METHOD FOR REPRODUCIBLY FABRICATING AND USING STABLE THERMAL-FIELD EMISSION CATHODES

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ABSTRACT
Methods are disclosed for reproducibly fabricating and using <100> thermal-field built-up field emission cathodes through the selective thermal elimination of contaminants and preservation of specific surface oxides in conjunction with the application of an E-field to promote faceting of certain planes while discouraging faceting of the [100] planes. An example in which <100> oriented tungsten wire is used as a starting material is treated comprehensively.

45 Claims, 9 Drawing Figures
PREPARE CATHODE ASSEMBLY USING A METALLIC WIRE HAVING A (100) BODY-CENTERED CUBIC CRYSTALINE STRUCTURE, THE END OF WHICH IS FORMED INTO AN EMITTER TIP WITH AN EFFECTIVE RADIUS BETWEEN 500 AND 4000 Å. SPOT WELD WIRE TO HAIRPIN FILAMENT SUPPORT STRUCTURE.

HEAT THE CATHODE IN PARTIALLY EVACUATED CHAMBER TO DESORB CONTAMINATES INCLUDING CARBON (AS A VOLATILE OXIDE, CO₂). DO NOT EXCEED THE TEMPERATURE AT WHICH A THIN RESIDUAL LAYER OF OXIDE OF THE METALLIC WIRE WOULD BE REMOVED FROM THE EMITTER TIP.

WHILE HEATING CATHODE TO MAINTAIN SURFACE MOBILITY AT THE EMITTER TIP, APPLY AN E-FIELD TO INITIATE (100) BUILDUP.

MAINTAIN ANODE/CATHODE CURRENT BELOW PREDETERMINED MAXIMUM AS (100) BUILD-UP OCCURS. STABILIZE CURRENT AT DESIRED LEVEL.

TEST FOR BUILD-UP.

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OPERATE

TEST FOR EMISSION DETERIORATION.

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INTRODUCE LIMITED AMOUNT OF OXYGEN INTO CHAMBER.

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TEST FOR EMISSION DETERIORATION.

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fig. 1
CONSTRUCT CATHODE ASSEMBLY BY SPOT WELDING A (100) TUNGSTEN WIRE TO A HAIRPIN FILAMENT SUPPORT STRUCTURE.

ELECTROCHEMICALLY ETCH AND POLISH ONE END OF THE WIRE TO FORM A CONICALLY SHAPED EMITTER TIP HAVING AN EFFECTIVE RADIUS OF BETWEEN 500 AND 4000 Å.

MOUNT THE CATHODE ASSEMBLY ADJACENT AN ANODE IN AN ENVIRONMENTALLY CONTROLLABLE CHAMBER.

EVACUATE CHAMBER TO THE 10⁻⁸ TORR RANGE.

FLASH HEAT THE CATHODE ASSEMBLY A FEW TIMES TO THE 1700-1900°C RANGE (BUT LESS THAN APPROXIMATELY 2000°C) TO OUTGAS THE ASSEMBLY AND DESORB CONTAMINANTS (ESPECIALLY CARBON AS CO₂) WHILE NOT REMOVING THE RESIDUAL LAYER OF TUNGSTEN OXIDE PRESENT ON THE EMITTER TIP FROM PREVIOUS EXPOSURE TO OXYGEN.

WHILE HEATING THE CATHODE ASSEMBLY TO MAINTAIN SURFACE MOBILITY AT THE EMITTER TIP (1500-1800°C), APPLY SUFFICIENT VOLTAGE BETWEEN THE ANODE AND CATHODE TO CAUSE CURRENT FLOW OF APPROXIMATELY 10-20 μA.

AS CURRENT (OR IMAGE TRANSMISSION) INCREASES, INDICATING (100) BUILD-UP, LIMIT CURRENT TO BETWEEN 50 AND 100 μA BY DECREASING THE ANODE/CATHODE VOLTAGE. STABILIZE CURRENT TO DESIRED LEVEL.

TEST FOR (100) MODE BUILD-UP. HAS SHARP CURRENT INCREASE OCCURRED? ARE TRANSMISSION CHARACTERISTICS ADEQUATE?

NO

TEST FOR EMISSION DETERIORATION. HAS CURRENT LEVEL DROPPED? IS IMAGE TRANSMISSION POOR? (E.G. AFTER EQUIPMENT SHUTDOWN)?

NO

YES

INTRODUCE A SMALL PARTIAL PRESSURE OF OXYGEN INTO CHAMBER. HEAT FOR FEW SECONDS AT 1300-1400°C.

YES

OPERATE

fig. 9
METHOD FOR REPRODUCIBLY FABRICATING AND USING STABLE THERMAL-FIELD EMISSION CATHODES

This invention relates generally to field emission cathodes and, more particularly, to <100> oriented built-up thermal field emission cathodes and methods for fabricating and using the same.

The desirability of an electron source capable of issuing a finely focused beam of electrons having a stable high current density has become increasingly pronounced with the widespread use and refinement of such electron beam applications as scanning electron microscopes, electron microprobes, integrated circuit mask fabrication, and electron beam access computer memory devices. As a result, field emission cathodes have received renewed interest because maximum current densities much greater than those associated with thermionic cathodes may be achieved. Also, the virtual electron source of a field emission cathode may be much smaller rendering it possible to focus an electron beam into a correspondingly smaller spot size without the need for extensive additional demagnification or focusing apparatus. However, practical use of field emission cathodes has not been realized heretofore because of instability and limited life of such cathodes under practical vacuum conditions. Under commercially practical vacuum conditions, undesirably high flicker noise is experienced with field emission cathodes operated at room temperature.

