

[0129] Step 30 - Jets of water 700 are emitted from nozzles 705 connected to piping 710. The jets of water 700 are directed at the electrode module 10 and in particular directed to the tray-assemblies containing the reduced feedstock. The jets of water are preferably directed such that they wash any remaining salt encrusting the electrode module and the reduced feedstock away.

[0130] Step 31 - After washing, the electrode module 10 is raised into the transfer module 400.

[0131] Step 32 - the washed electrode module and the transfer module are moved to a position above an unloading station.

[0132] Step 33 - The washed electrode module is lowered into position at the unloading station.

[0133] Step 34 - The electrode tray-assemblies containing reduced products are removed from the electrode module 10.

[0134] Step 35 - The reduced product is removed from the tray-assemblies and packaged for further processing.

[0135] The specific embodiment of a method described above may not be used in all cases. For example different steps may be used or some steps may be omitted.

[0136] It is preferred that the reduction of the solid feedstock proceeds by an electro-deoxidation reaction such as the FFC process.

Claims

1. A method of electrolytically reducing a solid feedstock, comprising the steps of; positioning an electrode module (10) comprising at least one electrode in a first position for loading of feedstock, loading solid feedstock onto the electrode module (10), moving the electrode module (10) from the first position within a transfer module (400), lowering the electrode module (10) into a heating station (510), heating the electrode module (10) to a predetermined temperature, and raising the electrode module (10) back into the transfer module

(400) to be transferred to an electrolysis chamber (220) under controlled conditions, engaging the electrode module (10) with the electrolysis chamber (220) such that the feedstock is in contact with a molten salt within the electrolysis chamber (220), and applying a voltage to the electrode module (10) such that the solid feedstock is reduced.

2. A method according to claim 1 in which the electrode module (10) is transferred under an inert atmosphere within the transfer module (400).
3. A method according to any of claim 1 or 2 in which the electrode module (10) is sealed within the transfer chamber (400) by closing a closure, preferably in which the closure is a gate-valve (440).
4. A method according to any preceding claim in which an opening of the electrolysis chamber (220) is closed by an openable closure, preferably in which the openable closure is a gate-valve (300) openable to allow the electrode module (10) to pass into the electrolysis chamber (220).
5. A method according to any preceding claim further comprising the step of removing the electrode module (10) from the electrolysis chamber (220) after electrolysis to recover the reduced feedstock.
6. A method according to claim 5 in which the electrode module (10) is lifted from the electrolysis chamber (220) into the transfer module (400).
7. A method according to claim 6 in which the electrode module (10) is cooled under an inert atmosphere within the transfer module (400) after being removed from the electrolysis chamber (220).
8. A method according to claim 6 in which the electrode module (10) is transferred to a cooling station (600) to be cooled to a predetermined temperature, preferably in which the electrode module (10) is transferred to the cooling station (600) within a transfer module (400), lowered into the cooling station (600), cooled to the predetermined temperature, and raised back into the transfer module (400) to be transferred away from the cooling station (600).
9. A method according to any of claims 5 to 8 in which the electrode module (10) is further transferred to a washing station for washing salt from the reduced feedstock and/or in which the electrode module (10) is further transferred to an unloading station for unloading the reduced feedstock.
10. A method according to any preceding claim in which solid feedstock is loaded onto removable trays (30b)

separate from the electrode module (10), the removable trays (30b) then being coupled to the electrode module (10) to load feedstock onto the electrode module (10).

11. A method according to any preceding claim in which the feedstock is loaded such that it contacts a cathode structure (30) of the electrode module (10) and/or in which the electrode module (10) comprises one or more bipolar electrodes (40, 41, 42, 43, 44, 45, 46), and feedstock is loaded in contact with a cathodic surface of each bipolar electrode.
12. A method according to any preceding claim that proceeds by the electro-deoxidation of the solid feedstock.

Patentansprüche

1. Verfahren zur elektrolytischen Reduktion eines Ausgangsmaterials, das die folgenden Schritte umfasst:

Positionieren eines Elektrodenmoduls (10), das wenigstens eine Elektrode aufweist, in einer ersten Position zum Laden von Ausgangsmaterial; Laden von festem Ausgangsmaterial auf das Elektrodenmodul (10),
Bewegen des Elektrodenmoduls (10) von der ersten Position in einem Transportmodul (400),
Senken des Elektrodenmoduls (10) in eine Erhitzungsstation (510), Erhitzen des Elektrodenmoduls (10) auf eine vorbestimmte Temperatur und Zurückheben des Elektrodenmoduls (10) in das Transportmodul (400) zum Transport unter kontrollierten Bedingungen zu einer Elektrolysekammer (220),
Ineingriffbringen des Elektrodenmoduls (10) mit der Elektrolysekammer (220), so dass das Ausgangsmaterial mit einer Salzschnmelze in der Elektrolysekammer (220) in Kontakt ist, und
Anlegen einer Spannung an das Elektrodenmodul (10), so dass das feste Ausgangsmaterial reduziert wird.

2. Verfahren nach Anspruch 1, bei dem das Elektrodenmodul (10) unter einer inerten Atmosphäre im Transportmodul (400) transportiert wird.
3. Verfahren nach einem der Ansprüche 1 und 2, bei dem das Elektrodenmodul (10) durch Schließen eines Verschlusses dicht in der Transportkammer (400) eingeschlossen wird, vorzugsweise bei dem der Verschluss ein Absperrschieber (440) ist.
4. Verfahren nach einem der vorhergehenden Ansprüche, bei dem eine Öffnung der Elektrolysekammer (220) durch einen Verschluss, der geöffnet werden

kann, geschlossen wird, bei dem vorzugsweise der Verschluss, der geöffnet werden kann, ein Absperrschieber (300) ist, der geöffnet werden kann, um das Elektrodenmodul (10) sich in die Elektrolysekammer (220) bewegen zu lassen.

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5. Verfahren nach einem der vorhergehenden Ansprüche, das ferner den Schritt des Entferns des Elektrodenmoduls (10) aus der Elektrolysekammer (220) nach der Elektrolyse zur Entnahme des reduzierten Ausgangsmaterials aufweist.

6. Verfahren nach Anspruch 5, bei dem das Elektrodenmodul (10) aus der Elektrolysekammer (220) in das Transportmodul (400) gehoben wird.

7. Verfahren nach Anspruch 6, bei dem das Elektrodenmodul (10) unter einer inerten Atmosphäre in dem Transportmodul (400) gekühlt wird, nachdem es aus der Elektrolysekammer (220) entfernt worden ist.

8. Verfahren nach Anspruch 6, bei dem das Elektrodenmodul (10) zum Abkühlen auf eine vorbestimmte Temperatur zu einer Kühlstation (600) transportiert wird, bei dem vorzugsweise das Elektrodenmodul (10) in einem Transportmodul (400) zu der Kühlstation (600) transportiert wird, in die Kühlstation (600) gesenkt wird, auf die vorbestimmte Temperatur abgekühlt wird und wieder in das Transportmodul (400) gehoben wird, um von der Kühlstation (600) abtransportiert zu werden.

9. Verfahren nach einem der Ansprüche 5 bis 8, bei dem das Elektrodenmodul (10) ferner zu einer Waschstation zum Abwaschen von Salz von dem festen Ausgangsmaterial transportiert wird und/oder bei dem das Elektrodenmodul (10) ferner zu einer Entladestation zum Entladen des reduzierten Ausgangsmaterials transportiert wird.

10. Verfahren nach einem der vorhergehenden Ansprüche, bei dem das feste Ausgangsmaterial auf abnehmbare Schalen (30b) geladen wird, die vom Elektrodenmodul (10) separat sind, wobei die abnehmbaren Schalen (30b) dann zum Laden von Ausgangsmaterial auf das Elektrodenmodul (10) mit dem Elektrodenmodul (10) gekoppelt werden.

11. Verfahren nach einem der vorhergehenden Ansprüche, bei dem das Ausgangsmaterial so geladen wird, dass es mit einer Kathodenstruktur (30) des Elektrodenmoduls (10) in Kontakt ist, und/oder bei dem das Elektrodenmodul (10) eine oder mehr bipolare Elektroden (40, 41, 42, 43, 44, 45, 46) umfasst und Ausgangsmaterial in Kontakt mit einer kathodischen Oberfläche jeder bipolaren Elektrode geladen wird.

12. Verfahren nach einem der vorhergehenden Ansprüche, das nach der Elektrodesoxidation des festen Ausgangsmaterials verläuft.

Revendications

1. Procédé de réduction électrolytique d'une charge d'alimentation solide, comprenant les étapes suivantes :

le positionnement d'un module d'électrode (10) comprenant au moins une électrode à une première position de chargement d'une charge d'alimentation,
le chargement de la charge d'alimentation solide sur le module d'électrode (10),
le déplacement du module d'électrode (10) à partir de la première position au sein d'un module de transfert (400),
l'abaissement du module d'électrode (10) à l'intérieur d'une station de chauffage (510), la chauffe du module d'électrode (10) jusqu'à une température prédéterminée, et le relèvement du module d'électrode (10) à l'intérieur du module de transfert (400) en vue de son transfert vers une chambre d'électrolyse (220) dans des conditions contrôlées,
l'enclenchement du module d'électrode (10) avec la chambre d'électrolyse (220) de telle sorte que la charge d'alimentation vienne en contact avec un sel fondu au sein de la chambre d'électrolyse (220), et
l'application d'une tension au module d'électrode (10) de manière à réduire la charge d'alimentation solide.

