Science Ring

This logo symbolizes Scientific and Analytical instruments of Hitachi High-Tech Group. It is composed with an “S”, standing for “Science”, our technology core competency, and with a ring that represents close connection we make with our customers. This “Science Ring” shows how we are committed to create new values by strengthening ties between Science and Society.

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NOTICE: For correct operation, follow the instruction manual when using the instrument.
Specifications in this catalog are subject to change with or without notice, as Hitachi High-Technologies Corporation continues to develop the latest technologies and products for our customers.

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customercenter.ev@hitachi-hightech.com

Ion Milling System

IM4000 Series

IM4000
IM4000 PLUS
Flat Milling

SEM observation of metallographic microstructures or defects of various materials requires special sample preparation. Traditional mechanical sample preparation via grinding and polishing can result in deformation, flaws, and artifacts that obscure the true structure of the material. Hitachi offers an ion milling system that can eliminate mechanical stress induced in the sample. The IM4000 can quickly and effectively provide a damage-less flat milling method to enhance mechanically prepared materials.

Hybrid Model: Dual Milling Configuration Available

This ion milling system is equipped for both cross-section milling and flat milling. By switching the milling holder, it can be utilized for applications according to a wide range of purposes.

Processing Principle

Cross-section Milling

High quality preparation of structures below the sample surface for SEM observation is commonly reserved for focused ion beam systems. Other alternatives for preparing cross-sections rely on mechanical or cleaving methods, which often distort internal structures or induce damage. The Hitachi Ion Milling System utilizes a broad, low-energy Ar+ ion beam to produce wider, undistorted, cross-sections without applying mechanical stress to the sample.

Features and Applications

- High quality damage-less cross-sections for the analysis of structures below the surface
- Sample examples: Electronic components such as IC chips, PCB, LED (analysis of layers, interconnects, cracks, voids), metals (EBSD grain structure, EDS elemental analysis, coatings), polymers, papers, ceramics and glasses, powders, etc.
- Removable sample stage unit enables bench top optical alignment of the sample and for site specific ion milling (see explanation)
- Samples with maximum dimensions of 20 mm wide x 12 mm long x 7 mm thick can be milled
- Sample stub compatibility eliminates the need to change mounts between mechanical polishing, ion milling, and SEM observation (Hitachi models)

Flat Milling

SEM observation of metallographic microstructures or defects of various materials requires special sample preparation. Traditional mechanical sample preparation via grinding and polishing can result in deformation, flaws, and artifacts that obscure the true structure of the material. Hitachi offers an ion milling system that can eliminate mechanical stress induced in the sample. The IM4000 can quickly and effectively provide a damage-less flat milling method to enhance mechanically prepared materials.

Features and Applications

- Approximately 5 mm in diameter can be ion-milled uniformly
- Eliminate flaws and artifacts generated from traditional mechanical grinding and polishing techniques
- Diverse range of materials can be processed by flat ion milling
- Observation of crystal grain boundaries and multi-layer films: Relief ion milling by sputtering perpendicular to the sample surface can enhance topography of composite materials or crystal orientations for observation.
- Interface observation, X-ray analysis, EBSD analysis: Flat ion milling at an oblique angle minimizes the dependence between sputtering rate and crystal orientation, yielding reduced surface topography and a flatter sample surface.
- Allowable sample size up to 50 mm diameter x 25 mm height
- Multi-function stage: Multiple rotation speeds and stage oscillation modes provide even greater control to reduce artifacts and sputter flatter surfaces in difficult materials.
**Ion Milling System**

**IM4000 Series**

**Hybrid Model: Dual Milling Configuration Available**

This ion milling system is equipped for both cross-section milling and flat milling. By switching the milling holder, it can be utilized for applications according to a wide range of purposes.

<table>
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<tr>
<th>Cross-section Milling</th>
<th>Flat Milling</th>
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<tr>
<td>High quality preparation of structures below the sample surface for SEM observation is commonly reserved for focused ion beam systems. Other alternatives for preparing cross-sections rely on mechanical or cleaving methods, which often distort internal structures or induce damage. The Hitachi Ion Milling System utilizes a broad, low-energy Ar⁺ ion beam to produce wider, undistorted, cross-sections without applying mechanical stress to the sample.</td>
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**Processing Principle**

**Cross-section Milling**

- High quality, damage-less cross-sections for the analysis of structures below the surface
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- Allowable sample size up to 50 mm diameter x 25 mm height
- Multi-function stage: Multiple rotation speeds and stage oscillation modes provide even greater control to reduce artifacts and sputter flatter surfaces in difficult materials.

