

IB-2/IB-3/IB-5

ION COATER

**ION SPATTER COATING AND ION ETCHING
FOR TEM/SEM SPECIMENS**



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Ion spatter coating and ion etching for TEM/SEM specimens Producing a glow discharge in a vacuum as low as around 10^{-2} Torr and ionizing the residual gas, Model IB-2/3/5 ion coater can spatter metals on a specimen, clean and etch a specimen with the energy of the ionized gas. It is best suited to prepare a specimen for both TEM and SEM observations.

Friendlier design, easy to operate without special skill.

This ion coater makes available higher resolution, higher quality photos with the TEM, and enables internal structure observation as well as surface observation with the SEM. Furthermore, the processing in various gas atmospheres can expect new findings.

It provides wide applications as an electron microscope peripheral.



IB-2

- **Photo on the front**
Surface of trachea of rat (Pt. spatter coating)
Magnified by 3,000
Courtesy of Prof. Keichi Tanaka and Prof. Hitoshi Osatake, M.D. of Tottori University

FEATURES

- (1) Provides effective coating of metals such as Au, Au-Pd, Pt, Pt-Pd, etc.
- (2) Best suited to high-resolution coating.
The coating particles are 1/5 or less in area, 1/2 or less in thickness, compared with those by vacuum evaporation. Coated with these particles, a specimen can have sufficient conductivity and high emission efficiency of secondary electrons.
- (3) Speedy, efficient processing.
One processing phase including evacuation takes less than 10 minutes. Four to ten specimens can be treated in a single processing phase.
- (4) Easy to set the optimum degree of vacuum.
(Model IB-3/5)
The vacuum gauge having the limit switch is provided on the low vacuum side. Also, the needle valve for evacuation control is provided.
- (5) Quantitative coating controllable.
The ion meter can be used to adjust ionization voltage, and the timer can control the processing time.
- (6) Free from ionizing/thermal damage on coating a specimen.
The water-cooled specimen mount minimizes a rise in the temperature of a specimen in the etching mode.
- (7) The safety device is provided to avoid a high voltage shock.

- (8) Model IB-2 features the minimal functions to available at low price.
- (9) Model IB-3 is a standard version provided with the limit-switch-attached vacuum gauge on the low vacuum side to maintain the optimum condition.
- (10) Model IB-5 is an upgraded version capable of coating fine spatters such as Pt and PT-Pd particles for the high-resolution SEM observation, also it can perform efficient ion etching of metals, ceramics, biological hard tissues, etc.

Model IB-5 uniquely designed to perform discharge between the special electrodes for each of coating and etching. Particularly for the spatter coating, both the upper and lower electrodes are of doughnut shape and fixed so as to retain stable discharge. The specimen mount can move up/down between these electrodes, and the distance between specimen and target can be set arbitrarily. Further, insulation from these electrodes prevents a specimen from being damaged by ions or electrons. (Ref.: 34th Cong. on Electron Microscopy – 21-D1-15.)



IB-3



IB-5

EXAMPLE OF APPLICATION—1

1. SPATTER COATING (Conductive-coating evaporation for SEM specimen)

In vacuum of 0.1 to 0.05 Torr, a voltage of approx. 1,000V is applied across the coating metallic electrode (negative) and the specimen mount (positive). This causes the ionized gas molecules to strike the metallic electrode, springing out metallic particles from it (spattering). The sprung-out metallic particles fly toward a sample. During the fly, they collide with the residual gas molecules to turn their directions. The metallic particles arrive at the sample in many directions, therefore no gimbal is re-

quired. Also, the electromagnetic force applied allows the metallic particles to attach deep into the specimen. This characteristic gives advantage particularly to the application of a complicate-shaped specimen. Available as the metallic coating materials are Au, Au-Pd, Pt and Pt-Pd. Finer than the particles for vacuum evaporation and excellent in the secondary-electron emitting efficiency, the ion-spatter coating particles are suitable to a specimen to be prepared for high-resolution SEM observation.

