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Burck

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[54] **DEVICE FOR THINNING TECHNICAL AND MICROSCOPIC SPECIMENS UNDER LAMINAR FLOW CONDITIONS**

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[52] **U.S. Cl.**..... **204/224 R,** 134/198, 204/129.1, 204/237, 204/273

[51] **Int. Cl.**..... **B23p 1/04,** B08b 3/02

[58] **Field of Search** 204/224, 129.1, 238, 239, 204/237, 273; 134/198

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Assistant Examiner—D.R. Valentine
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[57] **ABSTRACT**

Device for electrochemically or chemically thinning technical and microscopic specimens, such as thin or microsections, under laminar flow conditions, comprising two jet nozzle systems on opposite sides of the specimen to be thinned, each nozzle consisting of a long, wide-lumen jet tube, each system having a central nozzle space which tapers inwardly toward the nozzle proper, with a concentric, annular channel disposed about each nozzle space, with a number of (preferably four) symmetrical overflow channels running from each annular channel to its nozzle space, a supply conduit linking each annular channel to a circulating pump. Polishing masks are applied to the specimen with the interposition of thin, soft sealing rings. The jet nozzle spaces may have windows, allowing observation during the thinning process, e.g. by the aid of a microscope.

14 Claims, 4 Drawing Figures

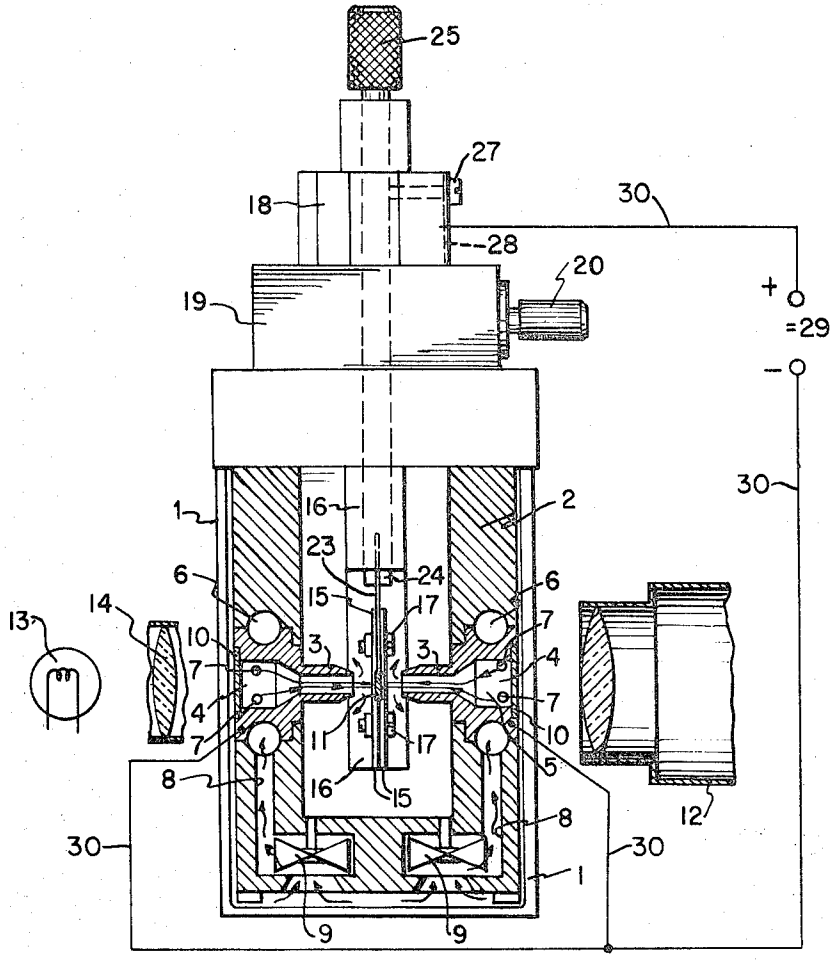


FIG. 2

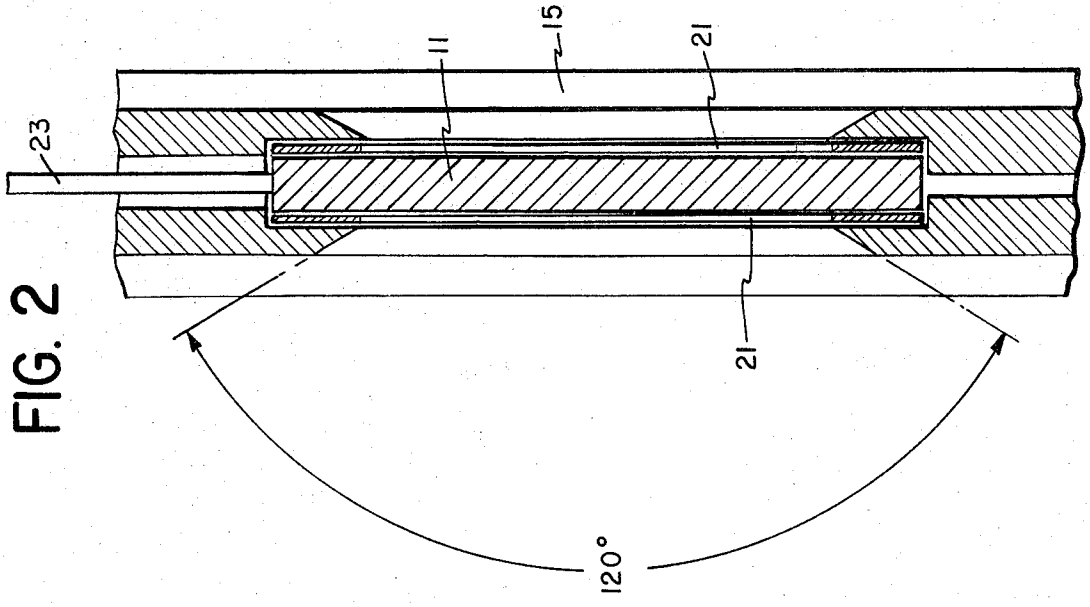

23
11
15
21
21
120°

FIG. 1

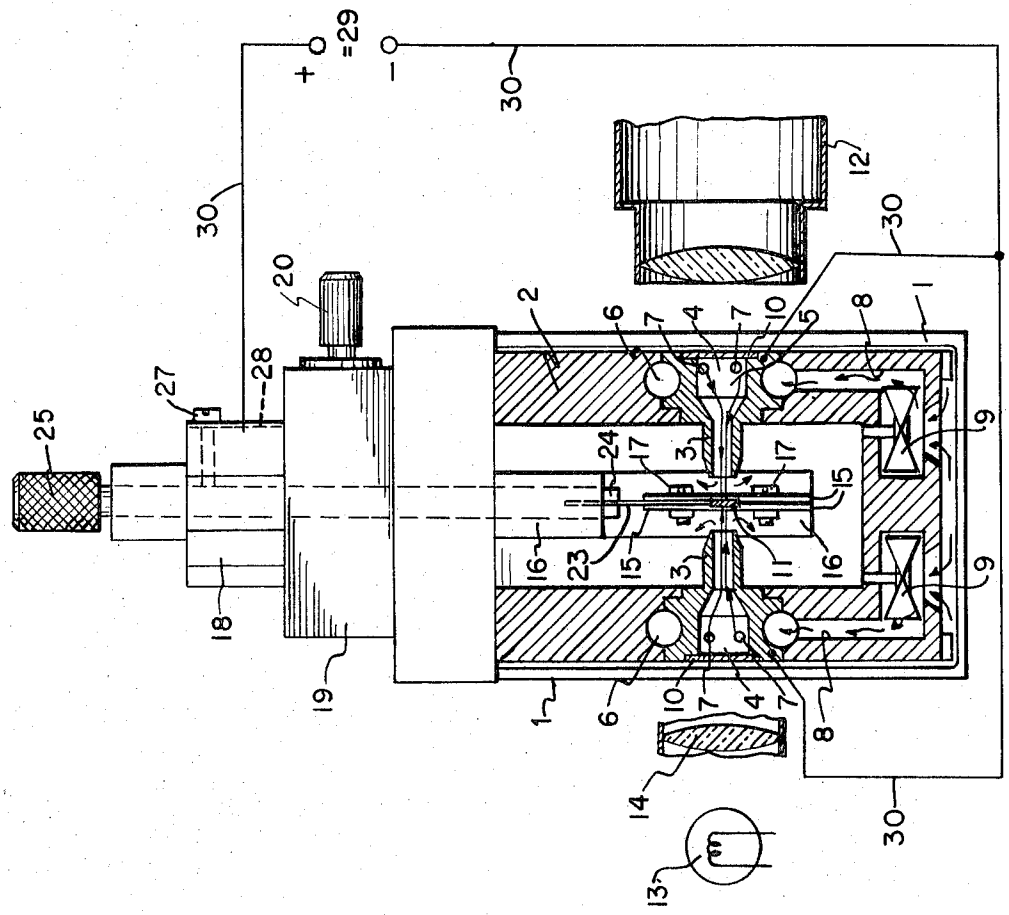

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FIG. 3

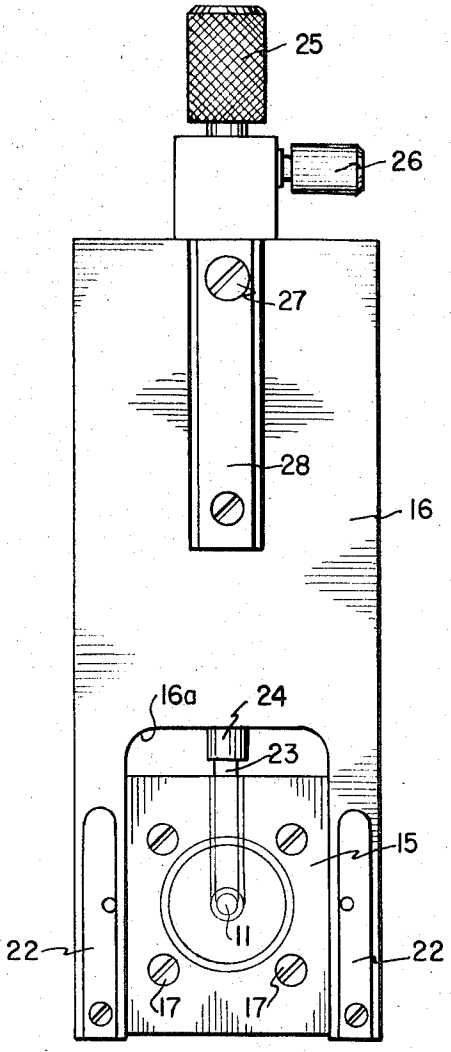
