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Villalobos

[11] **3,751,780**

[45] **Aug. 14, 1973**

[54] **ULTRA SHARP DIAMOND EDGES FOR ULTRA THIN SECTIONING AND AS POINT CATHODE**

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[22] Filed: **Dec. 22, 1971**

[21] Appl. No.: **211,138**

Related U.S. Application Data

[60] Division of Ser. No. 829,267, June 2, 1969, Pat. No. 3,646,841, Continuation-in-part of Ser. No. 466,877, June 22, 1965, Pat. No. 3,447,366.

[52] **U.S. Cl.** ..... **29/95 R,** 83/651 R, 83/915.5 R, 125/39 R

[51] **Int. Cl.** **B26d 1/00,** B28b 5/04

[58] **Field of Search** ..... 83/651, 915.5; 125/39; 29/95, 95 A

[56] **References Cited**

UNITED STATES PATENTS

3,060,781	10/1962	Fernandez et al.	83/651
3,271,036	9/1966	Kuzmick	125/39

FOREIGN PATENTS OR APPLICATIONS

519,256	12/1955	Canada	29/95

Primary Examiner—Harrison L. Hinson
Attorney—Robert E. Burns et al.

[57] **ABSTRACT**

Ultra sharp diamond edge is used for molecular and submolecular sectioning at ultra low temperature and as a high intensity point source for the emission of electrons rons and neutrons.