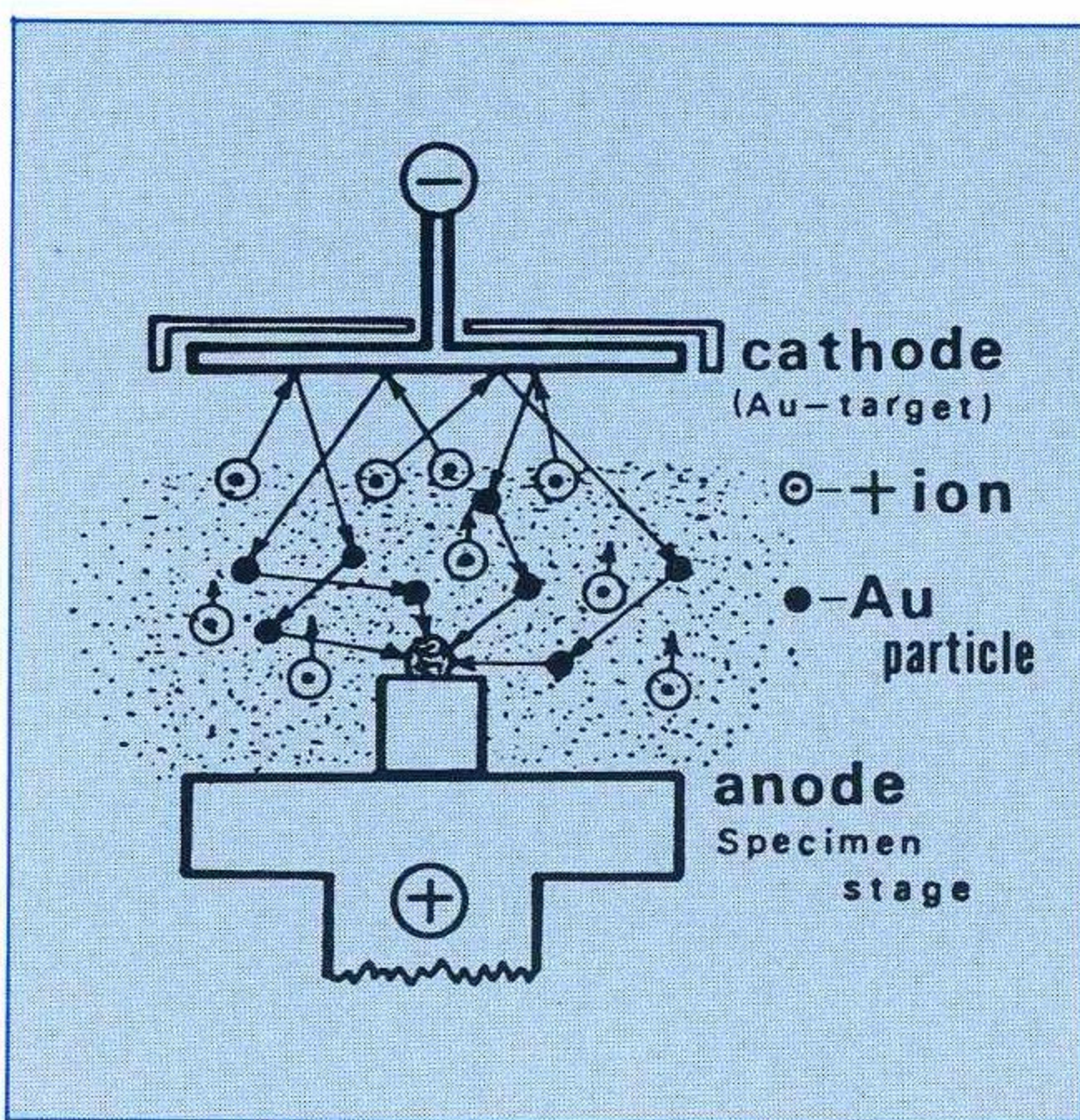


Fig. 1 Principle of Ion-Spatter Coating