2. Procédé selon la revendication 1 dans lequel le module d'électrode (10) est transféré sous une atmosphère inerte au sein du module de transfert (400).
3. Procédé selon l'une quelconque des revendications 1 ou 2 dans lequel le module d'électrode (10) est scellé au sein de la chambre de transfert (400) en fermant une fermeture, de préférence dans lequel la fermeture est un robinet vanne (440).
4. Procédé selon l'une quelconque des revendications précédentes dans lequel une ouverture de la chambre d'électrolyse (220) est fermée par une fermeture ouvrable, de préférence dans lequel la fermeture ouvrable est un robinet vanne (300) pouvant être ouvert pour permettre le passage du module d'électrode (10) à l'intérieur de la chambre d'électrolyse (220).
5. Procédé selon l'une quelconque des revendications précédentes comprenant en outre l'étape de retrait

du module d'électrode (10) de la chambre d'électrolyse (220) après l'électrolyse pour récupérer la charge d'alimentation réduite.

- 5 6. Procédé selon la revendication 5 dans lequel le module d'électrode (10) est relevé depuis la chambre d'électrolyse (220) à l'intérieur du module de transfert (400).
10 7. Procédé selon la revendication 6 dans lequel le module d'électrode (10) est refroidi sous une atmosphère inerte au sein du module de transfert (400) après son retrait de la chambre d'électrolyse (220).
15 8. Procédé selon la revendication 6 dans lequel le module d'électrode (10) est transféré à une station de refroidissement (600) en vue de son refroidissement à une température prédéterminée, de préférence dans lequel le module d'électrode (10) est transféré à la station de refroidissement (600) au sein d'un module de transfert (400), abaissé dans la station de refroidissement (600), refroidi à la température prédéterminée, et relevé à l'intérieur du module de transfert (400) pour le transférer hors de la station de refroidissement (600).
20 9. Procédé selon l'une quelconque des revendications 5 à 8 dans lequel le module d'électrode (10) est transféré en outre vers une station de lavage pour laver le sel de la charge d'alimentation réduite et/ou dans lequel le module d'électrode (10) est transféré en outre vers une station de déchargement pour décharger la charge d'alimentation réduite.
25 10. Procédé selon l'une quelconque des revendications précédentes dans lequel la charge d'alimentation solide est chargée sur des plateaux amovibles (30b) séparés du module d'électrode (10), les plateaux amovibles (30b) étant ensuite accouplés au module d'électrode (10) pour charger la charge d'alimentation sur le module d'électrode (10).
30 11. Procédé selon l'une quelconque des revendications précédentes dans lequel la charge d'alimentation est chargée de manière à faire contact avec une structure de cathode (30) du module d'électrode (10) et/ou dans lequel le module d'électrode (10) comprend une ou plusieurs électrodes bipolaires (40, 41, 42, 43, 44, 45, 46), et la charge d'alimentation est chargée en contact avec une surface cathodique de chaque électrode bipolaire.
35 12. Procédé selon l'une quelconque des revendications précédentes qui se déroule par l'électro-désoxydation de la charge d'alimentation solide.
40
45
50
55

