July 8, 1947. R. RÜHLE 2,423,729

VAPORIZATION OF SUBSTANCES IN A VACUUM

Filed Jan. 18, 1940

Fig. 1

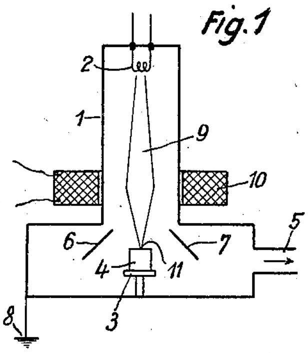


Fig. 2

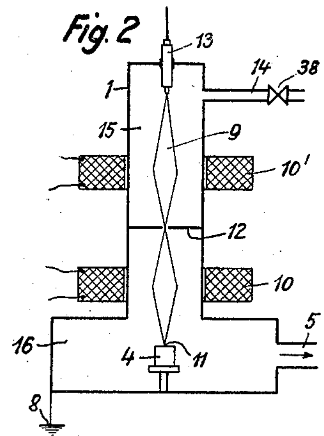


Fig. 3

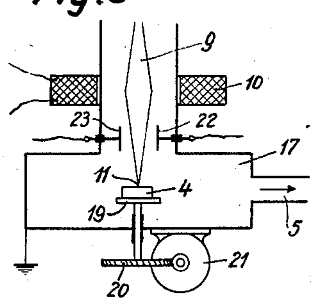
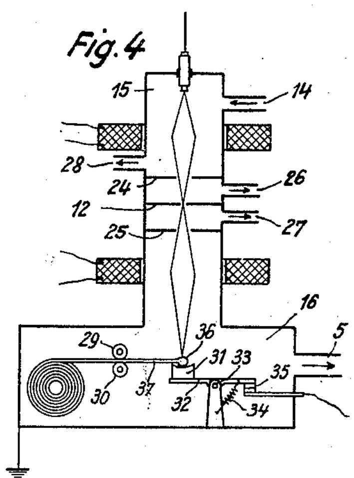


Fig. 4



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Patented July 8, 1947 2,423,729

UNITED STATES PATENT OFFICE

2,423,729

VAPORIZATION OF SUBSTANCES IN A VACUUM

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Application January 18, 1940, Serial No. 314,565
In Germany February 22, 1939

9 Claims. (Cl. 219—19)

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This invention relates to a method of and apparatus for vaporizing substances in a vacuum.

To produce metallic coatings on objects the metal to be used for coating, according to prevailing practice, is vaporized and deposited on the object to be treated. The necessary heat is supplied to the metal through the medium of a crucible which is brought to the required temperature in suitable manner, for instance by electric resistance heating.

It has been proposed to employ electronic rays emitted from a hot cathode instead of resistance heating for heating the crucible, but this method as well as the heating practice hitherto in general use are open to the objection that the crucible has to be heated first and heat is therefore indirectly applied to the substance to be treated. As the crucible has to be brought to a temperature exceeding that required for vaporization, the losses of heat due to the carrying off of a portion thereof by the crucible are, moreover, comparatively large.

The invention overcomes these troubles by placing the substance to be vaporized directly in the path of electronic rays which of themselves produce the heat needed for vaporization on striking the substance. As the supply of heat by conduction is eliminated, the temperature developed in the substance to be vaporized is higher than the ambient temperature. Compared with the known methods, during the application of which a certain amount of heat conducted away by the crucible or radiated in the wrong direction does not reach the substance to be vaporized, the method suggested by the invention causes the heat required for vaporization to be produced by electron bombardment at the very place where it is needed.

In further accordance with the invention the method and the yield obtainable by its application can be improved still more by placing the substance in the path of electronic rays that are concentrated or otherwise focused by suitable means. As the direction of non-focused rays emitted from the cathode cannot be controlled and a portion thereof is lost for vaporization, the invention provides for alignment of the rays and their concentration on the substance to be vaporized with the aid of electron-optical means whereby the intensity of radiation and thus the amount of energy consumed per unit area at the vaporization point are increased.

The substance to be vaporized is preferably introduced into the jet or beam of rays at a point where the beam has a relatively small cross section and the intensity per unit area is therefore very great, the most favorable position in this respect being obtained by placing the substance in the electron-optical image point of the emitting cathode.

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The cathode preferably comprises an electron-emitting source of high intensity, both hot and cold electrodes being usable which require, however, different gas pressures. If a hot cathode is employed, pressure on the filament must be extraordinarily low, since small quantities of residual gas will soon destroy the wire, slight traces of water vapor being for instance capable of causing decomposition of a tungsten wire within a very short time. In case of an unheated cathode the pressure prevailing in the space surrounding it should not be too low, as otherwise the ions required for the gas discharge will be lacking.

