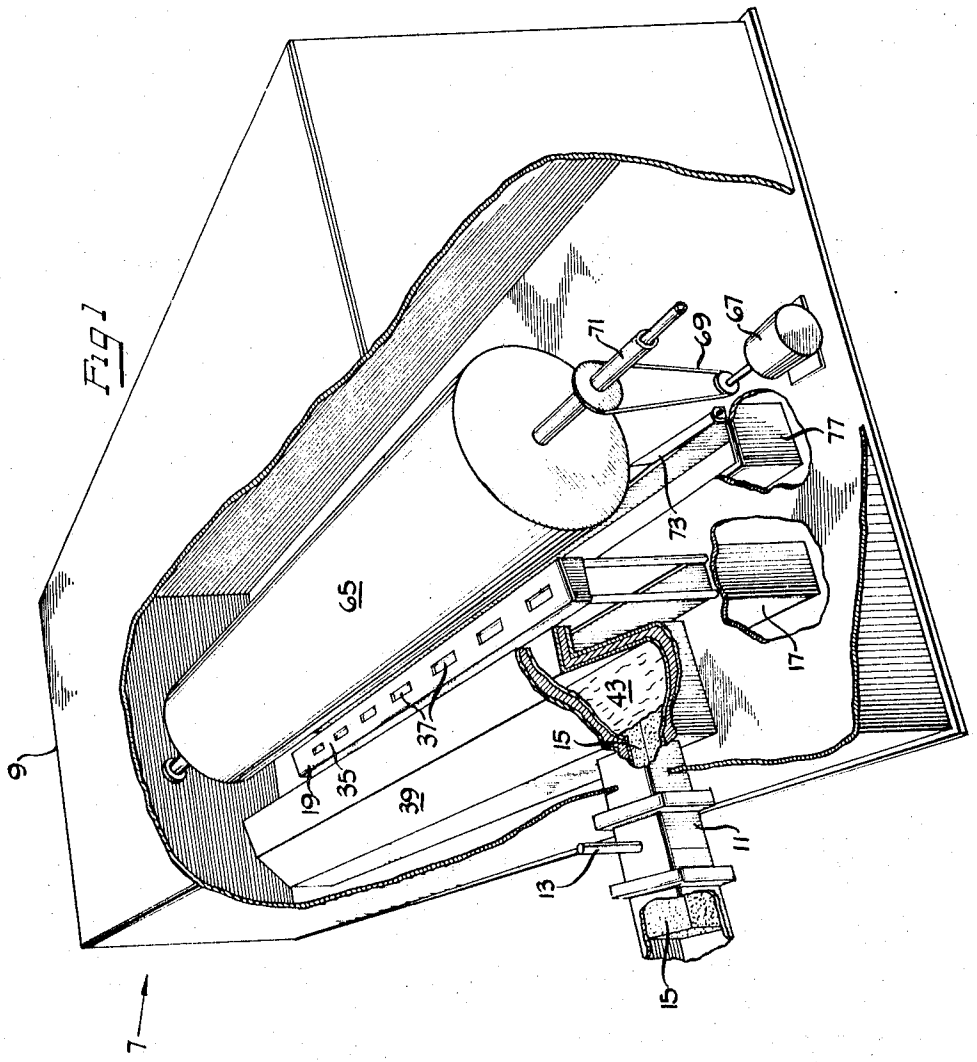
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MTR04 XR 3,364,296

Jan. 16, 1968 H. R. SMITH, JR 3,364,296

ELECTRON BEAM FURNACE

Original Filed June 12, 1963 2 Sheets-Sheet 1

Fig 1



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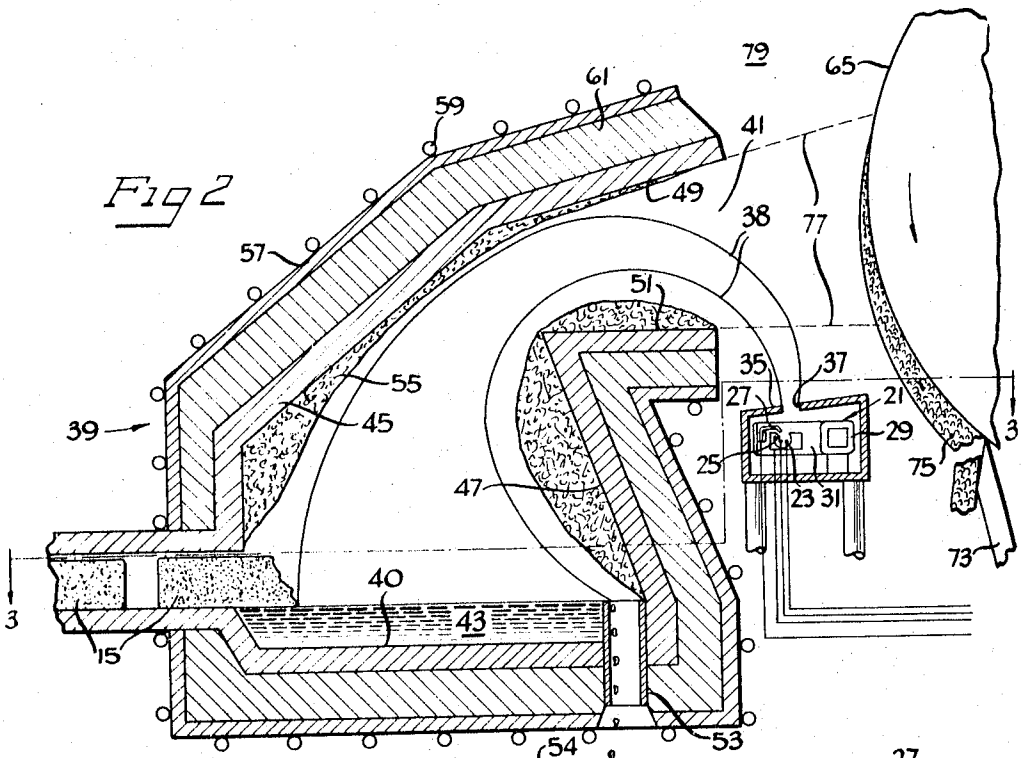
BY
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ELECTRON BEAM FURNACE