3 Claims, 10 Drawing Figures

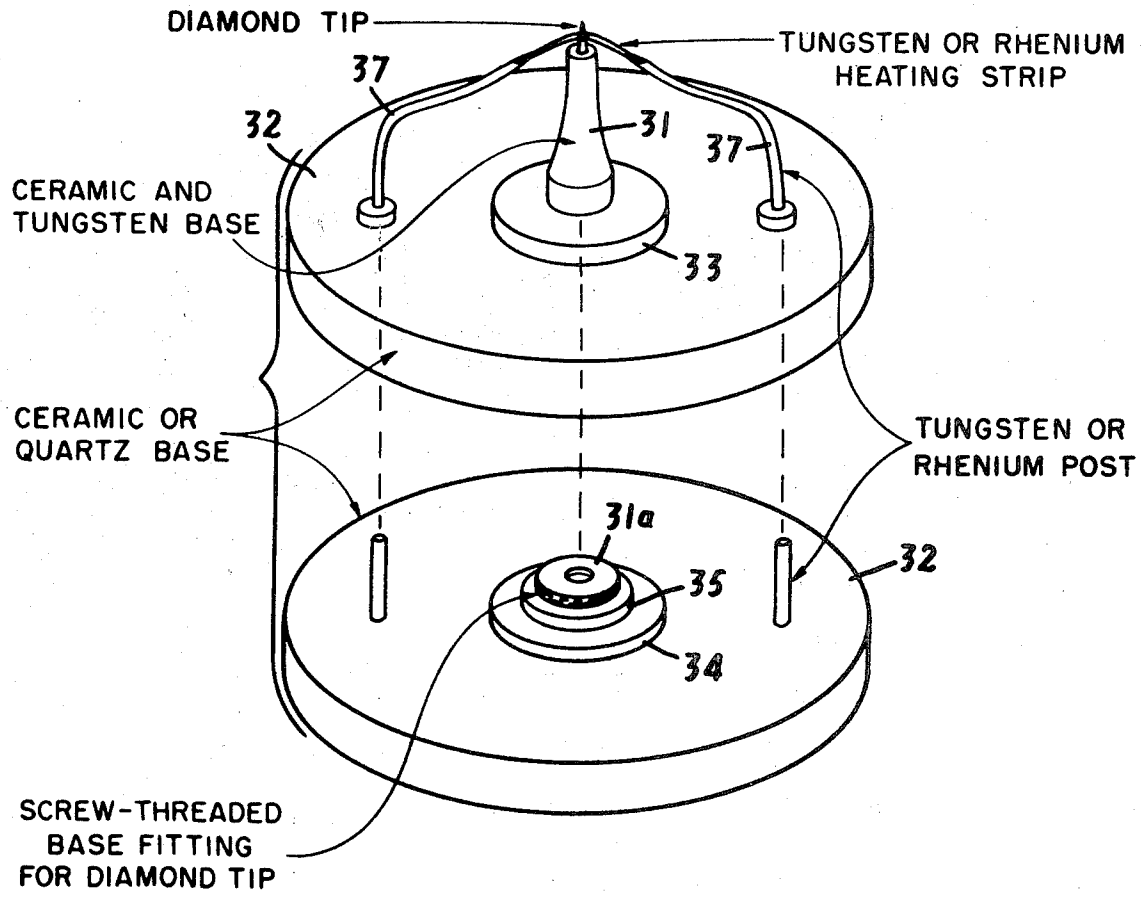


FIG.1C

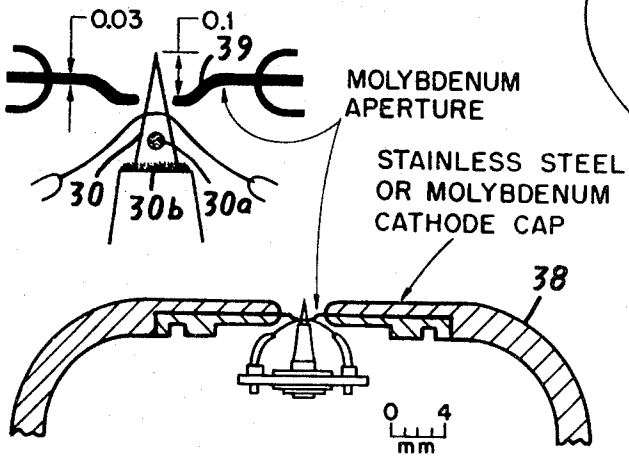


FIG. 1B

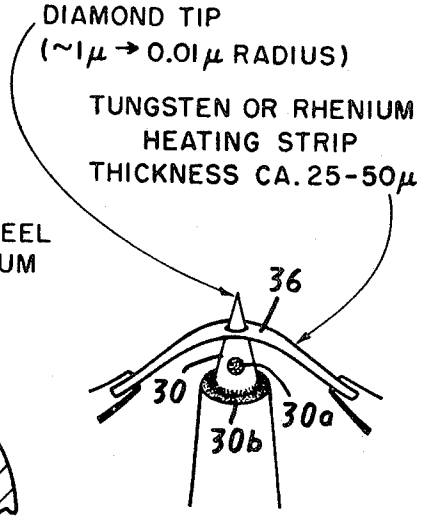


FIG. 1A

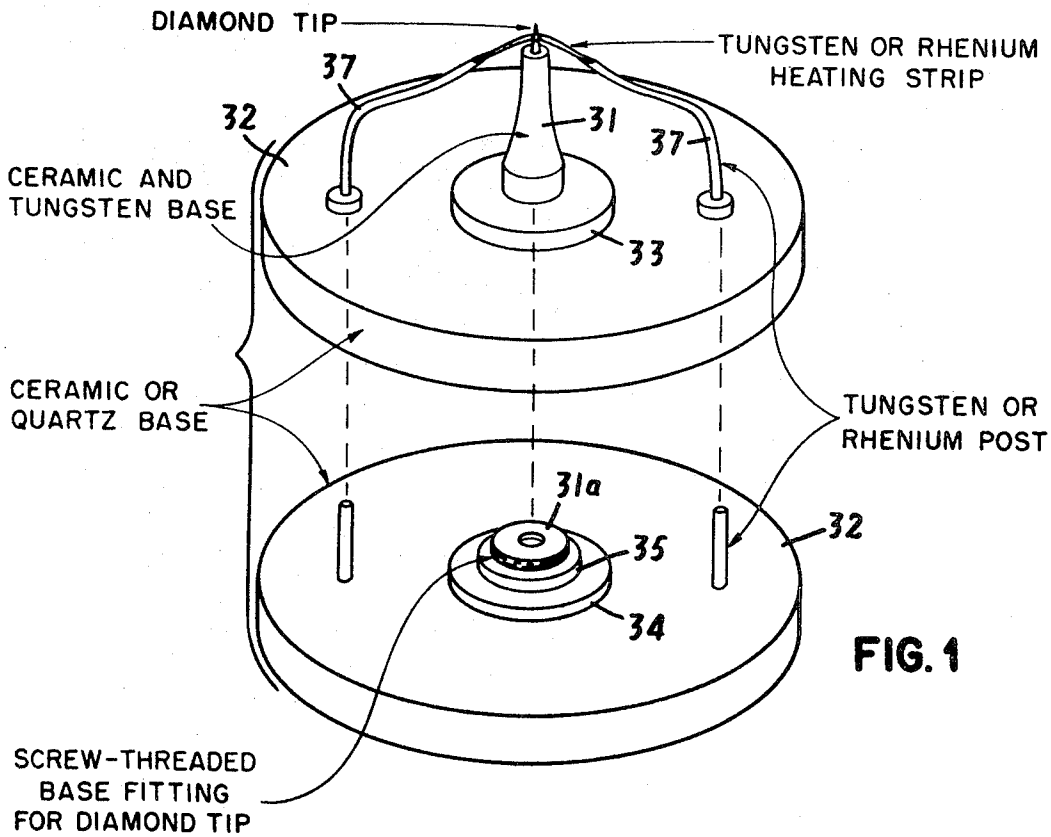


FIG. 1

Figure 2

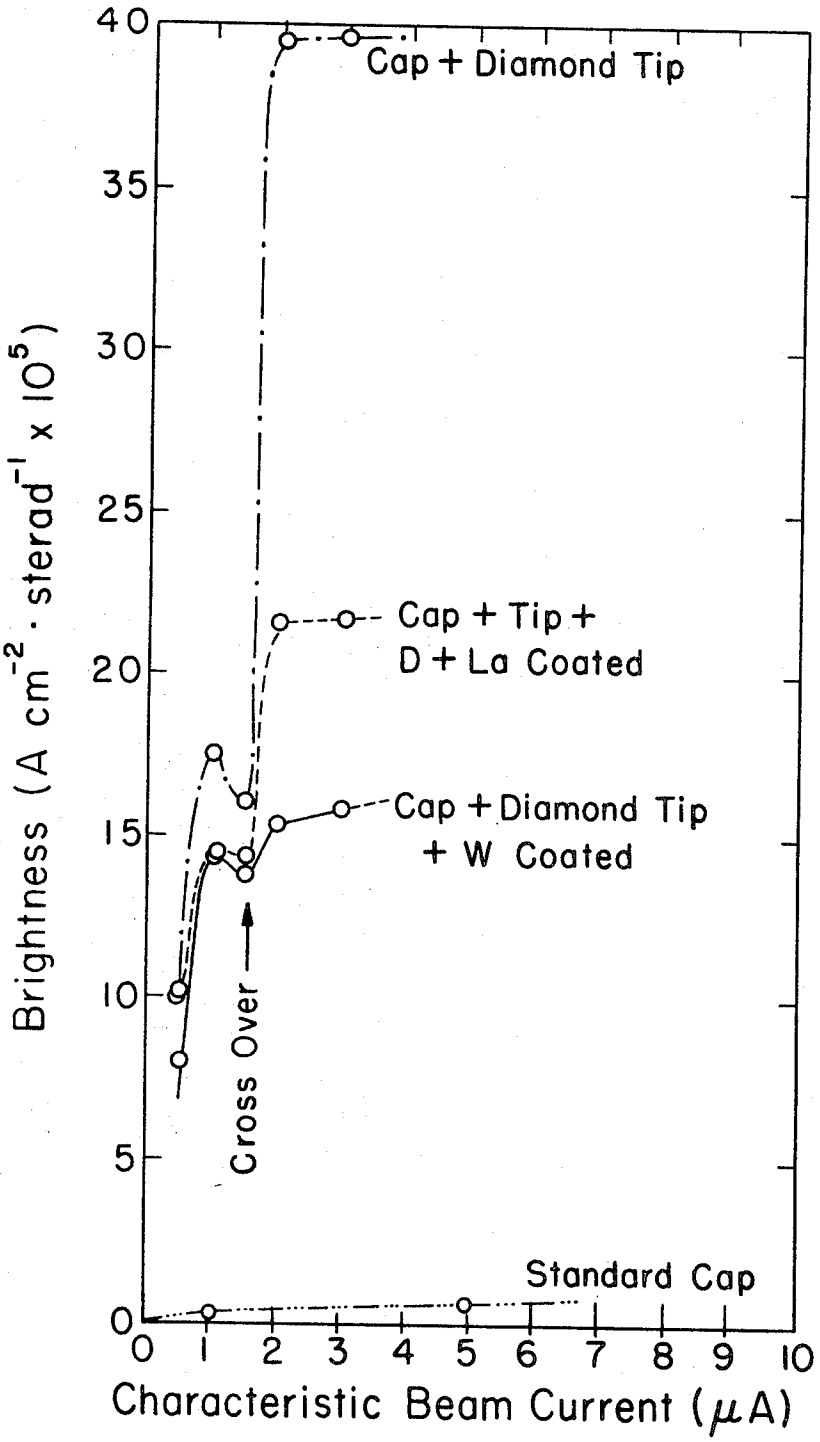

Cap + Diamond Tip
Cap + Tip + D + La Coated
Cap + Diamond Tip + W Coated
Standard Cap
Cross Over
Brightness (A cm^{-2} · sterad^{-1} x 10^5)
Characteristic Beam Current (μA)
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10
15
20
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35
40
0 1 2 3 4 5 6 7 8 9 10

