April 28, 1964 C. W. HANKS ET AL 3,131,051

PROCESS AND APPARATUS FOR REFINING LOOSELY COMPACTED REFRACTORY METALS IN AN ELECTRON BEAM FURNACE

Filed Sept. 23, 1959 2 Sheets-Sheet 1

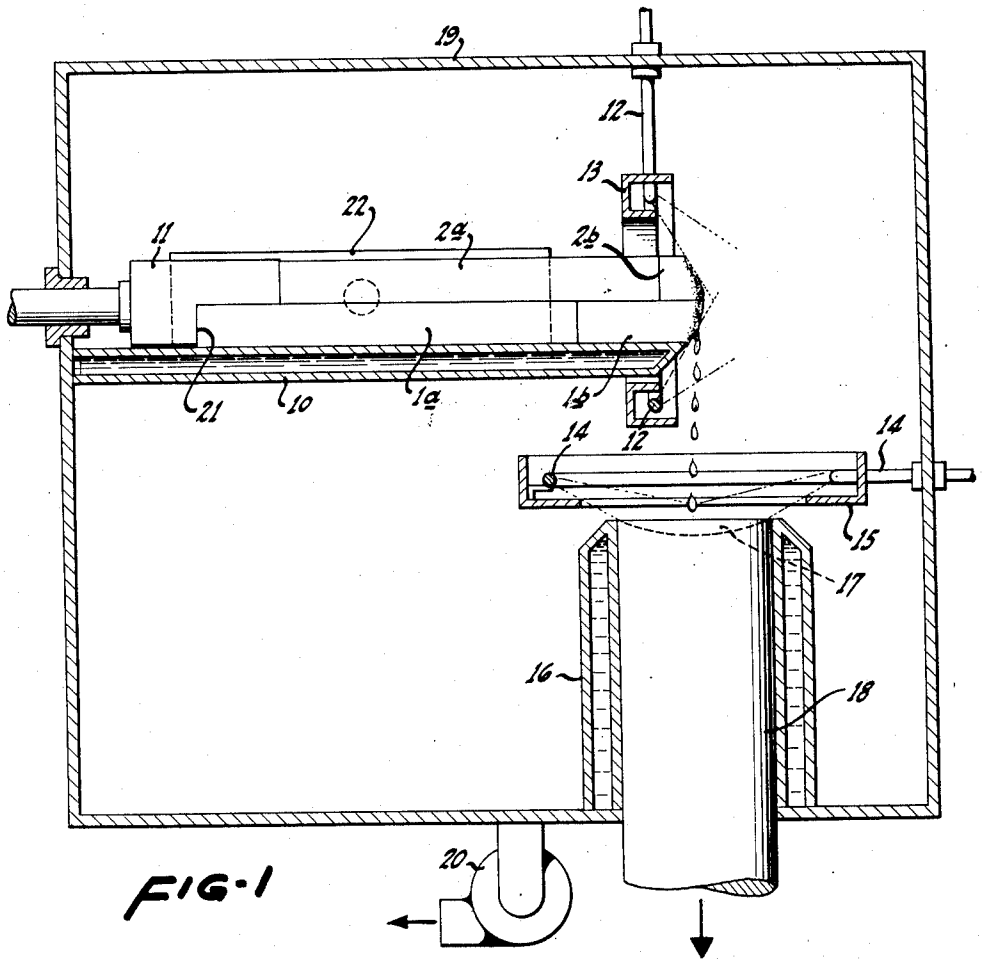


FIG-1

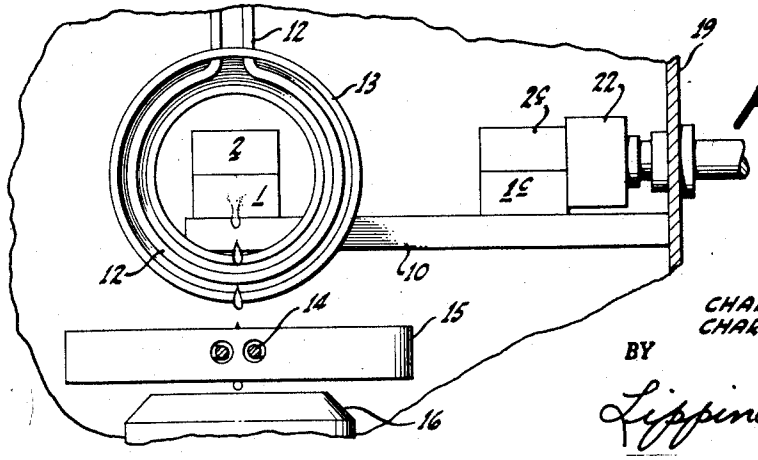


FIG-2

INVENTORS
CHARLES W. HANKS
CHARLES A. HUNT

BY

Lippincott & Ralls