Consideration has been given to the utilization of heated field emission cathodes to increase cathode life in less drastic vacations and to theoretically increase current density and other characteristics of the issued electron beam. The expected performance enhancement of thermal field emission cathodes, according to the best known prior art practice, follows the ability to balance surface tension with electric field stress forces to shape the cathode tip into a better emission shape, i.e., a very sharp point. However, investigation has revealed undesirable events which routinely occur when a thermal field emission cathode is fabricated and operated according to this approach. Appreciable emission can only be achieved if the E-field is increased beyond the "balance" value. But, when this value is exceeded, the geometry of the emitter tip changes randomly with time, causing current fluctuations. These changes in emitter geometry also drastically alter the emission spatial distribution which is intolerable for fine focus applications. Thus, thermal field cathodes, while theoretically affording numerous advantages, have found little practical application because it has generally been believed that no endform appropriate to high current density, narrow beam angle operation could be expected to reach a state of equilibrium with respect to further geometric alterations. Also, a necessity for operating in an extremely low pressure environment has required costly and unwieldy utilization and peripheral equipment.

Nonetheless, two additional important phenomena occur during thermal field operation which, in conjunction with the previously noted advantages, have resulted in continued investigation of thermal field emission cathodes as electron sources. First, the adsorption of residual gases on the emitter surface, which cause undesirable current reduction and noise, can be eliminated as a result of thermal desorption. Second, microscop surface roughness due to ion bombardment, which causes current density and spatial distribution instability, is inhibited because the micro-roughness sites are immediately smoothed by the surface tension of the heated cathode.

In view of the theoretical advantages discussed above, it will be readily apparent to those skilled in the art that it would be highly desirable to realize stable thermal field emission operation with a sufficiently small emitter tip radius to achieve high current density.

It is therefore a broad object of this invention to establish a process for fabricating thermal field, built-up field emission cathodes.

It is a corresponding object of this invention to provide a thermal field built-up field emission cathode with a tip of very small radius and stable geometric configuration.

It is another object of this invention to provide such a cathode which exhibits an extremely long life even when operating in vacuums substantially less drastic than is usual in the prior art.

Still another object of this invention is to provide such a cathode which issues a finely focused beam of electrons having very high current density, which beam may readily be refocused into a very small spot size with simple electrostatic or magnetic lens means.

A further object of this invention is to provide a method for building up such a cathode from a body centered cubic crystal lattice structure of tungsten, molybdenum or the like, having Miller indices <100> orientation in the axial direction.

It is a further object of this invention to provide a method whereby the foregoing objects are reliably and reproducibly achieved under elevated temperature conditions.

Another important object of this invention is to provide a method for making and repeatedly using such cathodes under practical and commercially economical vacuum conditions without the necessity of subjecting the cathodes to severe temperature conditions.

It is yet another object of this invention to provide a method for using such cathodes in conjunction with a wide range of D.C. or pulse operated cathode utilization devices and to thereby improve the overall operation of such devices by providing a stable, long life, high current density electron source.

The subject matter of the invention is particularly pointed out and distinctly claimed in the concluding portion of the specification. The invention, however, both as to organization and method of operation, may best be understood by reference to the following description taken in connection with the accompanying drawing of which:

FIG. 1 is a generalized flow chart setting forth the basic concepts of the present invention.

FIG. 2 is a representation of a body centered cubic structure illustrating the Miller indices and planes of interest in the crystal lattice of wire from which a field emission cathode according to the present invention may be fabricated.

FIG. 3 is a greatly magnified view of an emitter tip after initial preparation according to the prior art and representing one stage in the present invention.

FIG. 4 illustrates a hairpin cathode structure incorporating a field emission tip.
FIG. 5 illustrates the tip shown in FIG. 3 after sufficient heat has been applied to permit surface tension to affect the cathode tip shape.

FIG. 6 illustrates the tip shown in FIG. 5 after it has been built up pursuant to the method of the present invention.

FIG. 7 is a stylized view of that portion of the tip shown in FIG. 6 which lies about the plane 7—7.

FIG. 8 is a schematic illustration of a scanning electron microscope presented as an exemplary environment in which the present invention may be practiced.

FIG. 9 is a more detailed flowchart setting forth specific steps in fabricating, forming and using a cathode tip according to exemplary practice of the present invention.

Thermal field emission cathodes are fabricated and used in practicing the present invention from a single crystal <100> oriented wire. That is, crystallographic alignment of the cubic face of the single crystal unit cell is perpendicular to the axis of the wire. The <100> oriented wire may actually have the (100), (010) or (001) planes, according to the Miller indices notation, facing the direction of the wire length. Such <100> oriented wire of tungsten or a like metal, may be prepared by zone melting techniques known in the prior art.

In order to fully understand the present invention, it is necessary to have an elementary knowledge of Miller indices notation as it is utilized to characterize body centered cubic crystalline structures such as tungsten and molybdenum. Consider the body centered cubic structure illustrated in FIG. 2 in which the single unit cell 1 comprises nine atoms, eight of the atoms, 3, 4, 5, 6, 9, 10, 11, 12, being disposed at the corners of a "cube" and the ninth atom, 2, being disposed in the center of the "cube." To arrive at a basis for Miller indices designations, a line drawn between two vertically aligned atoms is deemed a Y axis, a line drawn between horizontally aligned atoms along a first side of the "cube" is deemed an X axis, and a line drawn between horizontally aligned atoms in a direction perpendicular to both the X and Y axes is deemed the Z axis. The three axes cross at a reference atom 4 in the example shown in FIG. 2.

Planes passing through the cubic structure may be defined by a three digit number. The first number designating the position in the "cube" at which the X axis is crossed, the second number indicating where the Y axis is crossed, and the third number indicating where the Z axis is crossed. This, as shown in FIG. 2, the (100) plane in FIG. 2 crosses the X axis at unity and includes four atoms comprising one side of the "cube" while never crossing either the Y axis or the Z axis of the specific molecule. Similarly, the (010) plane crosses only the Y axis and the (001) plane only the Z axis. It will be recognized that the designation of these planes is merely a matter of nomenclature such that the (100), (010) and (001) planes may be deemed the [100] family of planes.