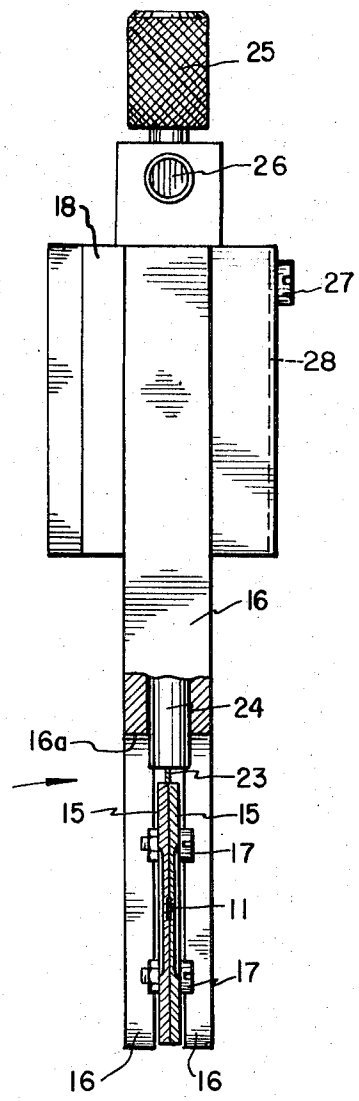

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FIG. 4


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DEVICE FOR THINNING TECHNICAL AND MICROSCOPIC SPECIMENS UNDER LAMINAR FLOW CONDITIONS

The invention relates to a device for electrochemically or chemically thinning technical and microscopic specimens or objects, such as metals or semiconductors, particularly irradiation anodes for micro-focus X-ray tubes, precision aperture diaphragms or objects through which rays pass for X-ray topography and electron microscopy by transmission, under laminar flow conditions, such specimens being generally designated thin or microsections. As is well known in this art, electrochemical thinning is being used for metallic specimens, while chemical thinning is suitable for semiconductors and electrically non-conductive specimens.

