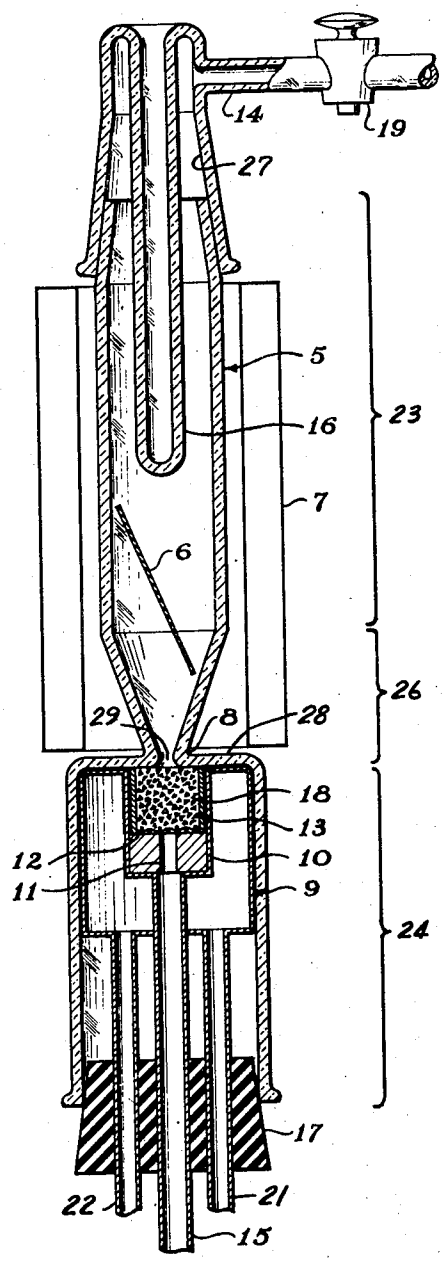
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APPARATUS FOR VAPOR COATING BASE MATERIAL IN POWDER FORM

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WITNESSES

Ralph Carlile Smith

Charles J. Murphy

INVENTORS.

Don S. Martin

René J. Prestwood

BY

Robert A. Saunders

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APPARATUS FOR VAPOR COATING BASE MATERIAL IN POWDER FORM

Rene J. Prestwood, St. Louis, Mo., and Don S. Martin, Ames, Iowa, assignors to the United States of America as represented by the United States Atomic Energy Commission

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1 Claim. (Cl. 118—48)

This invention relates to apparatus useful in the preparation of radioactive products and more particularly to apparatus useful in coating base materials with radioactive materials.

In many operations involving radioactive materials it is necessary that the radioactive material be in intimate contact with some base material and preferably coated on the surface thereof. For example, in the construction of a neutron source, polonium or some other alpha particle emitting substance is coated on the surface of beryllium or some other base material which emits neutrons in response to alpha particle bombardment. It has been determined that the efficiency of such a neutron source can be increased by depositing the radioactive material on a base material which is in a finely divided state. Similarly, for various radioactive products and/or for various purposes, it is desirable to deposit radioactive substances on finely divided base materials in contradistinction to deposition of continuous flat films of such substances.

Heretofore, it has been suggested that a radioactive coating could be deposited on base materials by the condensation of vaporized radioactive materials. Apparatus for accomplishing this has been constructed and the method found to be feasible. Such apparatus is described, for example, by Rona and Schmidt in Zeitschrift fur Physik, volume 48, pages 784–789, May 21, 1928, the particular embodiment comprising an elongated cylindrical quartz tube with a constricted portion near its midpoint. A suitable piece or other arrangement of radioactive material was disposed in the constricted portion of the tube. The base material was incorporated in a cup-shaped cavity in the end of a solid copper cylinder which was inserted in one enlarged section of the quartz tube and was positioned with the cup-shaped cavity adjacent to the constricted portion of the quartz tube. A suitable gas was admitted through the opposite enlarged section of the quartz tube, while heat from a Bunsen burner was applied to the constricted portion of the tube. As a result of the heating and of the reduced atmospheric pressure due to the Bernoulli effect, a portion of the radioactive material was vaporized and carried by the gas to the base material where the radioactive material condensed as a result of the cooling effected by the large heat capacity and conductivity of the copper cylinder. The excess gas escaped through the space between the copper cylinder and the wall of the quartz tube.

