

March 31, 1970

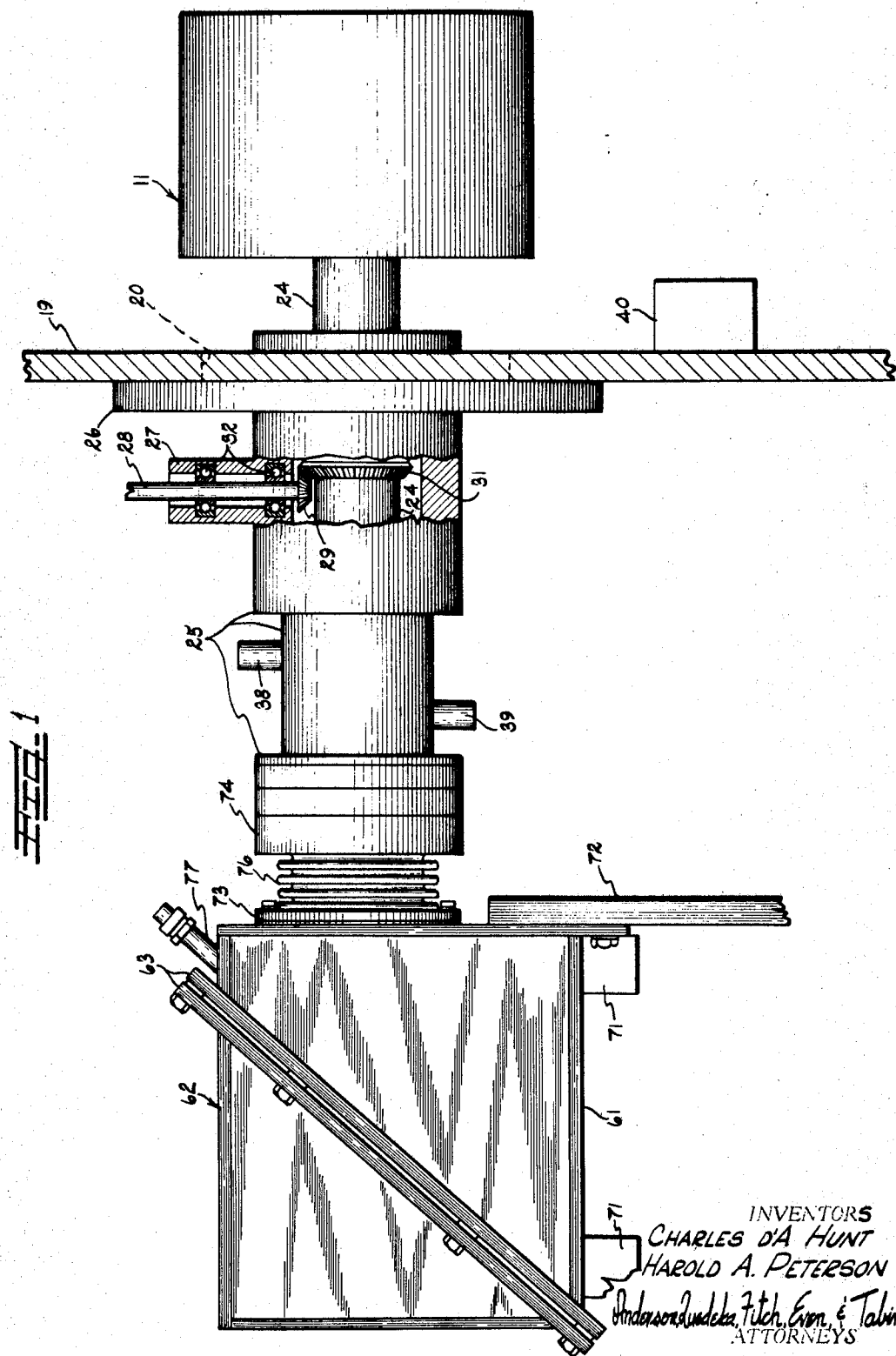
C. A'D. HUNT ET AL

3,504,094

METHOD AND APPARATUS FOR FEEDING PARTICULATE MATERIAL
TO A ROTATING VACUUM VAPORIZATION CRUCIBLE

Filed Oct. 31, 1966

4 Sheets-Sheet 1



INVENTORS

CHARLES D'A HUNT

HAROLD A. PETERSON

Anderson Lundgren, Fitch, Egan & Tabin
ATTORNEYS

March 31, 1970

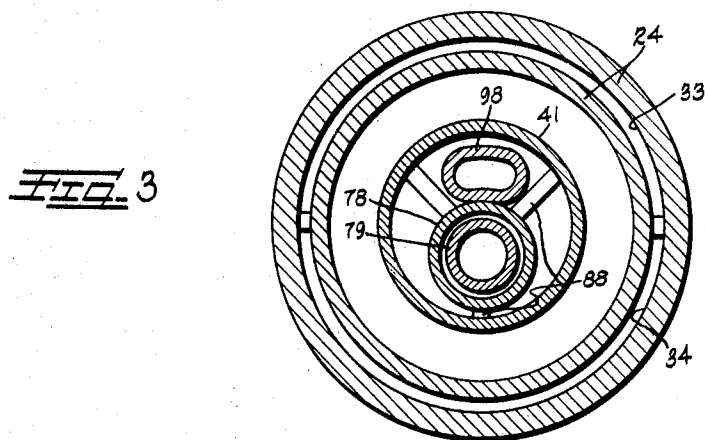
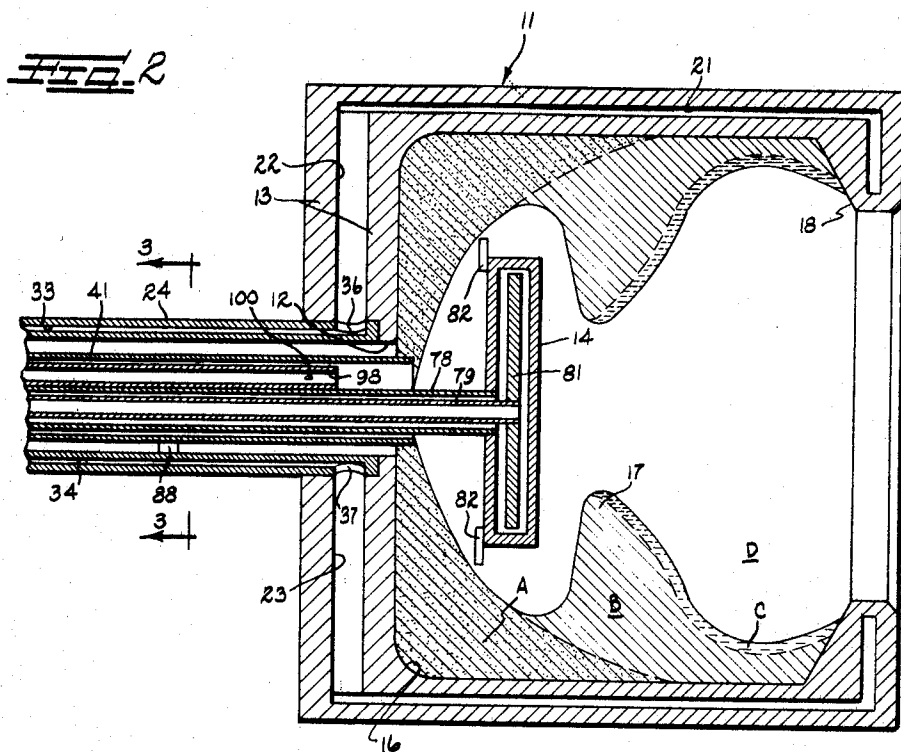
C. A'D. HUNT ET AL