ATTORNEYS

April 28, 1964 C. W. HANKS ET AL 3,131,051

PROCESS AND APPARATUS FOR REFINING LOOSELY COMPACTED REFRACTORY METALS IN AN ELECTRON BEAM FURNACE

Filed Sept. 23, 1959 2 Sheets-Sheet 2

FIG-3

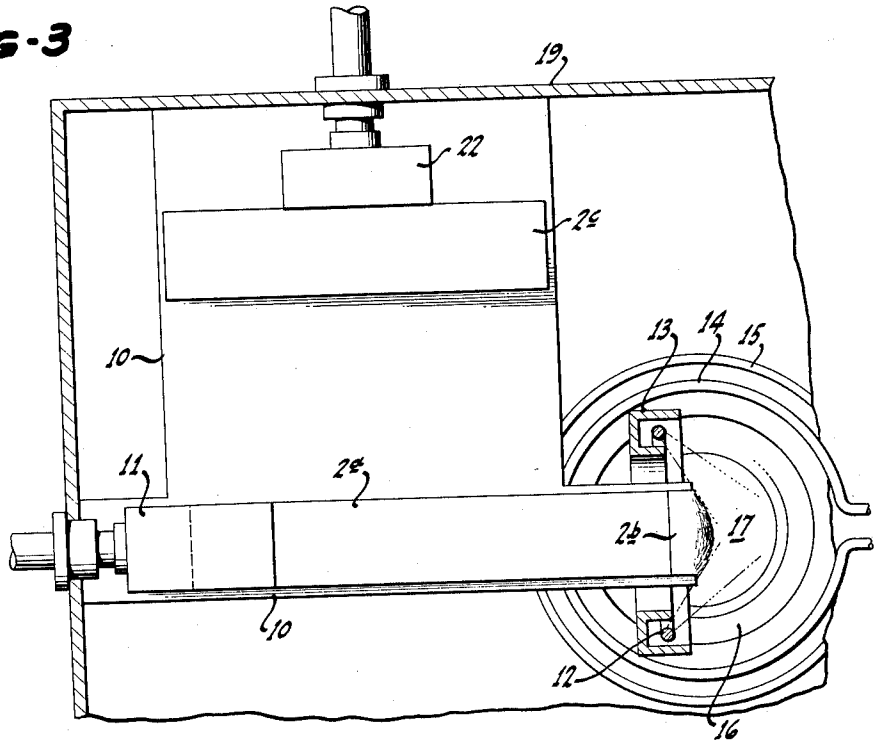


FIG-4

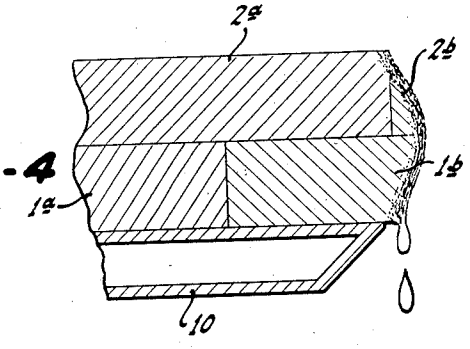
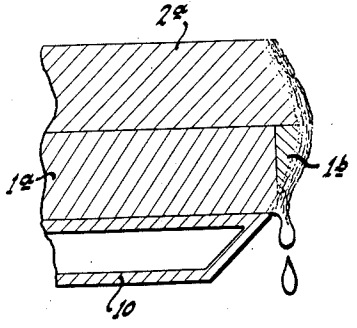


