

July 9, 1963

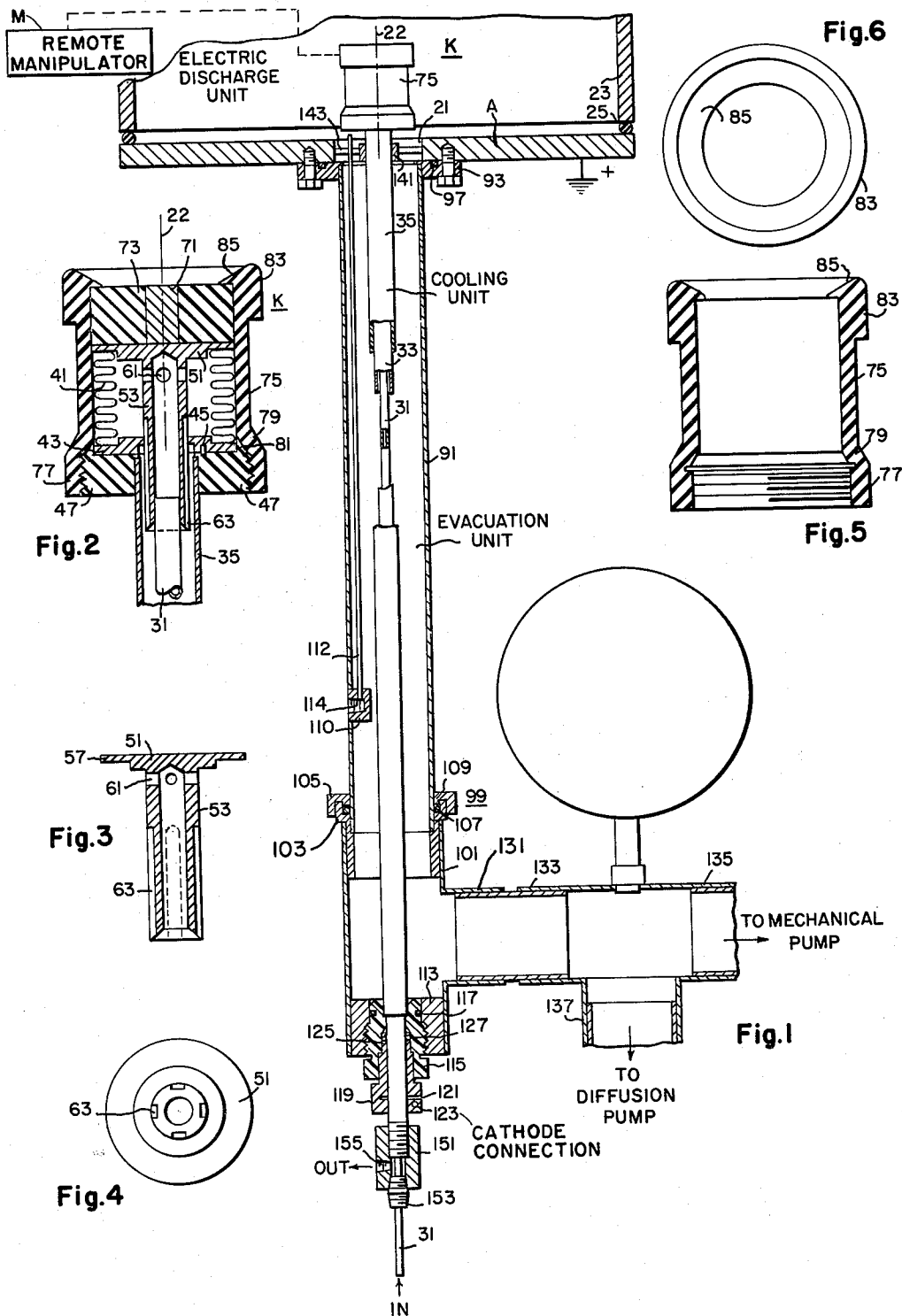
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APPARATUS AND METHOD FOR ETCHING OBJECTS