3,504,094

METHOD AND APPARATUS FOR FEEDING PARTICULATE MATERIAL
TO A ROTATING VACUUM VAPORIZATION CRUCIBLE

Filed Oct. 31, 1966

4 Sheets-Sheet 2



INVENTORS
CHARLES D'A HUNT
HAROLD A. PETERSON

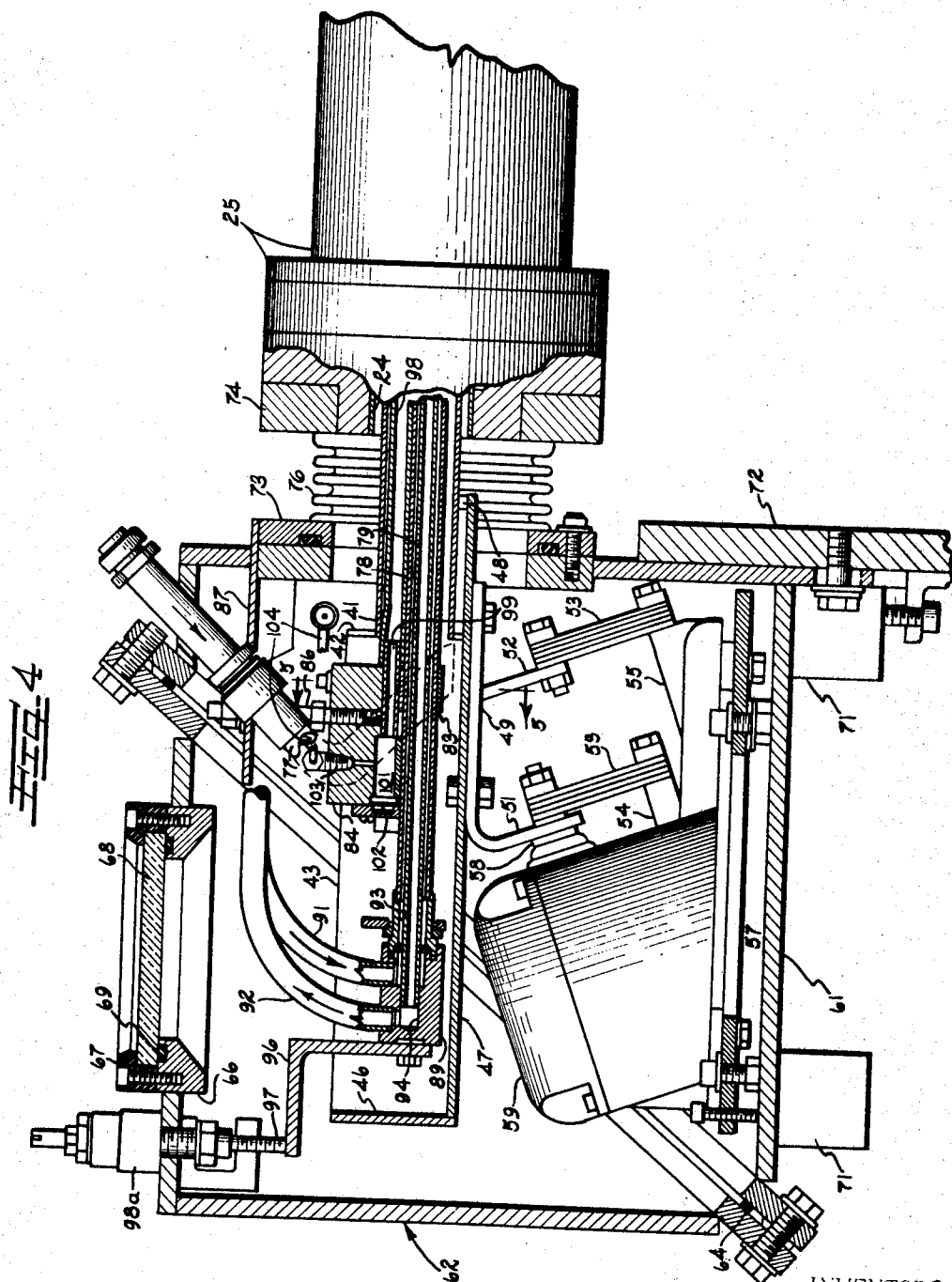
by Anderson, Quedeka, Fitch, Eron, & Talvin ATTORNEYS

March 31, 1970

C. A'D. HUNT ET AL
 METHOD AND APPARATUS FOR FEEDING PARTICULATE MATERIAL
 TO A ROTATING VACUUM VAPORIZATION CRUCIBLE
 Filed Oct. 31, 1966