FIG-5



INVENTORS
CHARLES W. HANKS
CHARLES d'A. HUNT

BY

Lippincott & Ralls
ATTORNEYS

United States Patent Office

3,131,051

Patented Apr. 28, 1964

3,131,051

PROCESS AND APPARATUS FOR REFINING LOOSELY COMPACTED REFRACTORY METALS IN AN ELECTRON BEAM FURNACE

Charles W. Hanks and Charles d'A. Hunt, Orinda, Calif., assignors to Stauffer Chemical Company, New York, N.Y., a corporation of Delaware

Filed Sept. 23, 1959, Ser. No. 841,828
2 Claims. (Cl. 75—10)

This invention relates to a process for refining loosely compacted refractory metals and apparatus for practicing the method. More particularly, the invention relates to a process for refining small compacts or bars by electron bombardment.

The peculiar problems attendant to refining refractory metals such as titanium, zirconium, molybdenum, tungsten, et al., have been attacked in a variety of ways. More recently it has been proposed that a source of electrons be directed towards a solid bar or compact of these more active metals in a high vacuum furnace. This particular approach to purifying refractory metals has met with great success since the electron beam provides a source of heat which is both sharply selective and virtually free of contaminating influence.

An exemplary electron beam furnace for purifying a compact or solid stock in a continuous or semi-continuous manner is disclosed and claimed in Patent No. 2,880,483, issued April 7, 1959, to Hanks et al. In this embodiment of an electron beam-high vacuum furnace, the melt stock is suspended vertically and a cathode source of electrons is caused to melt the lower end thereof which drips into a skull formed on top of the resolidified melt supported by a water-cooled crucible. The cast ingot, as the skull slowly solidifies from bottom to top is withdrawn at the lower end of the furnace to provide the refined refractory metal.

One of the problems associated with such refining of melt stock or compacts is that of forming the compacts or metal stock itself. Heretofore, in the prior art it has been necessary ordinarily to actually melt the refractory metal to form the stock to charge the electron beam furnace. Alternatively, if a compact was desired, an extremely heavy hydraulic press had to be used to obtain the high pressures necessary to form it. For example, in many applications, hydraulic presses having capabilities of 20 to 40 tons per square inch are needed to form compacts sufficiently strong to support their own weight (as required in a furnace such as that disclosed in the patent referred to above).

The present invention overcomes this substantial disadvantage by providing a method and apparatus which will work satisfactorily with loosely compacted powders, still provide a highly refined end product, and operate semicontinuously. The advantages of using loosely compacted bars of the raw material in an electron beam furnace are obvious. The equipment necessary to lightly compact refractory metal powders is a far cry from that required to develop 20 to 40 tons per square inch pressure. As a consequence, the cost of forming the loose compacts used in the present refining process represent a substantial saving.

Not only is the use of highly compressed bars expensive and wasteful of equipment, but a further problem arises since the compacts have to be welded together to form a continuous rod. Only in this way can the consumable electrode in the furnace be supported vertically in an electron furnace and, at the same time, be sufficiently dense to carry the high currents that are necessary to refine the refractory metal. The welding of compacts or solid bars together requires more equipment and additional expense, not to mention the more significant fact that it adds impurities to the melt stock. In order to minimize these impurities, it is usual practice to weld the compacts or solid bars together in the presence of an inert atmosphere. Even so, some impurities are added. As a result, to prepare the solid or compact stock for furnaces of the prior art not only requires a substantial outlay in equipment and added expense, but results in a stock charge which is generally inferior.

It should not be inferred from the above that there are no existing techniques available to refine granulated stock. On the contrary, there are furnaces which are charged with granules of refractory metals. These have been operated, however, in more or less non-continuous or batch-type operations because of the problem of recharging furnaces, particularly ones wherein a high vacuum is maintained. Another disadvantage attending the refining of metal from granular stock is mechanical grain movement. While these shifts of granules are not as harmful when pure metals are being refined, they are where alloys are involved. If the powder stock is alloyed, the grain movement can result in an end product that is not of uniform quality and therefore ill fitted for critical uses.

The present process and apparatus, which permits the use of relatively loose compacts or stock, overcomes the grain movement problem incident to alloy powder purification, the equipment expenses attributable to using highly compacted or solid bars, and eliminates the necessity for welding compacts together.