Original Filed June 12, 1963 2 Sheets-Sheet 2

Fig 2



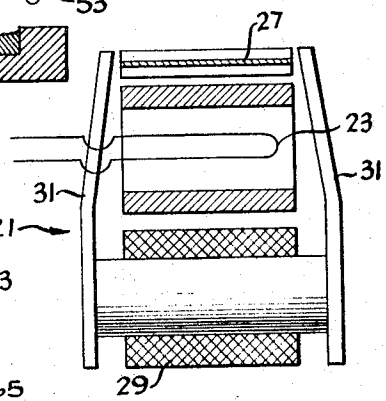


Fig 4

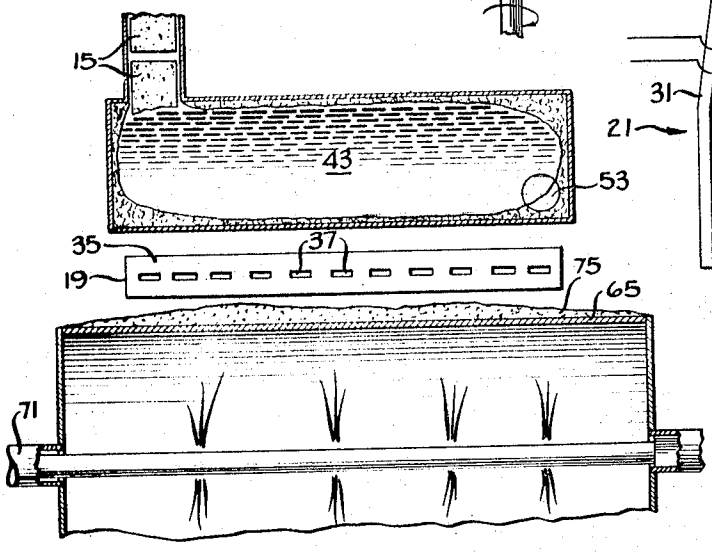


Fig 3

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United States Patent Office 3,364,296 Patented Jan. 16, 1968

3,364,296

ELECTRON BEAM FURNACE

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Original application June 12, 1963, Ser. No. 287,285, now Patent No. 3,307,936, dated Mar. 7, 1967. Divided and this application July 26, 1966, Ser. No. 596,353

2 Claims. (Cl. 13—31)

This application is a division of Ser. No. 287,285, filed June 12, 1963, now Patent No. 3,307,936 issued Mar. 7, 1967.

This invention relates to the production of relatively pure metals and metal alloys, and more particularly to an economical method and apparatus for the production of such relatively pure metals and metal alloys.

The purification of metals and alloys has long presented a problem to metallurgists; and, although processes for producing most metals and alloys have been generally set forth, these processes frequently involve apparatus and process conditions that prevent the production of particular metals and alloys at reasonable costs. Thus, the price of some metals and alloys has been so high that their use has been limited.

Technological and scientific advances of recent years have developed new uses for some of the heretofore expensive metals, if they could be produced at lower costs. In general, the new uses for these metals and alloys also require a higher degree of purity than has heretofore been possible.

Conventional processes for refining metals include electrolysis and distillation, among other techniques. Although electrolysis can be used to produce metals of fairly high purity, contamination of the metal by constitutents of the electrolyte, as well as by the materials of construction of the electrolytic cell, causes severe problems. In addition, electrolytic processes involve very high capital costs per unit of product output.

Distillation processes have also been used, but invariably involve very high operating costs because of the difficulty of supplying the required quantity of heat to the crude stock at a rate which is sufficient to cause a rapid distillation of the stock. Distillation processes are also generally disadvantageous due to the materials of construction problems associated with high temperature operations involving metals and metal compounds. Contamination of the distillate because of interaction between the melt and the container material also presents difficulty in conventional high temperature distillation operations. Generally, whenever the problem of producing a highly purified metal or alloy has been solved satisfactorily, the economics of producing the metal or alloy have been such as to seriously limit the use of these ultra pure products.