3,504,094

4 Sheets-Sheet 3



INVENTORS
 CHARLES D'A HUNT
 HAROLD A. PETERSON

BY Anderson, Quedeka, Fitch, Eron, & Talvin ATTORNEYS

March 31, 1970

C. A'D. HUNT ET AL

3,504,094

METHOD AND APPARATUS FOR FEEDING PARTICULATE MATERIAL
TO A ROTATING VACUUM VAPORIZATION CRUCIBLE

Filed Oct. 31, 1966

4 Sheets-Sheet 4

FIG. 5

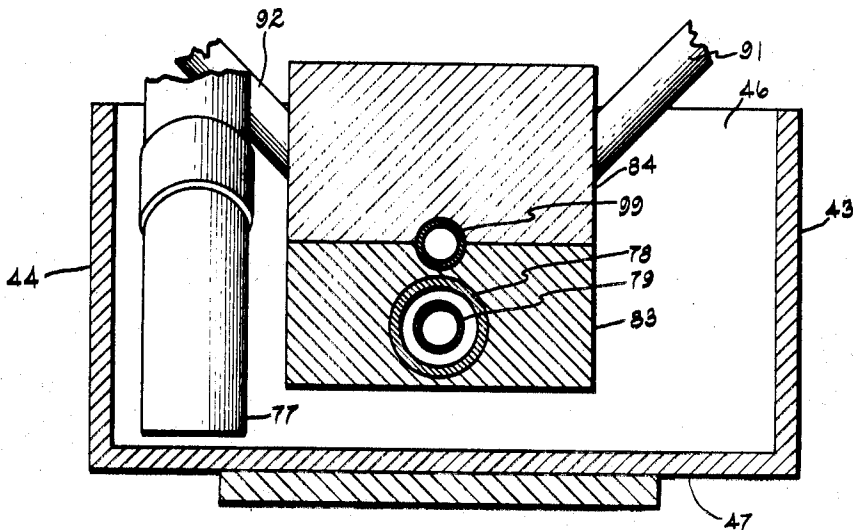
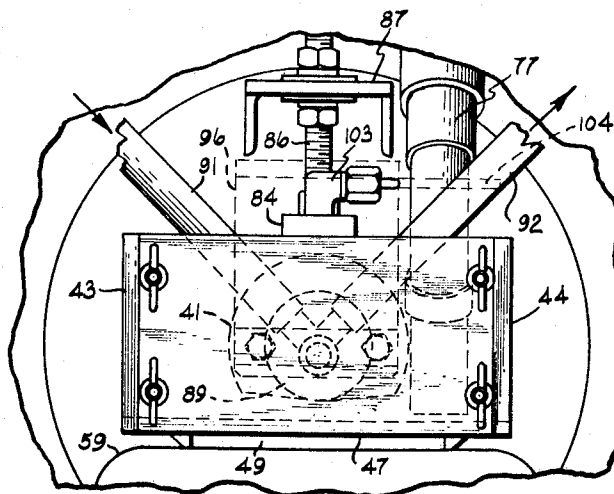


FIG. 6



INVENTORS
CHARLES D'A HUNT
HAROLD A. PETERSON
BY *Anderson, Ruedeka, Fitch, Even, & Talvin*
ATTORNEYS

1

3,504,094

METHOD AND APPARATUS FOR FEEDING PARTICULATE MATERIAL TO A ROTATING VACUUM VAPORIZATION CRUCIBLE

Charles d'A. Hunt and Harold A. Peterson, Orinda, Calif., assignors, by mesne assignments, to Air Reduction Company, Incorporated, a corporation of New York

Filed Oct. 31, 1966, Ser. No. 590,656

Int. Cl. H05b 7/18

U.S. Cl. 13—31

16 Claims

ABSTRACT OF THE DISCLOSURE

A method and apparatus for feeding particulate material into a rotating vapor source crucible in a vacuum deposition system is described. The material is introduced through an axial conduit in the crucible. The material disposed along the cylindrical wall of the crucible is melted and vaporized by an electron beam. A vapor barrier is positioned near the opening of the conduit into the crucible to prevent vapor from entering the conduit. The barrier may be comprised of a shield and an annular projection formed in the material itself.

This invention relates to the feeding of material into vapor source crucibles in vacuum deposition systems. More particularly, the invention relates to an improved method and apparatus for feeding particulate material into a vapor source crucible in such a system to replenish material vaporized in the crucible.

Vacuum deposition systems generally involve the condensation of a vapor of one material on a substrate of another material, performed in a relatively high vacuum environment. The materials may be of various types, such as metals and ceramics, and the substrate may be of any of a variety of thicknesses. The process may operate on a substrate comprising a continuously moving film, or the substrate may be of a more discrete form.

In a vacuum deposition system, the vapor is often produced by utilizing a crucible containing charge material which is melted and vaporized by high energy electron beams directed into the crucible and against the charge material. The production of vapor by such means provides good control over the thickness, density, and uniformity of the deposited vapor and also facilitates efficient utilization of materials.

For extended periods of operation, it is necessary to replenish the charge material which is vaporized in the crucible. The particular form or state of the replenishing material when introduced to the crucible depends upon such considerations as the ease of feeding, the melting characteristics, the purity, and the cost. For example, in connection with the vaporization of silica, replenishing material in the form of a solid fused silica rod has sometimes been utilized. Silica in this form is relatively easy to feed, but tends to be considerably more expensive than silica in the particulate form (i.e. sand). The feeding of particulate material, however, presents more difficulties in introducing the material to the crucible than does the fused rod form, especially if a substantially continuous feed of replenishing material is desired.