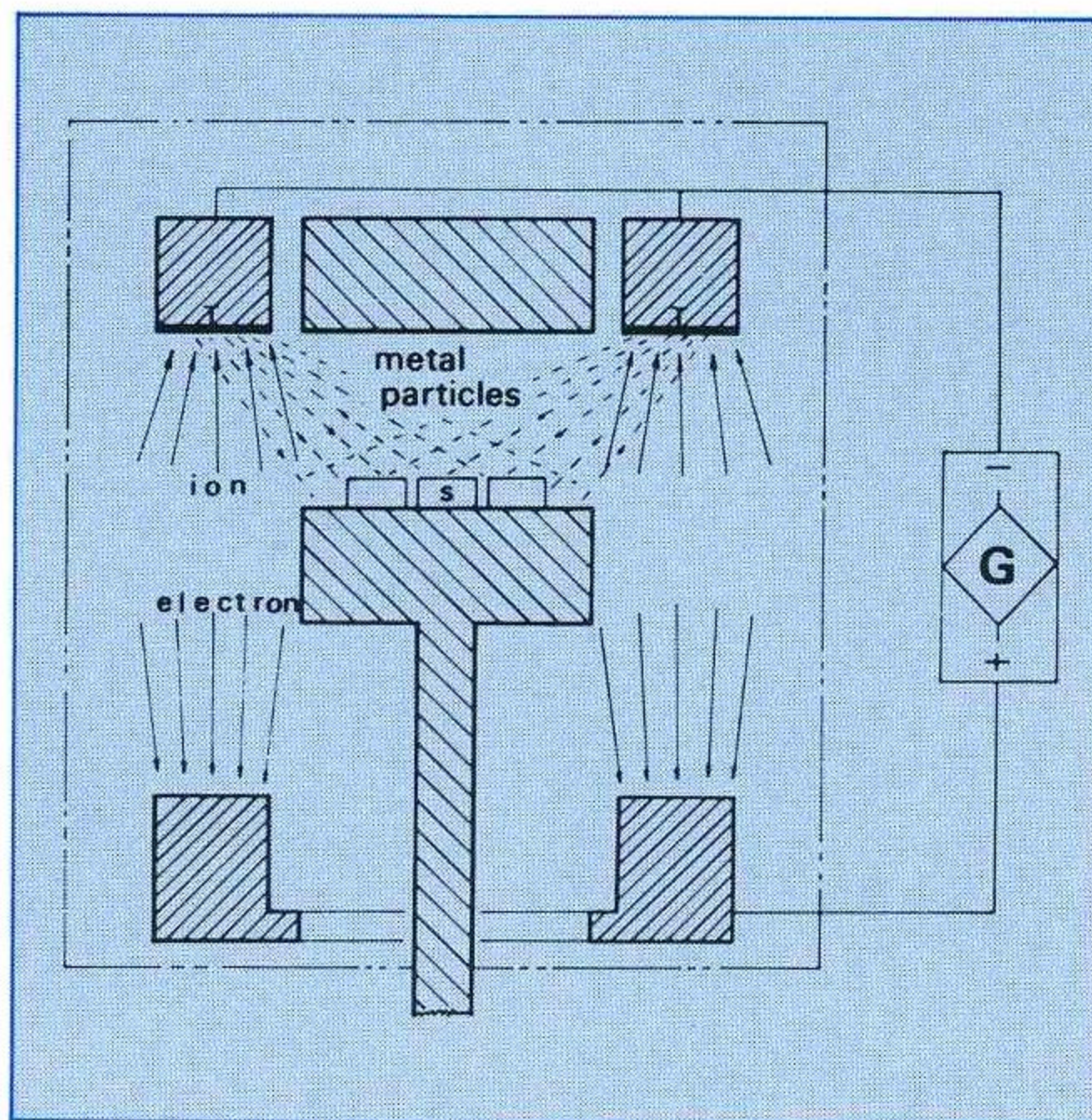


Fig. 2 Electrode Structure of Model IB-5
(T: Pt Target S: Sample G: Generator)