Any one of a number of planes passing through the unit cell 1 may be designated, according to the Miller indices, by dividing the distance between adjacent atoms lying along the three reference axes into increments. Hence, by way of example, the atom 3 is disposed in the (1) position along the X reference axis which also passes through the reference atom 4 with intermediate second, (2), and third, (3), positions spaced, respectively, at positions one-half and one-third of the distance to the (1) position. Similar designations are applied to positions between atom 5 along the Y axis and the atom 6 along the X axis. Thus, a (112) plane would pass through the atom 3 in the X axis, the atom 5 in the Y axis, and position (2) between the atom 6 and the reference atom 4 lying on the Z axis. This (112) plane would therefore pass through the unit cell 1 as generally indicated by the lines 7a, 7b, and 7c. As another example, a (310) plane would pass through position (3) between the atoms 3 and 4 and through the atom 5, but would never intercept the reference Z axis.

This plane, which is parallel to the reference Z axis, is represented by the lines 8a, 8b, 8c, and 8d. Those skilled in the art will perceive the manner in which the relationship of other planes to the basic body centered cubic crystalline structure of the unit cell 1 may be defined utilizing Miller indices notation.

Theoretical and prior art empirical investigations into the utilization of field emission cathodes reveal that the following relation holds approximately for a point cathode:

$$E = BV$$

where in which the factor B is approximately the reciprocal of five times the effective tip radius r of the cathode; V is the anode voltage and E is the electric field strength at the emitter surface. The density of current which may be drawn from a cathode at room temperature with a known radius of curvature may be calculated from the following equation:

$$J = \frac{a e^2}{4} \exp \left[-B \varphi^2 / 2E\right]$$

where J is the current density in amperes per square centimeter, E is the intensity of the electric field applied at the tip in volts per centimeter, \(\varphi\) is the work function of the tip material in electron volts, B is a constant equal to 6.83 \times 10^3 in the above units, and a is a function of \(e = 3.5 \times 10^{-7} \text{A/V}^2\) for tungsten with \(\varphi = 4.5 \text{eV}\).

Thus, it is apparent the maximum current density which should have the highest possible value, is limited by the relative dullness of the tip. That is, the smaller the effective tip radius r, the higher the current density so long as the other factors remain within practical constraints.

The best known prior art approach to initially preparing a built-up field emission cathode having a faceted shape has been to first electrochemically micropolish and etch tungsten wire into the shape generally depicted in FIG. 3. An effective tip radius in the range 500 – 3,000A. is achieved after flash heating to smooth and dull the tip under the influence of surface tension. The effect of such dulling is illustrated in FIG. 5, where the letter r indicates the effective tip radius of the emitter.

When the emitter tip is exposed to an E-field, emission is initiated from the low work function (or high B) regions of the emitter and usually is contained in a total angle of approximately 60° centered on the emitter apex. In order to initiate build-up of the emitter surface, the emitter temperature must be raised to a tem-
perature sufficient to insure surface mobility. The electrostatic force of the E-field causes a migration of surface atoms toward the emitter apex leading to emitter build-up.

The strength of the field at which a "balance" is achieved between surface tension dulling forces and the electrostatic build-up forces is given by the equation:

$$E_0 = \gamma r/\gamma r$$

where $\gamma$ is the surface tension which is approximately 2,900 dynes/cm for tungsten at 2,000°K. Although a "balanced" condition is achieved at $E_0$, a minimum field strength of $4 \times 10^7$ V/cm is required to achieve even marginally useful emission from the controls. Accordingly, it follows from equation (3) that $r$ cannot exceed approximately 800A. Thus, this approach (i.e., to operate at a balance point that retains the emitter shape of Fig. 5) has a distinct limitation in that the current density which can be achieved before radical geometric deformation of the tip (with a corresponding drastic change in local $r$) is strictly limited to a value which is orders of magnitude lower than is desirable or useable in most commercial applications. Also, because the E-field is not uniform over the emitting area, the condition specified in equation (3) occurs only at local regions of the emitting area. This leads to a generally unstable geometric condition except in the <100> build-up mode described herein.

It has been known for some time that heating a field emission cathode previously processed to the configuration illustrated in Fig. 3 in the presence of an electrical field approximating $4 \times 10^7$ V/cm (i.e., beyond the "balance" value) results in "build-up" of the emitter surface through surface migration at the tip. Therefore, in operation the emitter may be operated stably at room temperature provided that a sufficiently high vacuum condition is maintained.

The manner of fabricating a built-up field emitter according to this approach and operating at room temperature (including the limitations thereof), is treated in an article entitled "Electron Gun Using A Field Emission Source," by A. V. Crewe, D. N. Eggenberger, J. Wall, and L. M. Welser, The Review of Scientific Instruments, Vol. 39, 576-583, April 1968.

"Build-up" is a term of art describing a process by which surface migrating atoms are directed by externally imposed electrostatic forces to regions of minimum energy resulting in the lateral growth of planes having low Miller indices and ultimately leading to the development of large facets. One may refer to an article entitled "Activation Energy for the Surface Migration of Tungsten in the Presence of a High Electric Field" by P. C. Betler and F. M. Charbonnier, The Physical Review, Vol. 119, No. 1, 85-93, July 1, 1960, for a comprehensive discussion of the interrelated phenomena of build-up and faceting.

Various modes of tip build-up and faceting have been observed in tungsten cathodes. Build-up occurs when successively smaller overlaying planes extend outwardly from the emitter tip. Faceting of other planes necessarily accompanies such build-up to constitute the sides of the built-up emitter. One observed build-up mode leads to bright emission sites at the [112] faces due to faceting of the [110] and [100] planes. Another mode of build-up leads to bright emission from the [310] planes in which case faceting of the [100], [112], and [110] planes occur simultaneously.

