Model 1030

Automated Sample Prep (ASaP) System

Self-contained automatic plasma cleaning,
ion beam etching, reactive ion etching,
and ion beam sputter coating

FISCHIONE INSTRUMENTS

EXCELLENCE...MAGNIFIED

Model 1030 Automated Sample Preparation (ASaP) System

The powerful and flexible Model 1030 Automated Sample Prep (ASaP) System prepares samples that have been created by cleaving, grinding, cutting, or sectioning. The final sample configuration is ideal for analysis in a scanning electron microscope (SEM).

Processing with the ASaP significantly enhances the image quality and analytical data obtained from the sample.

Operation is fully programmable and automatic, completing sample preparation rapidly enough for high-throughput applications.

ASaP functions in one chamber
Plasma Cleaning (PC)
Ion Beam Etching (IBE)
Reactive Ion Etching (RIE)
Ion Beam Sputter Coating (IBSC)

Model 1030 Automated Sample Preparation (ASaP) System

Powerful automatic sample preparation

- Four functions in continuous vacuum.
- Rapid sample processing.
- Automatic operation with user-defined process sequence.
- Easy-to-use interface.
- Samples up to 1.0 in. (25mm) diameter and 1.0 in. (25mm) thick.
- Closed loop operation yields consistent results.
- Computer automation of instrument parameters.
- Automatic sample height detection.
- Rapidly pumped sample exchange load lock.
- Oil-free vacuum system.

User friendly interface

The sequence of processing steps can be loaded from memory using a predefined recipe or can be individually tailored for a given sample. Programming a new sequence is simple: click the buttons for the desired operations and build the recipe. The operator may choose any process step in any sequence, depending on the sample requirements. The ASaP provides plasma cleaning, planarization, surface feature enhancement, and surface coating using a variety of functions. Processing parameters can be individually adjusted for each step.

Once the sample is loaded and the sequence begins, the ASaP automatically advances through the complete process while the interface continuously indicates status.

At the conclusion of the process, the sample remains under a dry vacuum until the user acknowledges that the process is finished and removes the sample through the load lock.

Highly interactive user control with a real-time representation of both the process parameters and the operating status.

Automatic operation

The ASaP features a load lock for rapid sample exchange. The operator loads the sample into one of the specially designed holders. The holder is then attached to a transfer rod. Once the load lock door is closed and the load lock is evacuated, an automatic gate valve is opened and the sample holder is manually inserted into the ASaP's sample stage using the transfer rod. The sample holder can be continuously observed through a viewing window during transfer to and from the sample stage. A chamber light facilitates the transfer process.

Sample loading is complete when the laser and photo-detector automatically determine the height of the sample. This is done to ensure eucentric sample motion and proper sample placement throughout the process.

The operator then selects a stored recipe or creates a customized recipe using the interface. The ASaP's sophisticated control automatically adjusts all parameters to ensure reproducible sample processing. The operator clicks the Run button and the ASaP takes control. The sample automatically moves from process step to process step as determined by the program sequence. The stage control positions and manipulates the sample including changing the

continued

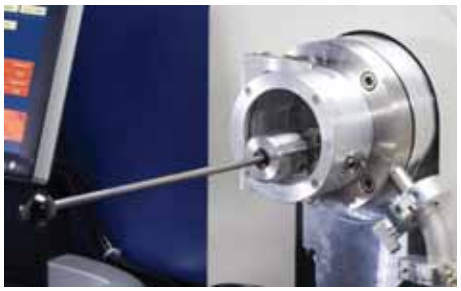
Model 1030 Automated Sample Preparation (ASaP) System

Automatic operation *(continued)*

ion beam's angle of incidence and controlling sample rotation or rocking. Optical encoders are used on all axes of motion for highly reliable performance.

At the conclusion of the process and after the load lock is vented, the sample can be rapidly transferred to the SEM, thus reducing sample contamination from ambient conditions.

Load lock open



Load lock closed

Advanced vacuum and gas flow technology

The ASaP's oil-free vacuum system consists of a turbomolecular drag pump backed by a multi-stage diaphragm pump. All process gas flows are regulated using mass flow control technology. A butterfly valve is used to regulate the pressure in the chamber in order to establish consistent operating parameters and ensure optimal pump life.

Today's microscopy needs advanced sample preparation

Scanning electron microscopy (SEM) is a powerful technique for analyzing a broad variety of materials on the nanometer scale. Field emission scanning electron microscopy (FESEM) yields resolution to better than one nanometer with field emission sources operating at low accelerating voltages.

Concurrent with the improvements in imaging and low voltage operation of the FESEM, feature sizes in the semiconductor and nanotechnology fields are ever decreasing. Also, today's advanced materials systems have more complex microstructures on progressively smaller scales.

