Osmium Plasma Coater

For coating of conductive thin films on scanning electron microscopy samples

Filgen, Inc.

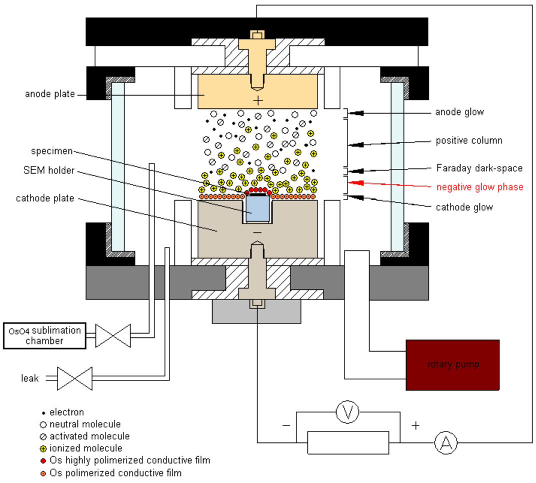
Overview

No charging. No grains. No heat damages.
An ultimate solution to obtain clear SEM images.

Osmium Plasma Coater

The Osmium Plasma Coater is a kind of plasma CVD coater which is utilizing a method for coating samples in the negative glow phase domain of DC plasma discharging. As conventional types of metal coaters, there are various kinds of so called sputter coater which use the heavy metals as coating material, such as gold, platinum, palladium etc. With these methods, the problem of granularity of coating itself is inevitable by any means. Also, if examined at high magnification, the specimens coated by these methods can not avoid charging, heat damage and contamination caused by exposure to strong electron beam with SEM, resulting in lower resolution of SEM image.

A gas reactor in which the anode and cathode plates are placed vertically is evacuated to a high vacuum, then sublimated osmium tetroxide (OsO_4) is introduced, and a DC glow discharge is generated under certain conditions, while maintaining a predetermined gas pressure. The osmium metal molecules excited by the collision of electrons instantaneously become plasma between the two electrodes. In particular, in the negative glow phase domain, there is strong light emission due to the fierce diffusion of the concentrated positively ionized metal molecules. As a result, on the surface of the specimen placed in this negative glow phase, the positive ion metal molecules uniformly adhere to the specimen surface and form a perfectly amorphous metal coating of molecular level, which molds the microstructure of the specimen surface faithfully.



Applications

Osmium thin film

- Conductive thin film for SEM specimen
- Prevention of contamination for SEM/TEM specimen
- Protective film for AFM specimen
- Conductive protective film for SPM specimen
- Protective film for SPM cantilever

Plasma-polymerized film (naphthalene)

- Protective film for FIB specimen
- Prevention for peeling sample from embedding resin
- Coating for fluorine resin (Os-PF hybrid coating)
- Support film for TEM grid
- Drift prevention for ultra-thin sections for TEM

Osmium ultra-thin film [optional]

- Observation of the ultrafine structure of insulators by FE-SEM
- Quantitative analysis of top surface of insulators by ESCA/AES
- Enhancement of conductivity for TEM specimen
- Static prevention treatment for AFM specimen
- Antistatic treatment for STM specimen
- Etching (Mixed gas method only)

Hydrophilizing treatment [optional]

- Pre-hydrophilization treatment for osmium coating on fluoro resin surface etc.
- Prevention for peeling sample from embedding resin
- Hydrophilization of the support film surface for TEM
- Hydrophilization of grids for TEM
- Hydrophilization of a diamond knife for ultramicrotome
- Improvement of wettability

Deep well electrode [optional]

- Coating for samples with a height

Multiple safety measures provide operator protection from OsO_4 exposure.

