I, HIROSHI ISHIZUKA,
of 19-2, Ebara 6-chome, Shinagawa-ku, Tokyo, Japan,
hereby apply for the grant of a Standard Patent for an invention entitled:

"VACUUM SEPARATOR"

which is described in the accompanying complete Specification.

Details of basic application:
Number: 71118/81
Country: Japan
Date: 12th May 1981

My address for service is:-

SHELSTON WATERS,
55 Clarence Street
SYDNEY, N.S.W. 2000

Dated this 29th day of April, 1982.

HIROSHI ISHIZUKA

by Robert K. Keats

Fellow Institute of Patent Attorneys of Australia
of SHELSTON WATERS

To: The Commissioner of Patents,

WODEN. A.C.T. 2606

File: D.B.134G
Fee: $69.00

MS
In support of the Convention Application for a patent for an invention entitled:

"VACUUM SEPARATOR"

[Insert Title of Invention]

(Name(s) of Applicant(s) of Invention)

[Here Insert Name(s) of Applicant(s)]

(Adress(es) of Applicant(s))

[Here Insert Address(es) of Applicant(s)]

I/WE

(Insert Full Name(s) and Address(es) of Inventor(s), if other than Applicant(s))

[Here Insert Full Name(s) and Address(es) of Inventor(s), if other than Applicant(s)]

I/WE

[Here Insert Name(s) of Inventor(s)]

do solemnly and sincerely declare as follows:

1. I am/We are the Applicant(s) for the Patent.

2. The basic Application(s) as defined by section 141 of the Act was/were made in Japan on the 12th day of May 1981.

3. I am/We are the actual Inventor(s) of the invention referred to in the basic Application [or where a person other than the Inventor(s) is/are the actual Inventor(s) of the invention and the facts upon which I am/we are entitled to make the Application are as follows:]

4. The basic Application(s) referred to in paragraph 2 of this Declaration was/were the first Application(s) made in a Convention country in respect of the invention, the subject of the Application DECLARED at Tokyo, Japan this 15th day of April 1982.

[Signature(s) of Applicant(s)]

To THE COMMISSIONER OF PATENTS.
In a vacuum separator for refractory metal from magnesium metal and magnesium chloride mixed therewith, comprising: a vertically elongated substantially cylindrical retort having, inside, a closed space, a lower section of which is capable of accommodating such mixture to be treated as held in a container and is provided with a heating means to evaporate the magnesium metal and magnesium chloride to a substantial part, and an upper section of which has a cooling means to provide, inside, a cylindrical face for condensing thereon magnesium metal and magnesium chloride which ascend as vapor, and a means for degassing the retort to an elevated level of vacuum, an improvement such that a heat shield unit is provided at an intermediate level between said upper and lower sections of the retort, said heat shield unit comprising, as a whole, opening so arranged as to block any direct sight of the surface of the retort lower section when holding the container from.../2
2. An improvement as recited in Claim 1, in which said heat shield unit comprises a plurality of circular...
The following statement is a full description of this invention, including the best method of performing it known to me/us:—

"VACUUM SEPARATION"
The present invention relates to a vacuum separator, or, an apparatus for removal of magnesium dichloride and magnesium metal coexisting with titanium or zirconium metals as recovered from a Kroll process wherein tetra-chloride of such metals is reduced with fused magnesium metal.

In the production of titanium or zirconium metal, the so-called Kroll process is generally utilized by which metal chloride is converted to metal sponge with fused magnesium as reducing agent which is charged usually in an amount in excess of the stoichiometry to complete the reduction, thus leaving magnesium metal as unconsumed as well as magnesium chloride byproduct when the process is ended. Thus, with the product metal accompanied with such inclusions, a mass as recovered from the reduction process is essentially subjected to vacuum separation, or a fractional distillation in a vacuum, for purification of the metal, by evaporating in a zone of vacuum separator such inclusions as magnesium metal and magnesium chloride at a temperature on the order of 1000°C, which then are condensed for recovery in another zone of the apparatus.

For such purification of the metal an elongated vertical arrangement is especially employed which basically comprises a retort to contain a crucible loaded with a reaction mass in the upper half which is placed in a furnace, while the lower half is provided with a cooling
means, as known, for example, from USP 3 663 001. This
design is generally employed as favorable because of
rather a high treatment efficiency since the metal and
chloride inclusions flow down from the upper zone to the
lower zone as cooled, in a liquid state as well as vaporous.