Several methods of material replenishment or feeding have heretofore been developed in order to take advantage of the lower cost of many materials, including silica, in the particulate form. One method of material replenishment or feeding using particulate material is by feeding the particulate charge material into the crucible through the mouth or open end thereof. This may be accomplished by batch feeding at intervals depending upon the

2

vaporization rate and the amount of each batch, or by a relatively continuous feed using a suitable conveyor. The former is not sufficiently sophisticated for many operations in that it involves interruption of the vapor deposition process in order to place the material in the crucible. Difficulties have been encountered in achieving relatively continuous feeding through the crucible mouth in that the conveyor apparatus may interfere with the vapor beam emanating from the mouth of the crucible and become heavily coated with condensate. Moreover, the high heat and vapor in the crucible may cause melting or fusing of the particulate material while still in the feeding apparatus, causing it to clog or jam and preventing it from reaching the crucible.

Under some circumstances, the nature the crucible may make the solution of material replenishment problems even more difficult. This is particularly true in certain vacuum deposition systems wherein the vapor source crucibles are cylindrical and rotate about substantially horizontal axes. Such a rotating crucible has particular advantage in coating vertically disposed substrates because the vapor beam emitted from the crucible moves in a generally horizontal path. Two or more of such crucibles may be arranged in banks for coating particularly large substrates. The reason for rotating the crucible is twofold. First, the molten charge material is distributed about the wall of the crucible by centrifugal force. The provides a symmetrical or uniform vapor beam of even density moving out of the crucible mouth. Second, although the electron beams may impinge upon only a small portion of the molten charge along the wall of the crucible at any given time, by rotating the crucible, all portions of the charge are under the electron beam the same length of time for each revolution. This effects a substantially uniform transfer of energy from the beam throughout the charge. In addition to being subject to the same problems discussed above in connection with crucible feeding generally, the motion of a rotating crucible and the fact of its substantially horizontal axial disposition present considerable difficulty in achieving an even feed of particulate material. An even feed is desirable to avoid such variations in the operating conditions in the crucible as produce excessive fluctuation in the density of the vapor beam emitted from the crucible.

It is, therefore, an object of this invention to provide an improved method and apparatus for feeding particulate material into a rotating crucible wherein the axis of rotation is substantially horizontal.

A more general object of this invention is to provide an improved method and apparatus for feeding particulate material into a vapor source crucible.

Another object of the invention is to provide a method and apparatus for feeding particulate material into a vapor source crucible other than through the open end of the crucible.

A further object of the invention is to provide an improved method and apparatus for feeding charge material in particulate form into a rotating crucible through a hollow drive shaft therefor.

Other objects and the various features of the invention will become apparent to those skilled in the art from the following description taken in connection with the accompanying drawings wherein:

FIGURE 1 is an elevational view, with part broken out, of a vapor source assembly incorporating the invention;

FIGURE 2 is an enlarged full section view of the crucible and associated portions of the assembly of FIGURE 1 and includes a schematic diagram of a cross section of the crucible contents;

FIGURE 3 is a sectional view taken along the line 3—3 of FIGURE 2;

FIGURE 4 is an enlarged full section view of a further portion of the vapor source assembly of FIGURE 1;

FIGURE 5 is a sectional view taken along the line 5—5 of FIGURE 4; and

FIGURE 6 is a rear elevational view of the portion of the apparatus shown in FIGURE 4 with parts thereof in phantom.

The invention provides a method and apparatus for feeding particulate material to be heated into a cylindrical vapor source crucible 11, which is rotating about its axis, in a vacuum deposition system. The material is fed into the crucible through an axial opening 12 in the closed end 13 of the cylindrical crucible. A beam of electrons is directed against the surface of the material in the crucible to cause melting and vaporization of the material at the surface thereof. A barrier 14 and 17 is formed in the crucible over the opening in the closed end thereof to prevent vapor in the crucible from entering the opening. This permits the flow of particulate material through the opening from becoming blocked due to condensed vapor causing fusion of the particulate material or clogging of the opening, or both. The barrier also helps prevent excessive radiation of heat to the particulate material flowing through the opening that might cause fusion of the material. The barrier is at least partially formed by the material in the crucible, which tends to build up an annular projection 17 extending, from the material along the cylindrical crucible wall, toward the axis of the crucible. The barrier may be partially formed by a shield 14 mounted in the crucible spaced from the opening, with the annular projection overlapping the periphery of the shield.

The apparatus of the invention comprises a conduit 41 having one end communicating with the interior of the crucible through the axial opening 12 in the closed end 13 thereof. Means 42, 43, 44, 46, 47 and 77 are provided for introducing particulate material to the conduit and a vibrator device 59 is connected to the conduit for producing vibrations to effect an even flow of such particulate material through the conduit into the crucible. In one form of the invention, the conduit extends through the hollow interior of a drive shaft 24 which rotates the crucible 11. The conduit is cantilevered from a support structure 42, 47, 48 and 49 and does not touch the drive shaft so that the conduit will not rotate with the drive shaft and so that the conduit may vibrate with respect to the drive shaft. In the illustrated embodiment, the shield 14 is a cooled disc supported in the crucible by means of an elongated tube structure 78, 79 extending through the conduit and constructed to carry coolant to and from the discs.

Referring in greater detail to the method of the invention, such method is practiced in connection with a rotating crucible such as the crucible 11 in the drawings. The crucible is cylindrical, rotating about its axis, and has an axial opening 12 in its closed end wall 13. The method of the invention is directed to feeding particulate material through such an opening into the crucible. The method will be described in connection with silica sand material, but it is to be understood that the invention may be utilized in connection with other types of particulate material. For example, quartz, aluminum oxide or chromium metal in particulate form may be fed into a rotating crucible according to the invention.

The interior of the crucible which the sand is entering is hot and is filled with vapor at a relatively high vapor pressure. If this vapor is permitted to enter the opening through which the sand is being introduced, it will condense on the walls of the opening and on the sand in the opening to cause fusion of the sand and possible obstruction to the free flow of sand into the crucible. Moreover, radiated heat from the hot vapor and molten material in the crucible, and stray electrons from the electron beams heating the charge material, may also cause the sand flowing through the opening to fuse and block the open-

ing. In accordance with the invention, a barrier is formed to protect the sand in the opening from vapor, radiated heat and stray electrons. The barrier may include a shield disposed in the crucible adjacent the end of the opening. The shield is spaced from the closed end wall of the crucible to permit sand to flow through the opening into the crucible.