Benefits

Osmium film

No grains: amorphous metal coating is formed
No heat damage: coating is proceed under room temp.
No electron beam damage: the melting temperature of Osmium is 2700 deg C
No contamination: coating is started from vacuum state

Plasma-polymerized film (naphthalene)

Strong film: withstand the gallium ions beams used in FIB
Heat resistant and insulated film
No grains: amorphous metal coating is formed
No heat damage: coating is proceed under room temp.
No electron beam damage
No contamination: coating is started from vacuum state

Usability

Automatic: whole process is completed just by pressing START button after settings
Intuitive: coating thickness is controlled by thickness setting, not by discharge time
Short coating time: a few nm/a few seconds
Easy to exchange Os ampoule: detachable reservoir, observation window (for checking of remaining amount of Os) and built-in ampoule cutter on reservoir

Safety

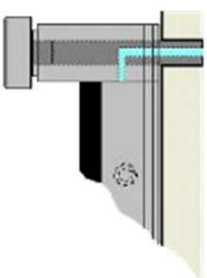
Fully automated: reduce chance of human error
Safety features on Os reservoir: gas port integrated locking pin and built-ion ampoule cutter
Interlocking system with reaction chamber: unable to open the chamber unless OsO_4 is exhausted, unable to introduce OsO_4 when the chamber open
Failsafe to prevent Os leak at power down
Osmium absorption filter: not require any ventilator

Safety features on osmium reservoir

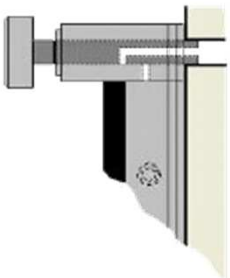
Filgen's OsO_4 reservoir is equipped with some safety measures including a mechanical interlocking system for safety operation.

- Gas-tight and robust design
- Safety interlocking system with gas port integrated locking pin (see below)
- Built-in ampoule cutter: can reduce risks for exposure to toxic OsO_4 gas.
- Observation window: can check remaining amount of OsO_4 crystals
- Detachable from main unit and can be stored in freezer

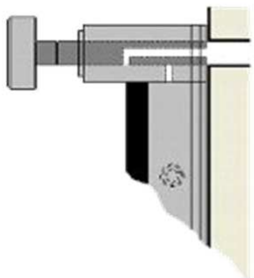
The gas port integrated locking pin of reservoir can simultaneously control status for reservoir locking and gas supply in safe.



Full-opened (during operation)
Reservoir: locked (undetachable) by manually inserted locking pin of reservoir
OsO_4 gas: can be introduced (depends on solenoid status)



Half-closed (before reservoir detach)
Reservoir: locked (undetachable) by manually inserted locking pin of reservoir
OsO_4 gas: blocked *residual gas in the inlet pipe must be evacuated at this step



Full-closed (after operation)
Reservoir: unlocked (detachable)
OsO_4 gas: blocked

Product line-up

Osmium film dedicated model #OPC60A

Coating type: Osmium
Thickness: several nanometer to hundreds nanometer

Osmium / Plasma-polymerized film model #OPC80T

Coating type: Osmium / PF (napthalene)
Thickness: several nanometer to hundreds nanometer

+

Optional units

Conductive ultra-thin film coating system

Coating type: Osmium
Thickness: 0.5 to 3.0 nanometer

Low-current method
Forming a film with very low discharge current by gradually introducing OsO_4 gas to highly vacuumed chamber with applying voltage. This enables to enhance reproducibility of coating thickness by lowering uncontrollable over current generated at initial phase of plasma discharge

Mixed-gas method
Forming a film slowly with relatively low concentration of OsO_4 gas by mixing an inactive gas to chamber. By discharging only with air or other inactive gas, it is also possible to etch your sample.

Hydrophilizing treatment system

Enhance adhesiveness of coating to your samples by applying this feature before coating

Deep well electrode

Enable to coat your samples that have a height up to 44 mm.

Accessories

Osmium reservoir

Detachable, equipped with safety features.

Transportation Container

Enable safer transportation for OsO_4 reservoir. Stainless steel made.