Filed Jan. 13, 1959

2 Sheets-Sheet 1



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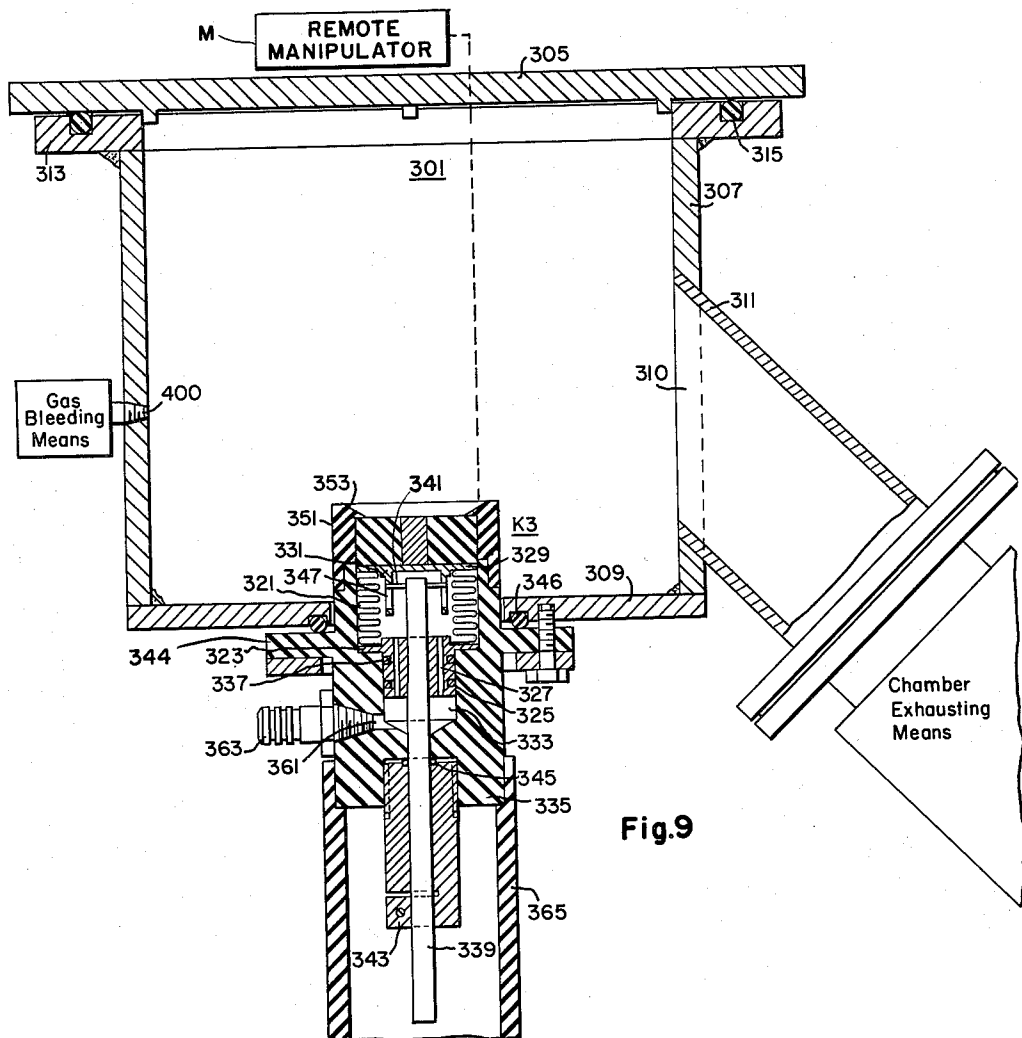
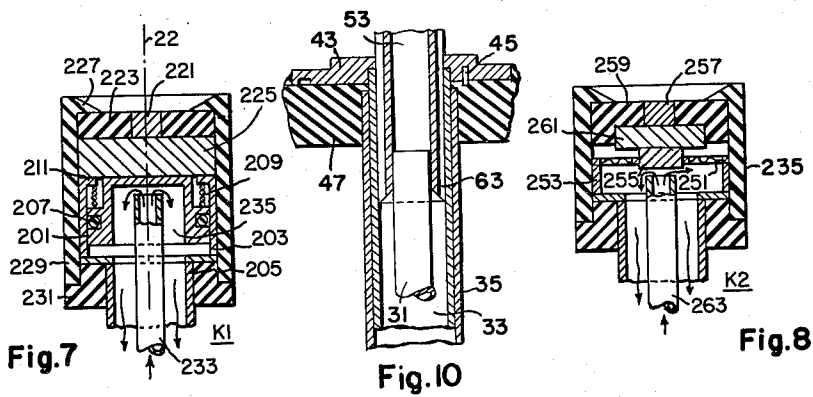
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APPARATUS AND METHOD FOR ETCHING OBJECTS