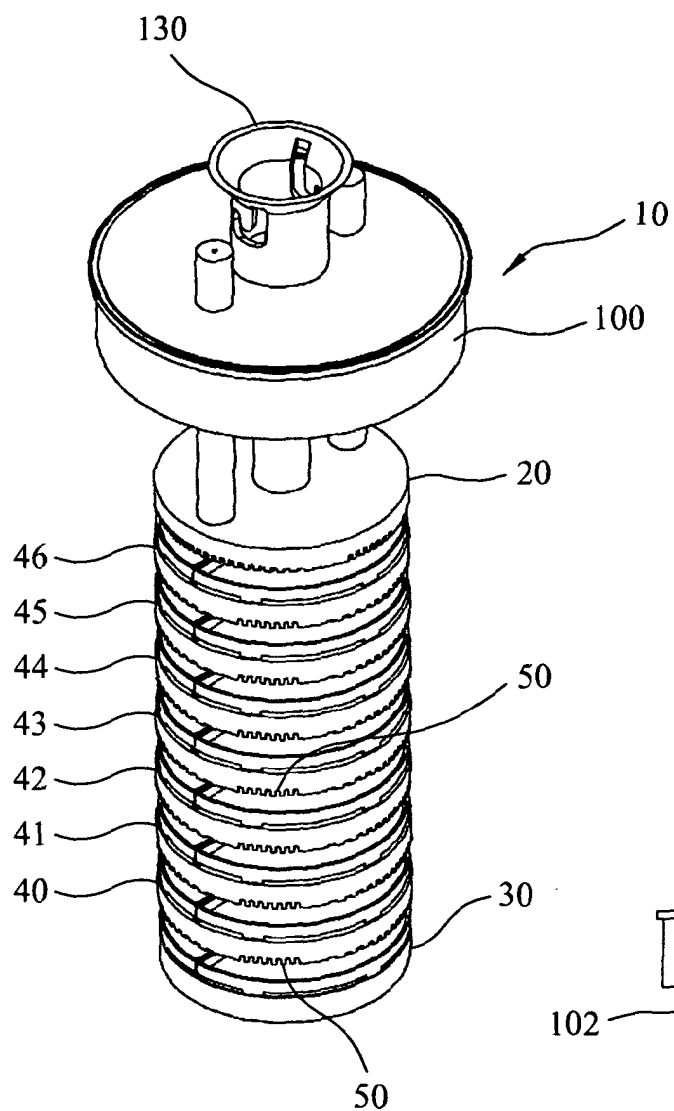


FIGURE 1

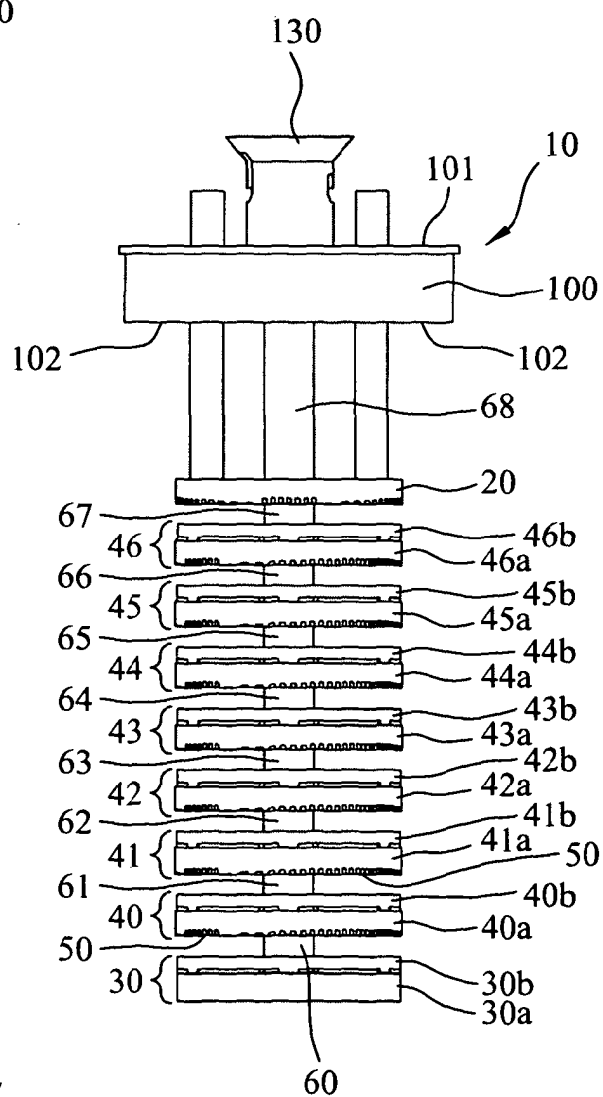


FIGURE 2

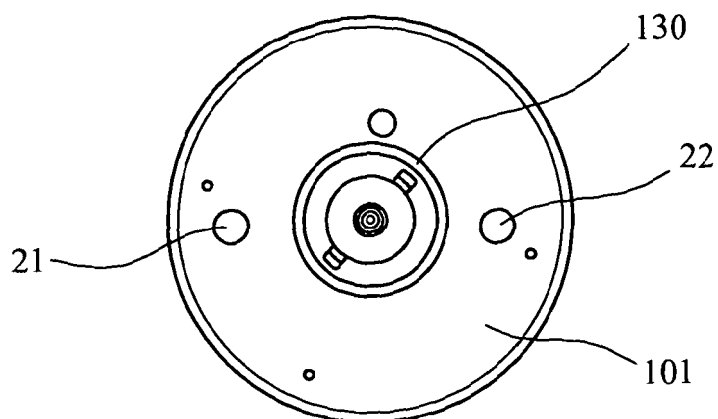


FIGURE 3