FIG. 3

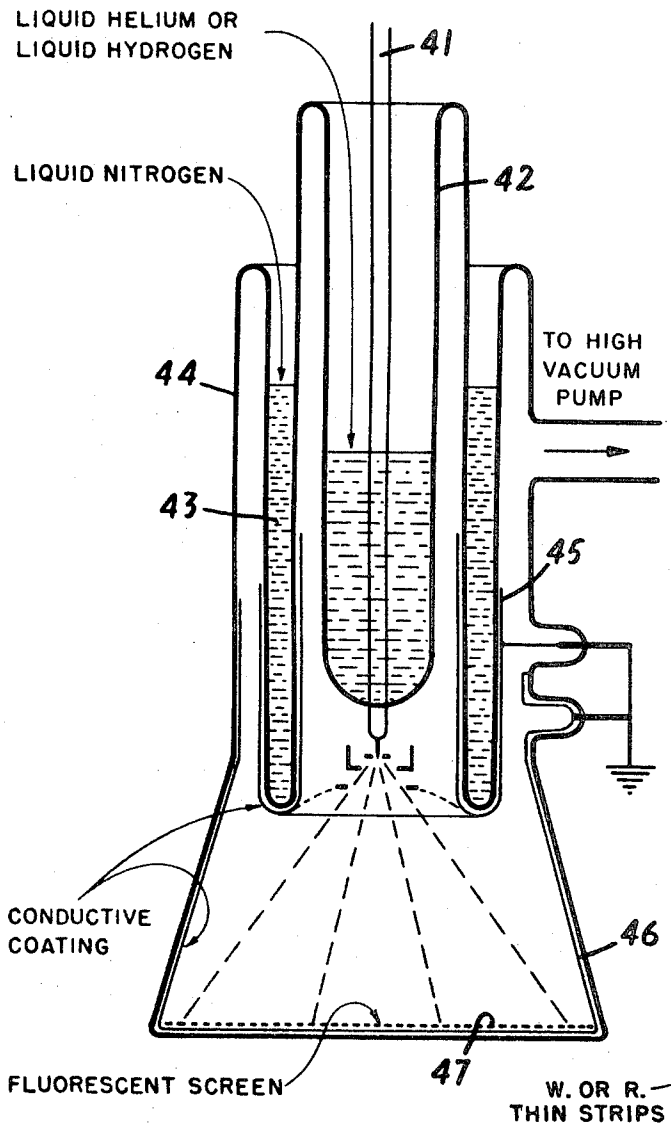


FIG. 3A

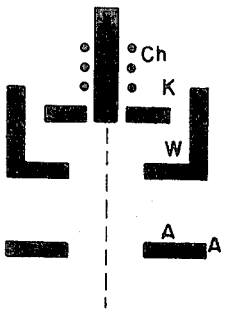


FIG. 3B

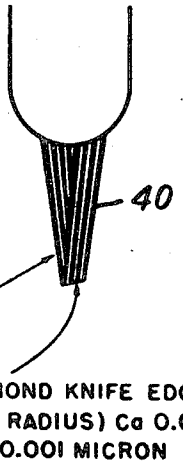


FIG. 4

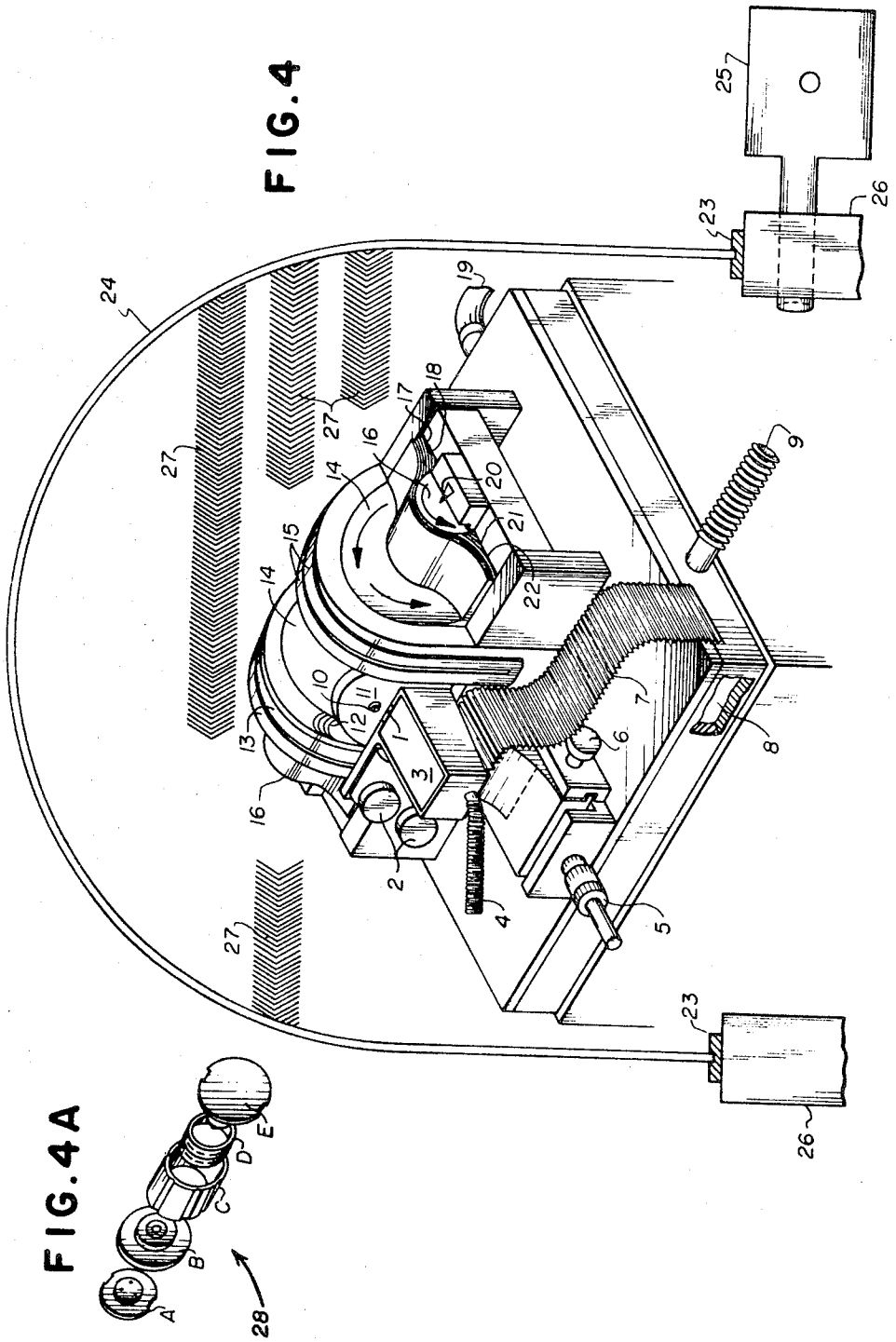

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FIG. 4A

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D
E
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ULTRA SHARP DIAMOND EDGES FOR ULTRA THIN SECTIONING AND AS POINT CATHODE

This application is a division of my application Ser. No. 829,267 filed June 2, 1969 which in turn was a continuation-in-part of application Ser. No. 466,877 filed June 22, 1965 now U.S. Pat. No. 3,447,366.

It is widely recognized that ultra sharp diamond knives and associated ultramicrotomy apparatus of the type developed and systematically introduced in research laboratories and in industry by the applicant have considerably advanced the state of prior art in the preparation of very thin slices of materials, and in ultraprecision machining. His pioneering work in this specialized field has resulted in the production of the highest quality metal surfaces presently attainable with finishes and tolerances in the low-microinch range.

This uprecedented, readily attainable microinch accuracy is now achieved directly by continuous turning operation using the diamond knife, without the traditional time-consuming and very costly additional work of lapping, buffing or polishing.

The cutting-edge sharpness radius of the order of 0.10 microinch, and the regularity of the diamond knife cutting edge are of decisive importance for this purpose, as compared with the meausred cutting-edge radius of well-sharpened conventional tools which is about 300 microinches. As shown by careful studies, this far greater cutting-edge radius and irregularity of the best standard tools results in the production of high frictional forces on the cutting edge which generates a poor surface finish by tearing and deforming the material plus accelerated wear and abrasion of the edge.

However, with a diamond cutting tool having an edge thickness of 0.001 to 0.01 micron results are consistently obtained that practically approach the idealized condition for optimum sectioning. Since the zone of compressive yielding and flow around the edge is reduced to a vertical length of a few microinches, more uniform flow and yielding of the material at the active tool edge is achieved with consequent improvement in the surface finish of the cut. Moreover, very precise sizing can be maintained because cut depths of only 5 to 10 microinches are readily made.

It is therefore essential to determine accurately the dimensions and properties of these ultra sharp cutting edges as an indispensable factor in the production and testing of the diamond knife, considered to be the most sophisticated cutting tool available today.

Prior to my work in this field, there were no adequate methods for direct imaging and measuring the cutting-edge sharpness radius of dimensions below the resolving power of the light microscope (0.2 to 0.4 micron). In fact, the active cutting region of our sharpest cutting tools remained "invisible" until suitable methods were devised which permitted successful application of the hundredfold higher resolving power of the electron microscope for direct observation of these submicroscopic structures. Direct visualization and precise measurement of the ultra sharp cutting edges was an integral part of research program in ultramicrotomy, and played a key role in the development of the diamond knife. The critical measurement and testing procedures for the cutting edges which have been developed in the course of this work represent the basis for a rational approach to the complex problems encountered in the new domain of ultraprecision machining and molecular sectioning.