The present invention employs a flat table or platform supported inside a high vacuum furnace to support two or more layers of alined, end-to-end oriented, loosely pressed compacts for movement in one direction. Means are provided, operable from a point outside the vacuum chamber, to move these layers of pressed bars along the platform in the one direction, and a cathode is provided adjacent one set of ends of the pressed bars to heat and melt them by electron bombardment. A crucible is provided in the furnace immediately beneath the ends of the pressed bars bombarded by the electrons. The crucible acts to hold a skull of molten metal in the top of the resolidified stock or to collect pellets of the refined metal, as desired. Auxiliary cathode heating means may be provided to maintain the skull in a molten state as the refined stock is slowly pulled from the lower side of the furnace.

Since the pressed bars are supported on a platform they do not need to support their own weight or for that matter to be integrally welded together. The pushing rod for moving the compacts toward the end of the platform where the electron source is operative is constructed so that adjacent layers of pressed bars are staggered and no end of a compact overlies the end of one on an adjacent layer. Thus, a small piece of a bar on the lower layer is retained and prevented from falling into the skull prior to melting, in part, by the pressure of bars in the upper layer. Conversely, the remains of an upper layer pressed bar is prevented from falling into the skull prior to being melted, in part, because of the support by a compact in the next lower layer. This staggered superposition of the compacts overcomes the principal disadvantage inherent in horizontally supporting and moving compacts.

Cooperating with the platform and external to the vacuum chamber is means mounted transverse to the normal movement of the pressed bars in the one direction for restocking the platform whenever the bars thereon have been substantially melted. In order to do this, the pusher for displacing the pressed bars toward the cathode heating source is retracted and the transverse member is caused to move two or more new layers of

pressed bars in line with the path of the first pusher member.

By the use of equipment as described above and by employing small compacts, a good even rate of melting is obtained which not only lowers the average D.-C. input to the cathode-anode heating circuit, but permits the vacuum pump to remove gases in a more even manner. Thus, with such smaller compacts, the discontinuity in gas formation as pure or alloyed metals are refined is minimized and the overall vacuum can be maintained at a more uniform figure without extreme variations.

Objects of the present invention, therefore, are to provide a method of refining refractory metals and apparatus for practicing the method which utilize smaller and more loosely compacted charges and, as a consequence thereof, the apparatus is simpler in construction, more economical to operate and provides a more highly refined product.

In accordance with one feature of the invention, the apparatus combines a platform for supporting two or more layers of small loosely compacted charges, means for maintaining the individual compacts of one layer displaced with respect to the ends of the compacts of the adjacent layers, a source of electrons to heat and melt the charges, means to move the layers adjacent the source of electrons in the overlapping relation, and means to recharge the apparatus at times.

Another feature of the invention pertains to the combination in a high-vacuum furnace of a platform supporting member for two or more layers of end-to-end oriented compacts, a source of electrons adjacent one end of the platform, means operable to displace the two or more layers of pressed compacts towards the source of electrons in overlapping relation, means for supporting the liquid stock adjacent the source of electrons and means to recharge the furnace at times.

Yet another feature of the present invention pertains to the combination of a platform supported in a furnace, two or more layers of loosely compacted charges laid end-to-end in each layer, a pushing rod to maintain the ends of compacts in each layer in overlapping relation to the ends of compacts in adjacent layers and to displace the layers as a unit towards one end of the platform, means adjacent the one end of the platform for bombarding the ends of the compacts with electrons to thereby cause them to melt, a mold supported beneath the one end of the platform to catch the molten charge, electron beam means for maintaining the upper part of the material in the mold in a molten state, means for maintaining a high vacuum in the furnace, and means cooperating with the platform to add layers of compacts intermediate the pusher rod and the one end of the platform as needed.

Broadly, the present process for refining loosely compacted refractory metals comprises the steps of supporting a plurality of compacts in end-to-end relation and in a plurality of layers, maintaining the compacts in each layer overlapping the compacts in adjacent layers, and bombarding one set of ends of the compacts with an electron source to melt the compacts.

More particularly, the process of the present invention might comprise the steps of supporting pressed compacts of refractory metal powder in a furnace in end-to-end relation and in a plurality of layers, maintaining each compact in each layer overlapping the compacts in adjacent layers, bombarding one set of ends of the compacts with a source of electrons to heat and melt them, displacing the plurality of layers of compacts in their overlapping relation towards the source of electrons as the ends of the compacts melt, collecting the melted refractory metal, and recharging the furnace with layers of compacts as needed.