The shield, as shown in the illustrated apparatus, may comprise a disc 14 supported independently of the rotating crucible so that it does not rotate. Suitable provision may be made for cooling the disc, as will be explained in greater detail subsequently. Alternatively to the non-rotating disc, the shield may comprise a plate or disc of high temperature material, such as molybdenum, secured to the closed end wall of the crucible and extending over the opening but spaced therefrom to permit sand flow therethrough. As explained in detail below, the material in the crucible will form part of the barrier, thus cooperating with the shield. As also explained subsequently, certain conditions may make a shield unnecessary in maintaining a barrier to vapor because the material in the crucible alone will suffice.

The detailed explanation of the method of the invention will begin at a point where sand first enters a rotating crucible having a shield therein. As the sand spills out from the opening in the closed end wall and into the crucible, it falls downwardly between the end wall of the crucible and the shield due to gravity. When the sand strikes the spinning walls of the crucible or when it strikes other sand which is moving with the crucible, the sand is fanned out around the center of the end wall of the crucible. Once having attained the general rotational speed of the crucible, the sand will be urged toward the cylindrical wall thereof by centrifugal force. Sand is continuously fed into the crucible until it fills up the annular corner 16 between the cylindrical wall and the closed end wall. The sand feed is permitted to continue until the sand is almost touching the periphery of the shield. When enough sand has entered the crucible that the annular corner of the crucible is filled up and the sand approaches the periphery of the shield, the electron beams are turned on to begin the melting process.

It has been found that the silica in the crucible will complete the barrier for the conduit by forming a seal or annular inward projection 17 of fused silica. This seal projects radially inward from the silica along the cylindrical crucible wall, and is positioned between the shield and the open end of the crucible. The seal operates to prevent the hot vapor in the crucible from entering the conduit by passing between the shield and the end wall of the crucible. The seal also prevents excessive buildup of condensate on the rear surface of the shield. In the event a cooled disc 14 is used as a shield, the cooling effect of the cooled disc on its surroundings helps keep the inner rim of the seal in the fused condition for structural rigidity.

Although the precise reason for the buildup of the seal or annular inward projection is not fully understood at this time, it is believed that the seal occurs due to the pushing of the granular sand from underneath the surface of the seal as a result of the sand flowing from the opening into the annular corner 16 between the end wall and the cylindrical wall of the crucible. The surface of the seal toward the electron beams will be melted by the beams and this molten material will flow down the slope of the seal, due to centrifugal force, and out along the cylindrical wall of the crucible to be distributed along the cylindrical wall between the seal and the annular lip 18 of the crucible. The vapor is formed off the surface of this molten silica.

Referring particularly to FIGURE 2, the solid and dotted curved lines define cross sectional regions of the silica in the crucible. In region A, the silica is in the particulate or granular form; in region B, it is in the fused form; region C, the molten form; and region D the

vaporous or gaseous form. Naturally, the regions A-D would not be as clearly defined in an actual cross section, and the representation of FIGURE 2 is schematic and for illustrative purposes only. It is believed that replenishment of the material in the crucible takes place in a "from the bottom up" manner. That is, the silica moves into the crucible from the opening 12 and then moves gradually upward from the annular corner 16 through regions A, B and C toward the vaporous region D. During this movement, the silica forms the seal 17 and changes from the granular form through the fused and molten forms into vapor.

The formation of the "skull," that is, the solid to liquid state gradient region of the material contained in the crucible, including the annular "seal," helps to thermally isolate the vaporous region D in the crucible from the cooled walls of the crucible. This maintains a relatively high thermal efficiency in the system. The seal 17 of the skull, as discussed above, also thermally isolates the sand being fed into the crucible (in region A) from the hot vapors and molten silica in the crucible (in regions C and D).

Under suitable operating conditions, it may be possible to achieve a completed barrier by means of the seal alone, rather than in combination with the shield (e.g., a stationary cooled disc or a rotating molybdenum plate). This will occur if the seal 17 is permitted to build up to such an extent that its inner diameter is small enough to prevent vapor from passing back to the opening in the end wall of the crucible. The major problem in obtaining such a barrier is during start up, but the use of a suitable pre-molded fused silica skull inserted in the crucible during start up will prevent vapor from reaching the opening.

The position of the annular seal should be maintained such that it is not sufficiently close to the open end of the crucible that the "focus" of the vapor beam is lost. Such occurs when the vaporous region D becomes too shallow and too many vapor particles having directional components transverse to the axis of the crucible escape therefrom. It is also necessary to prevent the annular seal from getting too far back in the crucible and hence too close to the shield. When this occurs, a reduction in "skull" thickness has occurred, reducing the overall thermal efficiency of the system. Furthermore, when a nonrotating disc 14 is used as a shield, there is a danger that the seal 17 will come in contact with the disc and will stick thereto, causing twisting and damage to the nonrotating disc.

In order to enable precise regulation of the shape and thickness of the "skull" including the position of the annular seal, the temperature in the opening 12 is sensed. A relatively higher sensed temperature indicates the seal 17 is relatively further back because of the larger amount of heat getting back to the opening due to the reduced "skull" thickness. To increase skull thickness and thereby reduce the sensed temperature, the feed rate of the sand is increased.

Ordinarily, a continuous flow of sand will not be maintained because such would replenish the material at too high a rate. Instead, the quantity of material in the crucible, and hence the skull thickness, is noted through observing sensed temperature readings and sand is added only when it is desired to increase the quantity of material in the crucible. When such quantity reaches the desired level, the addition of sand is suspended. By proper regulation of the periods of sand addition, an operator can maintain a fairly constant "skull" thickness and thus a fairly constant flow of vapor. The fact that the sand is not entering the crucible by falling directly onto the surface of the molten silica from which the vapor is being produced helps in keeping the vapor flow substantially constant and not subject to extreme fluctuation with each addition of material. It should be noted that, although the axis of rotation of the crucible is substantially horizontal, it need not be exactly horizontal. It is possible to perform the foregoing method with some deviation from the horizontal.