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Filed Jan. 13, 1959, Ser. No. 786,516
12 Claims. (Cl. 204—192)

This invention relates to the art of etching surfaces of material and has particular relationship to cathodic vacuum etching of specimens or samples of metals and alloys for metallographic and crystallographic studies.

In the etching of a specimen an ionizing electric discharge is produced between the specimen as a cathode and an anode electrode and a portion of the surface of the specimen is sputtered away by positive ion bombardment. The sputtering breaks away different parts of the sputtered surface differently exposing the crystal structure which may be observed under high magnification.

This invention arises from the discovery made in attempting to etch in accordance with the teachings of the prior art that for successful etching a number of conditions must be met. It is desirable that the discharge be concentrated on a relatively small area of the specimen. This area must be precisely positioned with reference to the anode electrode. It is desirable that the energy consumed in etching should be concentrated in the area to be etched and for this reason there should be firm electrical contact between the power supply and the specimen. It is also desirable that the specimen be cooled and the contact between the specimen and the cooling medium should be firm.

It is then an object of this invention to provide etching apparatus and a method of etching in the practice and operation of which the above-stated conditions shall be met.

The difficulty of meeting the above-stated conditions is increased by the circumstance that often the specimen to be etched is dangerously radioactive. Such a situation arises where an irradiated alloy including enriched uranium is being studied. Such specimens must be handled by remote control. It is then another object of this invention to provide etching apparatus readily operable by remote control which shall meet the above-stated conditions.

A more general object of this invention is to provide apparatus for establishing firm electrical or thermal contact between electrical or thermal conductors particularly suitable for cathodic etching but also having other uses.

Another general object of this invention is to provide a novel method of cathodic etching.

A further general object of this invention is to provide novel cathodic etching apparatus.

An incidental object of this invention is to provide a bellows assembly which may be readily cooled by a fluid such as water.

In accordance with one of the broader aspects of this invention the remote precise positioning of the surface of the specimen and the establishment remotely of firm thermal and electrical contact of the specimen with cooling means and an electrical power supply are all effected by pressure responsive apparatus which operates automatically when the space in which the etching is to be carried out is evacuated to position the surface to be etched and to establish the electrical and thermal contact.

In accordance with a specific but important aspect of this invention the positioning and the establishment of electrical and thermal contact is effected by a bellows or Sylphon. The bellows is mounted in the chamber within which the etching discharge takes place in such a way that

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when the chamber is evacuated there is a substantial pressure differential between the inside and the outside of the bellows and the bellows expands under this pressure differential. One end of the bellows is fixed; the other end which is movable as the bellows expands or contracts carries a thermal and electrical conductor. The specimen is so positioned that when the etching space is evacuated this conductor is moved by the expansion of the bellows into engagement with the specimen. The specimen is displaced until it reaches a stop position where the surface of the specimen which is to be etched is precisely positioned. At this point the conductor is in firm electrical and thermal contact with the specimen. Cooling water is supplied to the conductor and the conductor is connected to a power supply. Thus the bellows performs the triple function of precisely setting the surface to be etched and establishing firm electrical and thermal contact with the specimen. The bellows operates automatically when the pressure is reduced and thus where radioactive specimens are to be etched there is no necessity for complex remote operating mechanisms such as would be required for tightening a threaded holder.

