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ELECTRON BEAM ZONE REFINING

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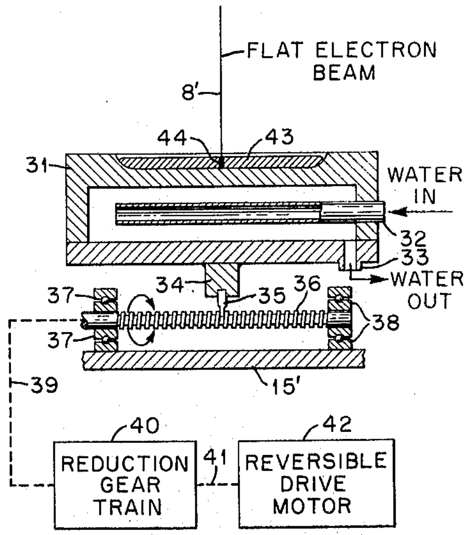

Fig. 2.

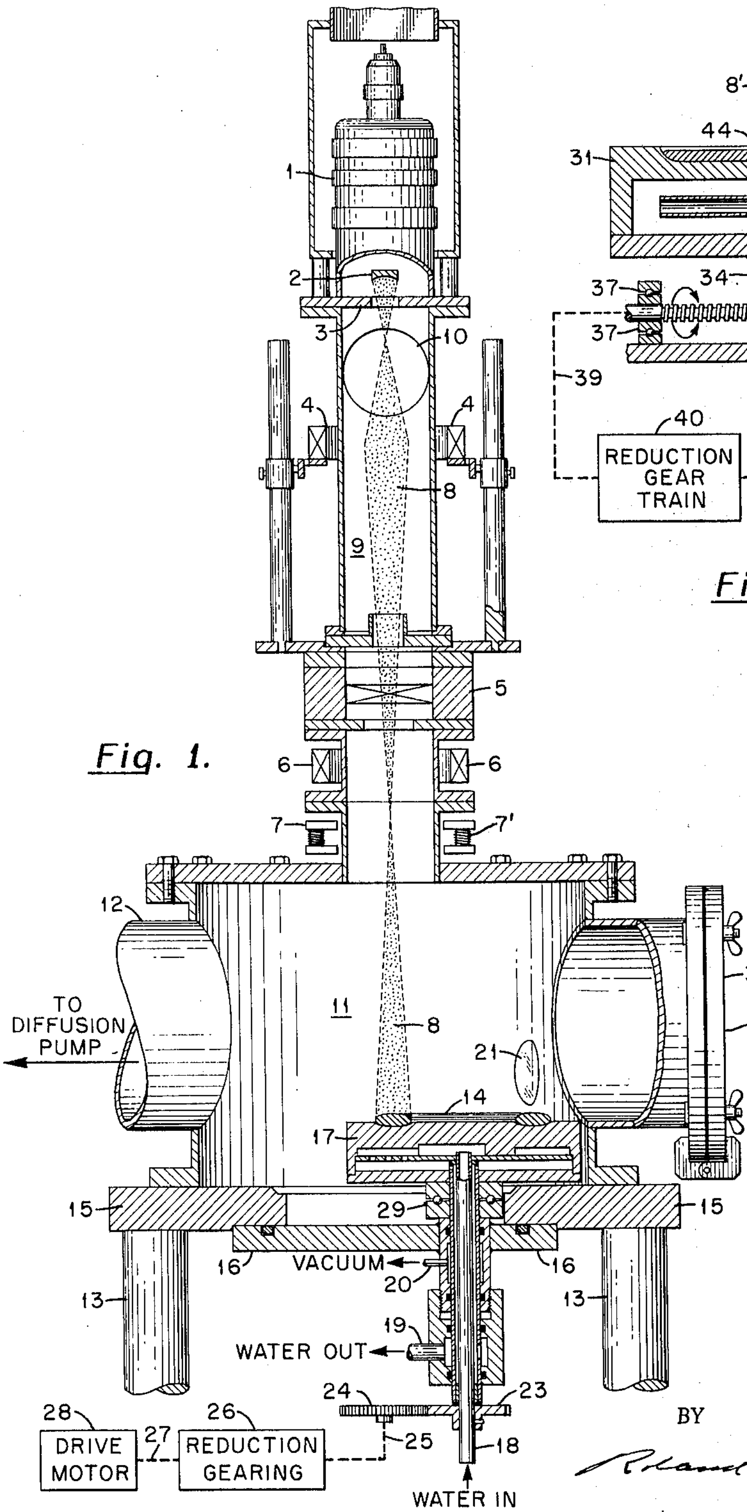

Fig. 1.