These and other objects and features of the present invention may be more fully understood when the following detailed description is read with reference to the drawings in which:

FIG. 1 is a schemtaic representation of furnace and charging apparatus in accordance with the present invention;

FIG. 2 is a partial end view of the charging apparatus of FIG. 1;

FIG. 3 is a partial plan view of the charging apparatus of FIG. 1;

FIG. 4 is a partial schematic of the compacts adjacent the melting zone when a small part of a compact on an upper layer remains; and

FIG. 5 is a partial schematic of the compacts adjacent the melting zone when a small part of a compact on a lower layer remains.

Turning to FIGS. 1, 2 and 3, the apparatus for performing the instant process may be seen to comprise a high vacuum furnace **19** having a water-cooled platform **10** therein upon which is supported layers **1** and **2** of loosely formed compacts of refractory material. A pushing member or pusher **11** is supported for movement along the surface of the horizonal platform **10** through an external force (not shown). Its displacement along a line (toward the right of the FIG. 1 drawing) causes the layers **1** and **2** of the compacts to move towards the right in overlapping relation. The overlap of the individual compacts **1***a*, **1***b*, **2***a*, **2***b*, etc., is insured by the stepped surface on the forward (right) end of the pusher **11**.

A circular cathode **12** is supported around the right end of the platform **10** and the compact layers **1** and **2** and an annular focusing member **13** cooperate therewith. The cathode **12** is focused to impinge upon the set of ends of the compacts adjacent the right end of the platform **10** in order to melt the powdered compacts which are connected to a source of positive potential (not shown) to act as an anode for the cathode **12**.

Immediately below the right set of ends of the compacts **1** and **2** is another circular cathode **14** which cooperates with a focus and shield member **15**. Below the cathode-shield combination **14—15** is a vertically disposed water-cooled crucible **16**.

With the compacts **1** and **2** constituting the anode of the cathode-anode electron beam circuit, the electrons flowing from cathode **12** cause the right set of ends of the compacts **1** and **2** to melt. The melt, in turn, drips into the uppermost part of the crucible **16** after passing through the cathode **14**. The cathode **14** is focused by member **15** to impinge upon the uppermost part of the solidified stock **18** supported in the crucible **16** thereby to maintain a preselected size skull or molten pool **17** of refractory metal. As the molten metal continuously drips into the skull **17** and solidifies, the stock **18** is withdrawn (by means not shown).

Since the pusher **11**, the platform **10**, cathodes **12** and **14** and the crucible **16** are all contained in the high vacuum furnace **19**, which is maintained at a vacuum approximating one micron of mercury by action of the vacuum pump **20**, relatively little or no contaminants are introduced into the refining process as the compacts **1** and **2** are pushed into the path of the electron flow from cathode **12**. The stock **1**, **2**, etc., melts and the refined bar stock **18** is withdrawn from the furnace as it does.

FIGS. 2 and 3 disclose the recharging mechanism for the refining apparatus of FIG. 1 most clearly. Whenever the compacts **1** and **2** are substantially melted, the pusher **11** is moved towards the left side of the furnace of FIG. 1 and additional layers of loosely formed compacts **1***c* and **2***c*, for example, are propelled along the platform **10** in a direction transverse to the line of movement of the pusher **11**. The recharging compacts **1***c*, **2***c* are moved in the line of movement of pusher rod **11** by a second pushing member **22** which is operated from a point external to the vacuum chamber **19** (by means not shown) whenever the compacts remaining become so small that the furnace requires recharging.

It will be noted, particularly with reference to FIG. 3, that it is immaterial whether or not the replacement charges **1***c* and **2***c* are displaced in overlapping relation

originally since the stepped front surface **21** on the pusher **11** will cause them to overlap as they are pushed along towards the right side of the platform **10**.