OsO_4 ampoules

With exclusive optional units, Filgen's OPC provide a variety of pretreatment methods for your EM samples

Specifications – base units

Type	Osmium / Plasma-polymerized film model	Osmium film dedicated model
Model #	OPC80T	OPC60A
Reaction chamber	Glass chamber, 160(ϕ) x 105(H) mm	Glass chamber, 120(ϕ) x 73(H) mm
Max. sample size	44(W) x 44(D) x 4(H) mm or 32(ϕ) x 14(H) mm	33(W) x 33(D) x 4(H) mm or 36(ϕ) x 14(H) mm
Supported SEM specimen mounts	10 mmϕ x 2 pcs and 15 mmϕ x 2 pcs and 32 mmϕ x 1 pc.	10 mmϕ x 7 pcs or 15 mmϕ x 4 pcs or 36 mmϕ x 1 pc.
Coating thickness	Several nanometers to hundreds of nanometers	
Settable min. thickness	Ultra-thin film mode: 0.1 nm / Normal mode: 1 nm	
Coating type	Osmium conductive film, Plasma-polymerized film (napthalene)	Osmium film
OsO_4 reservoir features	Detachable (capable to store in freezer) / Gas port integrated safety locking pin / Observation window / Built-in ampoule cutter	
Naphthalene reservoir features	Heater for effective sublimation / Observation window	-
Gas introduce/exhaust method	Automated control with vacuum gauge, solenoids, and vacuum pump	
Power requirements	100VAC (single-phase) 50/60Hz 12A (including supply to a vacuum pump)	100VAC (single-phase) 50/60Hz 10A (including supply to a vacuum pump)
Dimensions	450(w) x 410(D) x 390(H) mm	450(w) x 390(D) x 340(H) mm
Weight	Approx. 30kg	Approx. 20kg

Vacuum rotary pump (supplied with Osmium absorption filter)

Power requirements	100VAC (single-phase) 50Hz(60Hz) 550W	100VAC (single-phase) 50Hz(60Hz) 200W
Full-load current	9.0A (8.4A)	5.6A (4.8A)
Actual pumping speed	200L(240L) /min	50L(60L) /min
Dimensions	170(W) x 520(L) x 560(H) mm	170(W) x 400(L) x 580(H) mm
Weight	Approx. 31kg	Approx. 18kg

Specifications – optional units

Conductive ultra-thin film coating system (low-current method)

Coating type	Osmium conductive ultra-thin film
Coating thickness	0.5～3.0 nm
Gas introduce method	Needle valve

Conductive ultra-thin film coating system (mixed-gas method)

Coating type	Osmium conductive ultra-thin film	-
Gas introduce method	Mass-flow controller	-
Supported gas	N_2、Ar、He、O_2	-
Dimensions	570(w)×410(D)×390(H)mm	-

*This option cannot be installed along with hydrophilizing treatment system

Hydrophilizing treatment system

Gas introduce method	Needle valve	
Supported gas	Air	
Dimensions	570(w) x 410(D) x 390(H) mm	570(w) x 390(D) x 340(H) mm

*This option cannot be installed along with Conductive ultra-thin film coating system (mixed-gas method)

Deep well electrode

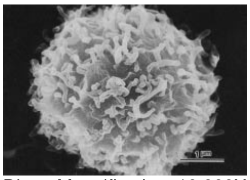
Reaction chamber	Glass chamber, 160(ϕ) x 125(H) mm	Glass chamber, 120(ϕ) x 103(H) mm
Max. sample size	44(W) x 44(D) x 4(H)mm or 46(ϕ) x 44(H) mm	33(W) x 33(D) x 4(H) mm or 36(ϕ) x 44(H) mm
Supported SEM specimen mounts	10 mmϕ x 16 pcs or 15 mmϕ x 7 pcs or 46 mmϕ x 1 pc.	10 mmϕ x 7 pcs or 15 mmϕ x 4 pcs or 36 mmϕ x 1 pc.
Dimensions	450(w) x 410(D) x 410(H) mm	450(w) x 390(D) x 370(H) mm