Conversely, the possibility of working with stable cutting edges of submicroscopic dimensions, and of studying their interaction with selected specimens under controled conditions has opened up a new field of investigation, disclosing new phenomena which are operative predominantly at the level of molecular organization.

Thus, for example, with a diamond cutting-edge sharpness radius measuring only 10 to 100 Angstrom units, which corresponds to some 6 to 60 carbon atoms strongly covalently bonded, it is actually possible to section or "cut up" certain long-chain organic polymers into their constitutent chemical subunits of molecular dimensions. By using the special ultramicrotome apparatus described earlier operating at very low temperatures it has been possible to cut starch macromolecules into their constitutent sugar molecules endowed with quite different chemical properties.

This controlled modification of molecular structures represents a new form of manipulation, extending well beyond the established definition of mechanical sectioning, and leading into the realm of a novel modality of "preparative chemistry and physical chemistry" performed under conditions of minimum perturbation in a low entropy environment.

The methods described in this application were used primarily in connection with the production and testing of diamond knives; since diamond is the hardest naturally occurring material, and its crystalline structure makes it possible to reproducibly obtain stable cutting edges of molecular dimensions, using appropriate techniques and apparatus, of the type described in my U.S. Pat. Nos. 2,961,908, No. 3,060,781, and 3,447,366.

However, the meaurement and testing procedures can likewise be applied to determine the sharpness and properties of the active cutting edges of all kinds of cutting tools, including glass knives for ultramicrotomy, razor blades and related steel cutting edges, the wide variety of well-sharpend machine tools, and in general all cutting tools used in science, industry and technology where optimum sharpness and regularity are essential. It should be pointed out that accurate determination of cutting-edge sharpness radius and related properties of active cutting tools is of fundamental significance as an integral part of any manufacturing or production procedure designed to systematically achieve efficient cutting tools for sectioning all types of materials, in a controlled and economic way. This difficult field has been neglected in the past, partly owing to technical limitations inherent in light microscopy. However, now that ultraprecision machining and related disciplines are assuming vital importance in an era of increased automation, which depends for its success on absolute uniformity of operation and reproducibility of the finished product, the refined measurement techniques described here are of key operational value. In many ways a similar relationship can be invoked between the improvements in measuring techniques and the subsequent spectacular advances in ultrahigh vaccum technology. It was only after reliable devices and techniques for measuring ultra-high vacuum, like the Alpert gauge, became available that the whole field of attaining and monitoring ultrahigh vacuum environments came into being and could be systematically developed on a rational basis.

The invention relates generally to measurement and testing procedures for ultra sharp cutting edges. The

separate steps can be carried out individually in different stages, but are best applied sequentially in systematic combination, since they are mutually supplementary, and finally yield an integrated picture of the cutting edge configuration and properties.