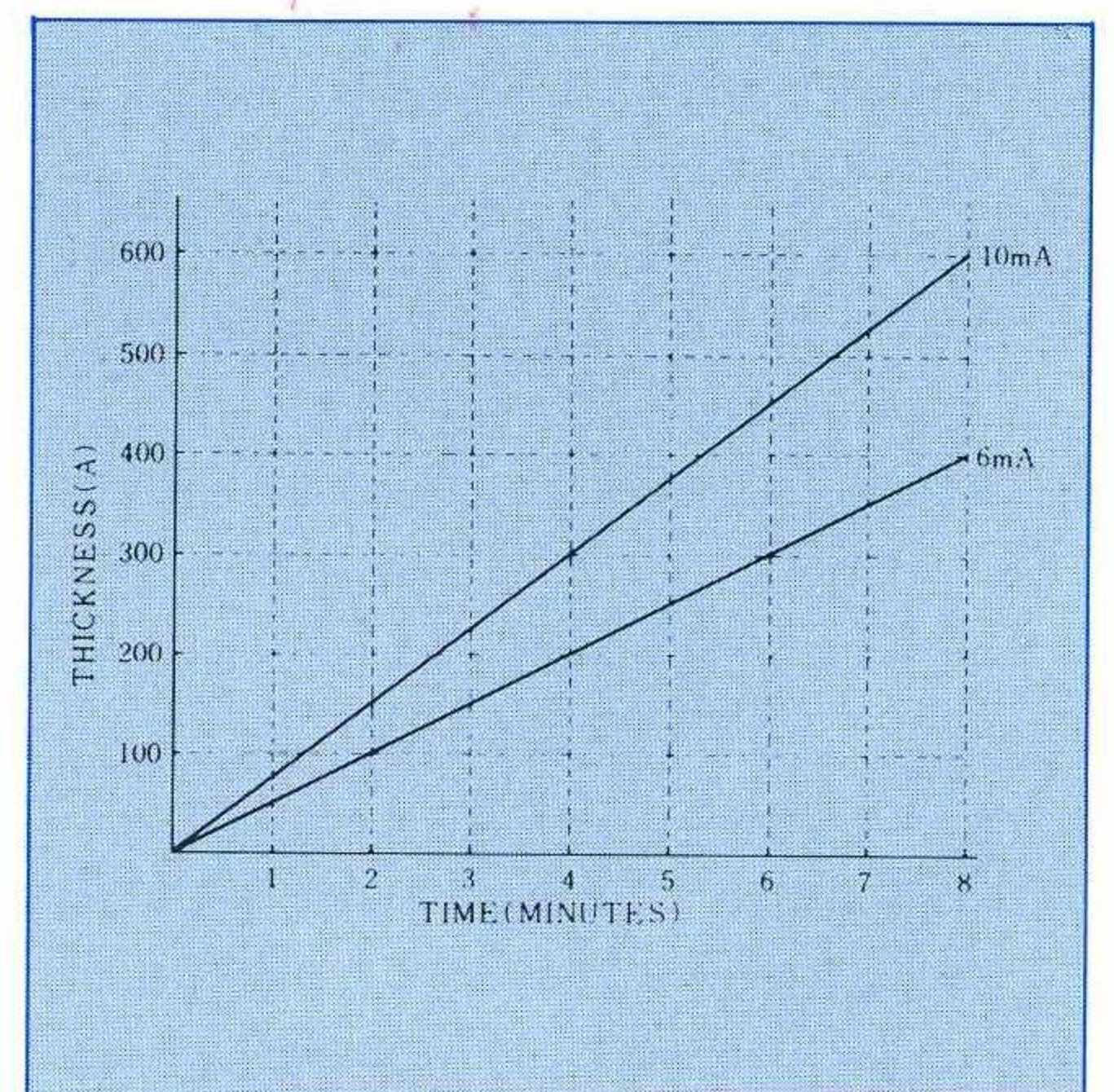
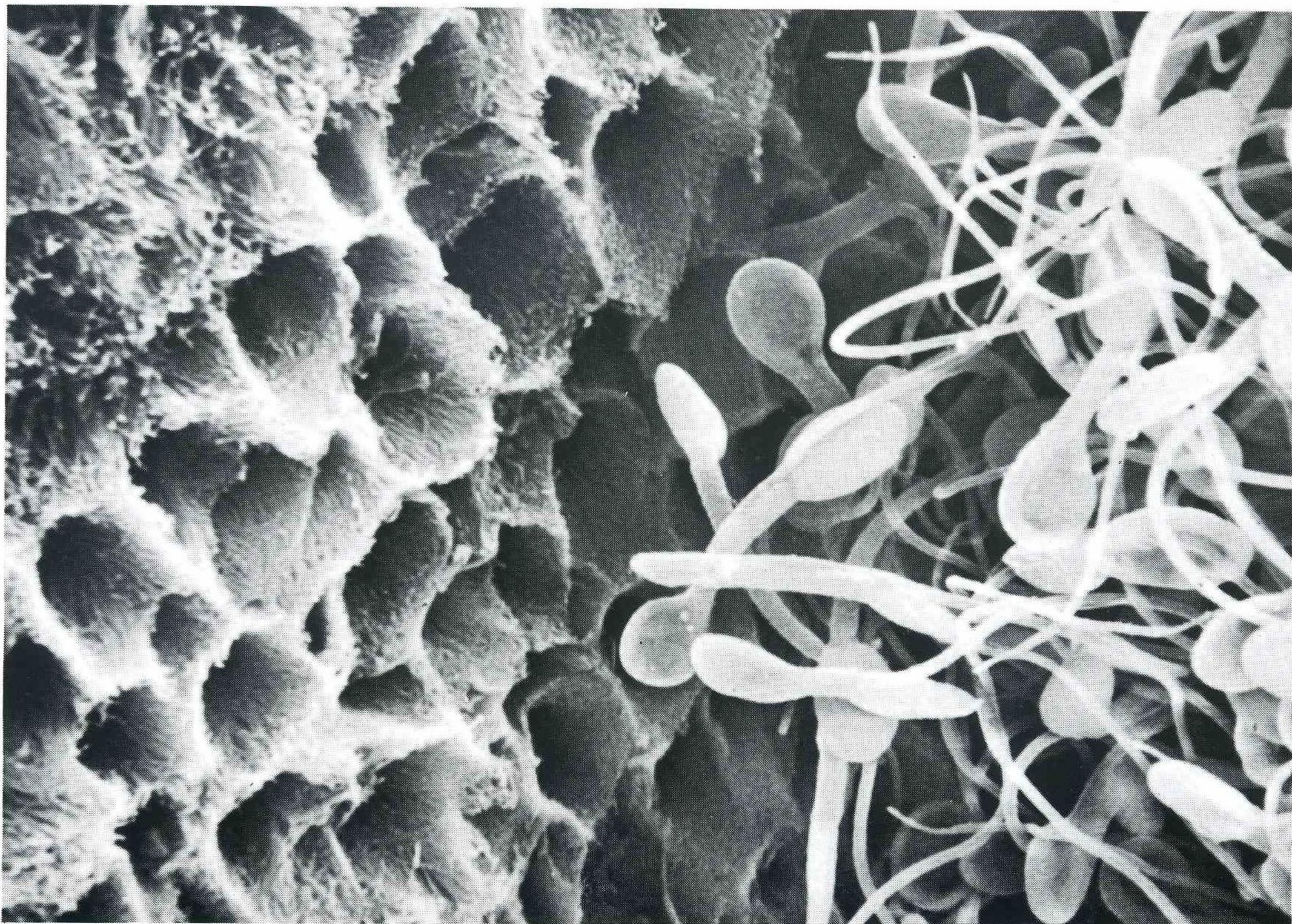