**Features and Applications**

- Specimen: Thermal Paper
- Cross-section by razor
  - 5 μm
- Cross-section by ion milling
  - 5 μm

**Specimen: Steel**

- After Mechanical Polishing
- After Flat Milling
  - 50 μm

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**Schematic diagram for processing during Cross-section milling**

- Ion gun
- Ion beam
- Specimen mask
- Specimen stub
- Specimen

**Schematic diagram for processing during Flat milling**

- Ion gun
- Rotation axis
- Flat Milling range
- Ion beam center
- Beam irradiation angle

- EBSD: Electron Back-Scattered Diffraction
**Function**

### Higher Milling Rate IM4000PLUS Available

Milling rate enhanced by higher ion beam current is now available in IM4000 series.

(Milling rate: 500 μm/hr, 50% greater than that of IM4000 @ Acc. Voltage 6 kV, Si sample)

### Ion Beam Intermittent Irradiation to Reduce Thermal Damage

Ion beam irradiation can be automatically switched on & off in order to minimize unnecessary specimen heating.

### Specimen Linkage with a Hitachi SEM

- A specimen can be transferred from the IM4000/IM4000PLUS to a Hitachi SEM without removing it from the specimen stub.
- Either the Flat Milling Holder or the Cross-section Holder can be fully mounted on a Hitachi SEM which has a draw-out specimen chamber.
- Additional milling can be done after SEM observation.
- The mask for Cross-section Milling can be fine tuned with a micrometer.

### Cooling Temperature Control Function

Ion milling with indirect LN2 cooling near the processing area of the specimen. This function is effective for temperature sensitive or beam-distorted materials. There is a temperature controller to prevent a specimen from cracking due to excessive cooling.

*Note: Not standard function of IM4000/IM4000PLUS, and available as optional accessory*
Ion Milling System

**Function**

**Higher Milling Rate IM4000PLUS Available**

Milling rate enhanced by higher ion beam current is now available in IM4000 series.

(Milling rate: 500 μm/hr, 50% greater than that of IM4000 @ Acc. Voltage 6 kV, Si sample)

- Overhang from the mask 100 μm, Accelerating voltage 6 kV

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**Ion Beam Intermittent Irradiation to Reduce Thermal Damage**

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**Specimen: Si Wafer**

IM4000PLUS fabrication result

**Specimen: Lead-contained Solder**

Continuous ion beam irradiation

Intermittent ion beam irradiation

**Specimen: Silicone Rubber**

Without cooling

With cooling

SEM: SU5000

Ex. SU3500

Hitachi FE-SEM

(ex. SU8200 Series)

Fabrication

Observation

Specimen stub

Flat milling holder, Cross-section Holder

Hitachi SEM with Draw-out Chamber

(ex. SU3500)

*Screen shows simulated image

Fabrication

Observation

IM4000

IM4000PLUS

IM4000PLUS with Cooling Temperature Control Function
**Air Protection Holder**

Air Protection Holder is used to keep a specimen isolated from the atmospheric environment. A specimen enclosed in the sealed cap can be transferred to another instrument where it can be released in an evacuated chamber. Thus, a specimen fabricated using the IM4000/IM4000PLUS can be loaded into a SEM, FIB, and/or SPM without exposing it to the atmospheric environment.

1. To be applied for Hitachi FE-SEM or FIB equipped with the specimen exchange chamber for Air Protection Holder.
2. Hitachi SPM equipped with the vacuum specimen chamber.

**Specimen:**
Li-ion battery negative electrode (after charged)

---

**Cross-section Milling Holder Fine Pitch Tuning**

Improved Fine Pitch Tuning Function is added on Cross-section Milling Holder for precise mask positioning. The mask will be positioned very precisely to the region of interest by using the Fine Pitch micrometer. The following shows the result generated when the mask is placed to the aimed position (the center of 20 μm pad) for TSV milling.

**Specimen:**
TSV (Through Si Via)

---

**Higher Beam Tolerance Mask**

It has been developed to correspond with the higher milling rate ion gun; it is twice as hard as the standard mask, thus enabling longer milling times for hard materials.

**Specimen:**
Cemented carbide drill, Milling time: 4 hours

---

**In-situ Optical Zoom Microscope**

Optical Zoom Microscope enables observation of the specimen during milling with magnifications of 15 to 100X. A trinocular type enables monitoring through CCD Camera (Optional).  

*Optional**
Air Protection Holder is used to keep a specimen isolated from the atmospheric environment. A specimen enclosed in the sealed cap can be transferred to another instrument where it can be released in an evacuated chamber. Thus, a specimen fabricated using the IM4000/IM4000PLUS can be loaded into a SEM, FIB, and/or SPM without exposing it to the atmospheric environment.