It is apparent that the efficiency of such apparatus is low since the rates of heating, cooling, and gas flow are not regulated. Excessive heating, for example, vaporizes an excess of radioactive material of which a large portion does not condense on the base material but rather is carried to other parts of the apparatus or lost from the system. On the other hand, too little heat does not vaporize a sufficient quantity of radioactive material and is wasteful of the carrier gas, or causes premature deposition of the radioactive material on the intervening parts of the apparatus rather than on the base material. Similarly, excessive gas flow would carry a large portion of the radioactive material beyond the base material while too little gas flow would cause premature deposition and poor penetration of the radioactive vapors into the cup-shaped cavity. Similar poor penetration or loss of radioactive material occurs if the rate of cooling is too large or too small. Even under the best of conditions, a finely divided base material cannot be uniformly coated, since the carrier gas does not penetrate to any great extent into the cup-shaped cavity causing deposition of the radioactive material on the first encountered base material. A further disadvantage of previously employed apparatus is that no provision is made for easily removing the radioactive product. Obviously any excessive handling during removal results in a loss of the radioactive material which would not only decrease the efficiency of operation but would also contaminate surrounding apparatus and jeopardize the health of the operator. Furthermore, breakage of the apparatus could occur as a result of a carelessly applied flame or similar localized heating, would also result in loss of radioactive material which would be a serious health hazard.

It is a primary object of the present invention to overcome the aforementioned disadvantages while maintaining the simplicity and convenience of the method.

It is a further object of this invention to provide a simple, convenient, and efficient apparatus for depositing a substantially uniform coating of a radioactive material on finely divided base metal powders.

Still further objects of this invention are: to deposit uniform radioactive coating on finely divided base metal powders by the condensation of radioactive vapors; to provide means whereby the variable parameters of the process are predetermined and controlled or regulated; to provide means for removing the radioactive product from the apparatus quickly and easily; to provide means for increasing the penetration of the bulk base material by the radioactive vapors; and to provide means whereby substantially all of the radioactive vapor formed within the apparatus is deposited on the base metal powder.

The objects of this invention are accomplished by an apparatus consisting of an elongated container containing a constricted portion with predetermined gas flow characteristics which separates the vaporizing section containing an elongated heater or other means of vaporizing the radioactive material from the condensing section containing a condenser with appropriate means for circulating cooling liquids surrounding a removable vessel holding the powders to be coated.

It has been found desirable to construct the elongated container member of a transparent material, e. g. quartz or Pyrex glass, to permit observation of the progress of the operation. In some embodiments, however, a suitable metal might be employed for increased durability of the apparatus or to act as a shield against harmful radiations from the radioactive material. It is also desirable in particular applications to construct the container member of two or more materials, for example, the inner walls of the elongated container might be constructed of a material chemically resistant to the radioactive vapors while the outer wall might be constructed of a material resistant to the transmission of radiations from the radioactive material.

The introduction of a jet-like constricted portion between the condensing zone and the vaporizing zone results in more efficient use of the radioactive material. That is, by employing the constricted portion of the container member to control the flow characteristics of the carrier gas through the container, as well as to focus or direct the stream over the base material in the condensing zone, less of the radioactive material is deposited on the walls of the container. Furthermore, improved penetration of the arrangement of granular base material is accomplished. Since the efficiency of operation of the jet depends upon the rate of gas flow, a suitable valve is preferably incorporated in the gas inlet line.