Fig. 3 Change of Au-Coating Thickness with Processing Time
(Au: 1,400V DC Pt: 3,000V DC)



Epididymis of Japanese monkey; Stereocilia and spermatozoa
Courtesy of Prof. Masahiro Murakami and Prof. Tatsuo Shimada, M.D., 2nd Anatomy Class, Kurume University

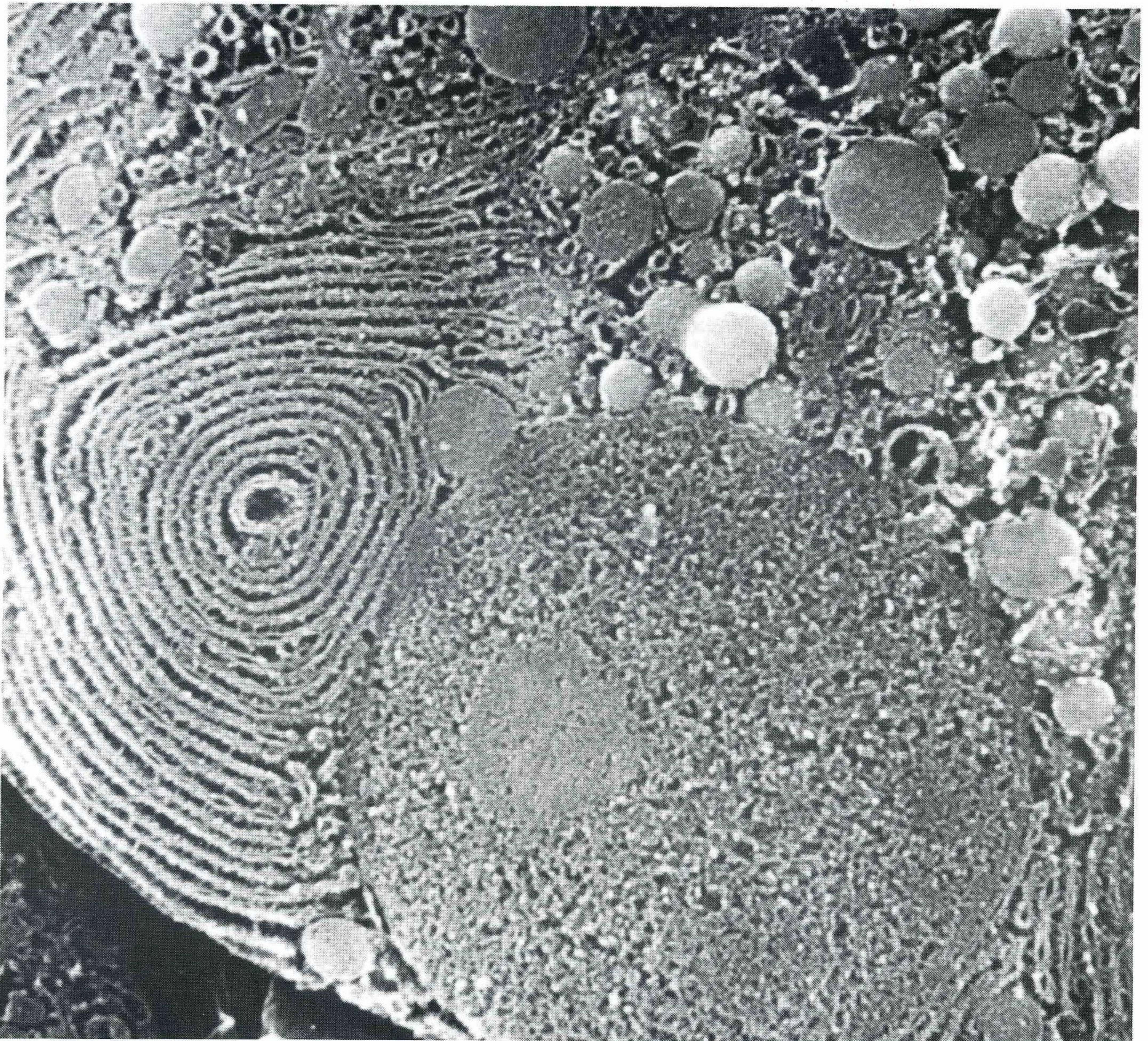
Fig. 4 Example of Biological Specimen

EXAMPLE OF APPLICATION—2

2. ION ETCHING

The ion etching is performed by striking a specimen with the ionized gas molecules (specimen being negative). For such a nonconductive specimen as a biological material, DC discharge may cause it to be charged positive by the gas ions, resulting in uneven etching. To avoid this, AC discharge can be performed during etching. (The AC etching mode is optional.)