INVENTOR.
Tsutomu Hikido

BY

Roland A. Anderson

ATTORNEY.

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ELECTRON BEAM ZONE REFINING

Tsutomu Hikido, Sunnyvale, Calif., assignor to the United States of America as represented by the United States Atomic Energy Commission

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This invention relates to an improved device for zone refining of metals and oxides by the use of an electron beam melting process.

It is well known that trace impurities exert a profound influence on the properties of many substances. In recent years, considerable effort has been put forth to obtain high purity metals and oxides, and to obtain crystal perfection. It is desirable to obtain or produce certain high purity materials so that their chemical and physical properties can be determined under ultra pure conditions. Zone refining also has application in growth of individual crystals and in preparing semi-conductor materials for transistors, solid state conductors, etc.

The technique of zone refining utilizes the freezing process which depends on the fact that a freezing crystal differs in composition from its liquid. Impurities will travel with or opposite to the movement of a molten zone depending on whether they raise or lower the melting point of the solid. Therefore, if a short molten zone is passed along the length of a solid, purification per pass of the molten zone is proportional to a distribution coefficient. Prior art methods for producing a molten zone have been by induction coil heating, electron bombardment, etc. For a discussion of the theory of and various methods for zone refining, reference is made to the book Zone Melting, W. G. Pfann, T. Wiley and Sons, Inc., 1958, and the book Zone Refining and Allied Techniques, N. L. Parr, George Newnes Limited, 1960.

In a conventional electron beam melting furnace, the electron beam normally produces a circular molten zone. Using such an electron beam, a typical columbium sample which was zone-melted lost slightly more than 24% of its original weight due to vaporization. In such a prior device, the circular molten zone does not provide for the sharpest possible demarcation between liquid and solid phases. In order to minimize vaporization losses, it is desirable to provide a molten zone as narrow as possible. Also in order to provide an efficient zone purification the demarcation between the liquid and solid phases should be made stable and compact and as sharp as possible.

Accordingly, it is a primary object of this invention to provide an electron beam zone refining furnace which includes means for minimizing the vaporization loss rate in a zone refining operation.

It is another object of this invention to provide an electron beam zone refining furnace in which a compact melting zone is provided that has a substantially straight liquid-solid interface.

These and other objects and advantages of this invention will become apparent upon a consideration of the following detailed specification and the accompanying drawing, wherein:

FIG. 1 is a cross sectional view of an electron furnace in which the principles of this invention may be carried out, and

FIG. 2 is a cross sectional view of a longitudinally movable hearth assembly that may be substituted for the rotatable hearth assembly of FIG. 1.

The above objects have been accomplished in the present invention by providing a pair of adjustable beam shaping electromagnets for changing the normal circular shape of the electron beam into a substantially thin rectangular shape. It has been determined that use of such

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a thin shaped beam has reduced the vaporization loss of a columbium sample unexpectedly to less than 4% as compared to a loss of about 24% when a conventional circular electron beam is used in a zone refining operation. Also, it has been determined that use of such a thin electron beam provides a molten zone that is substantially straight, stable and compact with a sharp demarcation between the liquid and solid phases.

FIGURE 1 illustrates one embodiment in which the principles of this invention may be carried out. In FIG. 1, an electron gun 1 is provided with a cathode 2 and an anode 3. The anode 3 is provided with a central opening for collimating the electron beam 8 from the cathode 2 and directing this electron beam into the evacuated chamber 9. An annular focus coil 4 is disposed around the chamber 9 for focusing the beam 8. The chamber 9 is connected by way of an opening 10 to a vacuum pump, not shown.