It is a principal object of the present invention to provide an economical method for the production of metals and metal alloys of high purity from low cost raw materials. It is another object of this invention to provide an economical method and apparatus for the production and purification of metals to a degree of purity which has been heretofore unobtainable except at high cost. A still further object of this invention is to provide a method for the production of pure metals and alloys which employs the use of naturally occurring ores and other available metallic compounds as starting materials. An additional object is to provide a continuous process for the production of pure metals and alloys. Yet another object of the present invention is to provide an apparatus for the production of pure metals and alloys which has a long life.

These and other objects of the invention are more

particularly set forth in the following detailed description and accompanying drawings of which:

FIGURE 1 is a partial perspective schematic view with portions broken away of one form of apparatus for carrying out the method of the present invention;

FIGURE 2 is a fragmentary, enlarged, vertical, cross sectional view of the apparatus of FIGURE 1;

FIGURE 3 is an enlarged, fragmentary horizontal cross sectional view taken along the line 3—3 of FIGURE 2; and

FIGURE 4 is an enlarged horizontal cross sectional view of one of the heating means shown in FIGURE 2.

In general, the method of the present invention comprises the vaporization of a metal or metal alloy to be produced in pure form from a carrier metal and impurities normally associated therewith, under conditions which provide very high rates of vaporization and long apparatus life. More specifically, the method includes the vaporization of a metal or metal alloy from a carrier metal and impurities normally associated therewith at a reduced pressure and at a temperature which is sufficient to vaporize the desired metal product and volatile impurities at the reduced pressure, but which is insufficient to vaporize the carrier metal and nonvolatile impurities. The carrier metal is present either as an alloying metal or as an additional material purposely added to the metal to be purified for the reasons set forth hereinafter. The vaporized highly purified metal product is recovered by condensation upon a condenser surface leaving a residue comprising the carrier metal and the nonvolatile impurities associated with the raw materials.

The production and purification of metals and metal alloys is, according to the present invention, preferably accomplished utilizing a method which includes the use of a very steep pressure gradient between two regions of the system, both of the regions being maintained at a low absolute pressure. The region of the apparatus in which the vaporization occurs is maintained at a pressure within the range of about 0.1 to 50 mm. of mercury and preferably within the range of about 5 to 10 mm. of mercury with the major portion of this pressure consisting of the pressure exerted by the vapors of the metal or alloy being vaporized. Surrounding this region of relatively higher pressure is a region of relatively lower pressure, i.e. high vacuum, of the order of 1 micron of mercury or less, preferably less than 0.1 micron of mercury.

The production of pure metals and alloys by vaporization techniques requires that the vaporization rate of the desired metals and/or alloys be substantially different from the vaporization rate of the carrier metal, nonvolatile impurities, and volatile impurities at the operating conditions. The provision of a relatively higher pressure region in which the vaporization takes place (both regions being maintained at an absolute pressure well below atmospheric pressure) allows high concentrations of energy input, as more fully described hereinafter, and the proper conditions for obtaining a well directed flow of condensible product vapor onto the condenser surface.

The process, as outlined, has been found to be particularly suitable for the purification of metals such as chromium, manganese, beryllium, calcium and aluminum, these metals generally being considered difficult to economically produce in ultra pure form. The carbides of these metals, which occur naturally in the form of ferroalloys, or which are easily made in conventional electric furnaces, have been found to be a convenient form of raw materials. Although it is difficult to recover highly purified metals from metal carbides which are in substantially pure form by vaporization techniques, when metal carbides are utilized as raw materials along with a carrier metal of, for example, iron, the method of the present invention provides a means by which the metal is readily separable

from the carbon and carrier metal and may be recovered in a highly purified form. When carbides such as those produced in an electric furnace are used as raw materials, the carrier metal should be added to the raw materials. The use of naturally occurring ferroalloys, which are essentially metal carbides dissolved in iron, as raw materials provides a suitable amount of iron carrier metal so that the ferroalloys can be purified according to the method of the present invention without the necessity of utilizing additional carrier metal. In some instances, however, additional carrier metal may be added to the ferroalloy raw material in order to provide a preferred composition in the molten bath of raw materials as set forth hereinafter. The raw materials, including the carrier metal used, can be compacted into suitable shapes for ease in handling prior to introduction into the purification apparatus, or can be placed in metal boxes or containers which become melted and form a portion of molten residue. The raw materials are preferably fed into the purification apparatus through a vacuum lock in batches so that the process can proceed continuously.

Impurities which are more volatile than the desired metal product will also be vaporized along with the metal product. These volatile impurities are eliminated from the final product by maintaining the condenser surface upon which the desired metal product is condensed at a temperature which is sufficient to condense the metal but which is insufficient to condense the volatile impurities, and by the continuing rapid and complete removal of the volatile impurities from within the purification apparatus.

