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Model 600 DuoMill



A close-up of the Model 600-CTMP DuoMill ® fitted with RIBE Octoguns ® and manual gas flow control valves.

History

Gatan shipped the first Model 600 DuoMill ® in January 1980. Since then, we have made many improvements to the original design. Our most recent developments are three systems for chemically assisted milling of semiconductors and other materials: a reactive ion beam etching (RIBE) Octogun ®, a chemically assisted ion beam etching (CAIBE) system and a dry etching (DE) system. Other developments include the Sector Speed Control Whisperlok &reg: the Sputter Shield, Electronic Gas Flow Control, Dual Laser Autoterminator and a digital Process Timer. There have been many other less obvious but equally significant advances, such as improved HT power supplies and more powerful Whisperlok ® motors. Gatan remains committed to producing a state-of-the-art instrument and for this reason the design of our Model 600 DuoMill ® continues to evolve.

The Basic System

The original concept for the DuoMill ® was an ion milling system for the preparation of high quality TEM specimens which minimized the time for specimen exchange and maintenance. This has been accomplished with two independent milling stations mounted on a single high-speed vacuum system (diffusion or turbomolecular pumping available). Each milling station has a Whisperlok ® specimen exchange system which functions without disturbing the vacuum or milling conditions of the other station and two Octogun ® ion guns (four altogether), each with eight selectable cathode apertures.

The Whisperlok Specimen Rotation and Exchange System

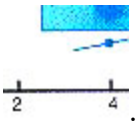
(US Patent 4,272.682) The Whisperlok ® quickly and gently moves the specimen between the normal milling position and an airlock for exchange. This is achieved without affecting the vacuum in the main work chamber or disturbing milling in the second station, so increasing specimen throughput and avoiding lost time in pumping the whole system down to a good vacuum before milling can commence again. A further advantage is the reduced stressing of the vacuum system since complete pump downs are only performed at initial startup after maintenance. The vertical motion of the specimen platform, pneumatically driven by the same gas source supplying the Octoguns ®, is actuated by a single switch. Once in the upper position, a button can be pressed to vent the airlock to atmosphere for specimen exchange or, after examination with the viewing microscope, the specimen can be returned to the lower position for further milling without venting the airlock. After specimen exchange, a second button is pressed to connect the airlock to the foreline for evacuation. In the lower working position, an electric motor rotates the specimen platform to ensure even milling.

The Octogun Ion Source

The Octogun is a cylindrical cold cathode/anode plasma gun producing a single ion beam perpendicular to the cylinder axis (below 6kV the beam is almost exclusively neutral atoms, dropping to ~85% neutrals at 10kV). The cathode has eight apertures with ignition occurring between the anode and the selected cathode aperture. The multiple aperture design greatly extends the interval between cathode exchange. General gun maintenance is particularly easy, each Octogun ® being comprised of only six parts. A fresh aperture is brought into alignment with the anode simply by rotating a knob at the end of the gun, outside the vacuum chamber. Each pair of Octoguns ® may be rotated +/- 40 degrees on a precision ball race (a special tool is supplied for this purpose), varying the useful sputtering angle from 9 degrees to 40 degrees. The guns are self aligning and the beam axes are typically within +/- 100 microns of the specimen range of available sputtering angles. High voltage is provided by two independent, continuously variable 0-10 kV power supplies each delivering up to a 2 mA beam

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current. Either voltage regulated or current limited modes may be selected. Normal procedure is to set a current limit and adjust the gas flow to set the voltage. Good thermal contact between the Octoguns ® and the specimen chamber allows the guns to function at high current without water cooling. The Octoguns ® normally operate with argon supplied at 8 psi (0.7 kg/cm^2). Gas flow is controlled by four sensitive metering valves. Under these conditions, the vacuum in the specimen chamber is typically ~8x10^-5 Torr (6.5x10^-3



Typical milling rate per workstation. The proportion of neutral atoms in the Octogun ® beam was determined by splitting the beam into its components with an electrostatic deflector and milling a gold film, deposited on a glass slide, at different voltages. Milling was stopped when the neutrals had completely removed the gold film. The proportion of the ions in the beam can be estimated from the amount of gold removed. At 6 kV ions are just detectable in the beam while at lower gun voltages ions are completely absent. The Low Energy Gun Kit (option) provides increased thinning rates below 4 kV.



As each cathode aperture wears out, a fresh aperture is simply moved into position by rotating an external knob. This is achieved without breaking the vacuum.

Electronic Gas Flow Control

When milling for long periods, erosion of the cathode apertures (particularly with a freshly selected aperture) causes a significant change in the gun current (voltage limited mode) or the gun voltage (current limited mode). To maintain constant milling conditions, the gas flow rate must be periodically adjusted. The Electronic Gas Flow Control Module replaces the standard manual gas flow regulators. A feedback circuit continuously monitors the Octogun ® current and automatically adjusts the gas flow via a piezoelectric valve, thereby keeping both voltage and current constant.