The electron beam 8 then passes through a 4-inch valve 5, through a second annular focusing coil 6, through a pair of beam shaping electromagnets 7, 7′, and then into a furnace chamber 11. Chamber 11 is connected by means of a tubular member 12 to a vacuum pump, not shown. The electromagnets 7, 7′ contain Armco iron cores with mild steel parallel end plates. The parallel end plates of each electromagnet are positioned, one on either side of the beam 8, and oriented normal to the beam axis. The coils 7, 7′ are turned 90° in the drawing from their actual position for the sake of clarity. A sight port 21 is provided in the wall of the chamber 11 so that the electron beam 8 may be observed while the currents to the electromagnetic shaping coils 7, 7′ are adjusted by means, not shown, to provide the desired thin rectangular shaped electron beam.

The chamber 11 is provided with a furnace bottom plate 15 which is supported by posts 13 which are affixed to a support plate, not shown. The plate 15 is provided with a circular opening through which a rotatable hearth assembly extends, as shown, and this assembly is supported by a furnace adapter plate 16. The hearth assembly includes a copper hearth 17, a thrust bearing 29, a vacuum connection 20, a water inlet pipe 18, a water outlet pipe 19, and a gear sprocket 23. The water is used for cooling the hearth 17 during a zone refining operation.

The hearth 17 is slowly rotated at a selected speed by means of a drive motor 28 which is coupled by means of a mechanical drive 27, a reduction gear train 26, a mechanical coupling 25, and a gear 24 to the gear 23 of the hearth assembly. The gear 23 is affixed to the tube 18 in a conventional manner. The speed of rotation of the hearth 17 is of a selected value in the range from 1/16–2 r.p.h. A typical speed is about 1/4 r.p.h., for example. The speed of rotation is determined by the type of material it is desired to zone refine.

The hearth 17 is provided with an annular cavity in which an annular metal sample 14 rests. The width of the metal sample 14 is made substantially equal to the length of the thin rectangular shaped beam 8 and this beam provides a molten zone normal to the surface of the sample 14. The molten zone is stable and compact with a sharp demarcation between the liquid and solid phases as the sample 14 is slowly rotated past the beam.

In the operation of the device of FIG. 1, the chambers 9 and 11 are evacuated to a pressure below 1×10^{-4} mm. Hg, normally about 1×10^{-5} mm. Hg, the electron gun is energized to provide an electron beam 8, and the currents to the shaping coils 7, 7′ are adjusted, while the molten zone in sample 14 is observed through the viewing port 21, until the beam 8 has acquired the desired thin rectangular shape next to the metal sample 14. The flattened electron beam 8 provides a very narrow molten zone in the metal sample 14 and as the sample 14 is slowly moved at a selected speed, the molten zone is caused to move along the sample. Thus, the sample **14** is zone refined by the flattened electron beam **8**. The number of passes of the sample **14** past the beam **8** is determined by the purity desired in the sample.

After the sample **14** has been zone refined, the electron gun is deenergized. The valve **5** is closed to maintain the vacuum in chamber **9**, and the sample **14** may then be removed from the chamber **11** after the chamber **11** is returned to atmospheric pressure by closing the valves connecting the chamber to the vacuum pump connected thereto and admitting air or inert gas through a valve, not shown. Removal of the sample **14** may be made by opening a door **30** to the chamber **11** to provide access to the interior thereof, or by lowering the hearth through the central opening in the bottom plate **15**. After a new sample is placed on the rotatable hearth **17**, the chamber **11** is resealed, the chamber **11** is evacuated again, the valve **5** is opened, the electron gun is again actuated, and the furnace is then ready to perform another zone refining operation.

The device of FIG. 1 is not restricted to the use of a rotatable hearth assembly. For example, a longitudinally movable hearth assembly may be used in the device of FIG. 1 in place of the one shown. FIG. 2 shows such a modification.