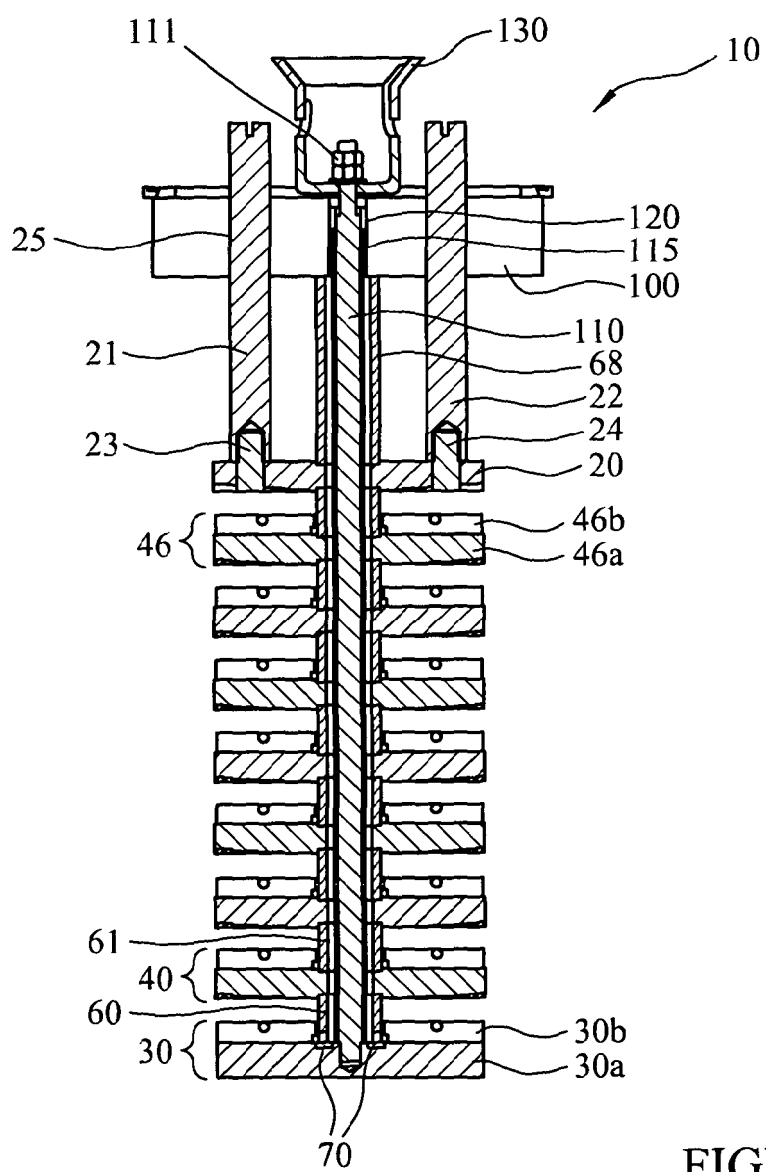


FIGURE 4

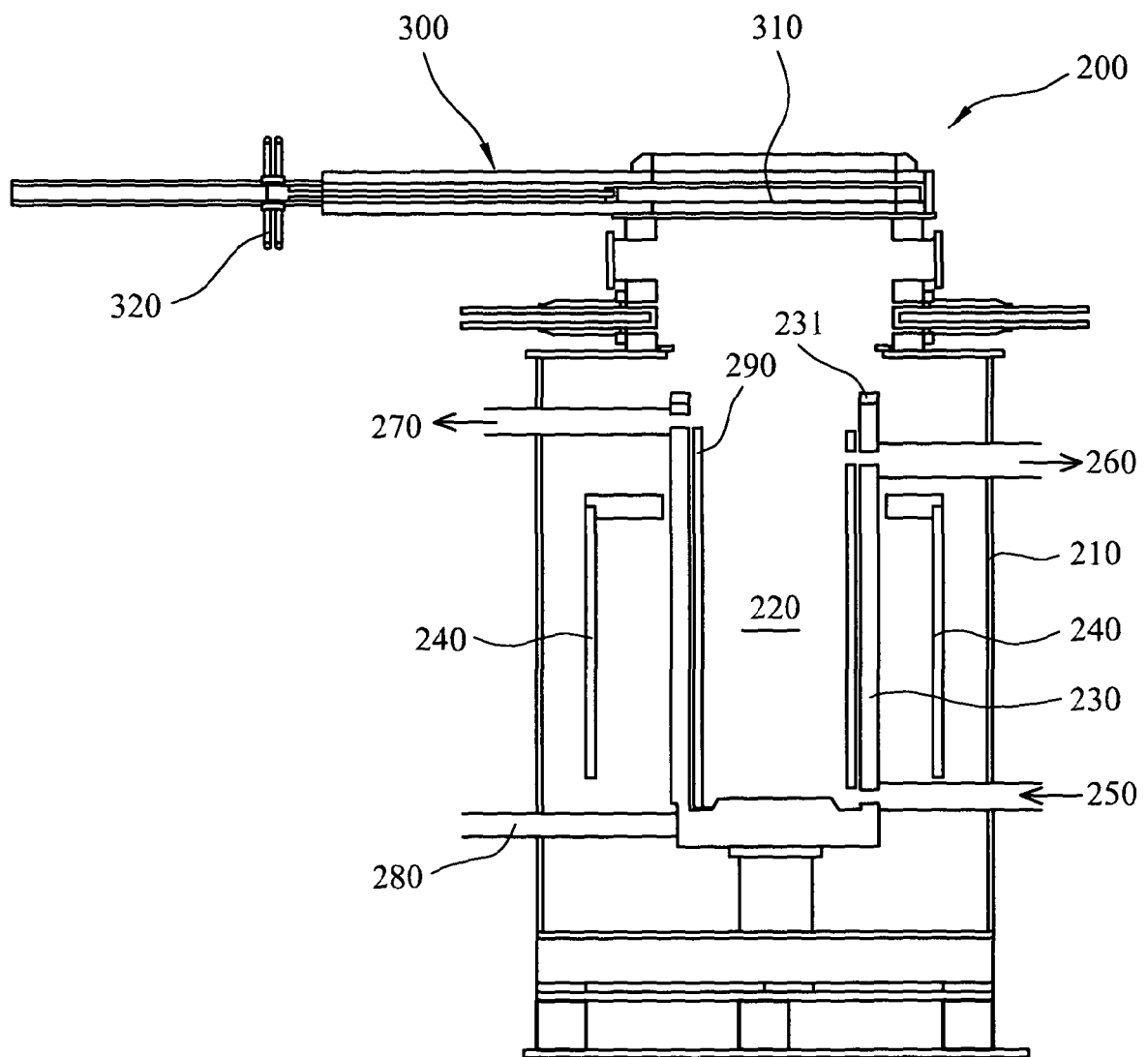


FIGURE 5

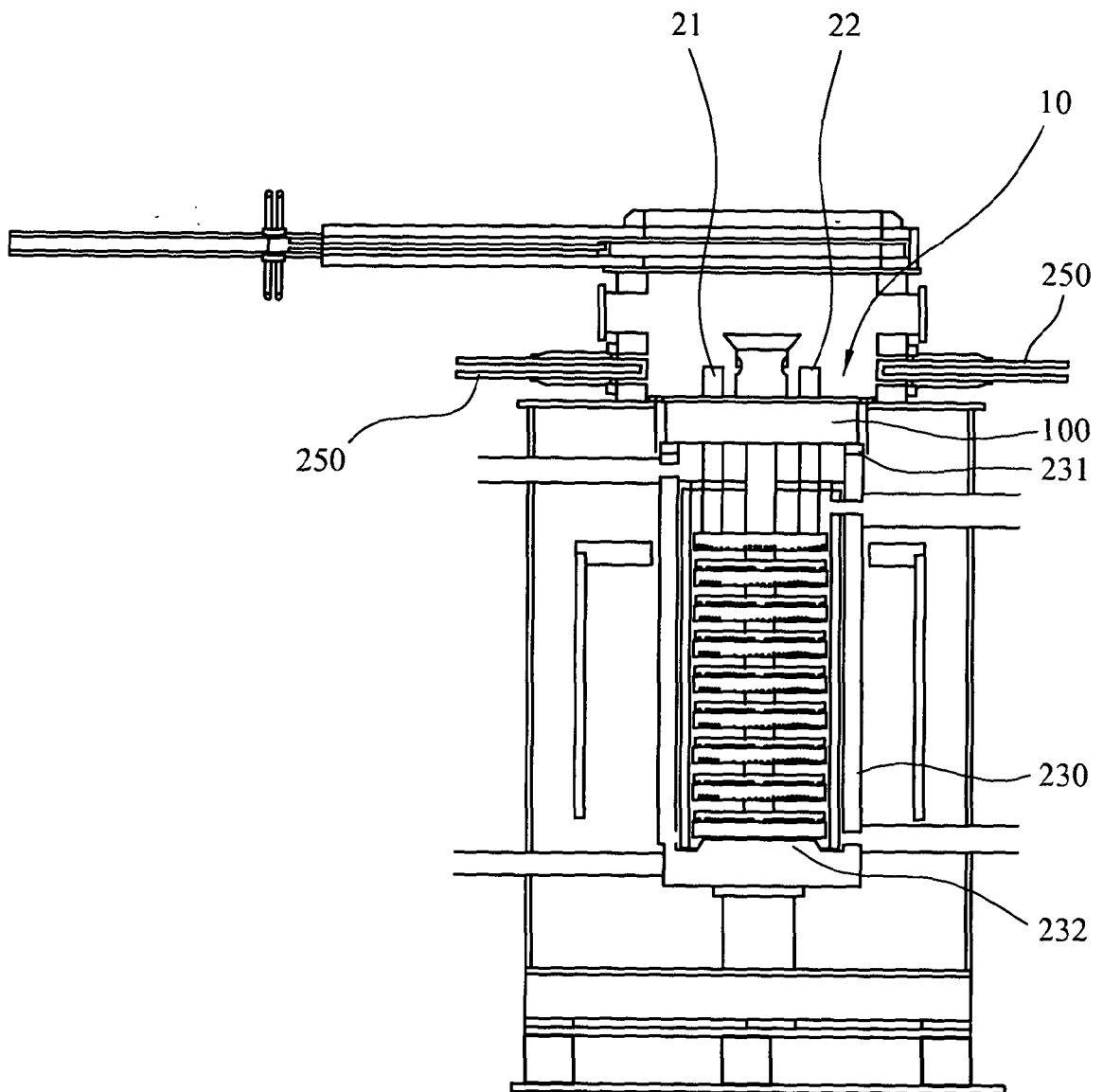
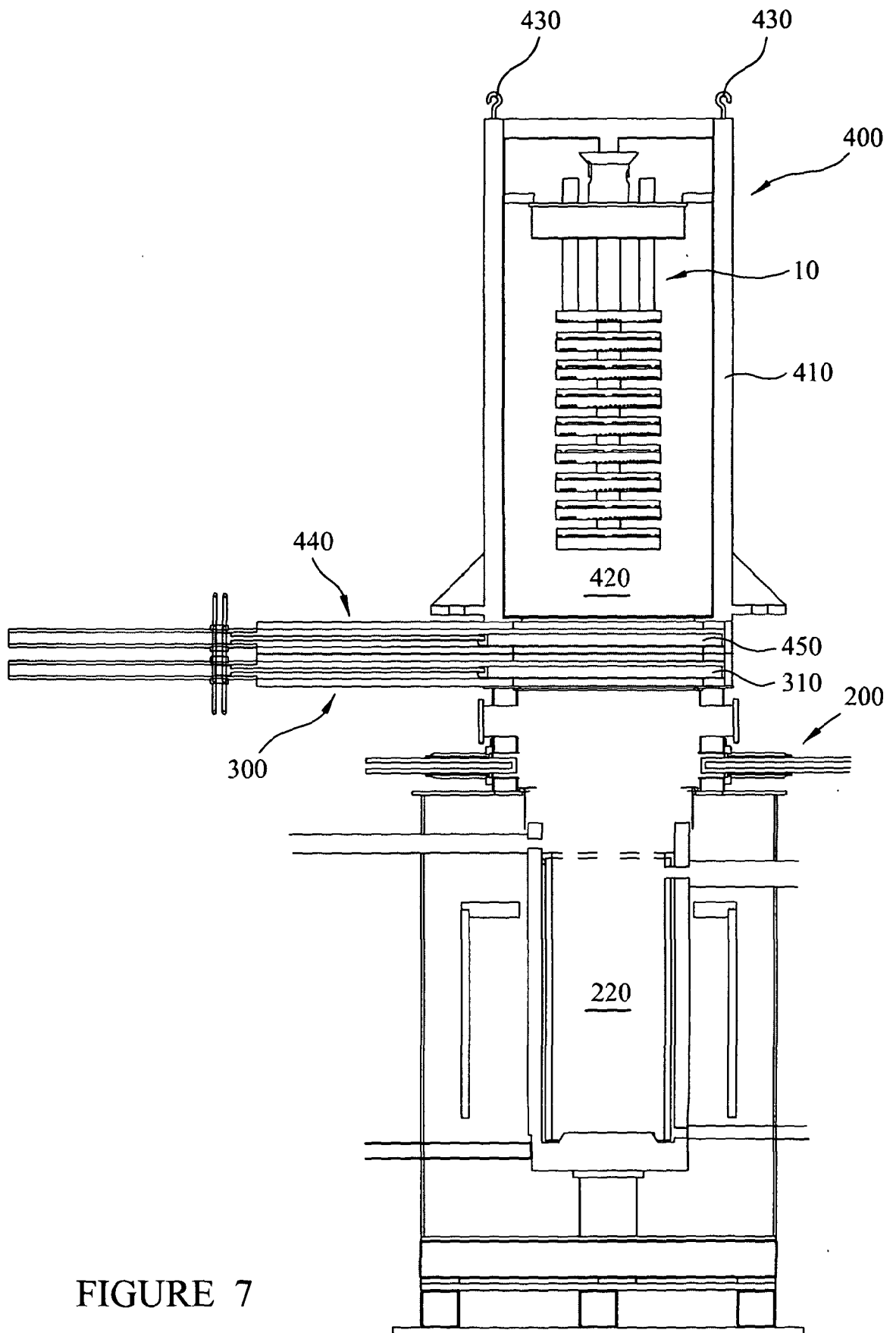