In accordance with a further specific aspect of this invention the specimen is embedded in an insulator in such a way that only the small surface to be etched is exposed and the surfaces through which electrical and thermal contact are made are not exposed. Thus the lines of force from the anode electrode are concentrated on the small exposed surface and heating of adjacent supporting parts by the ionic discharge is avoided. The surface to be etched is effectively cooled so that melting is avoided and the etching produces a surface satisfactory for metallographic and crystallographic studies.

The novel features considered characteristic of this invention are disclosed generally above. The invention itself both as to its organization and as to its methods of operation, together with additional objects and advantages thereof will be understood from the following description of specific embodiments taken in connection with the accompanying drawings, in which:

FIGURE 1 is a view partly in section showing etching apparatus in accordance with this invention;

FIG. 2 is a view in transverse section showing the cathode assembly of the apparatus shown in FIG. 1;

FIGS. 3 and 4 are views in longitudinal section and bottom elevation respectively of the conductor for establishing thermal and electrical contact of the assembly shown in FIG. 2;

FIGS. 5 and 6 are views in longitudinal section and in top elevation respectively of the holding ring of the assembly shown in FIG. 2;

FIGS. 7 and 8 are fragmental views in longitudinal section of modifications of the cathode assembly shown in FIG. 2;

FIG. 9 is a view partly in section showing a modification of the apparatus shown in FIG. 1; and

FIG. 10 is a view in section enlarged showing the lower portion of the specimen and Sylphon assembly of the apparatus shown in FIG. 1.

The apparatus shown in FIGS. 1 through 6 is a preferred embodiment of this invention which includes an Electric Discharge Unit, an Evacuation Unit and a Cooling Unit.

The Electric Discharge Unit includes a circular plate A of conducting material which serves as an anode and which has a circular opening 21 in its center. The anode A and the opening 21 are circularly symmetrical about the axis 22 of the anode. A bell jar 23 is mounted near the outer periphery of the anode A with its rim engaging an O-ring 25. The junction between the jar 23 and the anode A is gas tight.

The Electric Discharge Unit includes a cathode assem-

bly K which is centrally positioned within the jar 23. The positioning and mounting is effected by the cooperation of the assembly K with the inlet tube 31, the outlet tube 33 and the jacketing tube 35 of the Cooling Unit.

The cathode assembly K includes a bellows or Sylphon 41 of conducting material such as copper. One end of the bellows 41 is secured (by silver soldering, for example), to the shoulder of a washer 43. The washer 43 is positioned by a pin 45 on a supporting ring 47 of an insulating material such as Plexiglas, polyethylene or Teflon. The outlet tube 33 of the Cooling Unit is secured to the washer 43 (by silver soldering) and thus the one end of the bellows 41 is anchored.

The other end of the bellows 41 which is movable as the bellows 41 expands or contracts carries an electrically and thermally conducting plate 51 having a hollow stem 53 which is integral with the plate 51. The upper (with reference to FIG. 2) end surface of the bellows 41 is ground and engages and is secured to a shoulder of the plate 51. The outer surface 57 of the plate 51 is also ground. The seal between the plate 51 and the bellows 41 is vacuum tight.

Near the region where the stem 53 joins the plate 51 there are openings 61 through which the interior of the stem 53 and the inlet tube 31 are in communication with the interior of the bellows 41. The inlet tube 31 is slideable in the stem 53. Cooling water may thus flow into the bellows 41. From the end of the stem 53 longitudinal grooves 63 extend to a point above the washer 43. The tube 33 bounds the grooves 63 to the point where it is secured into the washer 43. Thus the cooling water in the bellows 41 returns through the annular space between the tubes 31 and 33 and effectively cools the stem 53 and the plate 51 with which it is integral.

The specimen 71 is partly enclosed in an insulator 73 to limit the area of the specimen subjected to the etching electric field. The insulator 73 should preferably be composed of a moldable plastic such as polyethylene, Plexiglas or Teflon and the specimen should be molded into the insulator. The insulator 73 is cylindrical having bases of the same diameter as the washer 43. The specimen 71 may be of any suitable cross section and is shown as cylindrical. The specimen 71 is coaxial with the insulator 73 and its bases are flush with the bases of the insulator.