The procedure embodies the following distinguishing features incorporated in the successive operational stages:

a. Direct examination of the ultra sharp cutting edge and its adjacent components, carried out step by step continuously at the different levels of magnification extending from the range of light microscopy to electron microscopy, electron diffraction and related electron optical techniques, in a non-destructive, highly accurate, and reproducible quantitative analytical way;

b. Supplementary examination of the surface characteristics of the cutting edge facettes and relationship to the active cutting radius by electron optical examination of a special type of high resolution indentation replica especially developed for this purpose;

c. Ultimate critical test of the performance and stability of ultrasharp cutting edges by actual cutting of ultrathin serial sections of selected specimens, such as metals, hard crystalline substances, macromolecular materials and biological specimens with a highly ordered internal structure. The quality and properties of the cutting edge can be accurately evaluated by combined electron microscopy, electron diffraction, physical, and physical-chemical studies of the resulting ultrathin sections and the exposed speciment block surface;

d. In special cases, and particularly for routine monitoring and inspection of large numbers of cutting tools with ultra sharp edges, certain procedures for examinations based on the characteristic electron optical, electrical, and ion-optical properties of these cutting edges can be adapted for automated inspection on an assembly line basis.

In the following description of the apparatus and method in accordance with my invention, reference is made to the accompanying drawings in which:

FIG. 1 is a schematic perspective illustration of a diamond tip point cathode source;

FIG. 1A is an enlargement of the diamond tip;

FIG. 1B illustratus the cathode point source and the cathode cap thereof in place;

FIG. 1C is an enlargement of the diamond tip with respect to the cathode cap;

FIG. 2 is a characteristic brightness curve of the point source illustrated in FIG. 1;

FIG. 3 is a schematic sectional view of a high intensity field-emission electron and ion source using an ultra sharp diamond edge; and

FIG. 3A is an enlargement view of the diamond point with respect to the grid caps and the arode of the structure of FIG. 3;

FIG. 3B is an enlarged view of the diamond knife edge of FIG. 3;

FIG. 4 is a schematic perspective view of apparatus for molecular and submolecular sectioning and FIG. 4A is an exploded view of a special cryogenic magnetic motor drive thereof.

a. Direct examination of the ultra sharp cutting-edge by high resolution light microscopy and electron microscopy, and related electron-optical techniques. edge

This procedure can best be illustrated by referring to a typical example such as a diamond knife of the type described in my U.S. Pat. No. 3,060,781. A representative diamond knife comprises a diamond body portion with a perfectly uniform and stable cutting edge of about 3 to 7 mm length, and with a cutting-edge sharpness radius of about 10 to 100 Angstrom units (0.001 to 0.01 micron). These diamond knives usually have facettes defining edges angles of about 40° to 50° for cutting plastic materials, and of 75° to 80° for cutting metals and other hard substances. However, contrary to certain claims, these edge angles do not define the active cutting-edge of the knife. Instead, the active cutting region is several hundred Angstrom units from the since line, ince it is built up of the regularly disposed crystalline unit layers of the diamond arranged in stable configuration to give smooth facets. In order to carry out the first series of optical tests the diamond knife embedded in its metal support must be mounted in a specially designed holder which permits it to be very accurately positioned and oriented at preselected optimum angles for direct examination by light microscopy, and subsequently by electron microscopy. A photomicrograph of the edge taken at the highest light microscope magnifications (1000 × to 2000 ×) shows a perfectly regular, straight and extremely smooth cutting edge facet. The actual cutting edge is not directly visible, since it lies beyond the resolving power of the ordinary light microscope. However, when examined under optimum conditions with point light sources of very high intensity (e.g., Xenon lamps, lasers, etc.) with the dark field "ultramicroscope" the active cutting edge can be faintly detected by virtue of its light-scattering properties. This allows detection of small cracks and irregularities in the edge, but still does not permit direct imaging of the critical active cutting edge. Nevertheless, improved light microscopy techniques such as interference microscopy permit accurate determination of the general quality of the cutting edge and adjoining facet. This is especially useful when comparing the uniform contours and regular spacing of the dark interference bands indicating a perfectly plane and smooth diamond edge and facet, with the curving and irregularly distorted interference fringes corresponding to the submicroscopic irregularities of a polished steel razor edge. Following these initial observations which can be readily carried out routinely during the various steps of the final polishing of the diamond or other type of ultra-sharp cutting edge, the decisive electron optical examination is carried out.

For this purpose the same holder can be used, suitably adapted for introduction into the high vacuum specimen chamber of an electron microscope. These studies require the use of a specially modified high resolution electron microscope operating at accelerating voltages of 40 to 100 kV in most cases (also in the low voltage range of 5 to 10 kV for certain applications), provided with a special pointed filament source with single-crystal tungsten tips of the type developed by the applicant. By means of these pointed sources highly coherent microbeam illumination (beam diameters ranging from 100 A to 10 microns) of low energy spread and very low intensity can be used, in conjunction with suitable high or ultrahigh vacuum specimen chambers preferably cooled with liquid nitrogen or hydrogen (vacuum of the order of 10^{-6} to 10^{-8} mm Hg) to prevent the deleterious contamination of the knife edge during illumination with the electron beam. Under these optimum conditions the properly oriented cutting edge can be very clearly seen directly and photo-

graphed at electron optical magnifications ranging from 2000 × to about 200,000×, with subsequent photographic enlargement to attain total magnifications of 500,000 to about 1,000,000 diameters. With a focused electron microbeam of adequately low intensity to avoid specimen irradiation damage the electron microscope can readily be employed to produce a distinct shadow image of the diamond knife edge and supporting region, at magnifications adequate to resolve fine details of the edge in order of a few hundred Angstroms. Imaging by reflection electron microscopy can also be readily accomplished by using oblique illumination and tilting the knife so that the incident electron beam makes a very small angle with its surface. This simple technique yields valuable data on the surface structure of the knife facet and of the active cutting edge, supplementing the results obtained by examination of the surface replicas.