Devices are known, such as so-called holder-type polishing devices, which essentially consist of one or two polishing masks made of polytetrafluoroethylene, the mask or masks closely hugging the disc-shaped specimens or objects to be thinned down from one or both sides. The masks have lip thicknesses of ≥ 0.2 millimeters at lip angles of about 30°. These masks hold the specimen in an electrolyte, the specimen being usually connected to the positive pole of a DC current source.

Devices are furthermore known, so-called jet polishing devices, which consist essentially of one one-sided or two double-sided jet nozzles facing the disc-shaped specimens to be thinned, substantially at right angles, having diameters of ≤ 1 millimeter, directly connected to the inlet or supply tube for the electrolyte. The object is usually connected to the positive pole of the DC source while the nozzle itself (or nozzles) is (are) linked to the negative pole. The thin electrolyte jets impinge upon the object to be thinned at very high flow velocities.

Combined holder-jet-polishing devices are also known which however merely constitute a simple combination of the just described two types of devices.

The objects already thinned to some extent with the known devices are usually further thinned by free immersion, such as with pincers or tweezers, into an electrolyte until the first perforation appears, usually in central regions of the objects. When making objects to be irradiated, such as for transmission electron microscopy, it is desirable to keep the obtained plate-like polishing recesses possibly flat, that is with only small lip angles at the edges to insure maximum areas that can be penetrated by the rays.

The known holder polishing devices have the disadvantage that the specimens or objects produced thereby for transmission electron microscopy have only small penetrable regions of about 10^2 to 10^3 μm^2 for 100 kilovolt electrons, in view of their relatively large lip angles about the edges. This is due to the considerable lip thicknesses of the polishing masks which are however indispensable for proper sealing in view of the material used for the polishing masks.

The considerable mask lip thicknesses furthermore lead to an adhesion of gas bubbles produced during the thinning process, namely at the edges of the polishing masks, resulting at these locations in undesirable perforations of the thinned objects.

In order to reduce or eliminate the indicated disadvantages, the specimens thinned with the known holder-type devices are treated usually only up to just before their perforation, the central areas being further thinned by free immersion with pincers into an electrolyte until the first perforations occur.

With the polishing procedure by the aid of pincers, however, material is also removed from the relatively thick, mechanically stabilizing edge of the thinned specimen, resulting in that the object is liable to be plastically or elastically deformed during its manipulation, an effect which is particularly harmful with objects for transmission electron microscopy, on account of the formation of artifacts.

Known jet polishing devices have the disadvantage that grooves or furrows appear on the bottom of the plate-like polishing recess, and consequently perforations at much too considerable thicknesses of the object being thinned. This is the result of turbulence in the electrolyte jet which results on account of the direct connection of the jet nozzles with the supply conduit for the electrolyte, and/or the small diameter of the nozzles themselves.

It is furthermore the result of the small nozzle diameters that relatively high electrolyzing voltages have to be applied for the electrochemical thinning between the specimens or objects, connected as the anode, and the nozzles, constituting the cathode, in order to attain the necessary polishing current density. This leads on the objects to be thinned to excessive gas and heat formation, and can also lead to sparks being formed which damage the objects. On the other hand, larger jet-nozzle diameters are not being adopted because then the polishing recesses become too large, thereby consuming the stabilizing edge of the object being thinned.

Jet polishing devices have the advantage over holder polishing arrangements that disturbing gas bubbles are rinsed away by the electrolyte jet from the surface of the specimen which is not obstructed by polishing masks. Since the edge of the object is not covered, the centering of the electrolyte jet has to be very carefully observed.

The slightest eccentricity results in non-centrically thinned and perforated objects with partially dissolved or consumed edges, which are difficult to irradiate when used for electron microscopy.