The only limitation placed on metals that can be used as carrier metals is their relative volatilities at the purification pressure and temperature when compared to the metal to be purified. Economically, iron, which has a low volatility, is generally preferred, but silicon and other metals of relatively low volatility are also operable. In some instances, such as the purification of aluminum, silicon is preferable, or may be used in conjunction with iron, as it is easier to form the carbide of aluminum in the presence of silicon, than in the presence of iron.

The composition of the carrier metal employed is chosen so that a molten pool of the carrier metal and product metal carbide will be formed during the purification process without the formation of a free carbon containing slag. If the mixture of the desired metal product and carrier metal is solid at the purification pressure and temperature, or if a free carbon containing slag is allowed to form, portions of the carrier metal, carbon or other impurities may be vaporized during the purification process due to localized superheating. Superheating will occur when solids are present in the distillation region since the solid metal or carbon cannot disperse the intense heat necessary to cause vaporization of the product as rapidly as can a molten pool. It has been found that when the raw feedstock mixture is maintained in a molten state, and when the residue of the carrier metal and other impurities remains in a molten condition after the desired metal product has been vaporized, the tendency for the carrier metal and other impurities to become vaporized is greatly diminished and a highly purified product can be obtained. The composition of the raw materials is preferably adjusted in iron content to provide a byproduct pig iron residue containing 6 percent to 7 percent carbon in order to prevent the formation of a free carbon containing slag on the melt.

The temperature of the molten pool within the higher pressure vaporization region is desirably maintained substantially, i.e., 200 degrees C. to 300 degrees C., above the melting point of the raw material in order to cause the rapid vaporization of the metal product without appreciable vaporization of any impurities in the form of a solution of raw materials. Due to the elevated operating temperature, the desired product is rapidly vaporized from the raw materials as soon as the raw materials enter the vaporization region. Thus, the composition of the raw materials is altered almost instantaneously as it enters the vaporization region, resulting in the formation of a higher melting point molten mixture containing a predominant amount of carrier materials which have a low vaporization rate. The low vaporization rate carrier materials are not appreciably vaporized at the operating conditions and thus do not contaminate the condensate product.

For example, the raw material feedstock for the production of manganese or chromium from a ferroalloy has a melting point of approximately 1350 degrees C. to 1400 degrees C. However, the average temperature of the molten pool within the higher pressure zone is preferably maintained at approximately 1600 degrees to 1700 degrees C., which is the melting point of pig iron containing 6 percent to 7 percent of carbon. The volatile product vaporizes from the raw material and carrier metal feedstock so rapidly that there is no chance for the molten pool to remain at the melting point of the original raw material mixture. Additionally, there is insignificant vaporization of the remaining low vaporization rate carrier materials from the raw materials solution which would contaminate the condensed purified product.

The only operable apparatus for economically carrying out the described process to the high degree of purity contemplated is an electron beam furnace, one form of which is shown in the attached drawings. An electron beam furnace can be made to operate at pressures of one-tenth of one micron of mercury or less and has the desirable characteristics of a complete absence of a reactive atmosphere and the absence of the introduction of any impurities into the metal to be purified by the electron beam during the purification process.

Referring now to the drawings, there is shown in FIGURE 1 a preferred form of an electron beam furnace **7**. The furnace **7** has a vacuum tight housing **9** and is formed with a vacuum lock inlet **11**, which is connected by pipe **13** to suitable pumps, not shown. A raw material feedstock **15** enters the furnace **7** through the vacuum lock inlet **11**. The furnace **7** is maintained under a high vacuum by one or more conventional diffusion pumps connected to the manifold **17** located in the bottom wall of housing **9**. The pumps have a high volumetric capacity and can be adjusted to provide any desired vacuum within the furnace **7**. The vacuum diffusion pumps handle only the volatile noncondensable impurities evolved during the distillation process, such as carbon monoxide, oxygen, and nitrogen, the large bulk of the vapor generated in the high pressure region during the distillation being condensed on the condenser substrate without affecting the overall pressure in the high vacuum region. The continuous withdrawal of the noncondensable gases from within the distillation apparatus by the vacuum pumps maintains the system at a satisfactory equilibrium pressure at the operating conditions.

Disposed directly above the diffusion pump manifold **17** is an electron beam gun housing **19** in which are housed a plurality of electron beam guns **21**. In order to economically produce metals and metal alloys by the above outlined process, the process must proceed via a rapid vaporization of the desired product from the feedstock. The rapid vaporization of the feedstock results in the formation of a high local pressure of distillate vapor surrounding the surface of the vaporizing feedstock. Electron beam heating provides the means for supplying heat at the required rates for rapid vaporization on a highly economic basis. The electron beam energy is particularly efficient since it is transferred directly to the interface of the feedstock where the phase change from solid to vapor or liquid to vapor is occurring. In order to operate at maximum efficiency, the electron beam generating apparatus must be maintained within the region of lower pressure, e.g., high vacuum, which is provided by the vacuum diffusion pumps and as shown in FIGURE 1, the electron guns are preferably located directly above the

diffusion pumps manifold 17 to provide a maximum vacuum.