Referring now more particularly to the drawings, a

specific embodiment of apparatus constructed according to the invention is illustrated. Although the illustrated embodiment is designed specifically for use in connection with silica (silicon dioxide— SiO_2) sand, it is to be understood that other feed materials in particulate form may also be utilized with little or no modification required. The vapor source assembly is disposed in a vacuum chamber housing 19, only a portion of which is shown. The substrate (not illustrated), upon which vapor is being deposited, is also disposed in the vacuum chamber housing. The vapor source assembly illustrated includes a cooled crucible 11 having a coolant chamber 21 in the cylindrical wall thereof. Suitable baffles, not shown, may be disposed in coolant chamber 21 for directing a desired flow of coolant. The crucible may be constructed of copper and the coolant circulated may be water. The crucible is of generally cylindrical shape, having an open end through which the vapor produced in the crucible escapes in the form of a vapor beam. The rim of the open end is surrounded by a lip 18. The opposite end of the cylindrical crucible is closed by an end wall 13. The closed end wall includes an inlet passage 22 and an outlet passage 23 through which the coolant passes into and from, respectively, the coolant chamber 21 in the crucible wall.

The closed end wall 13 of the crucible has an opening 12 therein, the axis of which is aligned with the axis of the cylindrical crucible. The opening 12 has two sections of different diameters, with the smaller of the two communicating with the interior of the crucible. The crucible is secured to a drive shaft 24 which mates in the larger section of the opening 12 in the end wall of the crucible and is suitably secured therein. The drive shaft extends from the crucible axially thereof and is journaled in and enclosed by a housing 25 for the source assembly. The housing for the source assembly is supported and sealed in an opening 20 in a wall of the vacuum chamber housing 19 by a mounting flange 26.

The vapor source assembly housing 25 is provided with an appendage 27 through which a power shaft 28 extends perpendicularly of the drive shaft 24. A bevel gear 29 on the power shaft engages an annular bevel gear 31 on the drive shaft for driving same. Ball bearings 32 are provided in the appendage 27 for journaling the power shaft.

The drive shaft 24 is constructed with two coolant flow passages 33 and 34 therein which extend along the drive shaft between the outer surface and the axis thereof. Each of the passages is slightly less than semiannular in cross section and one of the passages carries coolant to the crucible while the other carries coolant from the crucible. The passages in the drive shaft communicate with the coolant passages 22 and 23 in the crucible through openings 36 and 37, respectively, near the end of the drive shaft adjacent the crucible. Coolant is supplied to the coolant flow passages 33 and 34 in the drive shaft through a coolant inlet conduit 38 in the vapor source assembly housing 25. This coolant inlet conduit communicates with an annular coolant inlet chamber (not shown) and the passage 33 opens into such chamber to receive coolant therefrom. A similar annular coolant outlet chamber (not shown) in housing 25 communicates with the passage 34, and conduit 39 is provided for draining such outlet chamber. Coolant flow is maintained while the drive shaft and crucible are rotating.

The charge material in the crucible is melted by one or more electron beam guns 40 mounted in vacuum chamber housing 19. The guns are provided with suitable directing magnets (not shown). High energy electron beams from the guns 40 are directed into the crucible 11 to impinge upon the charge material held by centrifugal force against the inner side of the cylindrical walls of the crucible and thereby effect melting and vaporization of the charge material. By way of example, three electron beam guns may be used to produce three independent electron beams directed to impinge on equal areas spaced

120° apart around the crucible cylindrical wall. The areas should preferably be equidistant from the closed end wall of the crucible.

Silica sand enters the crucible through an elongated tubular conduit 41 which extends through the hollow drive shaft 24 and the opening 12 in the end wall 13 of the crucible. The conduit terminates about evenly with the inner side of the end wall 13. As will be explained subsequently, the conduit is suspended within the hollow drive shaft and does not contact the drive shaft. Accordingly, the rotating drive shaft will not interfere with the nonrotating conduit. The conduit extends from the crucible 11 completely through the drive shaft and out the rear end of the source assembly housing 25, terminating in an opening in the front wall 42 of a reservoir for the sand. The conduit is suitably secured in such opening to the front wall, as by welding. The front wall is part of a reservoir for sand consisting of a pair of side walls 43 and 44 and a back wall 46, all extending upwardly from a base plate 47. The base plate projects beyond the front wall of the reservoir and carries a support 48 thereon which engages the underside of the conduit and helps to cantilever the conduit in the hollow drive shaft.

The base plate 47 of the reservoir is secured to a bracket 49 having a pair of arms 51 and 52 extending downwardly from the underside of the base plate. The arms of the bracket are attached through a plurality of leaf springs 53 to a corresponding pair of brackets 54 and 55 fixed on a supporting plate 57. The leaf springs are of a suitable resilient material such as Fiberglas. The rearward arm 51 of the bracket 49 attached to the base plate is in contact with the armature 58 of a solenoid type vibrator 59. The vibrator, together with the leaf springs, cause a vibration in the reservoir and, consequently, the conduit 41. This vibration consists first of a forward lifting motion and then a substantially direct return so that the sand in the reservoir and in the conduit is urged forward in "hops" toward the crucible. The support plate 57, for the vibrator and supporting brackets for the leaf springs, is bolted to the floor 61 of a housing 62 for the vibrator, reservoir and associated apparatus.

The last named housing 62 is external of the vacuum chamber housing 19 and is for containing the sand reservoir and associated elements in a vacuum corresponding to the vacuum maintained in the vacuum chamber housing. This is necessary because of the direct communication through the conduit and hollow drive shaft 24 from the crucible to the sand reservoir. Alternatively, the vacuum chamber housing could be made large enough to contain all the feeder apparatus. The housing 62 comprises a box separable in two halves along diagonal flanges 63 extending from the upper edge closest to the crucible to the lower opposite edge. The two halves of the reservoir housing are bolted together along the respective flanges 63 and are sealed around their periphery by a suitable sealing gasket 64.

A window for viewing the operation of the apparatus and the level of sand in the sand reservoir is provided in the top of the reservoir housing. This window is shown comprised of a lower rectangular frame 66 welded in an opening in the top of the reservoir housing 62 and an upper rectangular frame 67 bolted to the lower frame. A transparent sheet 68 is held between the two frames 66 and 67 and may be comprised of heat resistant glass such as Pyrex. A sealing gasket 69 is provided between the sheet 68 and the lower frame.