It has been found preferable in the practice of the invention to heat the vaporizing portion of the elongated container member by means of an electrical resistance furnace element which substantially surrounds the vaporizing zone. The temperature of the furnace may readily be varied merely by changing the electrical input to the resistance element. It has also been found desirable to employ means such as a temperature sensitive device, e. g., a thermocouple, in reactive association with the vaporizing zone which may be used in con-

junction with indicating means or in conjunction with apparatus for the automatic control of the external heating means. Although an electrical resistance furnace element is well adapted for automatic control, it is not intended that the invention be limited to this one means for heating since there are other heating methods which could be used conveniently. Induced heating of the radioactive material within the vaporizing zone by external high frequency alternating electric currents would be suitable and would be amenable to automatic control. Various types of flames suitably equipped with control means might be desirable in some embodiments of the invention.

To facilitate the deposition of the radioactive material on the base metal powder, it has been found to be advantageous to circulate a coolant through the member in which the base metal powder is incorporated. The type of coolant used depends upon the temperature range desired in the condensing zone and although the number of coolants which might be conveniently used is almost limitless, water, air, steam, and low melting point alloys have been satisfactorily employed. Since the rate of condensation of the radioactive material is partially dependent upon the rate of flow of coolant, it has been found desirable to incorporate methods of controlling the flow of coolant such as a rotometer or a valve in the coolant supply line. In some embodiments of the invention it is desirable to employ a temperature sensitive device in association with the condensing zone to permit automatic control of the rate of coolant flow.

Another system for regulating the rate of coolant flow comprises an ionization chamber or some other radiation sensitive device located adjacent to the carrier gas exit line. If the rate of cooling decreases to the extent that a portion of the radioactive vapor is carried beyond the base material, the ionization chamber detects the presence of the radioactive material in the exit gas and gives a suitable indication that the rate of coolant flow should be increased. This can be effected either manually or automatically.

The tubular shape of the member in which the base metal powder is incorporated allows the carrier gas to pass through the volume occupied by the bulk base material thereby increasing the penetration of said material by the radioactive vapor and producing a more uniform deposition. This tubular member is adapted to be easily removed from the condensing portion of the elongated container member and means is afforded for easily extracting the radioactive product from the tubular member.

In order to further the understanding of the invention reference is made to a presently preferred embodiment shown in cross section in Figure 1 of the drawings made a part of this specification. As shown on the drawing the apparatus comprises three main sections in a continuous quartz tube 5. The first main section 23 is a vaporizing zone which includes a source foil 6 consisting of a coating (e. g. electrodeposited coating) of the radioactive material on a suitable base such as gold or platinum, and a heating means 7, which is preferably a resistance furnace element. The second main section 26 is a converging portion of the quartz tube defining a jet 8 which increases the velocity and controls the flow characteristics of the carrier gas and which enhances penetration of the bulk base metal powder. The third section 24 is the condensing zone in which the coating deposition takes place, and which includes a chambered condenser unit 9 through which a coolant, preferably steam or water, is circulated. The condenser unit 9 is a hollow container of U-shaped cross section and has a cup-shaped re-entrant portion 10 in which the base metal powder is retained by a bored plug 11, a platinum gauze 12 and a tubular platinum sleeve 13. The condenser assembly fits into an enlarged section of the quartz tube so that the re-entrant portion 10 is positioned adjacent to the jet 8. Elongated coolant ducts 21 and 22 for the ingress and egress of coolant are connected with the chambered condenser unit 9. Chambered condenser unit 9 is of U-shape in diametrical cross section. This condenser unit is supported adjacent an apertured disc-shaped end wall 28 of third section 24. The aperture in the disc-shaped end wall converges inwardly from the plane of the surface of the end wall which faces inwardly of the third section to thereby provide jet 8. The plane of least diameter of the aperture is substantially in the plane of the outer surface of end wall 28. The walls defining the aperture 29 connect and merge with the small end of converging second section 26, thereby forming a venturi or venturi section or venturi duct.

A gas inlet 14 and control valve 19 lets gas into the vaporizing zone and a gas outlet 15 communicates with the bottom of the cup-shaped re-entrant portion 10. An elongated re-entrant well 16 extending into the vaporizing zone permits insertion of a thermocouple or other temperature sensitive device which, with suitable auxiliary means, can be used for the regulation of the heating means 7. A rubber stopper 17 is employed to seal the unit.