However, the most important infromation is obtained by straightforward transmission electron microscopy with microbeam illumination at high magnifications, essentially because the active ultra sharp cutting-edge is ultrathin, and therefore it alone is electron transparent and can be selectively imaged. This remarkable effect which was discovered in the course of these studies has proved to be of extraordinary value as one of the most direct and powerful means of studying ultra sharp cutting edges at the highest electron-optical magnifications. It is readily understandable, since the ultra sharp cutting edges with a radius of merely 10 to 100 A are built up of a relatively small number of the crystalline unit layers of diamond to form an ultrathin wedge-shaped structure which is at most only a few hundred Angstrom units thick at the base, and therefore stands out selectively as an "electron transparent window" against the rest of the knife base which is electron-opaque. The fact that the ultrathin active cutting-edge of an ultra sharp cutting tool is essentially an "electron-transparent window" is of unique operational value because it permits us to study directly all structural details clearly visible in this most important region under excellent conditions which minimize specimen radiation damage. This effect is also clearly discernible when using electron diffraction techniques, since there is a well-defined transition from a reflection-type electron diffraction pattern to a "transmission electron diffraction pattern" precisely along the boundaries of the ultrathin cutting-edge. Although these characteristic electron-optical effects are most clearly recorded in diamond knife cutting-edges, they have also been observed under specially controlled conditions in other types of ultra sharp cutting edges in glass and steel razor edges, as well as other kinds of related cutting tools. This "ultrasharp knife cutting edge electron transmission effect" can serve as a basis for quantitative studies and selective detection of all types of cutting edges of these dimensions. After suitable calibration of the transmission electron microscope image or electron diffraction pattern using single-crystal graphite or epitaxially grown thin films as a standard for checking the thickness and related structural parameters, a conventional electron microscope can be readily adapted to serve as a routine monitoring and detecting device for these cutting edges of molecular thickness.

Other similar types of "molecular cutting-edge detectors" based on the selective electron-transmission affect are suitable for automated routine inspection and control of ultra sharp cutting edges on an assembly line basis. Some of these devices, which will be the subject of a separate application, are considerably simplified by making use of a collimated electron source (e.g., radioisotope β-emitter sources) suitably arranged so that the electrons selectively transmitted through the ultrathin cutting-edge are then recorded on a sensitive indicator, such as an image intensifier or photographic emulsion.

b. Examination of ultra sharp cutting-edges by high resolution replica techniques.

Ordinary replica techniques which provide an indirect view of a surface mold or impression on thin plastic and metal films are very difficult to apply to the study of ultra sharp cutting edges, because the active cutting edge is largely obliterated or can actually be damaged during the required stripping process, and there is considerable distortion of the initial structural relationships. It was therefore necessary to develop a simple and reliable replica technique which would yield an accurate impression of the active cutting edge and adjoining knife facet suitable for examination by high resolution electron microscopy. Among the numerous variants that were developed the following "high resolution indentation-replica technique" applied preferably on thin films was found to be the most useful method for accurately determining the cutting-edge sharpness radius, and the surface detail of the adjoining knife facets, including the orientation and size of the abrasive particle tracks in the final polishing process of the ultra sharp diamond knife edges.

It is based on the precise "indentation track" of the active cutting edge which is produced by perpendicular indentation of the knife on ultrathin (about 50 to 200 A) carbon-plastic films floating on a liquid surface or supported on a smooth soft support. The atomically smooth and perfectly plane films are produced by high-vacuum evaporation of carbon on a freshly cleaved mica surface, followed by coating with a collodion or Formvar film. The composite films are then stripped off from the mica by simple floating on water, on glycerol, or on gelatin or plastic substrates. The diamond knife or other type of ultra sharp cutting tool is mounted on a special holder connected with a sensitive balance to permit controlled ultra-low-load indentation of the smooth surface film, when the edge descends perpendicular on this ultrathin surface. The resulting indents (of which several can be made in rapid succession without damaging the sensitive knife edge) are readily visible with phase-contrast or dark-field microscopy. However, the actual active cutting edge can only be seen by direct examination of the replica (with subsequent carbon or platinum shadow-casting if necessary) by high resolution electron microscopy. The cutting edge sharpness radius stands out clearly and can be accurately measured, allowing for the contribution of the shadowing material. Adjacent to this cutting edge replica, the individual crystalline unit layers of the diamond facet can be clearly discerned. Profiles of the cutting edge can also be studied by means of stereoscopic pairs recorded with a special tilting state in the electron microscope. The replica technique can be further refined by carrying out the whole procedure in an ultrahigh vacuum chamber (10^{-7} to 10^{-9} mm Hg) indenting a freshly prepared ultrathin carbon film evaporated directly on a cooled, clean glycerol surface, followed immediately by carbon or carbon-platinum

shadow casting of the resulting indents of the ultra sharp cutting edges.

Large number of measurements performed on these replicas clearly show that the active cutting-edge sharpness radius of diamond knives are exceptionally regular with dimensions of the order of 10 to 30 A in the best knives. Moreover, those active cutting edges are remarkably stable after prolonged used, as determined by combined application of the replica and the other measurement techniques described here.

These variants of the replication techniques include direct evaporation of NaCl-carbon films on to the suitably oriented diamond or other ultra sharp cutting edges in an ultrahigh vacuum, followed by stripping off in an ultrafiltered water surface. "Shadowgrams" of the ultra sharp cutting-edges can also be obtained by collimated heavy metal shadow-casting of the cutting tools resting on a freshly cleaned mica surface.

c. Determination of properties and stability of ultra sharp cutting edges by cutting of ultrathin serial sections of metals and selected specimens, followed by examination of the sections and surface of the specimen block.

All of the previously described methods give valuable data on the dimensions and configuration of the ultra sharp cutting edges, but the most critical test for their stability and performance can only be provided by actual ultrathin sectioning. For this purpose a special instrument, such as the ultramicrotome described in my U.S. Pat. Nos. 2,961,908 and No. 3,091,144 preferably operating at very low temperatures is employed. Metal specimens, such as single-crystals of aluminum, copper, gold, zinc, or platinum with a shaped "pyramid or cone" tip, preferably only 10 to 100 microns in diameter, are mounted on the specimen holder of the very accurate ultramicrotome with a continuously adjustable feed, and a measured reproducibility

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The resulting ultrathin sections of metals are about 50 to 200 A, as determined by combined electron microscopy and electron diffraction studies, using control films and crystalline lamellae of known thickness for accurate calibration. The structure of the metal sections is a very sensitive indicator of the dimensions and quality of the ultra sharp cutting edges, since a "blunt" knife will result in section failure, edge deflection will result in irregular and compressed sections, etc. The corresponding block surface from which the sections have been cut exhibits an exceptional degree of finish (in the microinch range) which is superior to that obtained by any of the known mechanical polishing procedures. Nevertheless, even slight imperfections in the cutting edge show up as minute "tracks" in both sections and block surface.

One of the most sensitive criteria for determination of edge thickness and quality is provided by the preparation of ultrathin serial sections of macromolecular components and highly ordered biological tissues with a precise "built-in period" which serves as an internal calibration for the section thickness. Thus, an ultrathin section of about 90 A thickness as revealed in an electron micrograph of the highly regular paracrystalline lattice of insect wing inclusions depicting individual protein molecules with a regular spacing of 80 to 90 A. It is actually possible to section a virus particle, like the tobacco mosaic virus, of molecular dimensions into two or more than 80 A.