The specimen assembly 71—73 is held on the washer 51 by a clamping ring 75 of insulating material such as Plexiglas. The ring 75 has an internally threaded portion 77 at one end which terminates in a tapering portion 79. The supporting ring 47 has an external thread and a bevelled shoulder 81 adapted to engage the internal surface of the portion 79. The clamping ring is screwed onto the supporting ring until the internal surface of the portion 79 engages the shoulder 81. At the other end the clamping ring 75 terminates in an enlarged portion 83 from which an internal lip or overhang 85 extends. The specimen assembly 71—73 is held between the lip 85 and the plate 51. With the ring 75 screwed onto the ring 47 so that the parts 79 and 81 engage, the overhanging surface of the lip 85 sets the position of the specimen 71. When so held the specimen 71 is coaxial with the anode A so that the lines of force of the electric field between the anode A and the cathode K are concentrated in the small exposed area of the specimen.

When the specimen 71 is radioactive the clamping ring 75 may be screwed on the ring 47 by a master-slave manipulator M. But such a manipulator does not avail adequate force to screw on the ring 75 with the tightness required to effect adequate thermal and electrical contact and to set the surface to be etched precisely. The necessary firm contacts and precise seating of the specimen assembly 71—73 against the lip 85 is effected by the compressive action of the bellows 41 when it expands on the evacuation of the bell jar 23.

The Evacuation Unit includes a tube 91 of a metal

such as copper having a supporting flange 93 at one end. The flange 93 is bolted to the anode A with the junction between the flange and the anode sealed gas-tight by an O-ring 97 in a groove in the flange. At the other end the tube 91 is joined by a packing-gland 99 to one of the arms 101 of a T-joint. A ring 103 threaded externally at its upper end extends from the arm 101. The thread is engaged by a nut 105. An O-ring 107 is compressed by a gland ring 109 between the nut 105 and the ring 103.

The tube 91 includes an internal boss 110 from which a tube 112 extends into the space under the bell jar 23. The boss 110 has an inlet hole 114 which communicates with the tube 112 and through which a gas such as argon may be bled into the jar 23. The other arm 111 of the T-joint has at its end a stepped ring 113 of copper having an internal thread engaged by an external thread of an insulating tube 115 which may be composed of Teflon. An O-ring 117 is provided between the ring 113 and the tube 115. The internal thread of the tube 115 is engaged by a hollow bolt 119 of a conductor such as brass which has at its outer end a slot 121 and a screw 123 for connecting the negative hot terminal of the supply.

The insulating Teflon tube 35 extends into a counter-bore in the insulating tube 115 and the water return conductor 33 extends through the bolt 119. An O-ring 125 and a ring 127 are provided for sealing the joint between 33 and 115. The Teflon tube 35 and the ring 115 serve to insulate the tube 33 which is at a high potential from the tube 91 which is grounded.

The stem 131 of the T-joint is connected to an arm 133 of another T-joint. The other arm 135 of this joint is connected to a mechanical pump (not shown) and the stem 137 is connected to a diffusion pump. Between the arms 133 and 135 the T-joint carries a vacuum switch 35 which operates to preclude the application of potential to the anode A and the cathode K until the bell-jar 23 is evacuated.

The Cooling Unit includes the tubes 31, 33, 35. The tube 33 is secured to the washer 43 and to the bolt 119. The tube 35 is secured to the insulating tube 115 and is supported by the hub 141 of a spider 143 in the opening of the anode A. The tube 33 at its projecting end is threaded and the thread engages the internal thread of a block 151. At its projecting end the block 151 is also internally threaded and engages the external thread of a split fitting 153 which secures the tube 31. The tube 31 is thus suspended from the tube 33. The block 151 is provided with an exit opening 155 which communicates with the annular space between the tubes 31 and 33 and serves as the water outlet. At least six feet of insulating tubing (for example Tygon, not shown) should be connected between the outlet 155 and the water sink. About six feet of insulating tubing should also be connected between the end of tube 31 and the water supply.