High-Speed Milling

The Octogun ® milling rates are high. Even so, it can still take many hours to thin a specimen. This time is reduced considerably by careful pre-preparation of the specimen with the Gatan Model 646 Dimple Grinder. This instrument can rapidly grind and polish a dimple in the specimen, producing a central region 5 microns thick. Final thinning to electron transparency is undertaken in the DuoMill ® in 0.5-2 hours (depending on material).

Laser Autoterminator

Two digital process timers on the DuoMill ® can terminate milling at either specimen station after a set period. The optional Laser Autoterminator comprises one or two laser sources, photodetectors and a control unit which automatically terminates milling when the photodetector senses that a preset intensity level has been exceeded. The system is sufficiently sensitive to operate with translucent and transparent specimens (a special filter is supplied for this purpose). The control unit accepts one or two laser heads and independently controls termination of milling at either specimen station.



The Laser Autoterminator Control Unit operates with one or two Laser Head assemblies (which seat over the airlock capsule) to provide automatic termination of milling.

Sputter Shield

If allowed, material sputtered from the specimen could be deposited on the airlock viewing port and over a period of time this would make it difficult to see the specimen and might affect the function of the Laser Autoterminator. A sputter shield in the airlock assembly minimizes this problem. Operation of the autoterminator is unaffected.



Close-up views of a cut away DuoMill ® illustrates the operation of the Whisperlok ®. The lower, working piston (left) the sputter shield prevents material from the specimen coating the airlock capsule window. Simply pressing the "Raise" switch the specimen holder is gently moved up into the airlock (center). Once in position, the "Air" button is pressed to admit air into the airlock capsule which can be removed (right).

High Efficiency Specimen Cold Stage

The damage introduced into many specimens by ion thinning is appreciably reduced by cooling. A liquid nitrogen cooled version of the Whisperlok ®, providing a highly efficient transfer of heat away from the specimen, is available for this purpose. A specially designed copper specimen holder screws directly on to a conductor contained within the Whisperlok ® piston. A braid at the end of the conductor hangs into a liquid nitrogen dewar. The Cold Stage Whisperlok ® is fully compatible with the Laser Autoterminator and replaces the standard Whisperlok ®, operating like it in all respects. It may also be operated at room temperature if cooling is not required.



GaAs/Si interface prepared with the Model 600 DuoMill ®. Micrograph taken by Robert Hull on the Atomic Resolution Microscope, National EM Center, Berkley. Sample grown by S. M. Kuch, Stanford University.

Liquid Nitrogen Cold Trap

To minimize hydrocarbon contamination on the specimen, particularly when thinning cooled specimens, a Liquid Nitrogen Cold Trap is available. The Cold Trap is particularly effective on the diffusion pumped versions of the DuoMill ® but also improves the results on turbomolecular pumped systems.



SiC whisker reinforced Al2O3 composite prepared with Model 600 DuoMill ®. Material courtesy John Porter, Rockwell International Science Center. Magnification ~x60,000.

Single-Sided Milling

Certain specimens e.g. layered materials, integrated circuits, require thinning from one side only, so as to preserve the layer of interest. This is possible with the standard DuoMill ® by turning on only one of the two available Octoguns ®. However, this results in a halving of the thinning rate. The Single-Sided Milling Attachment consists of a modified gun mount in which the two Octoguns ® are 150 degrees apart instead of being diametrically opposed. When positioned symmetrically about the specimen platform, this allows both Octoguns to mill the specimen from above at a 15 degree sputtering angle thereby attaining the same thinning rate achievable with double sided milling. When milling a specimen from one side only, whether with a single Octogun ® or with two Octoguns ® on the Single-Sided Milling Attachment, shrouds should be installed on the specimen platform to prevent material sputtered from one side of the specimen being deposited on the other side. The use of the specimen cold stage will greatly improve the effectiveness of the shrouds.



InP after N+ implantation. The cross section was prepared with the RIBE Octoguns ®, charged with iodine crystals, and show the implanted surface region (~4 microns thick).



The high resolution InP (110) image, showing multiple twinning, is taken from one of the needles protruding into the amorphous region (material courtesy F. Xiong, Caltech).