Yet another, and a very important, mode of build-up causes bright emission sites at the [100] planes because of faceting of the [110], [112], and [310] planes. By far the greatest reduction in effective local tip radius $r$ and enhancement of beam angular confinement occurs with the <100> build-up in <100> -built-up site by 90°. The decrease in the E-field with apex angle at 90° is sufficient to preclude significant emission from the 90° (100) built-up sites. In contrast, other built-up endforms result in the plurality of emission sites within an angular distribution sufficiently close to the longitudinal axis of the emitter to allow for significant emission from all sites. This results in a wide angle of emission.

The highly desirable <100> build-up has heretofore been observed only occasionally and transitorily as may be more readily understood with reference to an article entitled "Angular Confinement of Field Electron and Ion Emission" by L. W. Swanson and L. C. Crouser, Journal of Applied Physics, Vol. 40, No. 12, 4741-4749, Nov., 1969. However, it has been generally believed that no field built-up endforms including <100> build-up from <100> oriented wire, could be expected to reach a state of equilibrium under conditions of thermal field emission because of two factors which have been thought unavoidable. First, the geometry of the emitter tip has changed apparently randomly with time causing severe current density fluctuations because of the change in local tip radius $r$. Second, these changes in emitter geometry also drastically alter the emission spatial distribution which is intolerable for fine focus applications in which it is necessary to confine the beam, by whatever means possible.

I have discovered, contrary to the prior art investigations and to the general belief among those skilled in the art, that <100> oriented built-up emitter tip endforms having extremely small effective radii may be reliably and reproducibly fabricated from <100> oriented wire and operated stably in the thermal field mode.

The reasons <100> field build-up occurs unexpectedly and preferentially under the conditions and procedures set forth below are not yet entirely clear; however, it is believed that three factors are of great importance in reliably achieving <100> build-up in accordance with the present invention. First, the slightest carbon contamination, which is almost unavoidably present with operation at tip temperatures in excess of 1,200°K, but below the temperature for thermal desorption of carbon, appears to preferentially lower the surface free energy of the [100] planes which leads to non,<100> build-up. Second, the presence of an oxide surface observed with tip temperatures below approximately 1,950°K appears to preferentially increase the surface free energy in the [100] planes over that of the [112] and [310] planes, thereby promoting <100> build-up. Third, the high degree of field localization as to the small area of the tip where $\beta$ is large allows the balance conditions (e.g. 3) to be satisfied at one point.
on the emitter surface with other areas of the surface not experiencing sufficient build-up fields to deform further.

The basic concept of the present invention may be preliminarily grasped with reference to the simplified flow chart of FIG. 1. A cathode hairpin assembly, FIG. 4, is prepared according to the previously referenced, known techniques. It will be understood that the hairpin assembly 20 illustrated in FIG. 4 may be fabricated from tungsten and incorporates a <100> oriented tungsten emitter wire 21 terminating in a tip of which FIG. 3 may be considered a greatly magnified view. The hairpin assembly is mounted in a partially evacuated chamber and heated sufficiently to desorb contaminants, including carbon as a volatile oxide thereof. However, care is taken to avoid exceeding a temperature at which a thin residual tungsten oxide layer would be thermally removed.

While maintaining the cathode assembly sufficiently hot to permit surface migration at the tip, a relatively strong E-field is applied to initiate <100> build-up which is, at this stage, the preferred mode because the effective surface free energy of the [100] planes has been raised while the effective surface free energy of the [100], [112] and [310] planes have been lowered. The [100] planes therefore prefer to build-up and the [110], [112] and [310] planes prefer to facet.

FIG. 6 illustrates an emitter tip which has been formed in accord with the instant invention. The arrow aligned with the axis of the wire indicates the <100> direction while any other arrow, perpendicular to the axis of the wire, indicates the <010> direction. At any given time the apex 53 of the emitter tip never converges in a perfect point but assumes a very slightly smattered configuration. The faceted [110] planes, 50, gradually merge into four [010] build-up planes 90° removed from the apex 53 of the wire and circumferentially spaced by 90° around the sides of the wire. These [010] build-up planes are little more than "bulges" in the sides of the wire since they are axially disposed at 90° with respect to the E-field.

FIG. 7 is an idealized view of that portion of the emitter tip located above plane 7—7 in FIG. 6. As can be seen, the emitter tip generally tetrahedral in shape with an apex 53 pointing in the <100> direction, four sloping sides (e.g. 50, 51) formed as a result of [110] faceting and a base 52 defined by [100] plane 7—7.

As build-up proceeds, the tip end 23 becomes smaller and the effective local tip radius r decreases correspondingly. As a result, current density and total current is observed to increase in accordance with equation (2) above. It is therefore necessary to adjust the stress of the E-field downwardly as the build-up proceeds to hold the total current below a value at which catastrophic or geometrically destructive vacuum arcing would occur. When the current stabilizes, <100> build-up to an exceedingly fine point has been achieved, and the cathode assembly is ready for operation.

The foregoing, of course, is only a brief description of the process to which the present invention is directed. In order to fully teach a specific embodiment to those skilled in the art, the following more detailed discussion may be studied in conjunction with the correspondingly more detailed flow chart of FIG. 9 and the schematic diagram of FIG. 8 which illustrates as an exemplary operational environment, a scanning electron microscope.

As previously noted, the first step in forming a <100> built-up thermal field cathode from <100> oriented wire is to form the tip 22 of the emitter 21 to the configuration illustrated in FIG. 3 by appropriate prior art techniques. This step may effectively be carried out by following the previously described electrochemical procedure.