In the space containing the substance to be vaporized the vacuum should be chosen so as to insure good vaporization and the production of a clean deposit. A poor vacuum will yield of course poor deposits whilst any excess will be uneconomical.

In still further accordance with the invention the source of electrons is disposed in a space in which the pressure differs from that found in the space where the substance to be vaporized is kept. When a filament is used, pressure in the cathode space must be lower than in the vapor space whilst in case of a cold cathode pressure conditions are reversed. Both spaces are preferably separated by a partition possessing a narrowly apertured diaphragm. In order to maintain the difference in pressure between the two spaces air is continuously supplied to the higher pressure space and removed by pumping from the lower pressure space. Through the diaphragm an air current is then produced which by its size determines the pressure ratio. Instead of air, a gas may be used which does not combine with the substance to be vaporized, the vapor and the deposited layer.

In order to insure a greater difference in pressure without the necessity of drawing off too large quantities of air a plurality of diaphragms may be installed, one after another, which act like a labyrinth packing, that is to say, the pressure heads in the spaces between the diaphragms are varied by degrees from one working space to the other. The spaces between the diaphragms may be connected to vacuum pumps if necessary, and if only one diaphragm is used, a suction connection may be provided near it for the higher pressure space to keep the air current flowing through the diaphragm as small as possible.

To permit the passage of as many electronic rays as possible through the narrow aperture of the diaphragm a system of electron lenses which refract all electronic rays emitted from the cathode in the direction of the diaphragm is arranged between the latter and the source of electrons, so that a first image point of the electronic rays is formed approximately in the plane of the diaphragm. The rays spreading behind the diaphragm are then concentrated by a second electron lens system and, at a suitable point, brought again into the narrowing jet of rays for the substance to be vaporized.

Owing to the extraordinary smallness of the part thereof struck by the rays, substances of low thermal conductivity may be directly inserted in the beam of rays in the form of blocks or similarly shaped bodies without requiring a crucible, etc. As the electronic rays would soon melt a hole in the substance, the position of the latter must be altered periodically. This can be effected by means of a mechanical drive of the members supporting the substance to be vaporized, or by turning or displacing the lenses to shift the point of impact of the rays, or both motions may be combined in such manner that the block to be vaporized is rotated and the focus of the rays is displaced in radial direction from the outside toward the inside, or vice versa, so that the substance is correspondingly vaporized in a helical line. Furthermore, the electronic rays may be strictly electrically controlled by providing in known manner electric or magnetic fields the variation of which serves to regulate the direction of the beam.

The substance to be vaporized may further be fed to the point of impact of the rays at uniform speed and practically without interruption. In case of fusible substances, as metals, the vessel, which is necessary in this arrangement, will then contain a certain amount of molten material the weight of which is utilized for regulating feed. For example, operation may be such that the increasing weight of the vessel actuates either a switch or a stopping device which interrupts feed until a portion of the molten metal is vaporized, whereupon the lightened vessel releases the switch or device in reversed direction and thereby starts feeding again.

The invention is diagrammatically illustrated in the accompanying drawings which show means for carrying out the method.

Figure 1 shows a vaporizing vessel provided with an electron lens system and a hot cathode.

Fig. 2 shows a vaporizing vessel provided with a diaphragm, two electron lens systems and a cold cathode.

Fig. 3 shows a vessel provided with a substance to be vaporized that is movably disposed and means for electrically influencing the course of the rays.

Fig. 4 shows a vaporizing vessel provided with a plurality of diaphragms and continued feed of material to the place of vaporization.

In the example shown in Fig. 1 the vacuum vessel **1** accommodates in its upper portion a filament **2** serving as cathode. In the enlarged lower portion of the vessel **1** a block **4** of material to be vaporized rests on a support **3**. **5** designates the discharge connection for a vacuum pump. The objects to be subjected to the coating action of the vapor are located at **6** and **7**. The vessel **1** is grounded at **8**. The electronic rays **9** emitted from the filament **2** in various directions are refracted by an electron lens having the form of a coil **10** toward the block **4** and impinge upon its surface at **11**.

The construction shown in Fig. 2 possesses, in addition to the electron lens **10**, a second electron lens **10'** and a diaphragm **12**. Projected from a cold cathode **13** at an angle of a few degrees, the electronic rays **9** are refracted by the electron lens **10'** toward the diaphragm **12** and focused in the rear of the latter by the electron lens **10** at **11** on the block **4** in the same way as shown in Fig. 1. Through a connection **14** air or a gas is continuously supplied in quantities that can be regulated by a valve **38**, and drawn off through the connection **5**. The pressure in the upper portion **15** of the vessel **1** is therefore greater than in the lower space **16**.