Technical data

Comparison data: Osmium plasma coat vs. Sputter coat

Sample: Human Lymphocyte

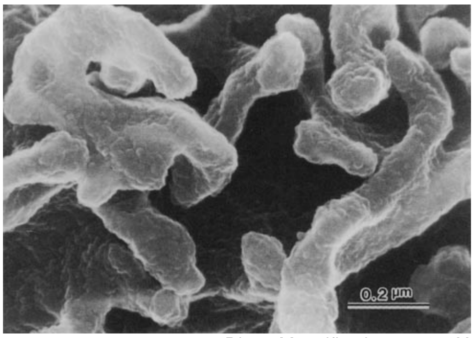
Filgen's OPC



Coating Device: OPC
Coating Material: Osmium
Coating Thickness: 5 nm
Accelerating Voltage: 15.0 kV

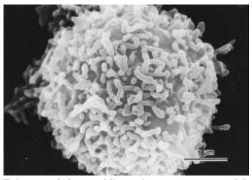
Direct Magnification: 10,000X

Highly smooth coating surface



Direct Magnification: 50,000X

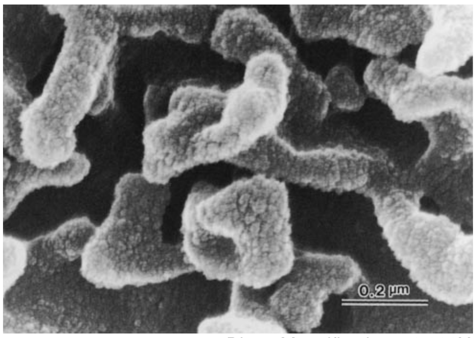
Ion beam sputter coater



Coating Device: Ion Beam Sputter
Coating Material: Pt-Pd
Coating Thickness: 8 nm
Accelerating Voltage: 15.0 kV

Direct Magnification: 10,000X

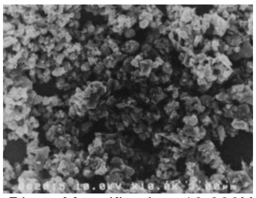
Grains of Pt-Pd are observed at higher magnification



Direct Magnification: 50,000X

Sample: Barium Titanate

Filgen's OPC

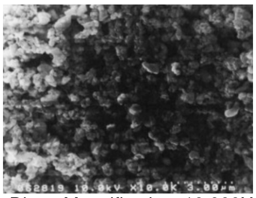


Direct Magnification: 10,000X

Direct Magnification: 100,000X

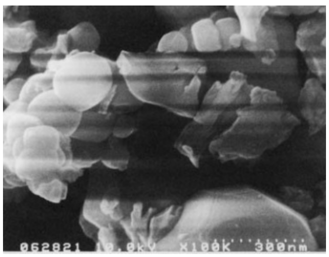
Coating Device: Osmium Plasma Coater
Coating Material: Osmium
Coating Thickness: 3 nm
Accelerating Voltage: 10.0 kV

Magnetron sputter coater



Direct Magnification: 10,000X

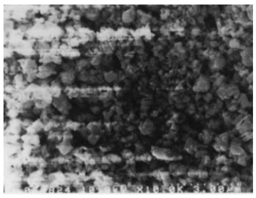
Horizontal bright and dark bands are observed due to charging



Direct Magnification: 100,000X

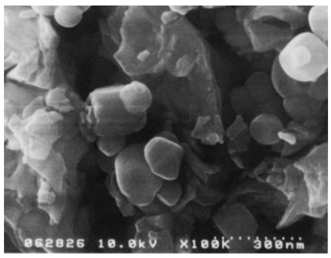
Coating Device: Magnetron Sputter
Coating Material: Pt-Pd
Coating Thickness: 3 nm
Accelerating Voltage: 10.0 kV