1 To be applied for Hitachi FE-SEM or FIB equipped with the specimen exchange chamber for Air Protection Holder.
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Li-ion battery negative electrode (after charged)

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In-situ Optical Zoom Microscope
Optical Zoom Microscope enables observation of the specimen during milling with magnifications of 15 to 100X. A trinocular type enables monitoring through CCD Camera (Optional).

* CCD Camera and the monitor will be prepared locally.
Application

Cross-section Milling

Specimen: Lead-free Solder

Specimen: Neodymium Magnet

Specimen: Lanthanum-doped Ceria

Specimen: Nano Pillar

Specimen: Thermal Paper

Specimen: Painted Film

Flat Milling

Flat milling on the surface of carbon fiber unveiled the buckling structure expected from spinning.

Specimen: PAN (Polyacrylonitrile) Carbon Fiber

Before ion milling

After ion milling

Metal microstructures are typically distorted when only mechanical grinding is performed; after ion milling it can be observed.

Specimen: Chrome-molybdenum Steel

Before ion milling

(FIB fabricated surface)

After ion milling

SEM: S-3400N

The dopant layer which can not be observed if only FIB fabricated, will be revealed after flat milling at the accelerating voltage of 0.5 kW.

Specimen: SRAM

Before ion milling

(FIB fabricated surface)

FIB fabricated & ion milling

SEM: SU8200 Series
**Application**

**Cross-section Milling**

Specimen: Lead-free Solder

Specimen: Neodymium Magnet

Specimen: Lanthanum-doped Ceria

Specimen: Nano Pillar

Specimen: Thermal Paper

Specimen: Painted Film

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Flat milling on the surface of carbon fiber unveiled the buckling structure expected from spinning.

Specimen: PAN (Polyacrylonitrile) Carbon Fiber

Before ion milling

After ion milling

Metal microstructures are typically distorted when only mechanical grinding is performed; after ion milling it can be observed.

Specimen: Chrome-molybdenum Steel

Before ion milling (Mechanical polishing surface)

After ion milling

The dopant layer which can not be observed if only FIB fabricated, will be revealed after flat milling at the accelerating voltage of 0.5 kV.

Specimen: SRAM

Before ion milling (FIB fabricated surface)

FIB fabricated & ion milling

**Specimen Courtesy:**
- Prof. Masahiko Yoshino, Tokyo Institute of Technology
- Prof. Katsunori Hanamura, Tokyo Institute of Technology
Application

Application for EBSD & SPM

EBSD

Structural observation by BSE Imaging and crystal orientation information by EBSD are combined for the analysis.

Specimen: Meteoric Iron, (Cross-section milling)

SPM

Flat milling a poorly mechanically polished sample can enable significantly clearer magnetic domain observation.

Specimen: Hot Worked Neodymium Magnet, (Flat milling)

The abnormal contrast indicated by the SEM images can be identified as a low resistance area by SSRM Image. 

Specimen: Lithium ion battery negative electrode, (Cross-section milling)

Major specification

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Gas used</td>
<td>Ar (argon) gas</td>
</tr>
<tr>
<td>Accelerating voltage</td>
<td>0 to 8 kV</td>
</tr>
<tr>
<td>Maximum milling rate (Material: Si)</td>
<td>200 μm/hr</td>
</tr>
<tr>
<td>Maximum sample size</td>
<td>20 (W) x 12 (D) x 7 (H) mm</td>
</tr>
<tr>
<td>Sample moving range</td>
<td>X±7 mm, Y 0 to 15 mm</td>
</tr>
<tr>
<td>Ion beam intermittent irradiation</td>
<td>–</td>
</tr>
<tr>
<td>Rotation speed</td>
<td>1 r/min, 25 r/min</td>
</tr>
<tr>
<td>Swing angle</td>
<td>±15°, ±30°, ±40°</td>
</tr>
<tr>
<td>Gas flow rate control system</td>
<td>Mass flow controller</td>
</tr>
<tr>
<td>Evacuation system</td>
<td>Turbo-molecular pump (33 L/s) + Rotary Pump (135 L/min at 50 Hz, 162 L/min at 60 Hz)</td>
</tr>
<tr>
<td>Weight</td>
<td>Main unit 48 kg + Rotary pump 28 kg</td>
</tr>
<tr>
<td>Cooling temperature control function</td>
<td>Indirectly heating by LEC. Range of set temperature : 0 to –100°C</td>
</tr>
</tbody>
</table>