The housing 62 rests on a pair of cross beams 71 and is bolted to a frame structure 72 extending upwardly from a suitable supporting base (not shown). A bellows arrangement comprised of a pair of annular plates 73 and 74 and an extensible tubular wall 76 extending therebetween joins the rear of the shaft housing 25 with the housing 62 and maintains a vacuum seal. The adjustable nature of the bellows allows greater tolerance in the po-

sitioning of the various elements and facilitates adjustment during assembly. Sand in the reservoir is replenished through a sand conducting pipe 77 by means of gravity feed from a vacuum sealed container, not shown, disposed above the reservoir exteriorly of housing 62. A hose (not shown) may extend between the sand container and the reservoir housing for maintaining equal pressures in the two so that a desired level of sand in the reservoir may be maintained through suitable adjustment of the level of the end of the pipe 77.

The shield in the illustrated embodiment is comprised of a cooled disc 14. Cooling and support for the disc is provided by an elongated tubular structure which extends through the conduit 41. The tubular structure is comprised of a pair of concentric cylindrical sleeves 78 and 79. The disc 14 is hollow and the outer sleeve 78 of the tubular structure terminates in an opening in the disc outer wall. The inner sleeve terminates in a baffle 81 suitably supported in the hollow disc. The flow of coolant is established through the space between the sleeves 78 and 79 of the tubular structure, into the disc 14, around the baffle 81, and back out through the smaller center sleeve 79.

To prevent sand from coming in contact with the relatively stationary disc 14 and thereby causing excessive buildup of sand behind the disc, the disc may be equipped with one or more plow blades 82 attached at the periphery of the disc on the rearward side thereof. These blades come in contact with the spinning sand and help keep the sand free of the disc and also help to maintain a flow of sand outwardly toward the cylindrical crucible wall.

The sleeves 78 and 79 of the tubular structure are supported in a block 83, passing through suitable openings therein. The supporting block 83 for the tubular structure is bolted to a larger block 84 which is hung, by means of a hanger bracket 86, from an inwardly projecting plate 87 attached to the housing 62. A spacer device or spider 88 is disposed toward the end of the tubular structure adjacent the disc 14 and serves to space the outer sleeve 78 of the tubular structure from the conduit 41. The end of the tubular structure opposite the spider 88 terminates in a suitable connecting block 89 providing connection between coolant inlet and outlet hoses 91 and 92 and the appropriate passages in the tubular structure. The inlet and outlet hoses for the coolant are attached to the block 89 and communicate with suitable passages 93 and 94 therein. Hoses 91 and 92 pass through suitable seals (not shown) in a wall of the housing 62.

The tubular structure, since it is in contact with the conduit 41 through the spider 88, will affect the vibrational characteristics of the conduit. In order to permit regulation of this affect, provision is made for "tuning" the apparatus to the desired vibrational characteristics. Tuning is accomplished by adjusting the pressure which the tubular structure exerts on the conduit through the spider. An L-shaped bracket 96 is secured to the fluid connecting block 89 and extends upwardly above the sand reservoir. An adjusting screw 97 passes through a suitable sealed fitting 98a in the top wall of the feeder housing and contacts the end of the L-shaped bracket opposite the fluid connecting block. The support block 83 and associated elements intermediate the ends of the tubular structure will act as a fulcrum such that adjustment of the screw 97 will change the force exerted on the spider by the tubular structure.

A pipe 98, in which a thermocouple 100 and the leads therefor (not shown) may be disposed for sensing temperature in the opening, extends along the edge of the tubular structure inside the conduit and terminates in a mating pipe 99 which extends through the support blocks 83 and 84. The mating pipe extending through the support blocks terminates in a chamber 101 within the larger block 84 and the thermocouple leads may be car-

ried out through one end of this chamber through a sealing plug 102. A gas bleed conduit 103 is disposed in the top of the larger block 84 and is attached through a tube 104 to a suitable supply of oxygen, not shown, exteriorly of housing 62. Oxygen at a predetermined rate is permitted to bleed into the chamber 101 and hence through the pipe 98 into the crucible. The amount of oxygen which is bled into the system is determined from empirical data and is selected such that the partial pressure of oxygen in the crucible is sufficiently high that decomposition of silicon dioxide to silicon monoxide plus oxygen ($2\text{SiO}_2 \rightarrow 2\text{SiO} + \text{O}_2$) is prevented.

In practicing the method of the invention in connection with SiO_2 sand and also with granulated chromium, in the foregoing apparatus, the following parameter values were found effective to produce a vapor flow rate of about 3 lbs. per hour:

Crucible inner diameter— $10\frac{3}{4}$ inches
 Crucible length (from closed end wall to inner side of lip)— $4\frac{3}{4}$ inches
 Disc diameter—4 inches
 Disc spacing from closed end wall— $\frac{5}{8}$ inch
 Material feed rate— $3\frac{1}{4}$ lbs. per hour
 Mean granule diameter of particles— $\frac{1}{32}$ inch
 Electron beam power (total for 3 equal beams)—35 kw.
 Area of beam impingement (for each of 3 beams 120° apart)—6 sq. inches

Satisfactory results were also obtained in producing a vapor flow rate of 11 lbs. per hour, using the following parameter values:

Crucible inner diameter— $10\frac{3}{4}$ inches
 Crucible length (from closed end wall to inner side of lip)— $4\frac{3}{4}$ inches
 Disc diameter—4 inches
 Disc spacing from closed end wall— $\frac{5}{8}$ inch
 Material feed rate—12 lbs. per hour
 Mean granule diameter of particles— $\frac{1}{32}$ inch
 Electron beam power (total for 3 equal beams)—50 kw.
 Area of beam impingement (for each of 3 beams 120° apart)—6 sq. inches

It will therefore be appreciated by those skilled in the art that the invention provides an improved method and apparatus for feeding particulate material into a vapor source crucible in a vacuum deposition system. More particularly, the invention has application to such a crucible which rotates about a substantially horizontal axis, and facilitates continuous operation of the vacuum deposition system. Careful regulation of the quantity of feed material entering the crucible and the rate of replenishment thereof may be attained. Various modifications and other embodiments of the invention will be apparent to those skilled in the art from the foregoing description and such other modifications and embodiments are intended to fall within the scope of the appendant claims.