FIGURE 6



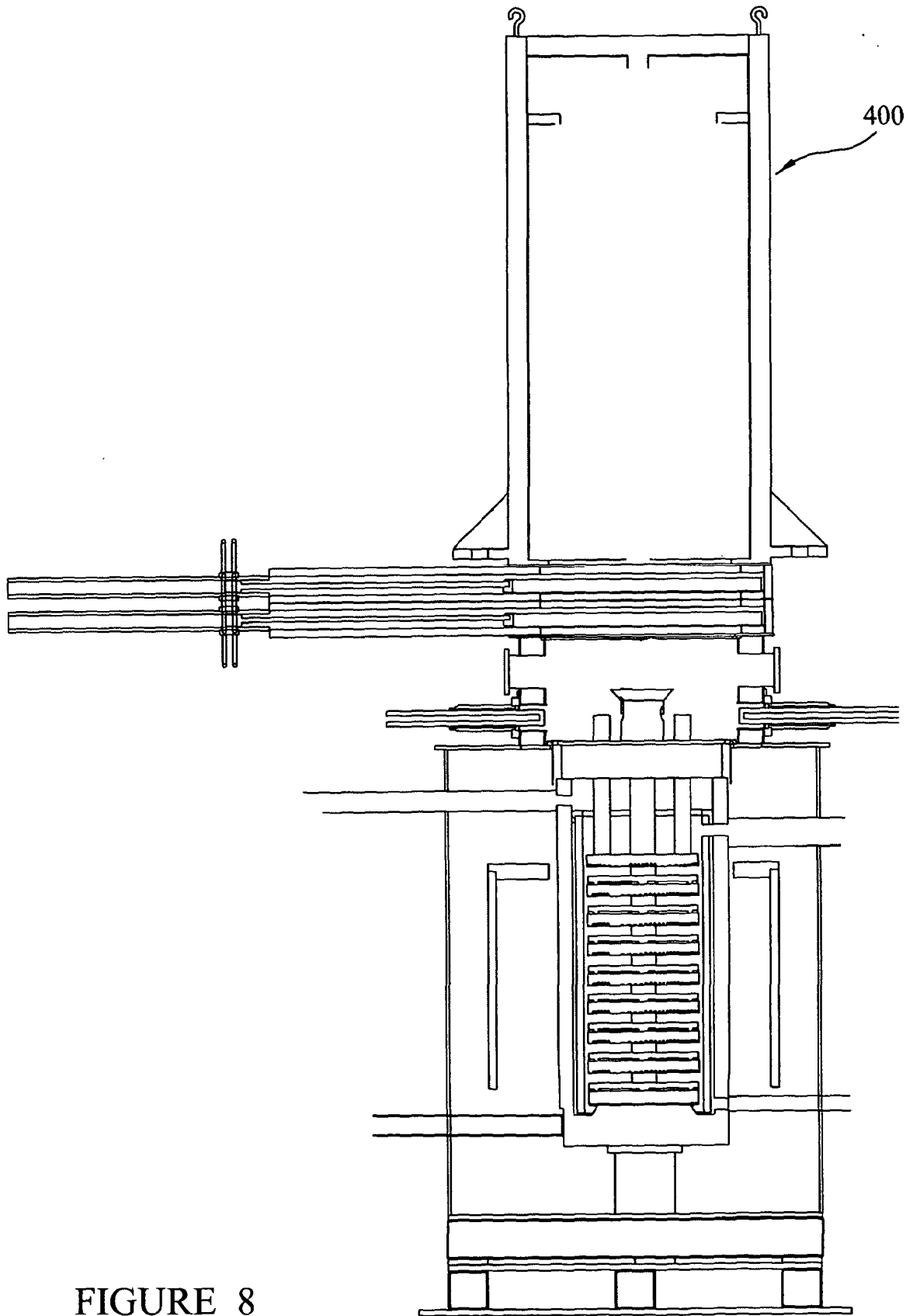


FIGURE 8

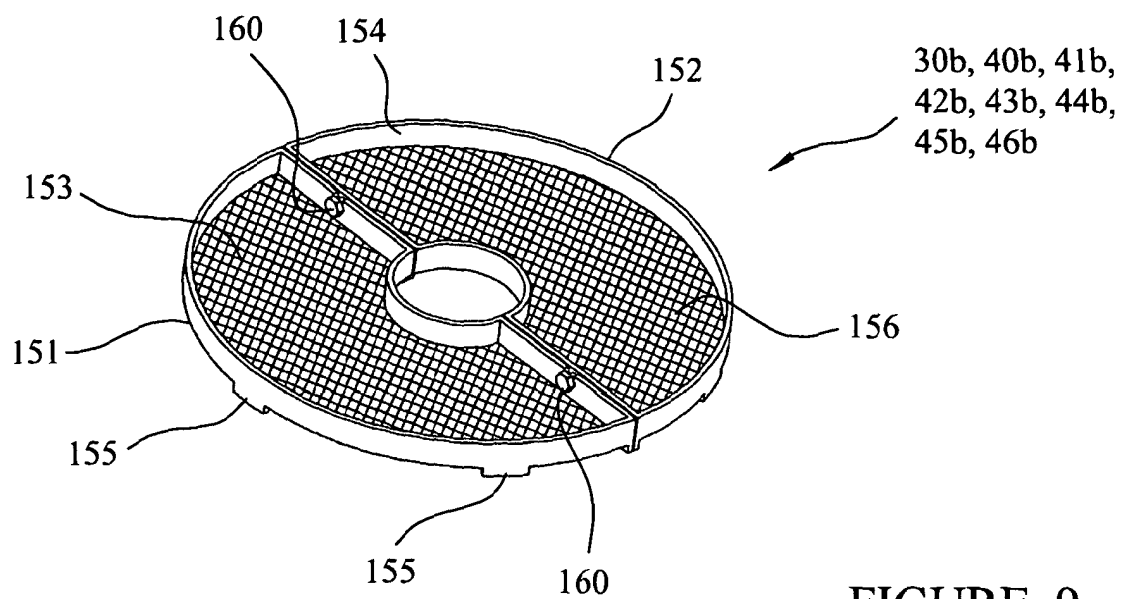


FIGURE 9

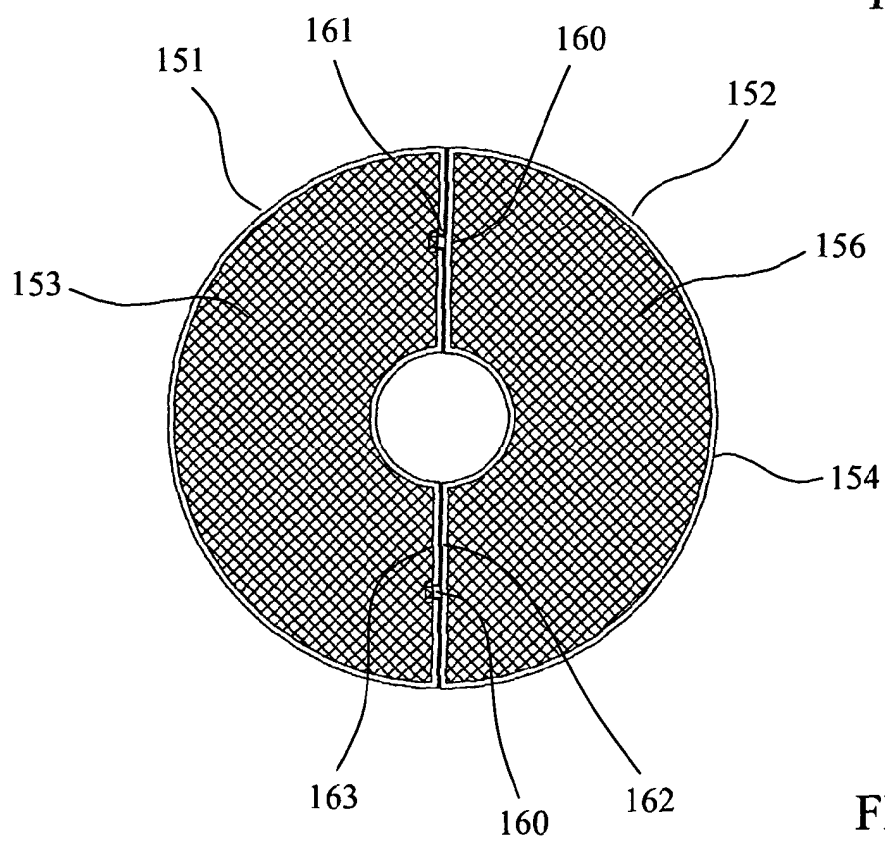


FIGURE 10

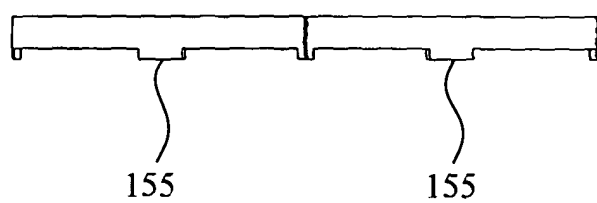