In the use of the apparatus shown in FIGS. 1 through 6 the specimen assembly 71—73 is placed on the plate 51 and the clamping ring 75 is screwed on until it engages the shoulder 81 of the ring 47. The bell jar is then evacuated to a low pressure which may be as low as one micron or lower and the bellows expands setting the specimen 71 and effecting the firm electrical and thermal connection to it. About 5000 volts D.C. is then impressed between the anode A and the cathode K and argon is bled in until a precise low pressure is reached. A discharge to the exposed surface of the specimen 71 then produces the desired etching.

FIGS. 7 and 8 show different cathode assemblies K1 and K2, respectively.

Cathode K1 includes a hollow piston 201 which is electrically and thermally conducting slidable within the hollow head 203 of an electrically and thermally conducting tube 205. The piston has a groove in which there is an O-ring 207 which engages the head 203 and seals the region of the tube 205 on one side of the piston from the region on the other side. The piston 201 is urged by a

spring 209 which engages a lip 211 on the head 203 towards the stem of the tube 205.

The specimen 221 is embedded in an insulator 223. The assembly 221-223 and an electrically conducting plastic cylindrical block 225 also having high heat conductivity are held between the end of the piston 201 and lips 227 of an insulating clamping ring 229 screwed onto an insulating supporting block 231. A tube 233 terminates in the opening 235 in the piston 201. Cooling water is supplied through tube 233 and returns through the annular space between tube 233 and tube 205.

The cathode K1 may be used in place of the cathode K in the assembly shown in FIG. 1. The specimen 221 should be symmetrical with respect to the centerline 22. In the apparatus with the cathode K1, the piston 201 establishes firm thermal and electrical contact and sets the specimen when the pressure in the jar 23 is reduced.

The cathode K2 includes a diaphragm 251 of conducting material which is sealed pressure tight between the end of a tube 253 and a cylindrical conducting block 255. The specimen 257 is embedded in the insulating C-shaped part 259 of a composite block which has a conducting part 261 extending from the specimen 251 within the insulating part 259 to a position below the part 259. The conducting part 261 engages the conducting block 255. Cooling water is projected on the block 255 from a tube 263 and returns through the space between 253 and 263. The cathode K2 includes a clamping ring 235 which functions like the rings 229 and 75. The cathode K2 may replace the cathode K1 in apparatus such as is shown in FIG. 1. In this case the diaphragm 251 effects the setting of the specimen 257, its energization and its cooling.

The apparatus shown in FIG. 9 includes a generally circular cup-shaped chamber 301 having a flat top 305. The walls 307 and base 309 of the chamber 301 are of conducting material and the base 309 has an opening in its center. From the wall 307 a flanged tube 311 extends. The chamber 301 is exhausted through the tube 311. The chamber 301 is flanged and the top 305 engages the flange 313; the joint between the top and the flange being sealed by an O-ring 315. When the chamber 301 is exhausted the top 305 is clamped pressure tight to the flange 315. The chamber 301 is grounded and serves as anode. The chamber 301 includes an opening 400 into which a tube is sealed. The opening 400 is generally opposite to the center 310 of the exhaust opening to which tube 311 is connected. Inert gas is bled in through the opening 400. During operation the pumps connected to 311 are operated continuously while inert gas is bled in through 400. Because of the pumping action inert gas flows continuously over the exposed surface 312 of the specimen.

The cathode assembly K3 includes a bellows 321 sealed at one end to a plate 323 from which a stem 325 extends. The stem 325 has longitudinal openings 327. At the other end the bellows 321 is sealed to a plate 329 from which a short hollow stem 331 extends.

The stem 325 is anchored near the open end of a cup-shaped cavity 333 in a flanged insulator 335. O-rings 337 are provided between the stem 325 and the wall of the cavity 333. A tube 339 of conducting material having a cross pin 341 extends through the stem 325 and the insulator 335. The tube 339 is anchored to a bolt 343 which screws into the insulator 335 and is sealed by an O-ring 345. The bolt 343 is slotted at the end to accommodate the negative hot terminal of the supply. The pin 341 extends into slots 347 in the stem 331 and limits the movement of the bellows 321 as it expands and contracts. The bellows is thus protected against being damaged by over-expansion or over-contraction.