One form of available electron gun which is particularly suitable for use in the electron beam furnace is illustrated by FIGURES 2 and 4. The electron gun 21 comprises an elongated emissive filamentary cathode 23, a cathode focusing structure 25 and an anode 27. The cathode 23 may be in the form of an elongated rod or hair-pin and is designed to project a wide and narrow electron beam. An electron focusing magnet including a coil 29 and pole pieces 31 straddles each of the electron guns as generally shown in FIGURE 4. A nonmagnetic protective shield 35 forms the upper wall of housing 19 to shield the electron guns from ions liberated during the purification process which might cause shorting of the guns. The shield 35 contains narrow slits 37 through which the electron beam is caused to pass by the focusing magnet pole pieces 31. The cathode 23 is heated to an electron emissive temperature by the passage of current therethrough and the emitted electrons are attracted by the anode 27 which is maintained at a positive potential with respect to the cathode. The attracted electrons are focused into a beam by the magnetic field established in the gap between the pole pieces 31. The beam, indicated generally by 38, is caused to form a curve and is compressed into a circle by causing a barrel shaped magnetic field to be established between the pole pieces 31. The shape of the electron beam may be adjusted by adjusting the field of the magnets.

Normally, the raw material feedstock 15 contains noncondensible volatile impurities as well as the desired volatile metal and metal alloys in impure form. The melting and vaporization of the feedstock that occurs as the electron beam 38 strikes the feedstock is accompanied by a great deal of physical splatter of the material being processed. The splatter impurities, i.e., carrier metal, carbon, etc., must not be allowed to strike the substrate upon which the desired product of high purity is being condensed. Because the splatter is molten, it does not rebound from the surfaces which it hits, but rather sticks to these surfaces and builds up a thick deposit.

In order to prevent the splatter from striking the condenser substrate, the vaporization zone is preferably maintained within an enclosure 39 which is provided with an exit 41 through which the product vapors exit from the enclosure 39 and through which the electron beams 38 enter the enclosure 39. The enclosure 39 is disposed within the housing 9 adjacent to the electron beam housing 19 and generally defines the high pressure region of the furnace 7. The low pressure region generally surrounds enclosure 39. The raw materials 15 which enter the furnace 7 through the vacuum lock inlet 11 enter the lower portion of enclosure 39, as seen in FIGURE 2, and are directed into a hearth 40. The hearth 40 may be cooled, as by water, if desired, causing a liner of solidified raw material to be formed on the surface of the hearth. Alternately, the hearth need not be cooled when it is constructed from a material such as graphite which is not vaporized at the operating conditions. The molten pool is saturated with carbon present in the raw materials and is not contaminated by solution of the portion of the graphite hearth. The barrel shaped magnetic field causes the electron beams 38 to enter the enclosure 39 through exit 41, curve downwardly within the enclosure and strike the hearth 40. The electron beams thus impinge against the raw materials 15 and melt the raw materials to form a molten bath 43 within the hearth 40.

The enclosure 39 is shaped so that it is relatively difficult for any splatter to strike the surface of the condenser substrate, preferably by forming the enclosure and exit so that the vapors will have to travel an arcuate path to escape from the enclosure. Additionally, the enclosure 39 is generally shaped to correspond to the shape of the curving focused electron beam entering the enclosure through the exit 41. In connection with this, enclosure 39 is generally arcuate in shape having converging side walls 45 and 47 which terminate in diverging walls 49 and 51 adjacent exit 41 to form a restriction therein. As can be seen in FIGURE 2, the exit 41 from the enclosure 39 is angularly displaced above the hearth 40 and vapors escaping from the molten bath in the hearth 40 must travel a generally arcuate path to escape from enclosure 39. An outlet weir 53 is maintained adjacent one corner of the hearth 40, forming an exit therefrom for the residue which is not vaporized by the action of the electron beams. The residue flows over the weir 53 and falls into an intermittently rotated ingot mold 54 positioned below weir 53. The mold 54 is cooled by suitable means, not shown, and the solidified residue is withdrawn from the mold 54 through a conventional vacuum lock, not shown.

As previously explained, the rapid vaporization of the product causes a portion of the molten bath to be thrown out of the bath. The liquid droplets strike the relatively cooler walls 45, 47, 49 and 51 of enclosure 39 and form a solidified splatter layer 55. The splatter 55 builds up until it enters the electron beam envelope 38 whereupon it is melted and flows back into the hearth.

The enclosure 39 can be constructed from any suitable material which can withstand the operating conditions of the furnace. In this connection, the walls of the enclosure 39 may be made from graphite or water cooled copper. The use of graphite provides an increased heat efficiency compared to the water cooled copper wall. However, the splatter layer 55 which builds up on the walls of the enclosure is a good insulator and when used in conjunction with a water cooled copper wall provides thermal characteristics closely approaching that of graphite. The use of water cooled copper walls has the advantage of eliminating the problems of erosion of the walls. However, when a thick deposit of splatter builds up on graphite walls the walls will not be eroded and graphite can safely be employed. It can be seen that the particular material chosen as the structural wall material is purely a matter of choice. In the hearth shown in FIGURE 2 the walls of the enclosure 39 may include a water cooled shell 57 which is cooled by water which is passed through coil 59. A layer of heat insulating material 61, i.e., asbestos, is provided between the water cooled jacket 57 and the inner wall 45 which is constructed of graphite.