Referring to FIG. 4, the <100> oriented tungsten emitter 21 may be on the order of 0.003 inch to 0.010 inch in diameter, and be formed through spot or electron beam welding to the tungsten hairpin support structure 20 which may have a diameter on the order of 0.010 inch. As shown in FIG. 8, the entire cathode structure 32 may be mounted in a tube embodiment including chamber 30 which may thereafter be evacuated to the 10⁻⁸ torr range by means of the vacuum pump 31 which communicates with the interior of the chamber 30. The temperature of the cathode structure 32 is then elevated sufficiently to outgas impurities and smooth the emitter surface as shown in FIG. 5. This is achieved by passing a pulse of current from a D.C. (or A.C.) energy source 35 through the tungsten hairpin 20 to bring the cathode structure 32 to a temperature of between 1,700° to 1,900°K during several short temperature flashes. The temperature to which the cathode structure 32 is raised may readily be controlled by adjusting current limiting resistor 34. The object of this step is to drive out all contaminants from the emitter tip 22 (FIG. 3) except for a thin residual oxide layer left on the emitter as a result of its inevitable prior exposure during initial preparation to free oxygen in the ambient atmosphere.

Any bulk carbon present in the surface region diffuses to the surface and combines with oxygen present within the initial oxide layer or within the chamber 30 to form oxides of carbon, principally carbon monoxide. The oxides of carbon are thermally desorbed at a rate dependent upon the temperature of the cathode structure 32 such that more rapid desorption may be achieved by heating the structure at higher temperatures within the range. However, an upper limit to the temperature which may be imposed on the cathode structure 32 during this and the subsequent steps will be noted below.

As shown in FIG. 6, the tip 22 endform has become hemispherical with an effective radius r due to surface tension acting upon the increasingly mobile heated cathode structure 32. It may be noted that, if sufficient carbon contamination is present in the <100> oriented tungsten wire, it may be necessary to introduce a small amount of supplementary oxygen into the chamber 30 while heating the emitter at temperatures between 1,300°K and 1,500°K in order to achieve a complete combination of the surface and bulk carbon with oxygen. If high purity tungsten wire is used, the oxygen present in the starting material is usually adequate to carry out the purging step. Generally there will be a sufficient amount of oxygen present, either in an adsorbed elemental form or in the form of tungsten oxide, to combine with the carbon and thereby form a volatile oxide. Carbon may also be thermally desorbed directly from the surface by heating to T > 2,500°K; this, however, will cause considerable dulling of the emitter which may be undesirable in many cases. This latter procedure may also require the subsequent step of in-
Introducing a small partial pressure of oxygen which calls for more elaborate equipment and this increases the ultimate cost of commercial equipment.

To insure complete outgassing of the tip, the cathode structure 32 may be flash heated several times to the higher temperature in the aforementioned range, but the temperature must not be allowed to exceed a temperature on the order of 1,950°K. More specifically, the temperature should not exceed that temperature in a specific environment at which a very thin residual layer of tungsten oxide, which normally overlays the cathode structure 32, including the tip 22, would be completely driven off leaving a clean tungsten tip. The tip condition sought is with all carbon contamination removed and a thin tungsten oxide residual layer remaining.

Field build-up of the tip 22 may now be instituted by applying a voltage stress between the emitter tip 22 and a nearby anode 36. The emitter must be maintained sufficiently hot (1,400° to 1,800°K) to assure surface mobility without, however, exceeding the previously noted upper temperature limit at which the tungsten oxide layer would be removed.

The E-field is increased until the “balance” value is exceeded whereupon stable and predictable build-up commences. Initial current at the start of the build-up process is typically about 20 μA. Total current is observed to remain constant for a time and then to increase rapidly with time, indicating <100> build-up. After build-up has occurred, it is desirable to limit the maximum total current to the 50–100 μA range by commensurately decreasing the cathode-anode voltage and hence the E-field. Total current stabilization indicates completion of build-up providing an exceedingly fine, axially aligned cathode tip 23 as shown in FIG. 6. In practical applications such as the scanning electron microscope of FIG. 8, high quality image transmission will indicate completed build-up. In general, beam transmission through a small aperture is the best indication of (100) build-up; similarly, tip degeneration is indicated by decreased transmission.

The object of the just described field build-up process is to achieve the pyramidal type structure shown in FIGS. 6 and 7. In obtaining <100> build-up of tungsten, the {100}, {112} and {310} planes form large facets, such as the facet 50 and 51 shown generally in FIG. 7. Because the partially built-up {010} planes shown in FIG. 6 are 90° removed from the principal E-field they produce no significant electron emission and substantially all of the longitudinally directed electron emission is from the tip apex 53 and is confined to a narrow angle.

It has been determined that, upon attaining the built-up endform illustrated in FIGS. 6 and 7, the emitter can be operated for very long periods of time in a temperature range of 1,200° to 1,700°K with a total current range of 1–300 μA in a pressure environment in the 10⁻⁷ – 10⁻⁸ torr range. A lower range of temperatures can be reliably used if the pressure is in the 10⁻⁹ torr range. Short periods of operation at even lower temperatures, including room temperature, can be realized, the length of the useful period being inversely dependent on the voltage applied to the emitter. As those skilled in the art will understand, the limit for minimum spot size in the focused beam is partly due to chromatic aberration which is related to the width of the energy distribution. Since the width of the energy distribution increases with temperature, it may indeed be desirable to operate at such low temperatures for short periods of time.

Should the emitter be heated above approximately 2,000°K in high vacuum, the oxide layer will be removed. A clean emitter which remains free of all traces of carbon contamination will, according to the laws of probability and as a function of the hierarchy of surface free energies associated with various families of planes undergoing <100> build-up when the foregoing procedures are followed. However, <100> build-up under such conditions is not predictable, and, of course, will not occur at all if carbon is present.

During the course of operation at low temperatures, or during inadvertent operation at too high a temperature, the emission properties of the emitter may deteriorate. It has been found that the original tip condition can be restored in situ by relaxing the E-field for 15 to 30 seconds at a temperature in the 1,600° to 1,700°K range and by then applying sufficient voltage to draw 10 to 20 μA total current. The ambient in which the emitter is working should also, of course, contain sufficient oxygen to restore the oxide layer and remove any surface carbon contaminants derived from the bulk or carbonaceous adsorbates. Current stabilization indicates reformation of the tip. Where emission deterioration is due to the collection of environmental contaminants on the cathode, it may be necessary to again perform the flash heating step before rebuilding the tip in situ, as has been alternatively indicated by the broken lines in FIGS. 1 and 9.