For such a conductive specimen as a metallic material, the DC ionization is effective. To realize the gas-varying etching for some kinds of specimens, various gases can be introduced through the vacuum control needle valve. The water-cooled specimen mount can prevent the temperature of a specimen from increasing due to ion bombardment.



- Specimen-Pancreas of dog; Exocrine gland cell (Magnified by 17,500)
 - Processing-Resin freezing and cutting → Critical point drying → Ion etching → Au spatter coating
- Courtesy of Prof. Keichi Tanaka and Prof. Tomonori Naguro, M.D., 2nd Anatomy Class, Tottori University

Fig. 5 Example of Ion Etching

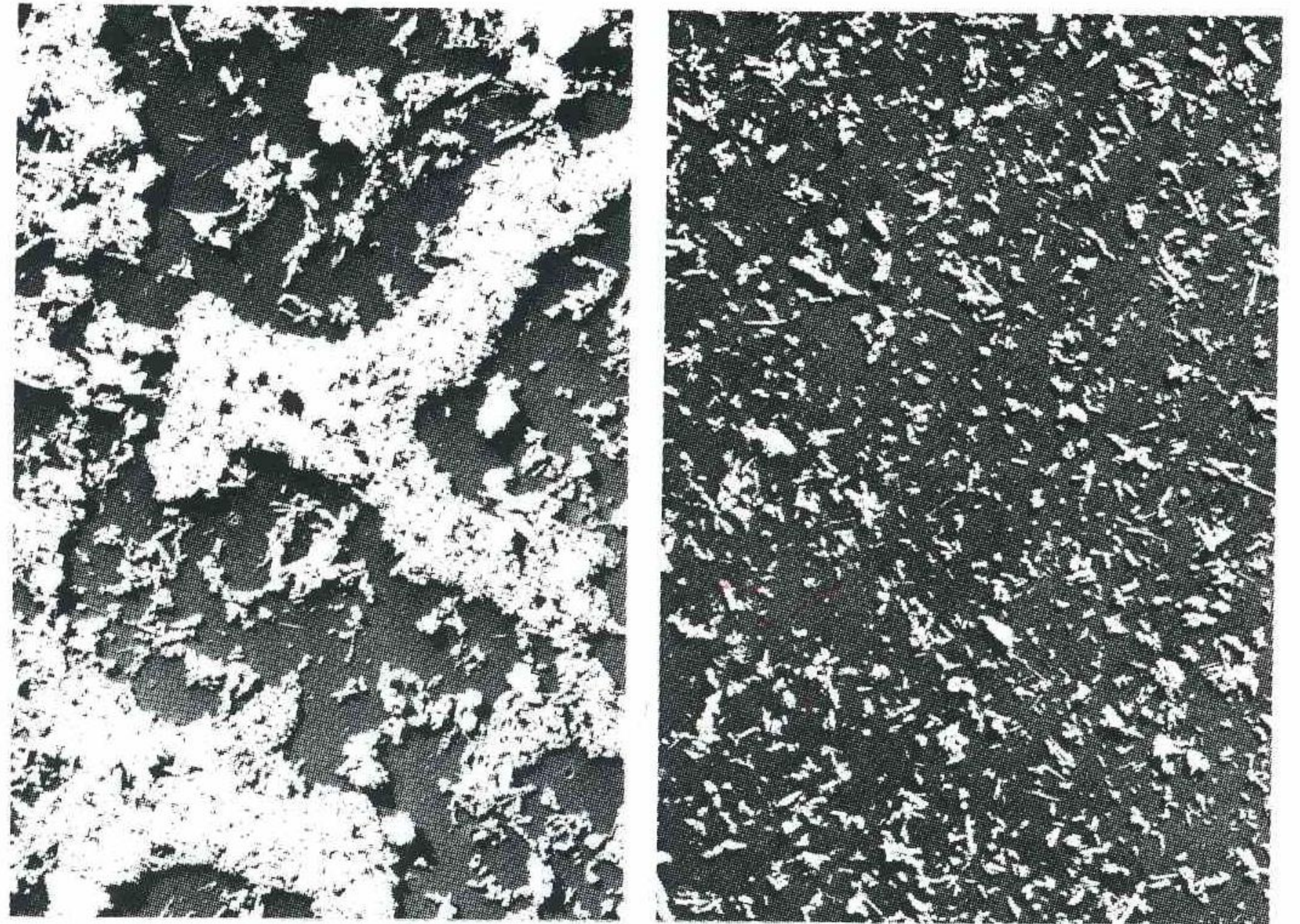
EXAMPLE OF APPLICATION—3

3. ION CLEANING (HYDROPHILIC PROCESSING)

With this ion coater, it is easy to make hydrophilic the supporting membrane of a specimen for TEM. Place the sheet mesh having a collodion membrane or carbonic reinforcing membrane on the slide glass, and set it on the specimen mount. With the supporting membrane thus prepared, flash 500V AC for five seconds in a vacuum of around 0.1 Torr. Thus, a perfectly hydrophilic membrane can be obtained.