What is claimed is:

1. In a vacuum deposition system, a method for feeding particulate material into a cylindrical vapor source crucible which is rotating about a substantially horizontal axis, comprising: introducing particulate material into the rotating crucible through an axially aligned opening in the closed end of the crucible, directing a beam of electrons against the surface of the material disposed along the cylindrical wall of the crucible to cause melting and vaporization of such material at the surface thereof, and controlling the addition of particulate material to the crucible in order to create a barrier formed by an annular projection of the material in the crucible proximate the opening in the closed end of the crucible to prevent vapor generated within the crucible from entering the opening, whereby a flow of particulate material into the crucible may be maintained.

2. A method according to claim 1 wherein the barrier

is formed partially of the material in the crucible, and partially by a shield disposed within the crucible spaced from the opening, the annular projection being formed on the opposite side of the shield from the opening and extending radially inward and overlapping the periphery of the shield.

3. A method in accordance with claim 2 wherein the addition of the particulate material to the crucible is controlled by sensing the temperature adjacent the opening in the closed end of the crucible.

4. A method according to claim 3 wherein particulate material entering the rotating crucible is permitted to substantially fill the annular corner between the cylindrical wall and the end wall of the crucible prior to the step of directing the beam of electrons, and wherein such corner is maintained substantially filled by the subsequent addition of further particulate material through the opening.

5. Apparatus for feeding particulate material into a rotating vapor source crucible in a vacuum deposition system, comprising, a crucible having a closed end and an open end and an axis of rotation extending through said closed and open ends, said crucible defining a vapor region adjacent the open end thereof and having an opening in the closed end thereof proximate the axis of rotation of said crucible, a conduit having one end communicating with the interior of the crucible through the opening in the closed end of said crucible, said conduit being adapted to receive particulate material from a supply source, means for moving particulate material through said conduit into said crucible, and a vapor barrier supported within said crucible spaced from the closed end of said crucible and extending transversely of the axis of rotation of said crucible between the closed end of said crucible and the vapor region of said crucible for substantially preventing vapor generated within the vapor region of said crucible from entering said conduit.

6. Apparatus according to claim 5 wherein said barrier includes a shield and means for supporting said shield in the crucible proximate said one end of said conduit.

7. Apparatus for feeding particulate material into a rotating vapor source crucible in a vacuum deposition system, comprising a crucible having a closed end and an open end and an axis of rotation extending through said closed and open ends, said crucible defining a vapor region adjacent the open end thereof and having an opening in the closed end thereof proximate the axis of rotation of said crucible, a hollow drive shaft supporting said crucible for rotation, said hollow drive shaft communicating with the interior of said crucible through the opening in the closed end of said crucible, an elongated conduit disposed within the hollow interior of said drive shaft and having one end in communication with the interior of the crucible, means for introducing particulate material into said conduit, means for moving particulate material through said conduit and into said crucible, and a vapor barrier supported within said crucible spaced from the closed end of said crucible and extending transversely of the axis of rotation of said crucible between the closed end of said crucible and the vapor region of said crucible for substantially preventing vapor generated within the vapor region of said crucible from entering said conduit.

8. Apparatus according to claim 7 wherein said conduit is cantilevered from a supporting structure at the end thereof opposite said one end.

9. Apparatus according to claim 8 wherein said barrier includes a shield and an elongated support structure for said shield extending through said conduit into said crucible.

10. Apparatus according to claim 9 wherein means are provided for supporting said elongated support structure intermediate its ends independently of said conduit, wherein a spider is disposed in said conduit between said means for supporting said elongated support and said crucible, said spider contacting both said conduit and said elongated support structure for maintaining a spaced

11

relation therebetween, wherein said moving means comprise a vibrator device connected to said conduit for producing vibrations therein, means for biasing said elongated support structure against said spider, and means for controlling the biasing force exerted on said spider by said elongated support structure for tuning the apparatus to a frequency having a desired relation to the frequency at which said vibrator device operates.

11. Apparatus according to claim 10 wherein said adjusting means for controlling the biasing force comprises means for applying force to the end of said elongated support structure which is on the opposite side of said elongated support structure supporting means from said shield, said supporting means thereby acting as a fulcrum.

12. Apparatus according to claim 9 wherein at least one plow blade is mounted on said shield at the periphery thereof for maintaining a space between said shield and particulate material in the crucible.

13. Apparatus according to claim 9 wherein a temperature sensing device is positioned within said conduit proximate said one end of said conduit.

14. Apparatus according to claim 9 wherein the particulate material is silicon dioxide, wherein an elongated pipe is positioned within said conduit, said pipe terminating proximate said one end of said conduit, and wherein means are provided for establishing a flow of oxygen through said pipe into the crucible at a rate to provide a sufficiently high partial pressure of oxygen in the crucible to prevent breakdown of the silicon dioxide into silicon monoxide.

12

15. Apparatus according to claim 5 wherein said barrier includes an annular projection of the material in the crucible extending inwardly from the sidewall of said crucible toward the axis of rotation.

16. Apparatus according to claim 5 wherein said barrier includes a shield and means extending through said conduit for supporting said shield in said crucible spaced from the closed end of said crucible and further includes an annular projection formed of the material in the crucible extending inwardly from the sidewall of said crucible toward the axis of rotation, said shield being positioned between said annular projection and the closed end of said crucible, said shield comprising a generally circular disc having a diameter greater than that of the diameter across the inner periphery of said annular projection.

References Cited

UNITED STATES PATENTS

1,193,783 8/1916 Hillhouse ----- 75—10

FOREIGN PATENTS

1,258,477 3/1961 France.

BERNARD A. GILHEANY, Primary Examiner

H. B. GILSON, Assistant Examiner

U.S. Cl. X.R.

13—9; 75—10