In a typical operation to coat beryllium with polomium for the preparation of a neutron source, with the condenser unit 9 in position and beryllium metal powder 18 incorporated in the cup-shaped portion 10, the source foil 6 on which polonium had been previously deposited, by well-known methods, is heated to about 1000° C. to 1100° C. by means of furnace 7 to effect vaporization of the radioactive material. The polonium melts at about 255° C. It has a vapor pressure of 4×10^{-4} millimeters of mercury at 290° C. and of 20 millimeters of mercuray at 760° C. Helium is started into the system through inlet 14 before or as soon as the temperature of the source foil 6 rises to 100° C. in order to minimize the radioactive material which is deposited within the apparatus. The helium flow is adjusted for optimum operating efficiency when the source foil 6 is heated to operating temperature. The gas carries the vaporized polonium through the jet 8 and through the metal powder 18, which is kept cool by steam or water flowing through the condenser unit 9. The vaporized polonium condenses on the beryllium particles, the gas passing out of the system through outlet 15. This outlet gas is routed through a suitable trap, not shown, which retains any radioactive material which is not deposited. After the flow has continued long enough to obtain the desired coating, the furnace 7 is turned off, but the helium flow is continued at a reduced rate until ten minutes after the source foil has cooled down below 100° C. When the coated powder is cool it is removed from the condenser by removing the stopper 17 and withdrawing the condenser unit 9 from the quartz tube. A suitable source container may then be slipped over the platinum sleeve 13, the unit inverted, and the coated powder pushed out by means of a rod inserted into outlet tube 15.

A rate of helium flow of about 50 milliliters per minute at a pressure of 600 millimeters of mercury and at room temperature with a jet 8 of 3 millimeters diameter will satisfactorily prepare in about two hours a polonium-beryllium neutron source comprising sufficient beryllium powder, which passes through 60 mesh and is held on 100 mesh screen, to fill a cylindrical container ½ inch in diameter and ½ inch deep.

Although two hours were taken in the example, the time needed for depositing the radioactive material depends on many variables such as the characteristics of the apparatus resulting from its design, and the conditions used in operating it. The size of jet 8 is one of the more important design variables. The rate of flow of the helium and the temperature of the source foil 6 are important operating variables. The temperature of the source foil 6 can be increased above 1100° C. and this will cause an acceleration in the rate of radioactive material deposited. However this will also cause a reduction in the efficiency of the operation.

Although specific apparatus and a specific operation have been described it is not intended that the invention be limited thereby. The elongated container member could be constructed of other materials and with other cross-sectional shapes and might conveniently be constructed in two or more separate sections which could be fastened together. Such a construction would be particularly useful in an embodiment of the invention in which it was desired to substitute various sized jets to achieve specific deposition effects. The radioactive material could be supported within the vaporizing zone by other means than that described, for example, it could be incorporated in a crucible which is disposed within the vaporizing zone. Many other modi-

fications of the apparatus could be made without affecting the spirit or scope of this invention.

What is claimed is:

Apparatus for coating base material in powder form by vapor deposition comprising an elongated receptacle having a first section, a second section and a third section, said receptacle first section being open at first and second ends and being of substantially uniform cross section and being adapted to support vaporizable material therein, heating means supported in surrounding relation to said receptacle first section, a gas duct connected to the first end of the receptacle first section, the receptacle second section being of venturi shape with the long portion of the venturi connected to the second end of the first section, the second end of the venturi being a disc-like portion the plane of which is normal to the axis of the receptacle venturi section and which includes a centrally located aperture flaring in the direction outward of the venturi section, the receptacle third section being of substantially uniform cross section and being connected at one end to the outer portion of the disc of the receptacle second section, a condenser having an outer configuration similar to the

configuration of the receptacle third section and having a cup-shaped re-entrant portion being supported in the receptacle third section with the cup-shaped re-entrant portion facing the venturi, a bored plug and gauze screen supported in the bottom of the cup-shaped re-entrant portion thereby providing in the remainder of the cup-shaped re-entrant portion a receptacle for the base material.

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