FIGURE 11

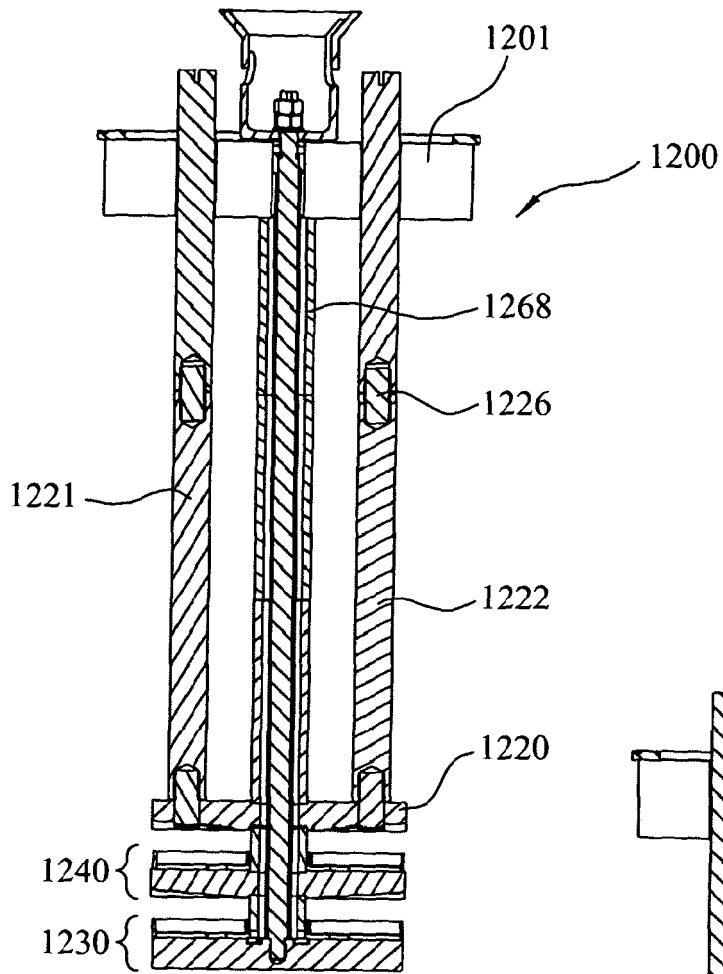


FIGURE 12

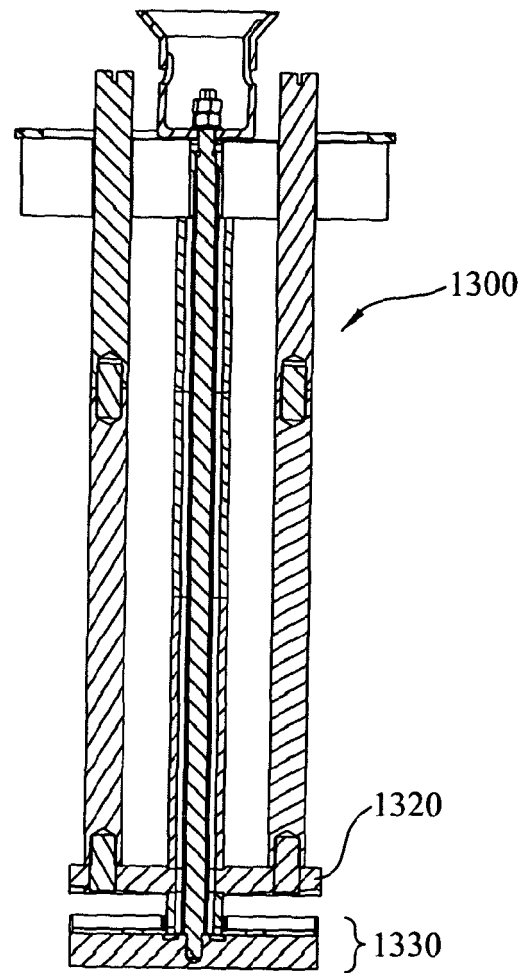


FIGURE 13

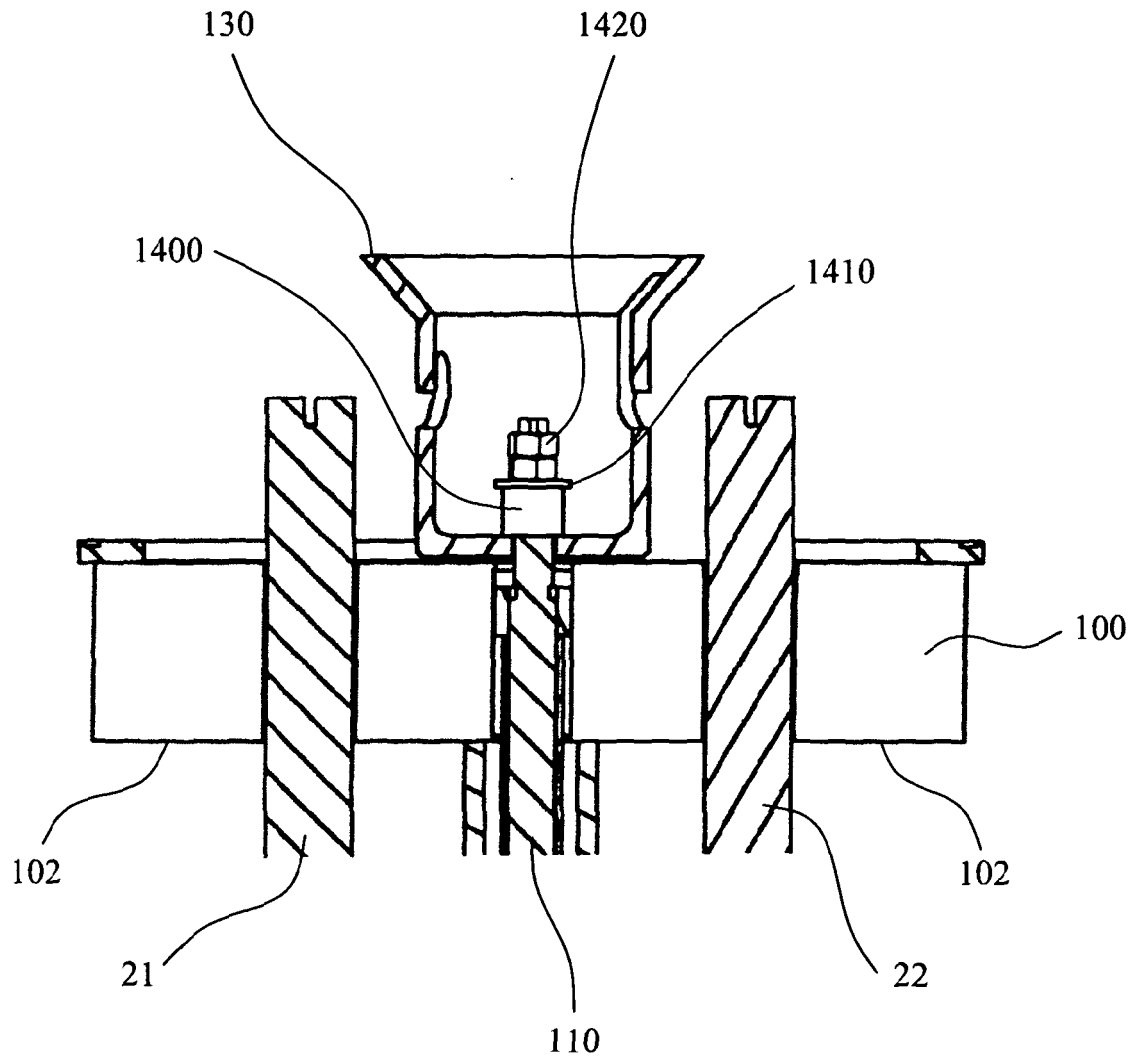


FIGURE 14