Ion beam sputter coater



Direct Magnification: 10,000X

Horizontal bright and dark bands are observed due to charging



Direct Magnification: 100,000X

Coating Device: Ion Beam Sputter
Coating Material: W
Coating Thickness: 1.5 nm
Accelerating Voltage: 10.0 kV

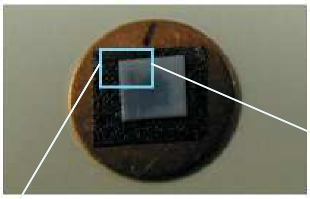
Hybrid coating with osmium film and plasma-polymerized film

By coating with plasma-polymerized film of naphthalene before osmium coating, a hybrid film which enables to protect samples, reduce peeling of resin block, as well as prevent surface charging is obtained. This hybrid film is suitable to prepare SEM samples of non-conductive hydrophobic material like fluororesin.

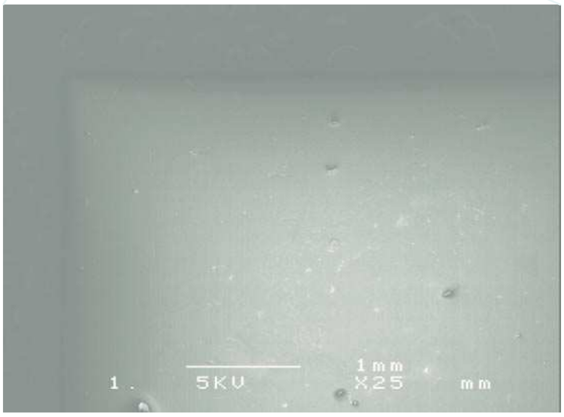
Comparison data: Osmium single coat vs. hybrid coat

Sample: fluororesin film, 0.3 mm thickness

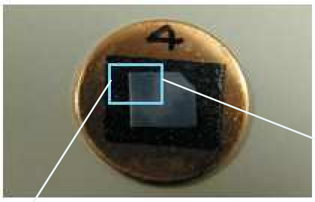
Osmium single coat



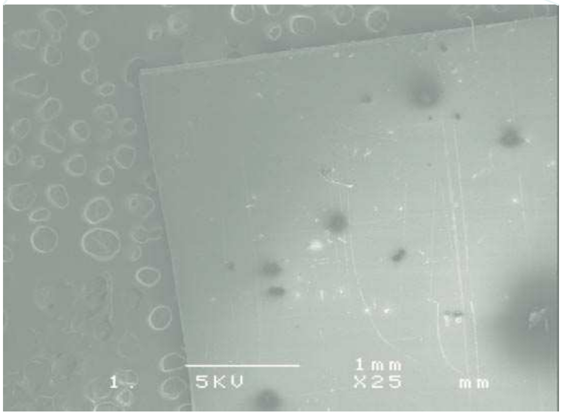
Osmium coat: 5 nm



Hybrid coat



PN coat: 2 nm
+
Osmium coat: 3 nm

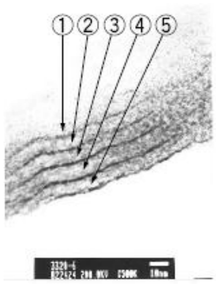


Evaluation for repeatability

Sample: epoxy block

Experimental protocol

1. Coat osmium and naphthalene alternately on an epoxy block,
   Naphthalene film: 10 nm, 6 layers
   Osmium film: 1.0 nm, 5 layers
2. Re-embed the sample by epoxy resin,
3. Prepare cross sections with ultra microtome, then observe with TEM.



Thickness
① 1.3nm
② 1.0nm
③ 1.1nm
④ 1.1nm
⑤ 1.1nm

For more details, please check our website: https://filgen.jp/Product/SI/English/OPC/technical.html

Filgen, Inc.
Scientific Instruments Dept.

1-1409 Jonoyama, Midori-ku, Nagoya 459-8011
JAPAN
TEL: +81 52-624-4388
Email: si-support@filgen.jp
https://filgen.jp/

(OCT 2019)