The exit 41 from the enclosure is desirably closely adjacent to a condenser substrate 65 so that all of the product vapors passing through the exit 41 will impinge against the surface of the condenser and will not escape into the low pressure region of the furnace surrounding the enclosure 39.

The condenser substrate 65 may be a rotating drum, driven by a suitable motor 67 and pulley 69. Alternately, the condenser may be in the form of moving strip or sheet. The substrate 65, which may be constructed of any suitable material, is, by any suitable means, such as a coolant gas or liquid introduced into the interior of the drum and withdrawn from the drum through concentric pipes 71, maintained at a temperature which is low enough to condense the metal product vapors on its surface but which is high enough not to condense any appreciable amount of the volatile impurities, such as sulphur, which may be present in the raw material feedstock. A scraper blade 73, or other suitable mechanical apparatus, is maintained adjacent the condenser surface 65 to mechanically remove the condensed metal from the surface of the condenser. The condensed metal 75 is removed from the surface of the condenser 65 in the form of flakes or chips which fall into a conventional vacuum outlet 77 which is positioned below scraper blade 73 and permits periodic removal of the condensate from the furnace.

The enclosure 39 surrounding the vaporization region of the furnace 7 generally confines the high pressure re-

gion of the apparatus to the area directly surrounding the molten feedstock. By confining the righ pressure region in such a manner, the pressure in the space between the surface of the molten pool **43** and the surface of the substrate **65** adjacent the exit **41** from the enclosure **39** is substantially that exerted by the vaporizing metal or alloy. The metal vapors flow out of the enclosure **39** through exit **41** in the form of a stream **77** which impinges against the surface of condenser **65**. The stream of vapors **77** is at substantially the same pressure as that established within the enclosure. The low pressure region **79** completely surrounds the stream **77** at the point where it strikes the condenser **65** so that the condensation of the product takes place adjacent the boundary between the vapor stream **77** which is at the relatively high pressure and the relatively low pressure within the region **79**. The large pressure gradient adjacent the point where the vapors condense causes the noncondensible vapors to be drawn into the low pressure region **79** and thus does not interfere with the rapid flow of product vapor through the exit **41** of the enclosure and against the condenser surface.

The use of an electron beam furnace which includes a region of relatively high pressure surrounding the raw materials being vaporized and a region of relatively low pressure surrounding the electron beam gun and the area of the condenser substrate where the vapors are condensed, in which there is an abrupt pressure gradient between the two pressures greatly increases the economics of the process. The pressure differential causes a rapid flow of metal vapors from the vaporization region into the condenser region increasing the amount of product obtained per unit of time. Further, the low pressure adjacent the electron beam guns causes them to operate at a higher efficiency, conserving electrical power.

It can be seen that an electron beam furnace overcomes the serious disadvantages of both the arc and induction furnaces which are believed not to be operable in the production of metals in the economical manner and to the degree of purity contemplated by the present invention. The electron beam furnace does not employ an electric arc which necessarily entails localized superheating both at the surface of the electrode and at the surface of the feedstock with the resultant vaporization of impurities from these surfaces. The electron beam furnace does not require the use of a nonconducting container for the molten feedstock as is necessary in induction furnaces, i.e., ceramics, which react with the molten feedstock at pressures below about 10 microns of Hg.

It has been found that some metals, such as aluminum and chromium, cannot be produced in a highly purified state in a single pass of the raw materials through a purification apparatus, such as the electron beam furnace described above. The relative volatilities of these metals and the respective carrier metals, e.g., iron and silicon, used in their purification are sufficiently close at the operating temperature and pressure that it is advantageous to perform the purification in a plurality of steps. In these instances a portion of the desired metal product is vaporized from the raw materials in a first pass through the furnace. The residue is then recycled and passed through the furnace a second, and if desired, a third time, etc., each pass through the furnace producing a product containing a higher degree of impurities than the product produced by previous passes through the furnace. The product obtained from the second and third passes through the electron beam furnace can then be further purified in the furnace to produce a product in a highly purified state.

It is also possible to further purify a distilled product obtained from the electron beam furnace by mixing the product with a further amount of carrier metal and revaporizing it in the furnace to obtain a further purified product. Likewise, products of varying purity may be obtained from a single batch of raw material by operating the furnace at varying temperatures and obtaining fractions of purified products in much the same manner as a conventional liquid distillation still.

