Precision Ion Polishing System (PIPS™)

Liquid Nitrogen (LN_2) Cold Stage

PIPS Sample Cooling Option

New technology and developments in new materials require changes in ion milling techniques to enhance sample quality and high-resolution TEM results. Gatan's Precision Ion Polishing System (PIPS™) continues to be an industry standard for TEM sample preparation. To enhance the performance and capabilities of the PIPS, a liquid nitrogen (LN_2) cooling option is now available.

Features and Benefits

- Dewar and conductor rod share PIPS vacuum
- Easy to fill Dewar with 3 - 4 hr capacity
- Sample temperature specification [e.g., minimum -120°C (+/- 25°C)]
- Electronic temperature regulation (-180°C to +100°C)
- Controller display monitors conductor temperature
- Fast cool down time (approximately 10 minutes)
- Fast warm up time before venting (approximately 10 minutes)
- Uses same standard PIPS DuoPost sample holders
- Through transmission illumination
- Built-in Dewar heater enables Dewar boil-off

Upgrade or Option

This new cold stage replaces the standard Whisperlok™ mechanism. It may be ordered with a new PIPS or as an upgrade for a PIPS in the field. The upgrade is designed to be installed by a user on-site.

NOTE: The upgrade option is not available for PIPS with vertical front panels manufactured before June 1992.

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PIPS Cold Stage

Specifications

Cooling Source

Liquid Nitrogen (LN_2)
Dewar Capacity: 250ml
Dewar lasting time: 3hrs – 4hrs
Dewar and conductor rod share chamber vacuum
Dewar heater for LN_2 boil-off

Electronic Controller

Controls

Rotary dial adjusts conductor temperature
Digital display monitors conductor temperature
Heater 1 regulates conductor temperature (-180°C to +100°C)
Heater 2 enables fast boil-off for Dewar to run the stage at room temperature if desired.
Dewar boil-off time about 45 minutes
Temperature sensor – Silicon diode

Power

Universal Voltage / Frequency 100VAC – 240VAC / 50-60Hz / 70W

Controller Dimensions

100mmH x 140mmW x 225mmD (4"H x 5.5"W x 9"D)
Shipping Weight – 23 lbs (10 kg)

Warranty

One year

Ordering Information

US customers order upgrade directly through Gatan Corporate Headquarters in Pleasanton, CA, USA. Telephone: 1.925.224.7314. International Customers must order upgrade through their local distributor.

Model No.	Description
691.CS	New PIPS ion mill with cold stage Whisperlok™ and electronic temperature controller
691.CS.UPG	Customer installed upgrade package to add sample cooling to an existing PIPS

Spares and Consumables

691.08450.FR	Cold Stage window (pkg. of 10)
691.17305	Moly disulfide O-ring lubricant, 1 gm
06985	Quad-seal #111, Cold stage x2

CONTACT

Gatan Inc.
Corporate Office
Western USA Sales
5933 Coronado Lane
Pleasanton, CA 94588
Tel. (925) 463 0200
Fax. (925) 463 0204
Contact: info@gatan.com

Eastern USA Sales
780 Commonwealth Drive
Warrendale, PA 15086
Tel. (724) 776 5260
Fax. (724) 776 3360
Contact: info@gatan.com

Gatan UK
25 Nuffield Way
Abingdon
Oxon, OX14 1RL
United Kingdom
Tel. +44 (0)1235 540160
Fax. +44 (0)1235 540169
Contact: ukinfo@gatan.com

Gatan GmbH
Ingolstädterstr. 12
D-80807 München
Germany
Tel. +49 89 358084-0
Fax. +49 89 358084-77
Contact: mfelsmann@gatan.com

Gatan France
3bis, Chemin du Haut Breuil
78113 GRANDCHAMP
FRANCE
Tel: +33 1 34944407
GSM: +33 6 80135139
Fax: +33 1 34871668
Contact: dmonville@gatan.com

Nippon Gatan
Hibarigaokaminamikan 6F
3-27-11 Yato-cho,
Nishi-Tokyo-shi
Tokyo 188-0001 Japan
Tel: +81 424 387230
Fax: +81 424 387228
Contact: miyamori@gatan.com

Gatan Singapore
10 Eunos Road 8
#12-06 Singapore Post Centre
Singapore 408600
Tel: (65) 6293 3160
Fax: (65) 6293 3307
Contact: wchuang@gatan.com

Gatan On-line
http://www.gatan.com
info@gatan.com

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TEM specimen preparation

Operation

The LN_2 Dewar assembly mounts into the existing vacuum manifold, replacing the PIPS vent-valve assembly or liquid-nitrogen trap. The new assembly helps improve the overall chamber vacuum. When the sample is lowered into the milling position, the sample mount (DuoPost™) makes thermal contact with the cold conductor, and milling can begin in approximately 10 minutes. When the sample is raised into the airlock, thermal contact is detached. The sample warms and can be vented in approximately 10 minutes. The electronic temperature controller displays the cold conductor temperature and drives two built-in warming heaters:

- Heater 1 regulates the cold conductor temperature (i.e. sample holder temperature). Regulation of the cold conductor is desirable for samples with a phase-transition temperature below -100°C, for example, that you want to avoid; the conductor temperature is then set to any temperature between -100°C and +100°C, prior to inserting the sample. Different users may want to mill at room temperature after the stage is cold. This is easily achieved by setting the conductor temperature to 23°C and waiting approximately 20 minutes.
- Heater 2 allows fast boil-off of liquid nitrogen in the Dewar. This is used if subsequent users do not want sample cooling.