Single-sided jet polishing devices require repeated turning over and centering of the specimens or objects, and consequently substantial expenditure in preparation and processing. Double-sided devices, on the other hand, result in all too fast occurring perforations which are difficult to control, so that they are only used for pre-thinning, followed by the above-described immersion-type pincer polishing, with its inherent disadvantages.

The known combined holder-jet-type devices also display the basic disadvantages of the described two kinds of arrangements since they have identically formed parts and systems. The disadvantages can be summarized as follows: only small ranges of specimens that can be irradiated for transmission electron microscopy; unwanted perforations of the specimens because the gas bubbles are not fully rinsed away by the electrolyte jet from the thick mask lips; grooves on the bottoms of the plate-shaped polishing recesses; excessive gas and heat formation as well sparks on the specimens being thinned; and/or the necessity of post-treatment by immersion polishing by the aid of pincers.

It is an object of the invention to provide a device with which relatively large-area technical and microscopic specimens or objects can be obtained, in a well reproducible manner and without substantial expenditures in preparation, having electrochemically or chemically thinned surfaces and mechanically stable, uniform edges.

It is furthermore an object of the invention to attain in a device for electrochemical or chemical thinning of technical and microscopic specimens laminar electrolyte flow, at substantial jet diameters, by the application of suitable structural expedients as will be explained.

In accordance with a major feature of the invention, the objects are solved in that two jet nozzle systems are provided on opposite sides of the specimen to be thinned, directed substantially perpendicular to its surfaces, each nozzle consisting of a long, wide-lumen jet tube, having a diameter larger than the inner lumen of polishing masks but at least **2** millimeters, each system comprising a rotation-symmetrical central nozzle space which tapers inwardly towards the respective jet nozzle.

A concentric, annular channel is disposed about each nozzle space with symmetrical, uniformly dimensioned overflow channels running from the annular channels to the respective nozzle spaces, a supply conduit linking each annular channel with a circulating pump.

In the described arrangement, direct connection is avoided between the pump and the nozzle spaces, thereby producing an important feature of the inventive device namely in that laminar electrolyte jet flow is insured, on one or both sides of the specimen, which is free from drift and turbulence, an expedient which is the prerequisite of a uniform, furrow-free thinning. Direct electrolyte supply cannot present these advantages.

The polishing masks, made from a hard plastic material, and having lip angles of about 30°, closely adjoin the specimens to be thinned, with the interposition of sealing rings of a soft plastic, the latter having thicknesses of less than 0.07 mm. The use of hard plastic for the masks makes for sufficient clamping power to seal the specimen edges against attack by the electrolyte, without however allowing deformation of the mask lips.

The lips can consequently be kept very thin. Sealing is accomplished by the rather thin and flat rings made from a soft plastic. The very thin mask lips do not disturb the laminarity of the electrolyte jets, as a result of their very low flow resistance, and do not allow any gas bubbles to adhere that could lead to unwanted perforation.

It is also a feature of the invention to keep the ratio of the diameter of the jet tubes above 1.7 in respect of the inner diameter of the polishing masks.

Preferably, the rear of each nozzle has a window to allow both the front and the rear sides or surfaces of the specimens to be thinned to be observed during the entire jet-thinning process, such as for example with a microscope.

The masks are to be made from polystyrol while the sealing rings are from polyethylene, thereby satisfying requirements both to their mechanical properties and to their durability and resistance against possible attack by the electrolyte, which could be hydrofluoric acid.

The polishing masks, with the specimen to be thinned therein, are preferably disposed in a specimen carrier which centers the specimen in respect of the jet nozzles by aid of a prism adapted to move along a guide, thereby dispensing with time-consuming adjusting operations.

It is possible to prepare with the device according to the invention, in a well repeatable and reproducible manner, and without substantial expenditures, that is in a routine operation, self-supporting foils for technical and microscopical specimens, such as thin or microsections, with a very high uniformity and without the danger of deformation.

The relatively slow electrolyte jets, having a streaming velocity of about **0.1** meter per second, have a cross-section as compared to the jets of the prior art, which is about 16-fold so that large-area specimens can be prepared.