Meanwhile, since the reduction process of such metal
chloride with magnesium is practised in batches, an increased
capacity treatable in each run is desirable as advantageous
in production economy. Obviously, use of a crucible of
increased dimensions for this purpose requires a vacuum
separator of accordingly increased capacity. With the
conventional techniques as said above, however, a substan-
tially increased cost is involved in plant construction
due to an essential separator which has a necessarily so
rigid and costly construction especially in the lower zone
that a considerable weight can be upheld of the crucible
of a substantially increased mass as well as a furnace en-
larged accordingly. Further cumbersome and rather dangerous
operation with possible human injury and/or equipment
breakdown are necessary when such massive furnace essentially
is moved up and down and transferred every run for mounting
and dismounting every run. In addition, with the top not
available for heater arrangement, the furnace should have
a substantially elongated construction for supplying from
around a sufficient heat to evaporate inclusion at the
bottom of the mass being treated. This also results in
Therefore, with an increased volume to be treated per batch, another separator arrangement is favored in which, as known from USP 4,105,192, for example, such retort has a lower half to be heated with a furnace around it and an upper half to be cooled so that a crucible placed on the bottom in the lower half is heated to evaporate out of metal product inclusions which are then caught as condensed in the upper zone. This construction is advantageous in that provision of a furnace of an increased weight is readily realized, in a simplified placement of a crucible as holding the metal product mixed with the inclusions, and in that the furnace bottom is available for heater provision so that a furnace of a substantially shortened design is effectively usable as a result of an efficient heat supply to the bottom of the crucible for removing the inclusions there. In case especially where another crucible as evacuated is used in the upper zone to deposit condensates of the magnesium metal and chloride, and where such deposited condensates are put in a Kroll process as attached to the crucible, the metal is used as reducing agent while the chloride can be discharged out of the retort together with in-situ formed magnesium chloride, thus saving labor and resulting recovery loss, involved with otherwise necessary step of scraping such condensates.

Although this arrangement thus exhibits some merits
in treatment of metal batches of an increased mass, conventional vacuum separators in use involve an inevitable drawback: condensates once formed on the wall in the cooling zone tend often to drop in the process into the lower zone, where they must be heated to be vaporous again, and as a result, a substantially increased treatment time is necessary relative to the first mentioned separator design by which the cooling zone is positioned over the evaporation zone.

The Inventor has observed that such condensate drop is most likely to be caused by the heat primarily radiated from the lower zone of the retort wall placed in a furnace, as this is seen to happen at a late stage where vaporization of magnesium metal and chloride is small and slow, so the vacuum level is high, and since this is also seen when temperature rise is rapid to fast evaporate the inclusions. This apparently results from low cooling efficiency through such a porous structure of condensates as well as a small adhesion to the cooled wall. A slowed heat supply however, is unfavorably results in an increased process time to lower metal productivity.

Therefore, the principal objective of the present invention is to provide a vacuum separator design removed of the drawbacks, as described above, involved with conventional techniques. Such objective has been fulfilled according to the invention, by providing a separator
arrangement wherein a mixed mass to be treated is placed in the lower section, with an adequate heat shield means to intercept primary heat radiation to a substantial part from below, such means being provided at an intermediate level between the lower and upper sections of the retort, thus allowing individual temperature regulation such that the lower section (evaporation section) may be kept at a temperature level over 900°C so as to effectively evaporate magnesium metal and chloride to separate from the metal product, while the upper section (condensation section) is at a temperature below 650°C so that such magnesium metal and magnesium chloride may not be fused again to fall apart from a condensation face which is conveniently provided on an evacuated crucible of a similar construction as used below placed in this section.