The hydrophilic supporting membrane allows uniform dispersion of specimen powders or virus particles suspended in water. If the sheet mesh with no supporting membrane is made hydrophilic, it becomes easy to pick up a slice or replica film. Also, it adheres closely to a specimen to eliminate movement under the electron beam radiation. (The AC discharge mode is optional.)

Fig. 6 Example of Hydrophilic Processing



6-a Supporting Membrane Before Hydrophilic Processing
6-b Supporting Membrane After Hydrophilic Processing
Hydrophilic processing effect of supporting membrane for fine particle dispersion
Specimen-Thepiolite clay (Pt-Pd shadowed)

HOW TO USE EIKO ION COATER

1. Ion Spatter Coating (Common to three models)

- 1) Place a specimen just under the target electrode, within 80% of the outside diameter of the electrode plate.
- 2) The standard clearance between target and specimen is 2.5 cm to 3 cm.
- 3) Perform evacuation down to about 0.1 Torr.
- 4) Set the timer to two minutes, turn on the H.V. switch, and rotate the H.V. control up to a reading of 9 or 10 (max.) gradually.
If the ammeter reads 5 mA or more and is likely to increase its indication, turn off the H.V. switch and continue evacuation further.
When the ammeter indicates less than 5 mA with the H.V. control in the vicinity of the maximum level, set the timer to two minutes again. And, loosen the needle valve gradually so that a reading of 8 mA can be obtained on the ammeter.
- 5) After keeping a current of 8 mA for about 30 seconds, close the needle valve slightly to return it to 5 or 6 mA. Use the remaining period for coating a specimen.
- 6) This makes available a coating film of approx. 100 Å. If it is desired to obtain other coating thickness than 100 Å, reduce evenly the conditioning periods in step 5).

2. Making TEM Specimen Supporting Membrane Hydrophilic (IB-2, IB-3)

- 1) Place on the slide glass the grid mesh with the supporting membrane facing up, and set it on the specimen mount.
Put the auxiliary mount on the lower electrode so that a distance to the upper electrode becomes 20 mm.
- 2) Replace the target on the upper electrode with the stainless target, close the lid, and perform evacuation.
- 3) Set the selector switch to the AC mode.
If the AC mode option is not provided, set it to the COATING mode. Notice that the ETCHING mode may damage the supporting membrane.
- 4) Set the H.V. control to 3 or 4.
- 5) When the limit switch of vacuum gauge is activated (approx. 0.2 Torr), press the FLASH switch for discharge for about ten seconds.
- 6) If a degree of vacuum is too hard to flow a discharge current, loosen the needle valve slightly while pressing the FLASH switch. Let the ammeter read 3 to 5 mA. (IB-2)
- 7) The extended flashing for more than ten seconds is ineffective. The supporting membrane which has lost hydrophilic characteristic can be processed repeatedly.
- 8) If the inside of the vacuum chamber is contaminated after coating many SEM specimens, it may cause contamination on the supporting membrane. If so, clean the chamber well before attempting the hydrophilic processing.

STRUCTURE

Vacuum control needle valve

Specimen mount (lower electrode)

Target (upper electrode)

Vacuum chamber

Mode selector switch

Timer

Ionization ammeter

H.V. control

Input switch



IB-2

Target (upper electrode)

Specimen mount (lower electrode)