FIGS. 4 and 5 illustrate more clearly how the overlapping relation of the charging compacts **1***a*, **1***b*, **2***a*, **2***b*, etc., prevent small remaining pieces of a compact from prematurely falling into the skull **17**. In FIG. 4 there is illustrated a small remaining part of a compact **2***b* and, as can be seen, it will not fall into the skull **17** due to the support of the lower compact **1***b* and the normal surface tension which exists between the surfaces thereof and the adjacent compact **2***a* on its own layer and its supporting compact **1***b*. In the FIG. 5, the opposite situation is illustrated wherein the small piece of compact **1***b* remaining is on the lower level **1**. Up until the last moment, this small portion **1***b* is supported intermediate compact **2***a* and the lip of the apron or platform **10**. Before it all melts the platform **10** no longer supports it, however, the surface tension between the contacting surfaces and the bond of molten metal flowing along its outer surface is sufficient to support it until cathode **12** has completed the job of reducing it to liquid form. Naturally enough, the platform **10** cannot extend to the focus of the electron beam because it would then be melted along with the compact.

It should be apparent that the present invention is not limited to two layers of compacts. It can use two or more layers, provided the front surface **21** of pusher **11** is formed to insure the overlap of compacts in adjacent layers as they approach the bombardment area.

Rather than use a skull **17** to collect the refined metals for removal from the furnace **19**, alternatively, the drops can be formed into pellets for further refining or mixing with other metals to obtain alloys, etc. The use of a skull **17** in a vertically movable melt stock **18** is purely exemplary and should not limit the scope of the present invention.

Beyond this, while there are no specific means indicated for operating the pusher **11** or recharging pusher **22**, it is apparent that these two could be operated either manually or by automatic means. Further, it can be seen that the pusher **22** can cooperate with the transverse part of platform **10** to provide a charging entrance for the vacuum furnace **19** which does not interfere with the maintenance of the high vacuum inside the furnace **19**.

These and numerous other modifications can be made by those skilled in the art without departing from the spirit and scope of the invention. Therefore, they are in no way intended to limit the scope of the present disclosure, which is only limited by the express language of the accompanying claims.

What is claimed is:

1. A high-vacuum furnace for melting rectangular blocks of compacted granular material arranged in a stack at least two layers deep, the blocks in each layer being aligned in an abutting end-to-end relationship, comprising a flat, horizontal platform, arranged to support the stack of blocks with one end of said stack protruding over one end of said platform, at least one thermionic electron-emitting cathode, means for accelerating and directing electrons from said cathode onto the protruding end of said stack acting as an anode to heat and melt the same, a crucible arranged under said projecting end to receive the molten material dripping therefrom, means for moving said stack lengthwise along said platform and including an externally operable pushing rod having an end thereof contacting the stack which has a stepped surface for maintaining individual blocks in adjacent layers of said stack in overlapping relationship to feed the protruding end of the stack into the electron bombardment zone as said end melts away, a vacuum container enclosing said platform, stack, cathode and crucible, and means for continually evacuating said container to a high vacuum.

2. A method of melting and casting granular material, which comprises compacting said material into a plurality of similar rectangular blocks, placing said blocks on a platform within a vacuum tank and arranging said blocks on said platform into a stack at least two layers deep, the blocks in each layer being aligned in an unconnected abutting end-to-end relationship and the blocks in adjacent layers overlapping one another in unconnected relationship, evacuating said tank to maintain a pressure not exceeding approximately one micron of mercury, moving said stack lengthwise along said platform until one end of the stack protrudes over one end of the platform, progressively melting away the protruding end of the stack by electron bombardment, progressively advancing the stack along the platform to replace said protruding end as its melts away, catching the molten material in a crucible as it drips from said protruding end, continuously heating the molten material in the top of said crucible by electron bombardment to maintain a molten pool thereof, continuously cooling said crucible to form a continuous solid ingot beneath the molten pool at the top thereof, and continuously withdrawing said ingot from the bottom of said crucible to maintain a substantially constant level of molten metal at the top thereot.

References Cited in the file of this patent

UNITED STATES PATENTS

1,069,326	Emberg et al.	Aug. 5, 1913
1,403,955	Hill	Jan. 17, 1922
2,708,158	Smith	May 10, 1955
2,762,856	Newcomb et al.	Sept. 11, 1956
2,880,483	Hanks et al.	Apr. 7, 1959
2,963,530	Hanks et al.	Dec. 6, 1960