According to the invention there is provided in a vacuum separator for refractory metal from magnesium metal and magnesium chloride mixed therewith, comprising:

a vertically elongated substantially cylindrical retort having, inside, a closed space, a lower section of which is capable of accommodating such mixture to be treated as held in a container and is provided with a heating means to evaporate the magnesium metal and magnesium chloride to a substantial part, and an upper section of which has a cooling means to provide, inside, a cylindrical face for condensing thereon magnesium metal and magnesium...
chloride which ascend as vapor, and a means for degassing
the retort to an elevated level of vacuum, an improvement
such that a heat shield unit is provided at an intermediate
level between said upper and lower section of the retort,
said heat shield unit comprising, as a whole, opening so
arranged as to block any direct sight of the surface of
the retort lower section when holding the container from
a substantial part of the condensation face in the upper
section, thus intercepting a substantial part of primary
heat radiation from the retort lower section, while allow-
ing passage of ascending vapor.

In the invention the heat shield unit may take
various setups as far as it meets the criteria: blocking
of primary heat radiation, to a substantial part, to the
condensation face from the retort wall as heated and passage
for ascending vapor of magnesium metal and chloride. The
unit basically consists of one circular plate with a central
opening, which is usually circular, too, with an additional
plate or an additional set of plates with a central bore,
similarly shaped or otherwise, placed with a substantial
distance between the top and bottom of the row, relative
to the dimensions and shape of the opening. Such additional
plates, partly or as a whole, may be replaced with a body
otherwise constructed, such as a solid disk, a cone or a
series of conical rings of a same or different diameters
with a cone atop, the disk or cone or other conical bodies
as a whole having a cross section larger than that of the opening and being arranged in adjacency to cover the opening. Each of such elements as said above consists of steel, and preferably of stainless steel. The plates to compose the unit favorably have a bottom side finish polished or ground for an improved reflectivity for heat radiation from below. Heat insulative of non-metallic material, such as carbon fiber is advantageously utilized as inserted between adjacent plates or as overlaid on the condenser-section end of the plate or plates.

Other features of the invention and advantages achieved thereby will be better understood from the following description taken in connection with the accompanying drawing which is given by way of example only and not for limiting the invention. Of the drawing:

Figure 1 illustrates a schematic view in section of a vacuum separator constructed according to the invention, while Figure 2 shows a few variations in particular of heat shield unit of the invention.

In the figures a vacuum separator, generally designated at 1, comprises an elongated substantially cylindrical retort 2 of steel partly placed in a furnace 3 having an electroresistive heating element 4 on the bottom as well as on the cylindrical wall. Encased in a metallic shell 5 and provided with a heat resistive packing 6
between the furnace 3 and retort 2, the interspace 7 formed therebetween is pressure regulatable with a conventional means (not shown). The retort 2 has divisibly connected lower portion 8 to provide an evaporation section, placed in the furnace 3, and an upper portion 9, to provide a condensation section, having therearound a jacket 10 to pass coolant water therethrough. The lower portion 8 accommodates such metallic product as titanium or zirconium in a sponge form mixed with magnesium metal and magnesium chloride as held on a perforated grate 11 in a crucible 12. The upper portion 9, which is arranged as protrudent from the furnace 3, may favorably accommodate another crucible 13 on a set of detachable stoppers 14 as evacuated and removed of the grate for condensing to deposit upcoming magnesium vapor of metal and magnesium chloride. The retort 2 has a pipe 15 for degassing connected at a top portion. The retort essentially has a heat shield unit 16 over the crucible 12 in a vicinity of the joint of two portions 8, 9. The unit 16, as illustrated in the figures, takes various setups. Figure 1 particularly shows a design which consists of a cone 17 of steel supported on and arranged so as to cover entirely a central round opening 18 of disks or circular plates 19, 20 of steel arranged at a spacing and inserted with a heat insulative 21, such as carbon fiber, therebetween. Such heat shield unit as a whole is detachable from the retort 2 when the latter
is divided. Figure 2 shows other variations at (a) to (c): a pair of steel disks 22, 23 with a similar central bore are rested on each stopper set 24, 25 at a space without any insert (a), a disk 26 with a central opening, resting on a stopper set 27, has an attachment thereto of a solid disk 28 of a cross section capable of covering the opening at the given space from below (b), and a set of a flat circular plate 29 and a flanged conical ring 30 each with a central bore, have therebetween a stuff of heat insulative material 31 and detachably placed on a stopper set 32. A typical example using such arrangement will be given.