Considering these factors, greater importance is placed on the quality of the sample surface.

Sample features of interest

Advanced FESEM samples in the physical sciences include semiconductor devices, nanotechnology structures, metal-matrix composites, thermal barrier coatings, and microelectromechanical systems (MEMS) components. In many cases, microstructural features or particle sizes are on the nanometer or sub-nanometer scale. Often, the features of interest are boundaries between dissimilar materials, for example an advanced semiconductor device containing Cu, Si, SiO_2, Ta, Ti, W, sub-nanometer oxide and nitride layers, and low-K dielectrics, often consisting of complex polymers.

Sample enhancement

Samples are typically created with processes that may damage the sample's microstructure such as cleaving, mechanical grinding, or focused ion beam (FIB) cutting.

Cleaving often creates irregular surface characteristics and delamination of various device layers.

Mechanical polishing can create micron-size scratches and subsurface damage or, in the case of soft sample materials such as copper, can smear and mask grain boundaries and create voids and inclusions.

Focused ion beam cutting has become a powerful tool for preparing electron microscopy samples; however, the highly energetic metal ions can cause irradiation damage.

The ASaP finishes the preparation so the sample will be ready for advanced FESEM analysis.

Plasma cleaning

Plasma cleaning prior to analysis to remove organic contamination has become widely accepted for transmission electron microscopy (TEM). This is especially useful for samples that are subjected to fine probe microanalysis using instruments with high-brightness electron sources.

Plasma cleaning has also become essential for effective imaging and analysis of FESEM samples. As resolution improves and accelerating voltages decrease, greater emphasis is placed on the surface characteristics of the sample. Hydrocarbon contamination often obscures imaging and makes it impossible to obtain meaningful analytical data.

The Model 1030 ASaP creates a capacitively coupled plasma (CCP) from an oxygen/argon gas mixture. Oxygen radicals produced in the plasma chemically react with the carbonaceous material, converting it to CO, CO_2, and H_2O. The plasma is highly effective in removing contamination caused by rastering of the electron beam during previous SEM observations or analysis on samples that have not been plasma cleaned.

Ion energies from the plasma are less than 12 eV. Because of the extremely low ion energies, organic contamination is successfully removed without altering the sample's properties.

Typical ASaP sequence

As-received state

▼

Plasma cleaning

▼

Planarization

▼

Surface feature enhancement

▼

Surface coating

▼

Finished sample

Model 1030 Automated Sample Preparation (ASaP) System

Planarization

Planarization sputters surface defects such as smearing and scratching and flattens the sample surface.

The Model 1030 ASaP incorporates ion beam etching (IBE) technology for planarization. A single hollow-anode discharge (HAD) ion source directs argon ions to the sample at angles ranging from 0° to 90°. The ion source's accelerating voltage and beam current parameters are easily adjusted to allow for either rapid material removal or more gradual surface polishing. A Faraday cup positioned in the ion source shutter monitors beam current, affording consistent ion milling performance.

For bulk materials, such as those used in electron backscattered diffraction (EBSD) studies, the sample can be rotated 360° with respect to the ion beam to obtain the best planarization. When preparing cross-sectional or layered materials, the sample can be rocked in relation to the ion beam to minimize shadowing or trenching of features. Rocking is particularly useful when the sample includes materials that sputter at widely different rates.

Surface feature enhancement

The Model 1030 ASaP offers multiple technologies to enhance the sample's surface characteristics either by exposing grain structures or by providing topographical differentiation between given layers or device microstructures.

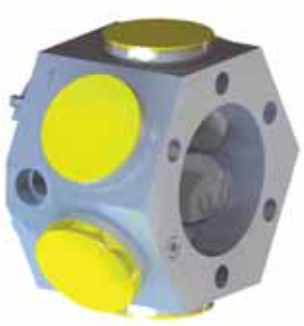
Ion beam etching (IBE) can apply energetic ions normal to the sample surface, removing material from the sample by momentum transfer. Since material removal rates vary with atomic number, selective milling of the sample is possible.

In addition, a parallel-plate reactive ion etch (RIE) system is included for selective treatment of device microstructures. In RIE, a process gas is introduced into the space between two plates electrically biased with RF power. A reactive plasma is formed and, through combined chemical and ion bombardment effects, sample material is rapidly and selectively removed. The ASaP allows up to six process gases to be introduced individually or blended for material-specific etching to "decorate" the sample surface. Each gas flow is regulated using mass flow control technology. The ASaP uses an RF power autotuning-matching network to ensure consistent and repeatable RIE processing conditions. Chamber vacuum is precisely controlled by a motorized butterfly valve positioned between the chamber and the turbo pump.