The insulator 335 is bolted to the base 309 and its end remote from the bolt 343 extends into the chamber 301, coaxial with the wall 307. The joint between base 309 and the flange 344 is sealed by an O-ring 346. The specimen assembly 349 is held between the lip 351

of a clamping ring 353 and the plate 329 secured to the bellows 321. The ring 353 screws onto the end of the insulator 335 which extends onto the chamber 301.

The insulator 335 has a lateral opening 361 which communicates with the cavity 333 and into which a fitting 363 is screwed. The cooling water delivered through tube 339 flows through the openings 327 to the cavity 333 and is removed through the fitting 363. The conductors 339 and 343 are enclosed in a plastic shell 365 which is screwed in insulator 335 and prevents personnel from coming into contact with the high voltage on 339 and 343.

The apparatus shown in FIG. 9 functions similarly to the apparatus shown in FIGS. 1 through 6, 7 and 8. The specimen assembly is initially held by the clamping ring 351, and top 305 is placed on the flange 313. The contraction of the bellows 321 is limited by pin 341. The chamber 301 is then exhausted. The bellows 321 expands establishing firm thermal and electrical contact with the specimen through the plate 329 which is cooled and sets the specimen. The expansion of the bellows 341 is limited by the pin 321. The exhausting of the chamber 301 also causes the top 305 to seal the junction between the top and the flange 313. After the exhausting is completed the required gas may be bled in through inlet 400. Because of the position of the inlet 400 relative to exhaust tube 311 which is continuously being pumped the specimen surface 312 is continuously bathed in fresh inert gas. A discharge is now produced between 307 and the specimen and the specimen is etched.

While preferred embodiments have been disclosed herein many modifications thereof are feasible.

We claim as our invention:

1. In cathodic vacuum etching of a radioactive body, the method of establishing electrical and thermal contact between said radioactive body and a cooled electrical and thermal conductor having a cooling surface of substantial area and having an electric potential impressed thereon, the said method comprising mounting said conductor so that said surface is at the pressure within said enclosure and another surface of said conductor is at atmospheric pressure, said conductor being mounted movable under any difference in pressure between said enclosure and the atmosphere, mechanically clamping said body adjacent said surface of said conductor by action of a remotely actuable manipulator, exhausting said chamber to establish said difference of pressure, under the action of said pressure difference causing said conductor to move to engage said body and to apply said pressure difference thereto to provide electrical and thermal contact between said conductor and said body.

2. In cathodic vacuum etching of a radioactive body, the method of establishing electrical contact between said radioactive body and a conductor having an electrical potential impressed thereon, the said method comprising mounting said conductor so that said surface is at the pressure within said enclosure and another surface of said conductor is at atmospheric pressure, said conductor being mounted movable under any difference in pressure between said enclosure and the atmosphere, mechanically clamping said body adjacent said conductor by action of a remotely actuable manipulator, exhausting said chamber to establish said difference of pressure between and under the action of said pressure difference causing said conductor to move to engage said body and to apply said pressure difference thereto to provide electrical contact between said conductor and said body.

3. In cathodic vacuum etching of a radioactive body, the method of establishing thermal contact between said radioactive body and a cooled thermal conductor having a cooling surface of substantial area, the said method comprising mounting said conductor so that said surface is at the pressure within said enclosure and another surface of said conductor is at atmospheric pres-

sure, said conductor being mounted movable under any difference in pressure between said enclosure and the atmosphere, mechanically clamping said body adjacent said surface by action of a remotely actuable manipulator, exhausting said chamber to establish said difference of pressure between the side of said conductor including said surface and the opposite side of said conductor, and under the action of said pressure difference causing said surface to move to engage said body and to apply said pressure difference thereto to provide thermal contact between said body and said surface.

4. Apparatus for etching a surface of a specimen comprising a chamber, means connected to said chamber for evacuating said chamber, an electrode within said chamber, pressure responsive means in said chamber operable on the evacuation of said chamber for positioning said specimen so that said surface is precisely positioned relative to said electrode for etching, cooling means for said specimen, means connected to said positioning means and rendered effective by the evacuation of said chamber for connecting said cooling means in effective cooling relationship with said specimen, and means connected to said electrode and to said positioning means for impressing an etching potential between said electrode and specimen.