In the case of initial or starting thicknesses of 0.3 mm, areas to be penetrated during transmission electron microscopy can be achieved in the range of 10^4 μm^2. The initial thicknesses of the specimens can range from 0.3 to 0.8 mm, as against a maximum of 0.3 mm with previously known arrrangements, without impairing the irradiation capacity of the electron-microscopic objects.

The aftertreatment of the thinned specimen, such as by pincer immersion, becomes superfluous. Consequently the danger of deformation and elastic spanning of the areas to be irradiated is eliminated. As a result of the considerable jet diameters, excessive gas and heat formation as well as sparking are avoided during the thinning process.

The inventive device is suitable for thinning semiconductor materials, as well as for thinning with hydrofluoric acid electrolytes.

Other objects and many of the attendant advantages of the invention will be readily appreciated as the same becomes better understood by reference to the following detailed description, when considered with the accompanying drawings, wherein

FIG. **1** is a vertical, longitudinal section of a preferred, exemplary embodiment of a device according to the invention for thinning specimens under laminar flow conditions;

FIG. **2** is a section, similar to that of FIG. **1**, on an enlarged scale, of a polishing mask containing a specimen to be thinned;

FIG. **3** is a front view of a specimen holder, as viewed at right angles in comparison to FIG. **1**, also on an enlarged scale; and

FIG. **4** is a side view of the specimen holder of FIG. **3**, that is viewed in the same direction as is FIG. **1**.

FIG. **1** shows the general arrangement of a preferred, exemplary embodiment of the inventive device, comprising a cuvette or vessel **1**, made for example of transparent polyvinyl chloride, and therein a jet cage **2**, preferably from the same material, having therein a pair of long, large-caliber jet tubes **3** protruding into a rotation-symmetrical central nozzle space **4**, constituting two jet nozzle systems **5**, made preferably of stainless steel, each of the latter being surrounded by an annular channel **6**, as shown. Central space **4** is connected with channel **6** by way of four radially symmetrical overflow channels **7** of substantially identical dimensions. Each of the annular channels **6** is connected by way of a supply channel **8** with its own centrifugal or

other circulating pump **9**. There is no direct supply of or connection for the electrolyte from either pump **9** to the jet systems **5**, as in known arrangements, thereby ensuring controlled laminar flow conditions, without untoward electrolyte drift or turbulence.

The rear of each jet nozzle **5**, as viewed from the center of the arrangement, where a specimen or object **11** to be thinned is disposed, has a window **10** of transparent polystyrol (also termed polystyrene) and the like, allowing the surface of the specimen to be viewed by the aid of an extraneous microscope **12** (only partly shown) by the aid of a substantially axially aligned luminous source **13**, such as a micro-light, and an appropriate condensor **14**. Observation can of course be continued throughout the thinning process.

Specimen **11** is held between two polishing masks **15** (see also FIGS. **2, 3** and **4**) which are held in a carrier **16**, secured with appropriate screws **17**.

Carrier **16** has a guide prism **18** for centering the specimen **11** to be thinned between nozzles **5**, namely in a prism guide **19**, a knurled adjusting knob **20** allowing arresting in the terminal position.

FIG. **2** illustrates, strongly enlarged, the chucking of specimen **11** between rigid polishing masks **15**, preferably made from transparent polystyrol, with soft sealing rings **21** disposed therebetween, preferably of polyethylene, and having an exemplary thickness of 0.06 millimeters.

FIGS. **3** and **4** show that masks **15**, together with specimen **11**, can be pushed from below into a U-shaped recess **16*a*** of carrier **16**. Clamping springs **22** and the like are provided for immobilizing masks **15**.

Chemical thinning has to be used for non-conductive specimens and for semiconductors. For purposes of electrolytic thinning, suitable for metals, specimen **11** is in ohmic contact with a contact lip or tongue **23**, fixed on a tongue carrier **24**, which latter is vertically adjustable by the intermediary of a control knob **25**. In its terminal, contacting position carrier **24** is immobilized by means of a knurled knob **26** (see FIGS. **3, 4**).

A current conducting screw terminal **27** is provided in conjunction with a contact rail **28** for linking specimen **11** to be electrochemically thinned with the positive pole of a DC source **29**; the negative pole of the latter is connected with the stainless steel jet nozzle systems **5**, preferably by means of conventional insulated platinum wires, schematically shown at **30**.