Finally, by using the best ultra sharp cutting edges and low-temperature ultramicrotomy, it has been possible to produce serial sections having molecular or submolecular thickness, prepared from frozen macromolecular substances. A native crystal of the enzyme catalase with a regular spacing of 90 A has been cut into sections which are about 100 A thick; another catalase crystal has been sectioned into slices of about 45 A thickness. This can only be directly visualized by high resolution electron microscopy when working with extremely thin sections of these molecular dimensions.

An even more sensitive criterion for cutting tool sharpness can be obtained by sectioning long-chain polymers such as starch into their constituent sugar molecules, as mentioned previously. This true "molecular sectioning" results in characteristic chemical changes (such as the different specific chemical reactions of sugar molecules, prepared by controlled breaking up of the starch molecules) which can be correlated with the known structural parameters of these molecules, and in turn related to the cutting-edge sharpness of the knife.

It is important to emphasize that there is a very close correlation and excellent agreement between the results of the different methods described here for determination of the dimensions and properties of these ultra sharp cutting edges.

Apparatus for effecting ultramicrotomy at low temperatures to produce serial sections of long-chain polymers and other important macromolecules into their active constituent components is illustrated in FIG. 4. Because the diamond kinfe used in conjunction with the cryo-ultra-microtome, as shown in FIG. 4, can cut specimens as thin as 10 Angstroms to 100 Angstroms, it is now possible to literally effect chemical changes by cutting. Thus, by means of molecular sectioning, a starch molecule can be cut up in such a way that it becomes sugar. More significantly, it is now possible to cut the master molecule DNA into viable segments which can be subsequently spliced onto other DNA molecules.

In the apparatus shown in FIG. 4, a diamond knife 1 of an edge thickness 0.001 to 0.01 mm is attached to a special holder 3, provided with adjusting screws 2 and can be cooled down to temperatures of about 1° to 30° above absolute zero by cryogenic fluid supplied through a tube 4 to a cavity in the holder to provide a bath for picking up the ultrathin serial sections. The holder can be precisely adjusted by a micrometer 5. Lateral movements of the holder can also be controlled by a screw 6. A flexible cooling strip 7, for example copper, ensures the required low temperature which is critical for biological specimens. The entire assembly base 8 is filled with a cryogenic fluid such as liquid nitrogen or helium through a flexible tube 9. The specimen 10 is affixed to a holder 11 which is also cooled through a device 12 and is attached to a highly precise cylindrical metal rotor 14 which is kept cold through special cooling coils 13, 15. A conventional motor drive operating in a high vacuum can also be attached to the rotor at 13, 15. Special diamond or equivalent point bearings 20 maintain the constancy of the rotor ends 16. Provisions are made by means of copper or other cooling strips 17, 18 to keep the bearings at the required low temperature. The bearing holders 20, 21 and 22 are needed for extra stability. The entire ultramicrotome is preferably enclosed in a high vacuum enclosure 24 with seals 23. An ultrahigh vacuum can be maintained in this enclosure by using an ion pump 25 in conjunction with cryogenic pumping. Since the system is very sensitive to temperature changes, radiation shields 27 are provided. The rotor can also be driven by a special cryogenic magnetic motor drive 28 comprising pick off pins A, an upper bearing B, a stator shell C, rotor D and lower bearing E. The entire assembly is mounted on a vibration proof base 26.

With the cooling means illustrated and described, it is possible to cool the specimen and its holder and the diamond knife and its holder and also the whole environment of the specimen and knife down to a temperature of the order of 0.001° above absolute zero. By cutting with a diamond knife having an edge thickness of 0.001 to 0.01 mm and by carrying out the cutting at a temperature at or below 20°K (the temperature of liquid gydrogen) it is possible to obtain much thinner cuts than heretofore possible. Preferably the cutting is effected at a temperature below 5°K. While liquid helium provides a temperature of 4.2°K, it has been found possible to achieve still lower temperatures by using helium 4 and helium 3 isotopes, preferably in dilution type refrigeration.

The adjusting screw 2 and micrometer 5 are used to effect accurate initial positioning of the diamond knife relative to the specimen carried by the holder 11 on the rotor. Means is incorporated in the mounting of the specimen holder for moving the specimen toward the knife holder by minute increments to cut successive ultrathin slices of the specimen as the rotor rotates. Such means may utilize the thermal expansion of a metal member, for example a member of INVAR alloy. The thermally expansive member may be sufficiently heated by light shining on it. Preferably, however, movement of the specimen is effected by magnetostrictive means. Thus, the specimen mounting may comprise a bar or other member of the rare earth dysprosium which deforms physically in response to a magnetic effect.

The described cryogenic ultramicrotome can be scaled down (as shown in the scale) from 50 to 1 centimeter approximately. The molecular or pauciatomic method has been throughly tested and can be used to section any type of material ranging from biological specimens to metals, catalysts etc. This type of "bio engineering" which has been made possible by the introduction of the diamond knife, has numerous other applications extending into the micro electronic industry (e.g., production of micro coils and micro transformers or capacitors by precision slicing). The key feature of this approach is given by the capabilities of the electron microscope which not only make it possible to see directly the critical domain of atoms and molecules but also to be able to "design" matter in this pivotal range which was hitherto inaccessible.

d. Examination of ultra sharp edges by ion-optical, field emission and electrical measurement techniques.

One of the most interesting analytical approaches in the study of ultra sharp cutting edges, particularly of diamond, which is being actively investigated by the applicant, derives from the application of point-projection microscopes. This type of microscope, as first described by E. Mueller, consists of a sharp metal cathode (g.g. very fine tungsten point with a radius of only a few hundred Angstroms) located in a highly evacuated or gas-filled tube with the fluorescent screen and an interposed anode ring at a specified distance. With a sufficiently high potential (usually of 10 to 20 kV) between anode and point, electrons or ionized helium atoms will be drawin from the point and imaged directly on the screen displaying the atomic structure of the point at magnifications of several million. The magnification can be readily calculated as a function of the radius of the cathode and the distance to the screen. A diamond ultrasharp edge of 10 to 100 A which is coated with tungsten or platinum by high vacuum evaporation can be actually considered as an extended point source in such a point projection microscope. Experiments demonstrate in fact that such a diamond knife edge is an excellent point source which gives exceptionally bright images both in the field emission and in the field ion microscope. From these images the dimensions of the emitting area which correspond to the active cutting edge of the knife can be determined. Moreover, as shown by the classic work of E. Mueller, this technique permits in principle a very accurate determination of the atomic structure of these

emitting tips, investigation of crystalline defects and other important properties.