Referring once again to FIG. 8, scanning electron beam apparatus is illustrated in which a narrow beam 40 issuing from the cathode structure 32 may be focused on a grounded specimen 41 by means of a single electrostatic (or electromagnetic) lens 42. The cathode structure 32, anode 36, lens 42 and grounded specimen 41 are maintained at progressively higher potentials (-10,000 volts, -8,000 volts with -800 volts and 0 volts, respectively) as indicated by sources 33, 38 and 44. The sweep generator 45 of a display tube 46 is coupled into deflection system 47 to cause the beam 40 to scan the specimen 41 for detection by the detector 48 and display on the tube 46. In such an exemplary environment, if the tip is inadvertently (or if the (100) build-up endform deteriorates, it may be reformed in accordance with the above procedures without removal from the system. When the scanning electron microscope of FIG. 8 is turned off, the emitter temperature should be first reduced, to “freeze” the built-up configuration, and thereafter the anode/cathode voltage is turned off. When the electron microscope is to be used again, the voltage is first turned back on and then the cathode is heated to operating temperature. In the event that the foregoing "shut down" procedure is not followed, tip build-up will be lost but may be very early restored by repeating the heating and voltage application steps of the process.

One of the most significant properties observed during operation of a tip formed in accordance with the present invention is its ability to operate with extremely high current densities for long periods of time. This feature orders of magnitude higher than that considered possible with prior art field emission cathodes. The manifest savings in vacuum and other peripheral and directly related equipment provides a drastic economy, not only in operation, but also in initial investment.
As previously noted, molybdenum also has a body centered cubic crystalline structure, and corresponding results have been achieved with <100> oriented molybdenum wire using essentially the same process with somewhat lower temperatures. For example, the surface tension (γ) for molybdenum is on the order of 2,200 dynes/cm at 1,700K which alters the E-field intensity at which the "balance" point is achieved according to equation (3). The behavior of a molybdenum emitter fabricated according to the above-described process generally follows that of tungsten. It is again fundamentally necessary to drive off all carbon contamination while leaving a thin residual oxide layer such that the work function of the [100] planes is raised and that of the [110], [112], and [310] planes are lowered, thereby rendering <100> build-up preferential. Certain other metals, such as tantalum, may be expected to respond favorably according to the present process. However, tungsten is generally preferred as a starting material because of such properties as a high melting point, a low vapor pressure, relatively high electrical and thermal conductivity, and high mechanical strength.

Thus, while the principles of the invention have now been made clear in an illustrative embodiment, there will now be immediately obvious to those skilled in the art many modifications of procedure, structure, arrangement, proportions, the elements, materials, and components used in the practice of the inventions which are particularly adapted for specific environments and operating requirements without departing from those principles.

I claim:

1. A method for reproducibly fabricating a thermal field emission cathode capable of stable long-term operation under conditions of high current density emission in a relatively relaxed vacuum environment, said method including the steps of:
   a. forming a generally conical tip at one end of a Miller indices <100> oriented metal wire composed of a material having a body centered cubic crystalline structure;
   b. heating said metal wire in a partially evacuated enclosure to a first temperature, which first temperature is sufficiently high to result in the desorption of surface contaminants, including carbon as a volatile oxide thereof; while not exceeding a second temperature at which substantially all residual surface oxide of the metal would be desorbed;
   c. heating said metal wire in the partially evacuated enclosure to a third temperature, the third temperature being less than the second temperature, but sufficiently high to assure surface mobility at the wire tip;
   d. applying an electrical potential between the wire tip and an adjacent electrode, the applied potential being adequate to develop a field of sufficient magnitude to initiate build-up of the [100] planes and corresponding faceting of the [110] and [112] planes;
   steps c and d being performed in overlapping sequence whereby a stable thermal field emission cathode is formed having an axially disposed, generally tetrahedral endform, the apex of which is characterized by a relatively small effective radius of curvature.

2. The method of claim 1 in which magnitude of the field is not allowed to exceed a value at which arcing occurs between the tip and the electrode.

3. The method of claim 2 in which steps c and d are initiated in reverse sequential order.

4. The method of claim 2 in which steps c and d are carried out substantially simultaneously.

5. A process for fabricating a field electron cathode, disposed near an electrode in a partially evacuated, environment controllable chamber, from <100> oriented wire having an end thereof brought to a terminal radius less than approximately 5000A, said process comprising the sequentially interchangeable and overlapping steps of:
   a. heating the wire sufficiently to diffuse contaminants to the wire surface; and
   b. applying sufficient voltage between the wire and the electrode to draw a predetermined minimum total current;
   said process further comprising the subsequent step of:
   c. maintaining the total current flow between the wire and the electrode at a value less than a predetermined maximum until total current stabilization occurs.

6. The process of claim 5 in which the <100> oriented wire is selected from the group including tungsten and molybdenum.

7. The process of claim 5 in which the <100> oriented wire is tungsten and step a is carried out by heating the tungsten wire to a temperature sufficient to diffuse any bulk carbon to the wire surface such that the carbon combines with oxygen to form volatile oxides of carbon, which volatile oxides of carbon are thermally desorbed.

8. The process of claim 7 in which the temperature of the tungsten wire is always kept below a temperature at which a surface layer of tungsten oxide on the end of the wire would be removed.

9. The process of claim 7 in which the temperature of the tungsten wire is not allowed to exceed 1,900K.

10. The process of claim 7 in which the tungsten wire is heated to a temperature of at least 1,200K.

11. The process of claim 7 including an additional step in which supplementary oxygen is introduced into the chamber after steps a and b have been carried out whereby substantially all bulk carbon in said wire is thermally desorbed as a constituent the volatile oxides of carbon.