Vacuum chamber

Ionization ammeter

Vacuum gauge

Timer

H.V. control

Mode selector switch

Vacuum control needle valve

Input switch



IB-3

Etching terminal

Vacuum gauge

Coating terminal

Timer

Upper electrode

Ionization ammeter

Vacuum chamber

Mode selector switch

Lower electrode

H.V. control

Specimen mount up/down control

Flash button

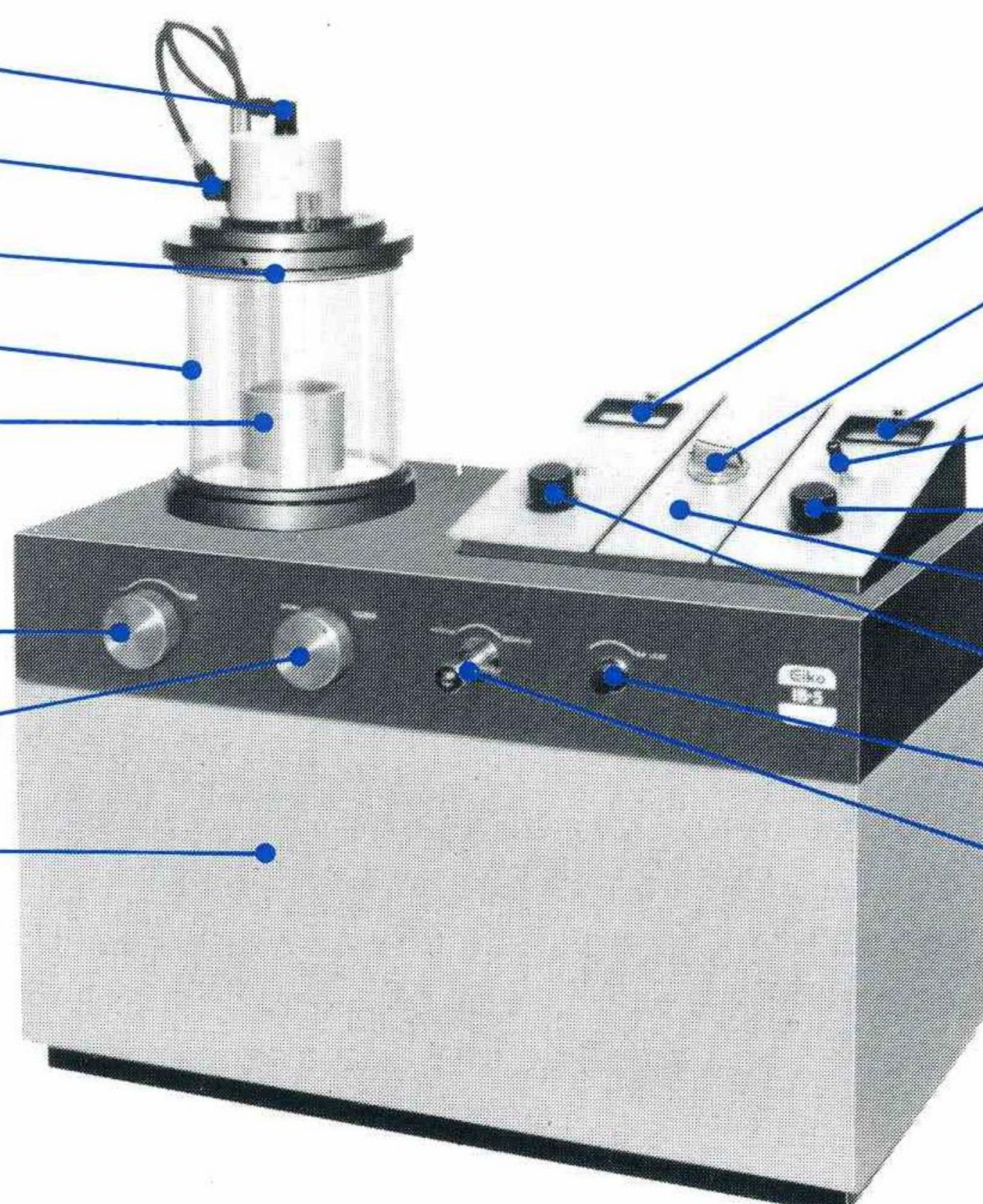
Vacuum valve control

Input switch

Rotary pump

R.P. air leak

Needle valve



IB-5

SPECIFICATIONS

	IB-2	IB-3	IB-5 (In coating mode)
Vacuum chamber	130 mm ϕ x 110 mmH	Same as left	150 mm ϕ x 160 mmH
Upper electrode (Target)	50 mm ϕ (Round)	Same as left	40 to 70 mm ϕ (Doughnut-shaped)
Lower electrode	52 mm ϕ	Same as left	50 to 70 mm ϕ (Doughnut-shaped)
Specimen mount	On lower electrode	Same as left	40 mm ϕ , Independent, Up/down movable
Cooling of specimen mount	Water cooling	Same as left	Same as left
Clearance between target and specimen mount	20 mm, 35 mm	Same as left	20, 35, 45, 55 mm; 4 steps variable
Ionization voltage	D.C. 0 ~ 1,400V	Same as left	D.C. 0 ~ 3,000V
Ionization current	10 mA (max) on ammeter attached	Same as left	Same as left
Coating electrode	Au ; Au - Pd , Pt - Pd	Au ; Au - Pd , Pt - Pd	Pt ; Pt - Pd , Au - Pd , Au
Operating vacuum	0.05 ~ 0.2 Torr	Same as left	0.01 ~ 0.2 Torr
Vacuum regulation	Needle valve controlled	Same as left	Same as left
Vacuum gauge	Not provided	Pirani gauge (with limit switch on low vacuum side)	Same as left
Vacuum pump	20 ℓ /min R.P. (Separate)	Same as left	50 ℓ /min R.P. (Contained)
Dimensions	384 (W) x 220 (D) x 345 (H)	384 (W) x 220 (D) x 390 (H)	600 (W) x 450 (D) x 670 (H)
Weight of main unit	14 kg	17 kg	25 kg
Rotary pump	9kg	9 kg	11kg

- Notes:**
1. With Model IB-5 in the etching mode, the interelectrode clearance should be 80 mm fixedly, and the upper and lower (specimen mount) electrodes be 40 mm ϕ .
 2. The coating target electrode is optional.
 3. The AC discharge function (AC mode) is optional in all of these three models.

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