Options

- Air protection specimen holder
- Cross-section milling holder (FP)
- Binocular type, Tri-eye (for CCD)

Installation Requirements

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Room Temperature</td>
<td>15 to 30°C</td>
</tr>
<tr>
<td>Humidity</td>
<td>45 to 85% without moisture condensation</td>
</tr>
<tr>
<td>Power supply</td>
<td>AC100 V (±10%), 50/60 Hz, 1250 W</td>
</tr>
<tr>
<td>Grounding</td>
<td>100Ω or less</td>
</tr>
</tbody>
</table>

Products prepared by customer

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ar gas</td>
<td>99.99% purity</td>
</tr>
<tr>
<td>Ar gas pressure</td>
<td>0.03 to 0.05 MPa</td>
</tr>
<tr>
<td>Ar gas tubing</td>
<td>1/8 inch SUS piping (1/8 Swagelock-compatible)</td>
</tr>
<tr>
<td>Oxygen content meter</td>
<td>19% oxygen concentration</td>
</tr>
</tbody>
</table>

System layout (unit: mm)

- IM4000 / IM4000PLUS with cooling temperature control unit
- IM4000 IM4000PLUS IM4000 IM4000PLUS

- Cross-section Milling Flat Milling

- Ar (argon) gas

- Turbo-molecular pump (33 L/s) + Rotary Pump (135 L/min at 50 Hz, 162 L/min at 60 Hz)
- Main unit 48 kg + Rotary pump 28 kg

- Indirectly heating by LEC. Range of set temperature : 0 to –100°C
Application for EBSD & SPM

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Structural observation by BSE imaging and crystal orientation information by EBSD are combined for the analysis.

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Major specification

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<tr>
<th>Item</th>
<th>IM4000</th>
<th>IM4000PLUS</th>
<th>IM4000</th>
<th>IM4000PLUS</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cross-section milling</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Accelerating voltage</td>
<td>0 to 8 kV</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maximum milling rate (Material: Si)</td>
<td>≥ 300 µm/hr</td>
<td>≥ 500 µm/hr</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Maximum sample size</td>
<td>20 (W) x 12 (D) x 7 (H) mm</td>
<td>≥50 x 25 (H) mm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Sample moving range</td>
<td>X 7 mm, Y 0 to 15 mm</td>
<td>X 0 to 15 mm</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Ion beam intermittent irradiation</td>
<td>Standard function</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Rotation speed</td>
<td>1 r/min, 25 r/min</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Swing angle</td>
<td>±15°, ±30°, ±40°±4</td>
<td>±15°, ±30°</td>
<td>±30°, ±60°</td>
<td></td>
</tr>
<tr>
<td>Mass flow controller</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Evacuation system</td>
<td>Turbo-molecular pump (31 L/min + Rotary Pump (135 L/min at 50 Hz, 142 L/min at 60 Hz)</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Dimension</td>
<td>136 (W) x 705 (D) x 312 (H) mm</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Weight</td>
<td>Main unit 48 kg + Rotary Pump 28 kg</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cooling temperature control function</td>
<td>Indirectly cooling by LN2. Range of set temperature: 0 to −100°C</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Options

<table>
<thead>
<tr>
<th>Item</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cross-section milling holder</td>
<td>Only cross-section milling</td>
</tr>
<tr>
<td>Cross-section milling holder (FP)</td>
<td>100 µm/rotate±6</td>
</tr>
<tr>
<td>Binocular type</td>
<td>Tri-eye (for CCD)</td>
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<tr>
<td>Ar gas tubing</td>
<td>1/8 inch SUS piping (1/8 Swagelock-compatible), Pressure regulation</td>
</tr>
<tr>
<td>Oxygen content meter</td>
<td>15% oxygen concentration</td>
</tr>
<tr>
<td>Recommended table</td>
<td>1000 (W) x 800 (D) x 700 (H) mm or more, Min. weight tolerance: 70 kg Minimum strength when installing only</td>
</tr>
</tbody>
</table>

System layout (unit: mm)

- IM4000 / IM4000PLUS with cooling temperature control unit
- Zoom stereo microscope unit
- Binocular type, Tri-eye (for CCD)
- Mass flow controller
- Turbo-molecular pump (33 L/S + Rotary Pump (135 L/min at 50 Hz, 142 L/min at 60 Hz)
- Main unit 48 kg + Rotary Pump 28 kg
- Indirectly cooling by LN2, Range of set temperature: 0 to −100°C

- Products prepared by customer
- Ar gas tubing connects Ar gas supply to the equipment. Pressure gauge regulator is required.
- Adequate ventilation and air quality measurements are required.
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