12. A method for fabricating a stable thermal field emission cathode for long-term usage under conditions of high current density emission in a relatively relaxed vacuum environment, the method including the steps of:
   a. forming a generally conical tip at one end of a Miller indices <100> oriented metal wire composed of a material having a body centered cubic crystalline structure;
   b. heating the metal wire in a partially evacuated enclosure to a first temperature, which first temperature is sufficiently high to cause the desorption of surface contaminants, including compounds of carbon, while not exceeding a second temperature at which the generally conical tip would undergo substantial geometric deformation;
c. heating the metal wire in the partially evacuated enclosure without exceeding a second temperature at which substantially all residual surface oxide of the metal would be desorbed; and

d. imposing an electrical potential between the wire tip and an adjacent electrode, the imposed potential being adequate to develop a field of sufficient magnitude at the wire tip to initiate build-up of the [100] planes and to initiate faceting of at least the [110] and [211] planes; steps c and d being performed in overlapping sequence, whereby a stable thermal field emission cathode is formed having an axially disposed, generally tetrahedral, endform, the apex of said endform being characterized by a relatively small effective radius of curvature.

13. The method of claim 12 in which the magnitude of the field is not allowed to exceed a value at which arcing occurs between the tip and the electrode.

14. The method of claim 13 in which steps c and d are initiated in reverse sequential order.

15. The method of claim 13 in which steps c and d are carried out substantially simultaneously.

16. The method of claim 13 in which step c is executed after introducing a small partial pressure of oxygen into the enclosure.

17. The method of claim 13 in which step d is executed after introducing a small partial pressure of oxygen into the enclosure.

18. A method for reproducibly fabricating a stable thermal field emission cathode for long-term usage under conditions of high current density emission in a relatively relaxed vacuum environment, the method including the steps of:

a. forming a generally conical tip at one end of a Mil ler indices <100> oriented metal wire composed of a material having a body centered cubic crystalline structure;

b. heating said metal wire in a partially evacuated enclosure, to a first temperature sufficiently high to cause the desorption of surface contaminants, including carbon as a volatile oxide thereof, while not exceeding a second temperature at which substantially all residual surface oxide would be desorbed from the wire tip;

c. maintaining said metal wire in said partially evacuated enclosure at a third temperature, the third temperature being less than the second temperature but sufficiently high to assure surface mobility at the wire tip responsive to surface tension; and

d. imposing an electrical potential between the tip of the metal wire and an adjacent electrode, said potential being of sufficient magnitude to initiate build-up of the [100] planes and to initiate faceting of at least the [110] and [211] planes, said potential being less than that potential at which geometrically destructive vacuum arcing occurs; said imposing step being sequentially interchangeable with said maintaining step;

whereby a stable thermal field emission cathode is formed having an axially disposed, generally tetrahedral, endform, the apex of the endform being characterized by a relatively small effective radius of curvature and a correspondingly small effective emission area.

19. The method of claim 18 in which steps c and d are initiated in reverse sequential order.

20. The method of claim 18 wherein the metal wire is of a material selected from the group of metals consisting of tungsten and molybdenum.

21. The method of claim 18 wherein the metal wire is composed of tungsten.

22. The method of claim 21 wherein the forming step is carried out in the presence of a partial pressure of oxygen sufficient to cause the surface formation of tungsten oxide layer at the tip of the metal wire.

23. The method of claim 22 wherein the first temperature is in the range of temperatures between 1,700° and 1,900°K.

24. The method of claim 23 wherein the second temperature is in the range of temperatures between 1,900° and 2,100°K.

25. The method of claim 24 wherein the third temperature is in the range of temperatures between 1,300° and 1,800°K.

26. The method of claim 18 wherein the pressure in the partially evacuated enclosure is in the range of pressures between 10⁻² and 10⁻⁶ torr.

27. A method for reproducibly fabricating a stable thermal field emission cathode for long-term usage under conditions of high current density emission in a relatively relaxed vacuum environment, the method including the steps of:

a. forming a generally conical tip at one end of a Miller indices <100> oriented metal wire composed of a material having a body centered cubic crystalline structure;

b. heating the metal wire in a partially evacuated enclosure to a first temperature, which first temperature is sufficiently high to bring about the desorption of substantially all surface contaminants, including carbon as a volatile oxide thereof, while not exceeding a second temperature at which the generally conical tip would undergo substantial geometric deformation;

c. heating the metal wire in the partially evacuated enclosure to a third temperature, which third temperature is lower than a temperature at which substantially all residual surface oxide of the metal would be desorbed, said metal wire being exposed to a limited partial pressure of oxygen sufficient to assure the presence of oxides on the surface of the metal wire;

and

d. imposing an electrical potential between the tip of the metal wire and an adjacent electrode, the potential being adequate to develop a field of sufficient magnitude to initiate build-up of the [100] planes and to initiate faceting of at least the [110] and [112] planes, the potential being lower than a potential at which geometrically destructive vacuum arcing occurs;

steps c and d being performed in overlapping sequence, whereby a stable thermal field emission cathode is formed having an axially disposed, generally tetrahedral, endform, the apex of which endform is characterized by a relatively small effective radius of curvature and a correspondingly small effective emission area.