As to the operation of the inventive device, it will be understood that specimen **11** is inserted and fixed in carrier **16**, and the latter inserted into the device, then the flow velocity of each electrolyte jet adjusted, first by sight, through the respective pumps **9**. The jet systems produce a drift- and turbulence-free electrolyte jet in the direction of the specimen as explained before. During the initial thinning process, the amount of the electrolyte filled into cuvette **1** is kept at a level below object carrier **16**.

The just described arrangement and operation ensure that the specimen is only pre-thinned until just before perforation. The specimen is then perforated after having stopped pumps **9** and added some more electrolyte into cuvette **1**, up to above specimen **11**. Upon perforation, carrier **16** with specimen **11** are removed and repeatedly rinsed by immersion in methyl alcohol. Still in the methyl alcohol, specimen **11** is removed from carrier **16**. Upon appropriate drying, the thinned and perforated specimen is ready for use.

It should be understood, of course, that the foregoing disclosure relates only to preferred embodiments of the invention and that it is intended to cover all changes and modifications of the example described which do not constitute departures from the spirit and scope of the invention.

What I claim is:

1. A device for selective chemical and electrochemical thinning of respective non-metallic and metallic, technical and microscopic specimens under laminar flow conditions of the applied electrolyte, the device having at least one jet nozzle directed toward the specimen so as to apply the electrolyte to its surface to be thinned and optionally perforated, the specimen being held in at least one mask, the device comprising, in combination, a wide-lumen jet tube associated with each said at least one jet nozzle, a central jet space being provided in an area adjoining said mask and tapering inwardly in the direction of exit of said jet tube, an annular channel surrounding said jet space, at least four symmetrically disposed overflow channels between said annular channel and the associated jet space, and pump means for conveying the electrolyte to said annular channel, by the intermediary of a supply channel, the arrangement of said annular channel and said at least four overflow channels between said at least one jet nozzle and said pump means providing means for producing controlled laminar flow of the electrolyte, with the absence of a direct supply between said at least one jet nozzle and said pump means making for the laminar flow conditions without drift and turbulence.

2. The device as defined in claim **1**, wherein the diameter of said jet tube is larger than the inner area of said at least one mask, in the order of at least 2 millimeters.

3. The device as defined in claim **1**, wherein said at least one mask has a lip angle of about 30°.

4. The device as defined in claim **1**, wherein the ratio between the diameter of said jet tube is above 1.7 in respect of the inner area of said at least one mask.

5. The device as defined in claim **1**, further comprising at least one window flanking a wall of said jet space to allow the inspection of the thinning procedure.

6. The device as defined in claim **1**, further comprising electrical contact means for connecting the poles of an extraneous DC source to the specimen as well as to said jet tube, for purposes of electrochemical thinning.

7. The device as defined in claim **1**, wherein said pump means and the flow conditions in said supply channel, said annular channel, said at least four overflow channels, said jet space and said jet tube are adjusted so as to yield a flow velocity of the electrolyte of about 0.1 meters per second at each said at least one jet nozzle.

8. The device as defined in claim **1**, wherein the initial thickness of the specimen to be thinned exceeds 0.3 mm and ranges up to 0.8 mm.

9. The device as defined in claim **1**, further comprising a sealing ring interposed between the specimen and said at least one mask.

10. The device as defined in claim **9**, wherein said at least one mask is made of a transparent, hard plastic such as polystyrene and said sealing ring is made of a

soft, elastic plastic such as polyethylene, the latter having a thickness of maximum 0.07 mm.

11. The device as defined in claim **1,** further comprising a carrier for said at least one mask and the specimen therein, a prism to which said carrier can be secured, and a guide along which said prism can be moved for positioning the specimen in front of and centered with respect to said at least one jet nozzle.

12. The device as defined in claim **11,** further comprising means for adjusting and securing the position of said carrier with respect to said guide.

13. The device as defined in claim **1,** further comprising a cuvette and a jet cage therein for lodging said jet tube and the associated elements of the device.

14. The device as defined in claim **13,** wherein said cuvette and said jet cage are made of transparent polyvinyl chloride while said at least one jet nozzle is made of steel.

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