In some instances an alloy is the desired product. When the desired alloy consists of a metal which can be purified by the process as described, and a metal which has a volatility such that it might be employed as a carrier metal, the alloy can be produced by the purification process herein described. Alloys of the desired metal containing silicon with extremely low carbon content are desirable and can be produced by conducting the purification in a manner to cause a specific portion of the carrier metal to be vaporized and carried over onto the condenser surface. The amount of carrier metal carried over into the alloy product is affected by the relative volatilities of the metals at the operating conditions as well as their relative concentrations within the furnace. At steady state conditions, that is, continuous feed of raw materials and continuous withdrawal of residue, the composition of the alloy vapor is affected by the relative amount of total material vaporized as compared to the amount of feed to the furnace. Although it is a complex function of many variables, in general, if more carrier metal is desired in the alloy product, a greater fraction of the raw material feedstock is allowed to vaporize by increasing the power input of the electron beam apparatus.

In some instances the feedstock of raw materials and carrier metals will have a composition such that the feedstock can be melted to form a molten pool without the vaporization of any significant amount of the desired product and without splattering of the residue. In these instances, an enclosure surrounding the distillation zone of high pressure is not required, although it is preferred to restrict the high pressure region to that area directly adjacent to the molten bath. It is possible to carry out the process in two stages, a first premelting stage in one section of an electron beam furnace where the feedstock is melted at a relatively high absolute pressure of about 50 microns of Hg; followed by vaporization of the product in a second stage surrounded by a region maintained at a low absolute pressure of less than 0.1 micron of Hg.

Various features of the process as described are set forth in the following examples.

Example I

High purity manganese is produced from a ferromanganese ore, containing about 35 percent manganese, or about one-half the amount found in commercial ferromanganese alloys. The alloy ore is charged batchwise in lump or powder form through a vacuum lock into a graphite crucible in an enclosed distillation region of an electron beam furnace. The ferromanganese alloy is heated by focused electron beams which are adjusted so that a temperature of from 1600 degrees to 1700 degrees C. is obtained within the graphite crucible. The pressure in the region surrounding the electron beam apparatus is maintained at 0.05 micron of mercury. The pressure within the vaporization region is maintained at 1 to 5 mm. of mercury. The alloy ore melts to form a molten pool in the vaporization region accompanied by rapid vaporization of manganese. The enclosure surrounding the vaporization region is shaped with an angular bend therein so as to prevent splatter of the molten residue onto a moving condenser surface positioned adjacent the exit from the enclosure and within the high vacuum region. The electron beam envelope melts the splatter that is solidified on the walls of the enclosure surrounding the vaporization region, preventing the accumulation of an excessive deposit of splatter.

The manganese vapor escaping through the exit in the enclosure surrounding the vaporization region is condensed on a rotating condenser surface positioned adjacent the exit. The condenser surface is maintained at a temperature within the range of from about 400 degrees to 500 degrees C. The temperature of the rotating condenser surface is adjusted to prevent condensation of the sulphur and

other highly volatile impurities present in the ferro-manganese ore.

A three way separation and purification is effected, the highly purified manganese being separated from both the more volatile impurities, such as sulphur, which do not condense at 400 degrees to 500 degrees C. and from the carbon and iron which do not volatilize at significant rates when compared to the rate of vaporization of manganese at a temperature of from 1600 degrees to 1700 degrees C.

The condensed purified manganese metal is scraped from the rotating condenser by a doctor blade and is removed from the furnace batchwise through a vacuum lock. The manganese so obtained contains less than 100 p.p.m. each of sulphur, phosphorous, oxygen, carbon and nitrogen and less than 0.5 percent of iron.

Example 2

Highly purified calcium is prepared from crude calcium carbide in a manner similar to that as set forth in Example 1. The calicum carbide raw material is fed into the electron beam furnace along with a sufficient amount of crude iron and/or scrap steel carrier metal to maintain a molten residue, and the calcium is evaporated from the calcium carbide at a temperature of from 1600 degrees to 1700 degrees C. The iron or steel is used as a low cost carrier metal for the byproduct carbon separated from the calcium, the iron being converted to a high carbon pig iron. The iron carrier metal, which is maintained in a molten state, provides a molten pool which prevents localized super-heating and hot spots on the calcium carbide which, if in the solid form, would cause the vaporization of carbon as a contaminate.

The calcium is collected from the rotating condenser in the same manner as was the manganese of Example 1 and a product is obtained which analyzes less than 100 p.p.m. each of sulphur, phosphorous, oxygen, carbon and nitrogen and less than 0.1 percent of iron.

Example 3

Highly purified aluminum containing silicon and small amounts of iron is produced from a solid solution of aluminum carbide dispersed in a combined iron and silicon carrier metal. The solid solution was prepared in an electric furnace from conventional aluminum ores, such as low grade clays and commercial ferro-silicon metal.

The pressure within the low pressure region of an electron beam furnace is reduced to less than 0.1 micron of mercury by diffusion pumps and the electron beams are adjusted to produce a molten pool of aluminum carbide, iron and silicon at a temperature of 1700 degrees to 1800 degrees C. in a vaporization region within the furnace which is maintained at a pressure of 1–5 mm. of mercury. The aluminum metal distills from the silicon and carbon residue as a vapor and is condensed on a rotating condenser maintained at a temperature of 400 degrees C. in a manner as described in the preceding examples.