The temperature sensor for the cold stage is attached to heater 1 which is mounted to the cold conductor assembly, not to the sample or sample holder. Since the sample is mounted to the top of the rotating piston, it is difficult to monitor sample temperature; therefore, the temperature displayed on the controller is not the same as the sample temperature. Once sample perforation occurs, the thinnest (electron transparent) area is unable to effectively dissipate heat; heat transfer is very poor across extremely thin material sections. Therefore regardless of cooling, the localized temperature rise incurred when milling heat sensitive materials may still be relatively high.

NOTE: This essential rule applies to any ion mill with a cold stage.

Performance

Semiconductors

Typically, semiconductor compounds containing Indium develop Indium islands on the surface when ion milled with argon. The belief is that preferential sputtering enriches the surface with Indium and that heat generated by the ion beam melts the Indium which then agglomerates forming small globules on the surface. Studies have shown the agglomerates of Indium can be prevented by ion milling the sample with the use of a cold stage. Numerous users have also had success in ion milling Indium using other techniques but there are risks and/or higher costs associated with them:

- CAIBE system with iodine: This technique avoids surface enrichment of Indium at the start and chemically assists the ion beam with an iodine vapor stream at the point of contact of the ion beam.
- Substitution of argon with xenon (Xe): Indium island formation during ion milling can also be eliminated using this technique.

Applications

Examples of heat sensitive materials ion milled in PIPS with a cold stage upgrade courtesy of: Prof. Dave Smith and Mr. Changzhen Wang, Arizona State University (ASU); Mr. Weifeng Ye and Dr. Heiping Sun in Prof. Rachel Goldman's group in the Department of Materials Science and Engineering, Univ. of Michigan (EMAL).

Application 1: Arizona State University

Arizona State University (ASU) uses the Gatan Model 600 DuoMill for ion milling of all heat sensitive materials. The ASU users performed a comparative analysis between their DuoMill and their PIPS system with the new cold stage installed. "We can now run all our samples in the PIPS to our advantage. The cool down/warm up time is several times faster in the PIPS as is the milling rate."

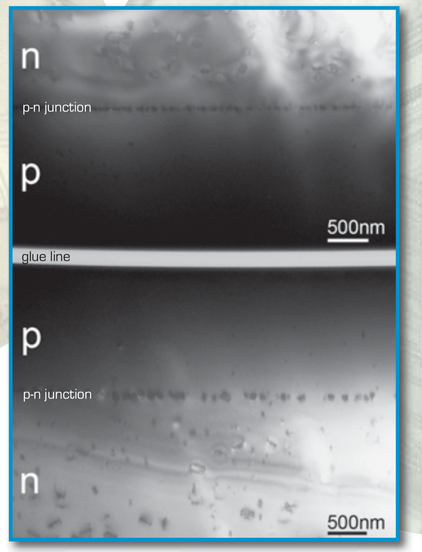

Figure 1: Back-to-back cross section of two epitaxial HgCdTe p-n Heterojunctions prepared together in an older Duo Mill milled with a cold stage but with insufficient time for cooling before commencement of ion-milling (different In concentrations in n-type layer). Note defects in the two n-type layers and defect pile-up at p-n junctions.

Figure 2: Same sample as in Figure 1 (top half only) after being re-thinned in the DuoMill for 10 minutes but with sufficient time allowed for cooling (about 90 minutes). No defects visible anywhere.

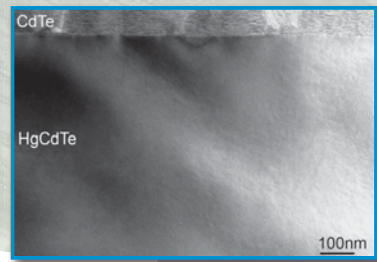

Figure 3: Low magnification electron micrograph showing cross-section of HgCdTe/CdTe heterostructure prepared in the ASU PIPS (circa 1998) with the new Cold Stage upgrade. The HgCdTe layer is completely free of any ion-milling defects.

Application 2: University of Michigan

Sample Preparation:
- Sample polished flat and parallel to about 40um; no dimpling was performed.
- Sample mounted to a Clamp Type DuoPost.
- Dewar was charged, sample lowered and milling started in approximately 10 minutes.
- Top and bottom guns maintained at same angles.
- Beam modulation was on at double sector.

PIPS Milling Parameters

Angle	Energy (keV)	Time (min)
8°	3.5	23
6°	3.5	15
4°	3.2	5

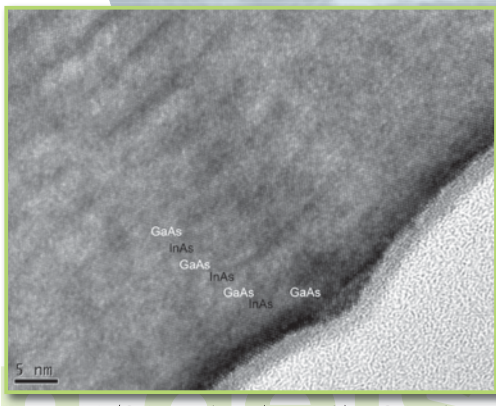

Figure 4: InAs/GaAs superlattice (10 periods) on GaAs substrate. Preparation parameters are indicated in chart.

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