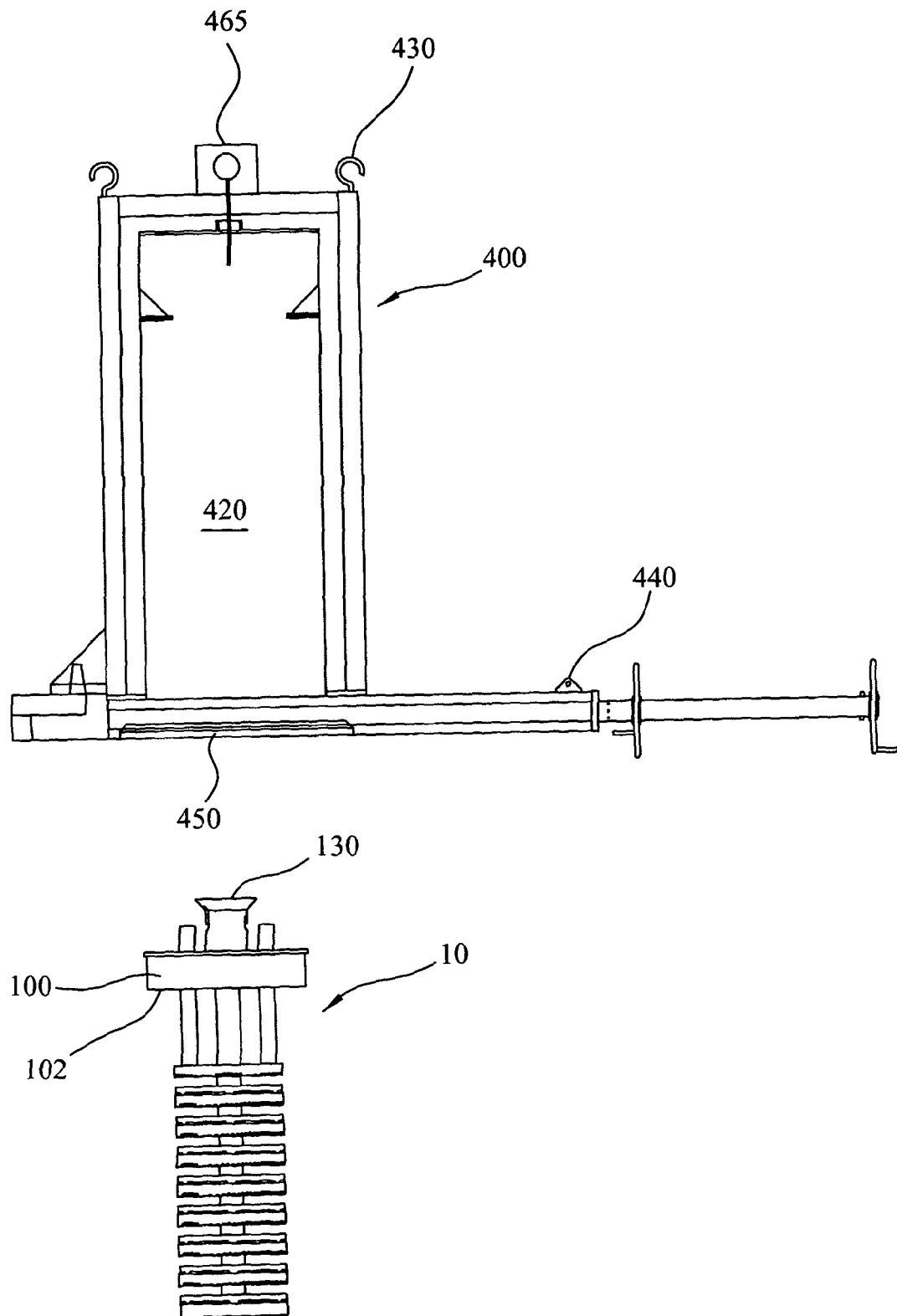


FIGURE 15

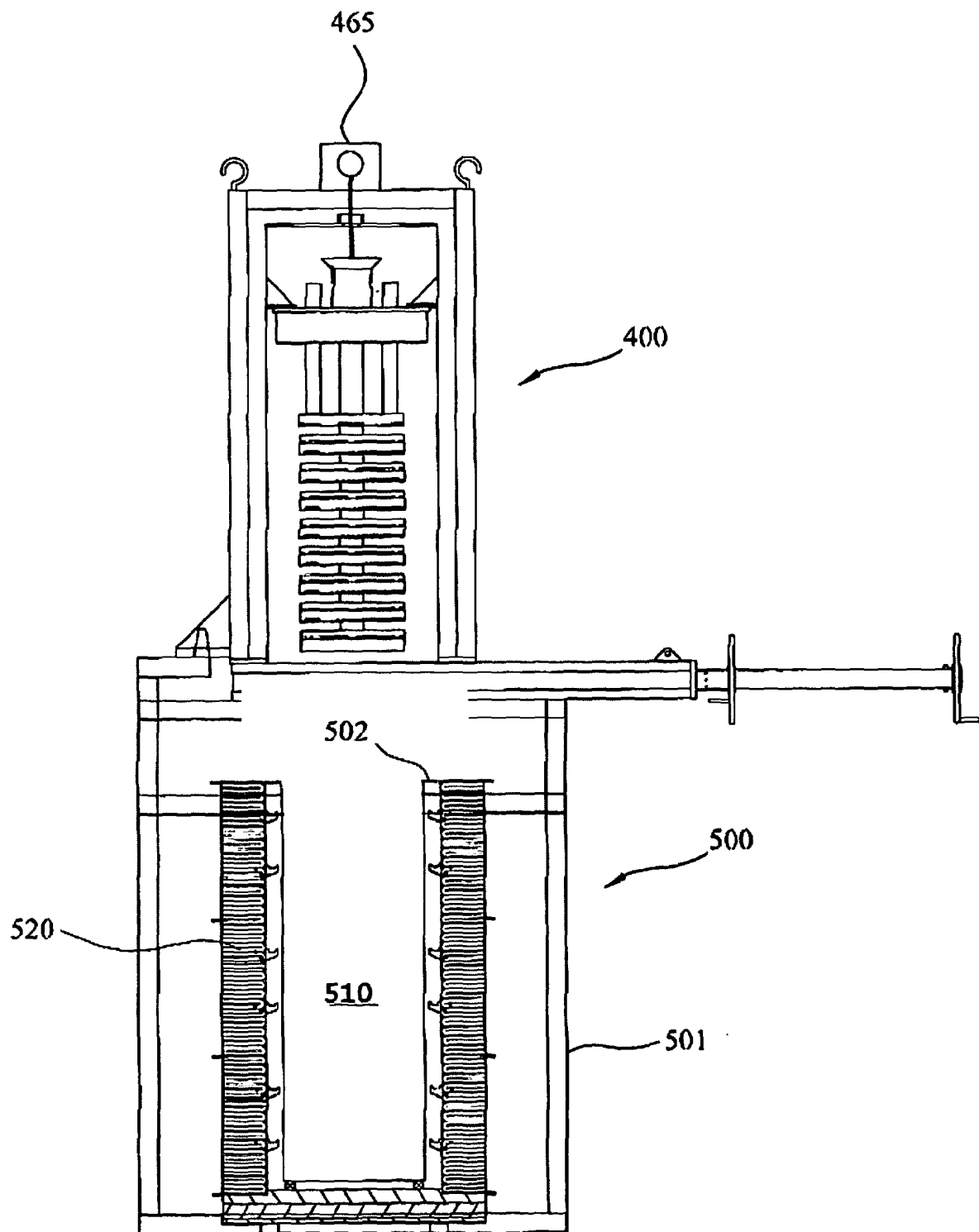


FIGURE 16

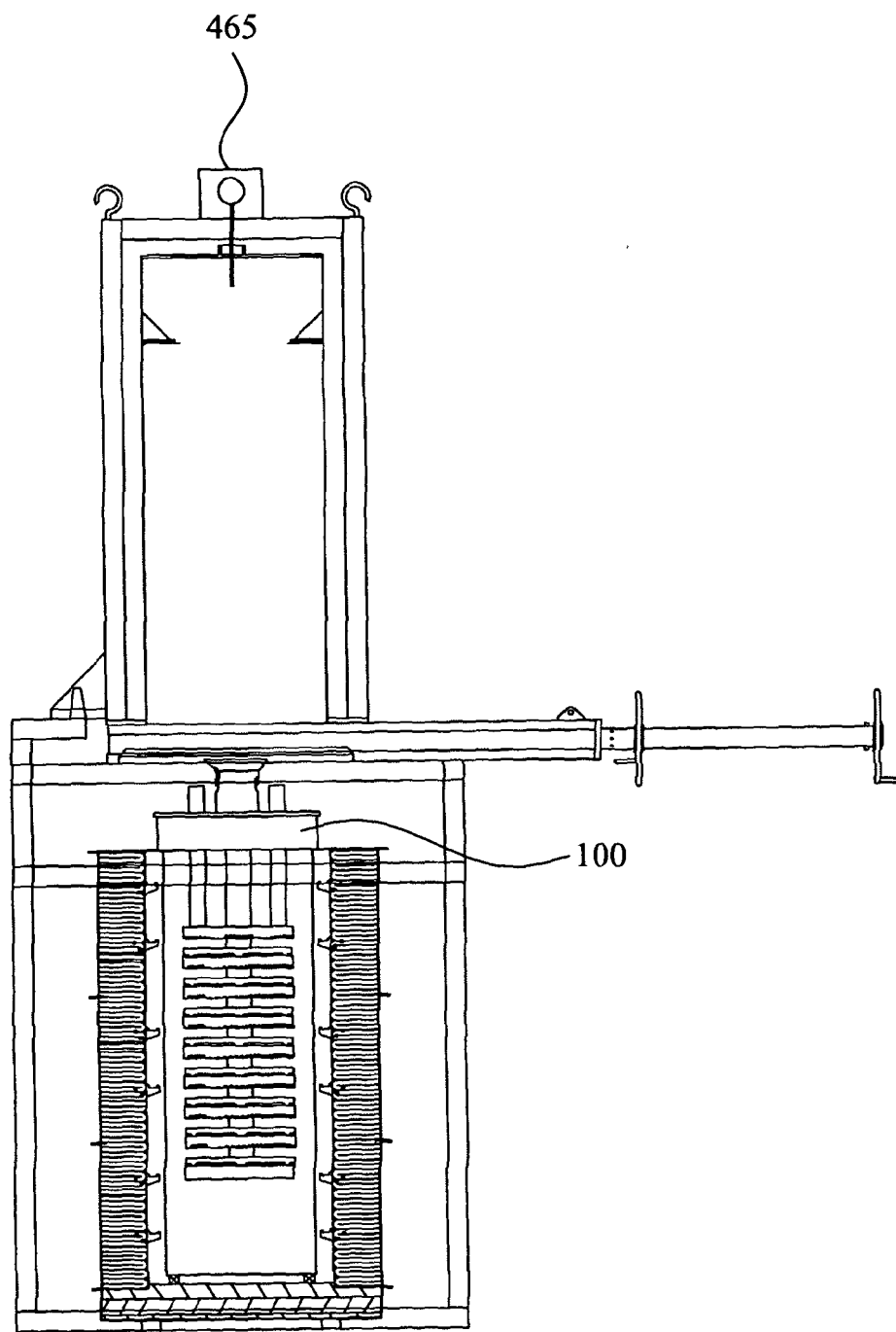


FIGURE 17

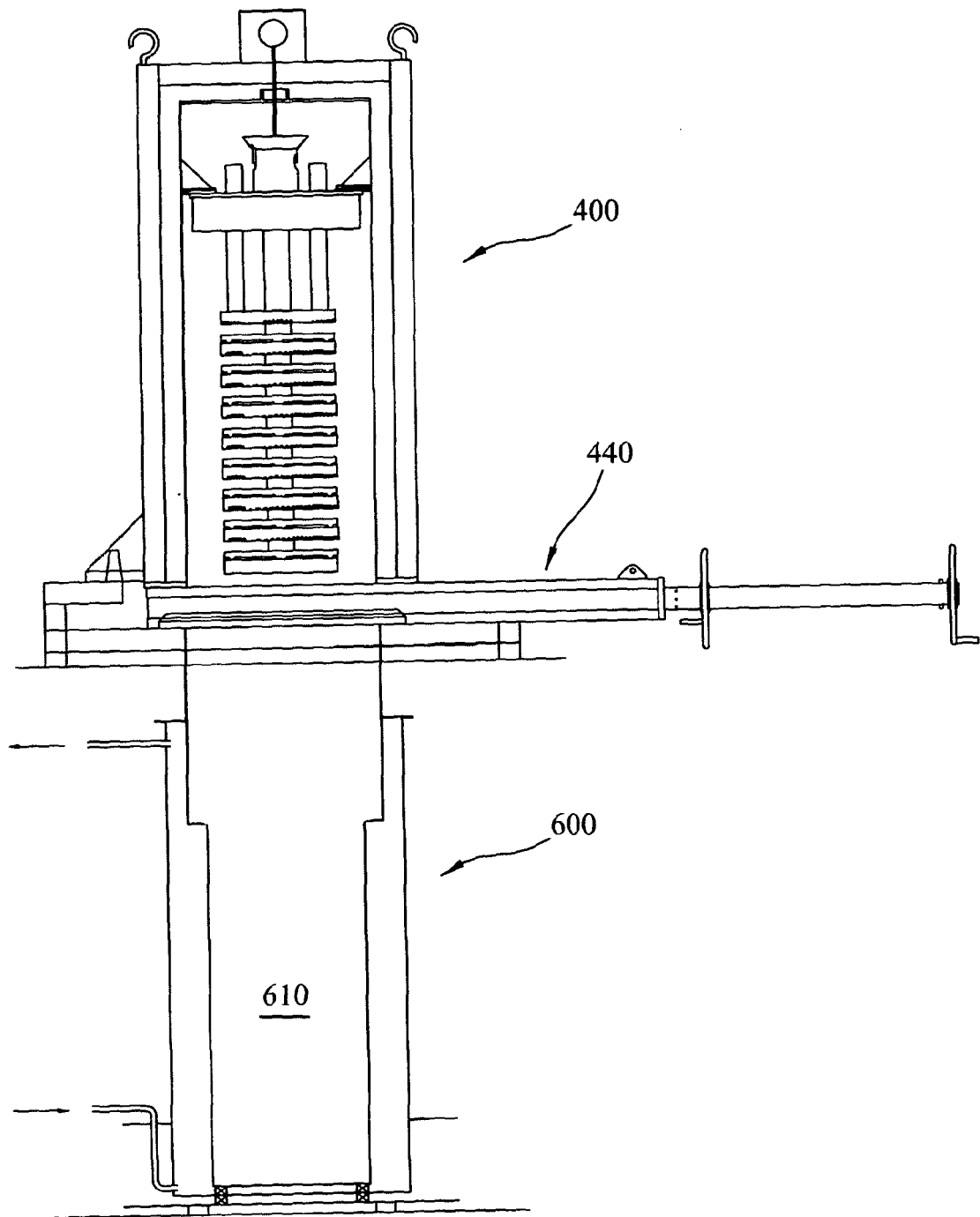


FIGURE 18