Systematic work in this field combined with investigation of the characteristic electron-optical and electrical field **9** properties exhibited by ultra sharp stable edges of these dimensions are of great potential value for atomated inspection of ultra sharp cutting edges of all kinds.

A new type of source for electrons, ions, X-rays, neutrons of controlled exceptionally high intensity and coherence is illustrated in FIG. **1.**

A diamond point or edge **30** having a tip radius of approximately 0.001 to 1 micron or up to about 10 microns and of a precisely determined shape acts a permanent substrate for sources of a suitable material such as tungsten, rhenium, lanthanum, barium, caesium and other related materials or suitable work function and physical properties for electron emission. As shown in drawing FIG. **1,** the diamond tip or edge is accurately mounted on a suitable ceramic or tungsten base **31** which is mounted on a ceramic base plate **32** so it can be precisely aligned. For example the base **31** is provided with a reduced threaded portion **31***a* which extends through an upper washer **33,** a hole in the base plate **32** and a lower washer **34** and is secured by a nut **35.** The hole in the base plate is sufficiently large to permit adjustment of the base **31** carrying the diamond tip whereupon the nut **35** is tightened to hold the diamond tip in adjusted position.

Associated with the diamond tip **30** is a heater and emitter **36** in the form of a strip or coil or other suitable configuration. The emitter-heater is shown by way of example as a strip extending between two posts **37** and having a hole through which the diamond tip extends. The strip **36** and also the posts **37** are preferably of tungsten or rhenium. The tungsten or rhenium strip **36** is heated to a high temperature by current supplied through the posts **37** in order to evaporate a thin film of this emitting material onto the diamond point.

The assembly is placed in a "cathode gun" (see FIG. **1**) of suitable shape and material and serves as an electron source which, in conjunction with an anode (not shown in drawing) is used to accelerate an electron beam of very high brightness, for example 10^4 to 10^6 amperes cm^{-2} $Sterad^{-1}$ as shown in FIG. **2.** As illustrated in FIG. **1,** the gun includes a cathode cap **38** of stainless steel or molybdenum having a molybdenum member **29** with an aperature through which the tip of the diamond projects. The aperture is of a size to receive the diamond tip closely without touching it and may, for example be of the order of 0.1 to 1 mm. The cap shields the anode from emission by the strip **36** and thereby assures that only the diamond tip emits. A bias voltage of, for example 100–1000 volts is applied between the emitter **36** and the cap **38** while an accelerating voltage of, for example 1000 to 100,000 volts is applied between the cap and the anode.

In order to increase the life of the cathode, a reservoir of the emitter is provided. This may, for example be in the form of emitter material preferably in paste forms introduced into a transverse hole **30***a* in the diamond tip or a ring of said material around the base of the tip as illustrated at **30***b*. The reverse material gradually migrates to the hotter tip as the emitter material on the tip is used up.

The characteristic beam current obtained with a cathode according to the invention in conventional high vacuum tubes, for example 10^{-4} to 10^{-6} mm Hg, used in electron microscopes and in television tubes and related devices is orders of magnitude higher than the normal thermionic sources. The unique advantages of this type of source are (*a*) permanent critical alignment since the diamond which acts as a substrate is not affected and the source can be readily replenished simply by evaporating a new film onto the diamond tip. Since diamond has the lowest thermal expansion coefficient and is the hardes substance known, it will withstand the great stresses normally involved. The expected life of cathodes in accordance with the invention is months or years, as compared with the average lifetime of the usual cathodes measured in several hundred hours only. This source can be applied to replace the existing cathode sources for electron microscopes, television tubes, X-ray tubes, etc. without appreciable modifications. In view of its exceptional stability and precise alignment, for example in an electron microscope where alignment has to be adjusted every time the tungsten filament source is replaced, it is particularly suitable for applications such as space probes, etc. requiring months and years of unattended use. The diamond tip may be a point or an edge according to the application of the cathode.

The thin film of emitter material on a diamond tip substrate in accordance with the invention permits critical centering and height adjustment of the emitter relative to the cap to assure stable bias voltage and emission control. This electrode and cap design is applicable to both cathode and anode structures, combining optimum micro-geometry and operational conditions to obtain space-charge-free emission and significant field enhancement. The emitter in accordance with the invention may operate as a thermionic emitter or as a temperature-field emitter.

A similar type of source as shown in FIG. **3** is used in a field emission electron and ion tube using either a diamond point or a diamond knife edge **40** with a tip radius of about 0.01 to 0.001 micron serving as the substrate for accurately deposited tungsten or rhenium thin film strips, and working in an ultra high vacuum, for example of the order of 10^{-8} to 10^{-12} mm Hg. This type of source becomes the equivalent of a "comb type source" representing many hundreds or thousands of individual field emission point sources.

Alternatively, the diamond tip may have a continuous coating. The tip **40** is mounted on a holder **41** shown in the form of a rod extending down through a vessel **42** cooled by a cryogenic fluid, for example liquid hydrogen or helium. The diamond tip **40** is surrounded by an emitter Ch of a suitable emitter material, for example tungsten or rhenium. The point of the diamond tip extends through a hole in a grid cap K, for example of molybdenum. A second grid cap W having a larger aperture is interposed between the diamond tip and an anode A.

The cathode-anode assembly is surrounded by an annular chamber **43** which is likewise cooled by a cryogenic fluid, for example liquid nitrogen, and is enclosed in an envelope in the form of a tube **44** in which a high vacuum is maintained. Shielding is provided by conductive coatings **45** and **46** connected to ground. A fluorescent tube **47** is provided at the end of this tube. It has been demonstrated that with an emitter of this kind on can obtain enormous current densities, for example of the order of 108 to amp./cm^2, thus exceeding other

conventional cathodes by a factor of a million. For example, by using this type of source in the T-F mode (temperature and electrical field emission) with accelerating voltages of about 10,000 to 200,000 or more, these point shaped cathodes operating in parallel can yield peak powers of 3MW or more corresponding to energies of 10^8 to 10^{15} ergs or more in microsecond bursts. Further modifications of this type of source for producint high energy and highly coherent electron beams, X-rays, neutrons, etc. may be made.

It will be understood that features of the embodiments illustrated in the drawings and herein described are mutually interchangeable in so far as they are compatible and that many modifications in size, materials and specific configuration and design may be made.

What I claim is:

1. A point cathode source comprising a base, a diamond tip mounted on said base and having a tip or edge with a radius of the order of 0.001 to 10 microns, said tip being coated with an emitting material.

2. A point cathode source according to claim 1, comprising a metallic heating strip associated with said tip.

3. A point cathode source according to claim 1, comprising a cap having an aperture and positioned over said base with a portion of said diamond tip extending through said aperture.

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