RIBE Octogun ®

Reactive **I**on **B**eam **E**tching It is standard practice in the integrated circuit industry to " etch" semiconductors with relative gas plasmas, derived principally from halogens e.g. chlorine and iodine. A similar technique is very effective in preparing TEM specimens from the same materials. The RIBE Octogun ® uses a halogen source, preferably in a crystalline form, contained within an insulted capsule within the gun body. With the source insulated from ground potential, damaging and hazardous electrical discharges between the source and the anode do not occur. The close contact between the source and the anode allows operation at room temperature without any heater. Since the whole assembly runs slightly warm, there is no condensation within the RIBE Octogun ® which could lead to blockages. Gas flow is precisely controlled by a manual valve and the high stability and high thinning rate achieved make electronic gas flow control unnecessary. The gun is rapidly switched to argon by closing the halogen supply valve and opening the argon valve. The corrosive properties of halogens mean that the RIBE Octoguns ® should only be operated on DuoMills ® with chemical pumping systems.



The simple, effective design of the RIBE Octogun ® makes cleaning and maintenance easy.

High-Current, Low-Energy Octogun ®

Some materials, especially certain ceramics and semiconductors, are particularly susceptible to damage during ion milling. While this damage is reduced by cooling the specimen, it is often high voltage dependent and may be minimized or eliminated by reducing the kV of the incident beam. Unfortunately, for a given gun design, the current attainable depends on kV and a reduction in kV inevitably reduces the maximum possible gun current. For operation at voltages below 4 kV, the Low Energy Gun Kit provides an easy, user-installable modification of the standard Octoguns ® to maximize the current attainable at lower voltages (a special version of the kit is available for the RIBE Octoguns ®). Please note that Octoguns &reg: so modified should not be operated above 4 kV or potentially damaging arcing may occur.



Cross section, Si/CoSi2/(111) Si, grown by MBE. Two face-to-face pieces of the same wafer are shown. Prepared with the Gatan Model 600 DuoMill ®. Micrograph courtesy of Ernest L. Hall and Nathan Lewis, GE Corporate Research and Development.

Cross-Sectional Specimen Preparation

Cross-sectional TEM specimens are important for determining the properties of e.g. layered materials. However, in many cases the components mill at different rates and one component may perforate while the other is still electron-opaque (e.g. heavy metal oxides and silicon). The most successful technique for overcoming this problem is to rotate the the specimen slowly when the ion beam is perpendicular to the layers (or glue-line) in the specimen and rapidly through the remaining sectors. The Sector Speed Control Whisperlok ® replaces the standard Whisperlok ® assembly. The specimen is moved at 2rpm through the selected sector(s) and at 10rpm through the remainder. The slow sector may be may be selected to cover 40 degrees, 60 degrees, or 80 degrees with a single sector or two diametrically opposed sectors, depending on the nature of the specimen. The specimen may also be rotated continuously at 2rpm or 10rpm. Room temperature and liquid nitrogen cooled versions are available. The double sector is used when milling cross-sectional specimens from both sides, the

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specimen being aligned so that the "glue-line" is perpendicular to the center line of the slow sectors. In specimens with a resistant surface layer on a more easily milled substrate, milling with one Octogun ® from the resistant side with a single slow sector is most effective. Precise alignment of the slow speed sectors with the ion beams is achieved with an optical encoder, preserving the zero point of the Sector Speed Control even in the event of a power failure. To assist in the preparation of TEM cross-sectional specimens, the Model 601-0700 Cross-Sectioning Kit, an accessory for the Gatan Model 601 Ultrasonic Disc Cutter, is recommended.



The Sector Speed Control Whisperlok ® is available in room temperature and LN2 cooled versions for the preparation of cross-sectional specimens.

Ion Beam Coater

For users who occasionally need to coat specimens but do not wish to purchase a dedicated coater unit, an ion beam coater attachment is available. A solid target version employs a range of targets supplied by Gatan while a second version can use as a target any suitable material in powder form.

Specimen Holders

In addition to the standard specimen holder and the high conductivity cold stage version, there is also a special specimen holder which allows the Cold Stage Whisperlok ® to be operated at room temperature.

Viewing Microscope

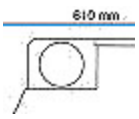
A small viewing microscope and illuminator are available to permit the examination of the specimen without removing the specimen from its holder or airlock.

Special Gas Conversion Kit

The Whisperlok ® specimen exchange system is driven pneumatically by argon, normally from the same source as that which supplies the Octoguns ®. The Octoguns ®, however, will operate with a variety of gases such as xenon, krypton, nitrogen, carbon dioxide or argon/oxygen mixtures (with less than 20% oxygen). In this case, it is probable that users will not want to have a single gas supply for both the Octoguns ® and the Whisperloks ®. The Special Gases Conversion Kit comprises the parts needed to modify the DuoMill ® to run from two independent gas supplies.

Spare Parts Kit

Two spare parts kits are available; the standard Spare Parts Kit, providing sufficient spares and consumables for approximately 6 moths operation, and the Special Spare Parts Kit, with spares and consumables for approximately 2-3 years operation. These kits ore particularly recommended to purchasers outside North America, Europe or Japan who might encounter shipping delays in obtaining spares.



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