28. The method of claim 27 in which steps c and d are initiated in reverse sequential order.

29. The method of claim 27 in which steps c and d are carried out substantially simultaneously.

30. A method for reproducibly fabricating a stable thermal field emission cathode for long-term usage under conditions of high current density emission in a
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relatively relaxed vacuum environment, the method including the steps of:

a. forming a generally conical tip at one end of a Miller indices \(<100\>\) oriented metal wire composed of a material having a body centered cubic crystaline structure;

b. heating the metal wire in a partially evacuated enclosure to a first temperature, which first temperature is sufficiently high to bring about the desorption of substantially all surface contaminants, including carbon as a volatile oxide thereof, while not exceeding a second temperature at which the generally conical tip would undergo substantial geometric deformation;

c. maintaining the metal wire in the partially evacuated enclosure at a third temperature, which third temperature is lower than a fourth temperature at which substantially all residual surface oxide would be desorbed;

d. forming oxides of the metal comprising the wire on the tip surface thereof; and

e. imposing an electrical potential between the tip of the metallic wire and an adjacent electrode, the potential being adequate to develop a field of sufficient magnitude to initiate build-up of the \([110]\) planes and to initiate faceting of at least the \([110]\) and \([112]\) planes, said potential being lower than a potential at which geometrically destructive vacuum arcing occurs;

whereby a stable thermal field emission cathode is formed having an axially disposed, generally tetrahedral, endform, the apex of which endform is characterized by a relatively small effective radius of curvature.

31. The method of claim 30 in which steps \(c\) and \(e\) are initiated in reverse sequential order.

32. The method of claim 30 in which steps \(c\) and \(e\) are carried out concurrently.

33. A method for reproducibly fabricating a stable thermal field emission cathode for long-term usage under conditions of high current density emission in a relatively relaxed vacuum environment, the method including the steps of:

a. forming a conical tip with a generally hemispherical endform at one end of a Miller indices \(<100>\) oriented tungsten wire having a diameter in the range of values between 0.005 and 0.010 inches;

b. heating the tungsten wire in a partially evacuated enclosure to a first temperature falling in the range of temperatures between 1,400\(^\circ\) and 1,900\(^\circ\)K, the enclosure having an internal pressure in the range of pressures between \(10^{-2}\) and \(10^{-6}\) torr;

c. maintaining the tungsten wire in the enclosure at a second temperature falling in the range of temperatures between 1,200\(^\circ\) and 1,900\(^\circ\)K; and

d. imposing an electrical potential between the tungsten wire and an adjacent electrode, the potential being of sufficient magnitude to cause an initial electrical current flow between the tungsten wire and the electrode in the range of currents between 10 and 20 microamperes; whereby a stable thermal field emission cathode is formed having an axially disposed, generally tetrahedral, endform, the apex of which endform is characterized by a relatively small effective radius of curvature.

34. The method of claim 33 in which steps \(c\) and \(d\) are initiated in reverse sequential order.

35. The method of claim 33 in which steps \(c\) and \(d\) are carried out substantially simultaneously.

36. A method for reproducibly fabricating a stable thermal field emission cathode for long-term usage under conditions of high current density emission in a relatively relaxed vacuum environment, the method including the steps of:

a. forming a conical tip with a generally hemispherical endform at one end of a Miller indices \(<100>\) oriented tungsten wire having a diameter in the range of values between 0.005 and 0.010 inches;

b. heating the tungsten wire in a partially evacuated enclosure to a first temperature in the range of temperatures between 1,400\(^\circ\)K and 1,900\(^\circ\)K, the enclosure having an internal pressure in the range of pressures between \(10^{-2}\) and \(10^{-9}\) torr;

c. maintaining the tungsten wire in the enclosure at a second temperature falling in the range of temperatures between 1,200\(^\circ\)K and 1,900\(^\circ\)K;

d. imposing an electrical potential between the tungsten wire and an adjacent electrode, the potential being of sufficient magnitude to cause initial electrical current flow between the tungsten wire and the electrode in the range of currents between 5 and 25 microamperes;

e. detecting substantially increased current flow between the tungsten wire and the electrode, the increased current flow being an indica of \([100]\) plane build-up and the associated facetterings of at least the \([110]\) and \([112]\) planes; and

f. adjusting the imposed potential to achieve a predetermined current flow rate between the wire and the electrode;

whereby a stable thermal field emission cathode is formed having an axially disposed, generally tetrahedral, endform, the apex of which endform is characterized by a relatively small effective radius of curvature.

37. The method of claim 36 in which steps \(c\) and \(d\) are initiated in reverse sequential order.

38. The method of claim 36 in which steps \(c\) and \(d\) are carried out substantially simultaneously.

39. A method for refabricating during operation a thermal field emission cathode fabricated from \(<100>\) oriented starting material, said method including the sequentially interchangeable and overlapping steps of:

a. heating the cathode in a partially evacuated enclosure to at least a first temperature, said first temperature being sufficiently high to assure surface mobility at the emission tip of said cathode, while not exceeding a second temperature at which substantially all residual surface oxide would be desorbed; and

b. imposing an electrical potential between the cathode and an adjacent electrode, said potential being of sufficient magnitude to initiate build-up of the \([100]\) planes and to initiate faceting of at least the \([110]\) and \([112]\) planes, the potential being lower than a potential at which geometrically destructive vacuum arcing occurs.

40. The method of claim 39 wherein said first temperature is within the range of temperatures between 1,500\(^\circ\) and 1,800\(^\circ\)K.

41. The method of claim 39 wherein said steps a and b are performed substantially simultaneously.
42. The method of claim 39 wherein said first temperature is sufficiently high to cause the disorption of surface contaminants including carbon as a volatile oxide thereof.

43. A method for refabrication during operation of a thermal field emission cathode of the type fabricated by the method set forth in claim 27, said refabrication method including the additional sequentially interchangeable and overlapping steps of:
  a. heating the cathode in a partially evacuated enclosure to at least a first temperature, said first temperature being sufficiently high to assure surface mobility at the emission tip of said cathode, while not exceeding a second temperature at which substantially all residual surface oxide would be desorbed; and
  b. imposing an electrical potential between the cathode and an adjacent electrode, said potential being of sufficient magnitude to initiate build-up of the [100] planes and to initiate faceting of at least the [110] and [112] planes, the potential being lower than a potential at which geometrically destructive vacuum arcing occurs.

44. A thermal field emission cathode fabricated by the method of claim 43.

45. A thermal field emission cathode fabricated by the method of claim 1.

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