In FIG. 2, the furnace bottom plate **15'** is not provided with a central opening as is the plate **15** of FIG. 1. Mounted on top of plate **15'** are a pair of bearing assemblies **37**, **38** which rotatably support a worm screw **36**. Worm screw **36** is rotated in one direction or the other by a reversible drive motor **42** which is coupled to the screw **36** by means of a mechanical coupling **41**, a reduction gear train **40**, and a mechanical coupling **39**.

A water cooled hearth **31** is provided with a straight dished out cavity for holding a metal sample **43** to be zone refined. The hearth **31** further includes a water inlet pipe **32** and a water outlet connection **33**. A support member **34** is attached to the underside of hearth **31**. Member **34** in turn is provided with a pawl member **35** which rides in the grooves of the worm gear **36**. Thus, when gear **36** rotates in one direction, the hearth **31** will move in a path parallel to the axis of gear **36**, and when gear **36** rotates in the other direction, the hearth **31** will move in a path just the opposite to that described above.

The hearth **31** is supported and guided by means, not shown, such that it moves in a straight line path in a direction parallel to the axis of the worm gear **36**. Thus, the hearth **31** is moved by means of the coupled motor **42** at a selected speed 1 to 36 inches/hour past the narrow rectangular shaped electron beam **8'** such that the molten zone **44** effected by the electron beam **8'** is caused to move from one end of the metal sample **43** to its other end. This movement of the molten zone may be repeated as many times as desired before the sample is removed and a new sample is substituted therefor to begin a new zone refining operation.

It should be understood that the electron beam furnace used with the device of FIG. 2 is the same as that used in FIG. 1, with the exception that the rotatable hearth assembly of FIG. 1 is replaced with the longitudinally movable hearth assembly of FIG. 2. The principles of operation of both assemblies are the same, the only difference being that one metal sample is rotated with respect to the electron beam and the other metal sample is moved in a straight line path with respect to the electron beam.

The device of FIG. 2 shows means for moving the

hearth **31** with respect to a stationary electron beam **8'**. In some zone refining applications, it may be desirable and preferred to maintain the hearth **31** stationary and move the electron beam **8'** with respect to hearth **31**. This could be done easily by removing the means for moving the hearth **31**, and providing an equivalent means for moving the electron beam assembly above the hearth **31** along a suitable opening in the top plate of the chamber **11**, or by deflecting the electron beam magnetically or electrically.

This invention has been described by way of illustration rather than limitation and it should be apparent that this invention is equally applicable in fields other than those described.

What is claimed is:

1. In a zone refining electron bombardment furnace including a melting chamber, an electron gun, means for collimating and focusing the electrons provided by said gun into a beam, means for directing said electron beam into said chamber, and means for evacuating said furnace, the improvement comprising a pair of electromagnetic shaping magnets mounted between said collimating-focusing means and said chamber, said magnets being placed one on either side of said beams and oriented normal to the axis of said beam, a water-cooled hearth disposed in said chamber, said hearth being provided with a cavity for receiving a sample to be zone refined, means for adjusting the current flow to said magnets for shaping said electron beam in a substantially flat rectangular shape, said flat beam being normal to the surface of said sample, and means for moving said hearth at a selected rate of speed such that a sample placed in said hearth cavity is moved along said rectangular shaped beam during a zone refining operation, said flat electron beam providing a narrow molten zone in said sample which moves along said sample during said operation, said short molten zone insuring a minimum of evaporation loss of said sample during said operation.

2. The electron beam furnace set forth in claim **1**, wherein said hearth is circular and rotatable, and said hearth cavity is annular, said sample moving along said flat electron beam during movement of said rotatable hearth by said hearth moving means.

3. The electron beam furnace set forth in claim **1**, wherein said hearth cavity is straight and elongated and said hearth being moved longitudinally by said moving means such that said molten zone is perpendicular to the surface of said sample and is moved longitudinally along the sample during said operation, by said movement of said hearth with respect to said electron beam.

4. The electron beam furnace set forth in claim **1**, wherein said chamber is provided with a viewing port for visual observation of said molten zone.

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ANTHONY BARTIS, *Acting Primary Examiner.*

JOSEPH V. TRUHE, *Examiner.*