Fig. 3 shows the lower portion **17** of a vacuum vessel in which the block **4** rests on a rotatable table **19** driven by a small motor **21** through a worm gear **20**. In the space **17** two plates **22**, **23** are provided between which the electronic ray **9** controlled by the coil **10** extends and to which voltage can be applied so as to permit displacement of the point of impact **11** on the block **4**.

In the vessel shown in Fig. 4 two additional diaphragms **24**, **25** are provided besides the central diaphragm **12**. The spaces between the three diaphragms are connected to air pumps, not shown, by the members **26**, **27** acting as suction connections, and in the upper space **15**, near the diaphragm **24**, another suction connection **28** serves for removing part of the air supplied through the connection **14** from this space to reduce the air current passing through the diaphragms. The air in the lower space **16** is drawn off at **5**. With the aid of two rolls **29**, **30** the substance **37** to be vaporized is continuously fed in the form of wire to a crucible **31** mounted on a lever **32** rotatably disposed at **33** and drawn up by a spring **34**. On the other arm of the lever **32** a switch **35** is provided which is normally closed. The crucible **31** is shown to contain a small amount of molten material **36**. When the feeding speed of the material is too high, an excessive quantity thereof will accumulate in the crucible **31** which thus becomes too heavy and by its weight moves the left arm of the lever **32** in downward direction against the action of the spring **34** with the result that the switch **35** is opened and the drive of the feed rolls **29**, **30** stopped. After sufficient material has vaporized from the crucible **31** and the weight thereof decreased, the crucible is drawn up by the spring **34** and the switch **35** closed so that feeding can continue.

I claim:

1. The method of vaporizing substances in a vacuum comprising producing electronic rays, subjecting vaporizable substances to the direct heating action of said electronic rays by placing the substances in the direct path of the rays, thereby causing the rays to impinge on the substance and develop the heat required for vaporization directly therein by said electronic rays, partitioning into two spaces the area between the point where the rays are produced and the position of the substances to be vaporized so as to inhibit interchange of pressure while permitting unimpeded passage of the electronic rays through said partition, and producing a constant air current between said spaces to maintain difference in pressure between them by the continual supply of air to one space and removal of air from the other space.

2. The method as claimed in claim 1, including separating said spaces by intervening spaces and inhibiting interchange of pressure between spaces while permitting unimpeded passage of electronic rays from one space to the other while producing a constant current of air between spaces and maintaining a gradual variation in pressure between adjacent spaces by supplying air to one space and removing air from an adjacent space.

3. The method as claimed in claim 1, including separating said spaces so as to inhibit interchange of pressure while permitting unimpeded passage of electronic rays from one space to the other, the electronic rays produced in one space being electron-optically refracted into the second space and electron-optically concentrated therein on the substance to be vaporized.

4. The method as claimed in claim 1, in which the substances to be vaporized are supplied in a uniform and continuous manner and the supply is automatically interrupted by the weight of molten substance when an excess of non-vaporized substances has accumulated at the vaporizing point.

5. The method of vaporizing a substance in a vacuum, comprising producing electronic rays with a source located within a zone maintained at sub-atmospheric pressure, placing a vaporizable substance within said zone in spaced relation to said source of electronic rays, and modifying the normal path of travel of rays emitted by said source by passing rays from the source through a field of force to converge and concentrate said rays onto the vaporizable substance whereby to heat the latter and cause it to vaporize.

6. The method of vaporizing a substance of a vaporizable character in a vacuum comprising producing electronic rays with a source located within a zone maintained at sub-atmospheric pressure, concentrating rays emitted by said source into a ray bundle and bringing the rays in the bundle to a focus by passing rays emitted by said source through a field of magnetic force, and subjecting a vaporizable substance within said zone to the action of the concentrated rays by placing the substance directly in the path of the rays in said bundle at a position where the bundle is of small cross section whereby to heat and vaporize the substance.

7. The method as set forth in claim 6 wherein the vaporizable substance is located substantially at the focal point of said rays.

8. The method as claimed in claim 1, including separating said spaces so as to inhibit interchange of pressure while permitting unimpeded passage of electronic rays from one space to the other, and directing a current of a gas non-combinable with the substances to be vaporized into both spaces to maintain a difference in pressure between them by the continual supply of gas to one space and removal of gas from the other space.

9. The method of vaporizing substances of low heat conductivity in a vacuum comprising producing electronic rays, subjecting a vaporizable substance to the heating action of said rays by directing the rays on to an area on a surface of said substance and into impact with that portion of the surface within said area whereby to cause the rays to heat and vaporize said substance, causing the path of travel of said rays to traverse the area of the surface, and additionally moving said substance with respect to the path of travel of the rays so as to bring said rays into impact with different areas on said surface.

RUDOLF RÜHLE.

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