Example

An electroresistive furnace of a substantially cylindrical form was used which measured 2 m in I.D. and 4 m in inside length, and had thereinside a substantial part of the lower portion of a retort which measured 1.6 m in I.D. and 32 mm in thickness and consisted of stainless steel. In the retort placed was a crucible of also a stainless steel, which had a 1.4 m in O.D., a 16 mm thickness and 2.4 m in entire length, and held some 4 (metric) tons of titanium sponge, as mixed with a minor amount of magnesium metal and chloride inclusions. As a heat shield unit two disks of, each, a 2 mm thickness and a circular central opening some 30 cm across were arranged over the crucible at a space of 10 cm from each other.
On such disks atop there were supported a solid disk 100 cm across and 2 mm thick, as spaced with leg members. The disks as well as the cone and leg members in this example each consisted of stainless steel. Over the shield unit a set of stoppers was attached to the retort and a blank crucible of a similar construction was seated thereon, and the upper half was mounted to connect with the lower half to build up a retort. The retort thus set up was degassed through the pipe atop, while it was heated in the lower half from outside to a temperature level between 950 and 1000°C at a rate of 50°C/hour, while cooling the upper portion which circulating water. A vacuum of $3 \times 10^{-3}$ Torr was reached in some 40 hours from the outset of degassing. Treatment was practised at the temperature for 60 hours to complete. This achievement consists a substantial improvement over conventional techniques, without employment of the heat shield unit of the invention, which typically takes some 90 hours for treatment of such amount of titanium metal.

As described above in detail, the invention, whereby condensates of magnesium metal and magnesium chloride are effectively prevented from falling into the crucible at the evaporation section, permits a substantial improvement in productivity as a result of raised separation efficiency of inclusions from sponge product of refractory metal.
1. In a vacuum separator for refractory metal from magnesium metal and magnesium chloride mixed therewith, comprising: a vertically elongated substantially cylindrical retort having, inside, a closed space, a lower section of which is capable of accommodating such mixture to be treated as held in a container and is provided with a heating means to evaporate the magnesium metal and magnesium chloride to a substantial part, and an upper section of which has a cooling means to provide, inside, a cylindrical face for condensing thereon magnesium metal and magnesium chloride which ascend as vapor, and a means for degassing the retort to an elevated level of vacuum, an improvement such that a heat shield unit is provided at an intermediate level between said upper and lower sections of the retort, said heat shield unit comprising, as a whole, opening so arranged as to block any direct sight of the surface of the retort lower section when holding the container from a substantial part of the condensation face in the upper section, thus intercepting a substantial part of primary heat radiation from the retort lower section, while allowing passage of ascending vapor.
2. An improvement as recited in Claim 1, in which said heat shield unit comprises a plurality of circular plates with a central opening, said plates being so arranged in a row that the plates at the top and bottom of the row may be substantially distant from each other relative to the opening.

3. An improvement as recited in Claim 2, in which at least one spaces between adjacent plates are stuffed with a mass of heat insulative of non-metallic material.

4. An improvement as recited in Claim 1, in which said plate has a layer of heat insulative of non-metallic material overlaid atop.

5. An improvement as recited in each of Claims 3 and 4, in which said non-metallic material basically consists of fibrous carbon.

6. An improvement as recited in Claim 1, in which said heat shield unit comprises at least one circular plates with a central opening and another body arranged in adjacence to cover such opening.

7. An improvement as recited in Claim 6, in which said body substantially consists of a flat solid disk of a cross section larger than that of the opening.
8. An improvement as recited in Claim 6, in which said body substantially consists of a cone with a horizontal cross section larger than that of the opening.

9. An improvement as recited in Claim 6, in which said body substantially consists of a series of conical rings with a cone atop.

10. An improvement as recited in Claim 6, in which said heat shield unit comprises at least two circular plates spaced from each other.

11. An improvement as recited in Claim 10, in which said plates have a mass of heat insulative of non-metallic material inserted therebetween.

12. An improvement as recited in Claim 11, in which said material basically consists of fibrous carbon.

13. An improvement as recited in each of Claims 2 and 6, in which said circular plate substantially consists of steel and has a bottom side finish ground.

DATED this 29th day of April, 1982.

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bottom of the mass being treated. This also results in
Although this arrangement thus exhibits some merits