5. Apparatus for etching a surface of a specimen comprising a chamber, means connected to said chamber for evacuating said chamber, an electrode within said chamber, pressure responsive means in said chamber operable on the evacuation of said chamber for positioning said specimen so that said surface is precisely positioned relative to said electrode for etching, cooling means for said specimen, means connected to said positioning means and rendered effective by the evacuation of said chamber for connecting said cooling means in effective cooling relationship with said specimen, and means connected to said electrode and to said positioning means and rendered effective by the evacuation of said chamber for impressing an etching potential substantially only between said electrode and specimen.

6. Etching apparatus for etching an object including a substantially gas-tight chamber, a portion of which constitutes a circularly symmetrical ring-shaped conductor, means for mounting said object in said chamber so that said object is substantially coaxial with said conductor, means connected to said chamber for setting the gas pressure therein, means connected to said conductor and to said mounting means for impressing a potential between said conductor and said object to produce an electrical discharge for etching between said conductor and object, and insulating means partially enclosing said object to limit the part of said object subject to the electrical field and the electrical discharge between said object and conductor to a predetermined area of said object.

7. Apparatus for cathodic etching of an object including a gas tight chamber, means mounting said object within said chamber, means connected to said chamber for maintaining in said chamber an ionizable gas at a low pressure, and power supply means connected to said object as a cathode and including an anode for producing an ionizing electric discharge in said chamber to said object as cathode, said object being embedded in an insulator which exposes only a predetermined limited area of the face of said object to be etched to said ionizing electric discharge and said anode surrounding said object so that said power supply means concentrates said discharge on said limited area.

8. Apparatus for cathodic etching of an object having opposite faces including a gas tight chamber, means mounting said object within said chamber, means connected to said chamber for maintaining in said chamber an ionizable gas at a low pressure, and power supply means connected to said object as a cathode for pro-

ducing an ionizing electric discharge in said chamber to said object as cathode, said object being embedded in a composite block including an electrically insulating part and an electrically conducting part, one of said opposite faces of said object being exposed to said discharge and the other being in electrical engagement with said conducting part, said power supply means being connected to said other of said opposite faces through said conducting part.

9. Apparatus for etching a surface of a radioactive specimen comprising a chamber, means connected to said chamber for evacuating said chamber, an electrode in said chamber, specimen constraining means within said chamber, when constraining said specimen setting said specimen so that said surface is precisely positioned for etching, mounting means for said specimen in said chamber adjacent said constraining means, pressure responsive means connected to said mounting means and actuable on the evacuation of said chamber for moving said mounting means and said constraining means relative to each other so that said constraining means is in constraining engagement with said specimen, and means connected to said electrodes to be connected to said specimen for producing an electric discharge for etching at said surface.

10. Apparatus for etching a surface of a specimen comprising a chamber, means connected to said chamber for evacuating said chamber, an electrode within said chamber, pressure responsive means in said chamber operable on the evacuation of said chamber for positioning said specimen so that said surface is precisely set relative to said electrode for etching, and means connected to said electrode and said positioning means for impressing an etching potential between said electrode and specimen.

11. Apparatus for etching a surface of a specimen comprising a chamber, means connected to said chamber for evacuating said chamber, an electrode within said chamber, pressure responsive means in said chamber operable on the evacuation of said chamber for positioning said specimen so that said surface is precisely positioned relative to said electrode for etching, and means connected to said electrode and to be connected to said specimen for impressing an etching potential between said electrode and specimen.

12. Apparatus for etching a surface of a specimen comprising a chamber, means connected to said chamber for evacuating said chamber, an electrode within said chamber, pressure responsive means in said chamber operable on the evacuation of said chamber for positioning said specimen so that said surface is precisely positioned relative to said electrode for etching, and means connected to said electrode and to said positioning means and rendered effective by the evacuation of said chamber for impressing an etching potential substantially only between said electrode and specimen.

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