In order to produce aluminum of commercial purity the entire amount of aluminum is not removed from the aluminum carbide in a single pass through the furnace and a portion of the silicon and iron carrier metal residue containing undistilled aluminum is recycled to the electric furnace for the formation of a further amount of aluminum carbide and returned to the electron beam furnace. Approximately one-half of the amount of aluminum present in the feed material remains in the silicon and iron carrier metal which is recycled, and, depending upon the nonvolatile impurities present in the ore, a definite fraction of the recycled carrier metal is discarded to prevent excessive buildup of nonvolatile impurities within the carrier metal.

A purified aluminum alloy is recovered from the rotating condenser surface which analyzes less than 100 p.p.m. each of sulphur, phosphorous and carbon. The silicon content of the alloy is approximately 10 percent by weight and the iron content is 1 to 2 percent by weight.

Example 4

Low carbon content chromium containing 2 to 20 percent of iron is prepared in the electron beam furnace from a high carbon ferro-chrome alloy, containing about 35 percent chromium, or about one-half the amount present in commercial ferro-chrome. The low grade ferro-chrome was made directly from low grade North American ores, as opposed to the African ores, which are presently employed to produce commercial grade ferro-chrome alloys.

The ferro-chrome alloy is charged to an electron beam furnace in the same manner as previously described. The pressure within the vaporization region of the furnace is maintained at 1–5 mm. of mercury and the pressure in the vacuum region surrounding the vaporization region is maintained at 0.1 micron of mercury. The electron beam apparatus is adjusted so that a temperature of from 1600 degrees C. to 1700 degrees C. is maintained within the molten pool in the hearth. The relative volatilities of iron and chromium causes a measurable amount of iron to be vaporized simultaneously with the chromium, the relative amount of iron vaporized depending upon the residual concentration of chromium desired in the molten residue. The chromium and iron vapors are condensed on a rotating drum in the manner as previously described, the chromium product having the same low levels of impurities as did the manganese obtained in Example 1 and from 2 to 3 percent of iron.

Since the commercial value of chromium increases as the iron content decreases, the residue containing from 10 percent to 25 percent chromium is recycled through the furnace a second time, yielding a product having from 10 to 20 percent of iron and a residue containing less than 2 percent chromium. The product of each pass analyzes less than 100 p.p.m. of sulphur, phosphorous, carbon, oxygen and nitrogen. The chromium product obtained from either pass through the electron beam furnace can be further purified by subsequent treatment in the furnace, if desired, or can be utilized as is.

Example 5

The low iron chromium product of Example 4, procured from the first pass of the ferro-chrome alloy through the electron beam furnace, is remelted and subjected to two more passes through the furnace in the manner as described in the previous examples, the residue in each case being discarded. A chromium product analyzing less than 0.2 percent iron is obtained.

Example 6

Beryllium was produced in an electron beam furnace from beryllium carbide and iron carrier metal in a manner identical to that described in Example 2. Since beryllium is not as volatile as calcium, the product obtained was reprocessed through the furnace a second and third time to produce a high purity beryllium metal having less than 100 p.p.m. each of sulphur, phosphorous, oxygen, carbon and nitrogen and less than 0.2 percent of iron.

It can be seen that a process has been provided which produces metals in a higher degree of purity than has heretofore been obtainable and does so in a continuous process which is both economical and convenient. It is understood that other metals whose partial pressures at high vacuum are such that they can be separated from impurities and carrier metals by vaporization thereof are also operable and within the scope of the invention. The metals need not be in the form of carbides, as other metal compounds, such as borides and silicides, can also be separated by distillation and can, therefore, be used in place of metal carbides.

Various of the features of the present invention are set forth in the following claims.

What is claimed is:

1. An electron beam furnace for the production of highly purified metals comprising, a vacuum tight housing, means creating a vacuum within said housing, a hearth within said housing, means for admitting raw materials into said hearth, an enclosure having an exit therein surrounding said hearth, electron beam heating means disposed within said housing adjacent said exit, said electron beam heating means adapted to project a beam of electrons through said exit and onto said hearth, a condenser positioned adjacent said exit for condensing metal vapors, said exit being positioned in said enclosure out of the line of sight of said hearth.

2. An electron beam furnace for the production of highly purified metals comprising, a vacuum tight housing, a hearth within said housing, an enclosure having an exit therein surrounding said hearth, a condenser positioned adjacent said exit, said enclosure and the space between said exit and said condenser defining a first region of reduced pressure within said housing, means for admitting raw materials into said hearth, means for creating a vacuum within said housing, electron beam heating means disposed adjacent said exit and adapted to project a beam of electrons through said exit onto said hearth, said vacuum means establishing a second region of reduced pressure substantially surrounding said first region and having a lower pressure than said first region so that a pressure gradient occurs between said first region and said second region adjacent said exit, said exit being positioned in said enclosure in relation to said hearth so that the metal vapors are required to travel a non-linear path from said hearth to said exit.

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