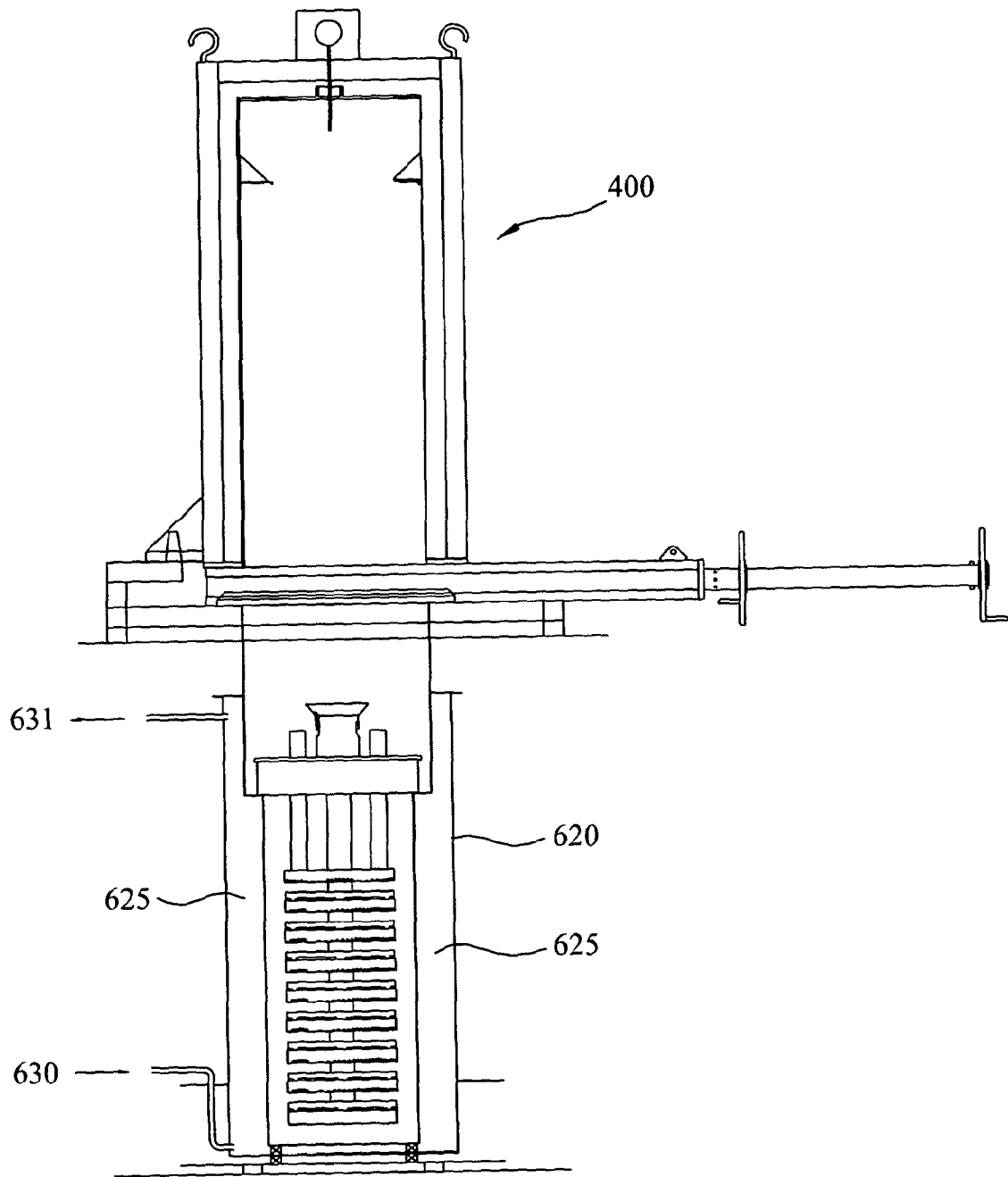


FIGURE 19

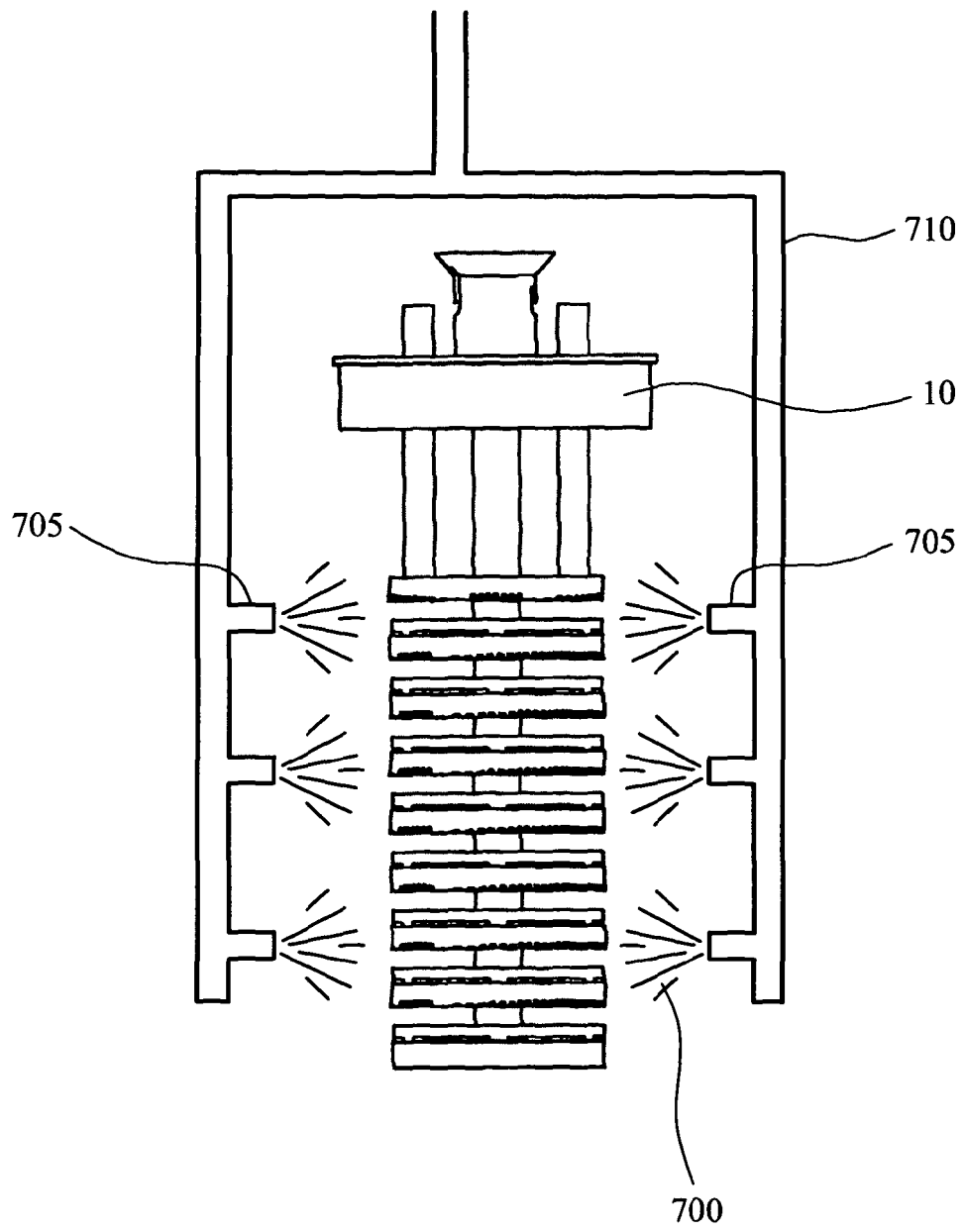


FIGURE 20

REFERENCES CITED IN THE DESCRIPTION

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