Specific concentrations of CF_4 and O_2 are extremely effective for processing many types of semiconductor samples, and in particular for the preferential etching of Si versus SiO_2. Ti-based microstructures can also be selectively etched using these gases.

To avoid thermally induced damage during the RIE process, the ASaP contains an integral liquid nitrogen cooling system.

Sample coating



Target Carousel

To eliminate the effects of charging on image quality in the SEM, the Model 1030 ASaP employs high-resolution ion beam assisted sputter coating (IBSC) technology. Six targets are included in a carousel, which is interchangeable through the load lock; therefore, there is no need to break vacuum when replacing a worn target. The ASaP allows the user to select the desired coating target material for a given application.

If required, multiple coatings can be applied to a given sample. The ASaP automatically positions the target with respect to the ion beam. The rate of deposition and the desired coating thickness are selectable by the user.

Typical coating materials are W, Cr, Pt, Ir, Ta, and C, although others can be easily substituted as required.

Sample holders

Sample Holders

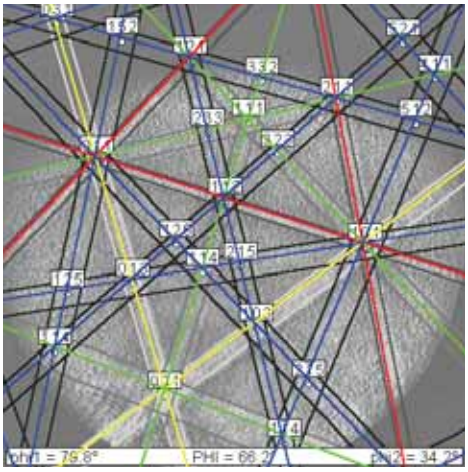
A complete series of sample holders greatly enhance the applicability of the ASaP. They are designed to accept both planar and cross-sectional samples and provide proper orientation for various treatments.

Sample observation

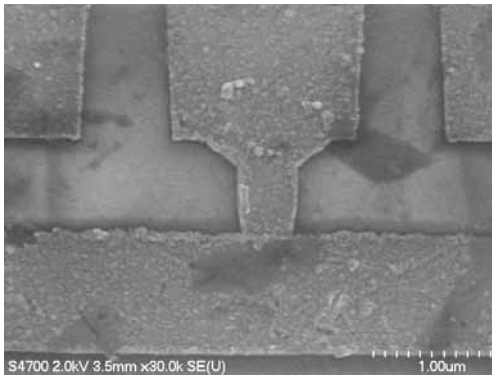
To provide feedback relative to the effectiveness and progress of the preparation process, the sample can be viewed without breaking vacuum. The ASaP contains a high-magnification microscope and CCD camera (1,000X). To view the sample, the user includes an inspection step in the recipe. The process pauses automatically and the sample stage moves into the viewing position. The image is displayed on the ASaP's monitor. The microscope parameters of magnification, focus, and illumination intensity are adjustable through the interface.

Model 1030 Automated Sample Preparation (ASaP) System

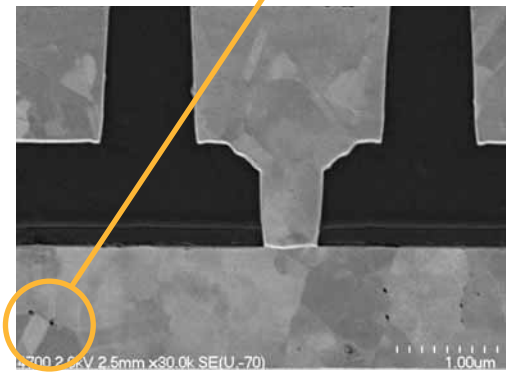
Results



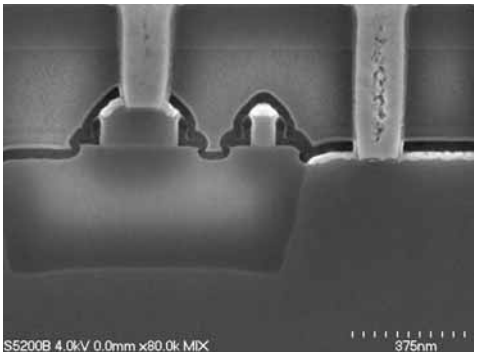
Indexed pattern obtained from an Al sample by electron backscattered diffraction (EBSD) following low angle argon ion beam etching (IBE) at a voltage of 2kV, a current of 5mA, and an incident milling angle of 10°. The sample was fully rotated (360°) during ion beam etching. Prior to sample treatment in the Model 1030, the pattern could not be indexed. Following IBE, a high-quality pattern with a 90% confidence index was obtained.



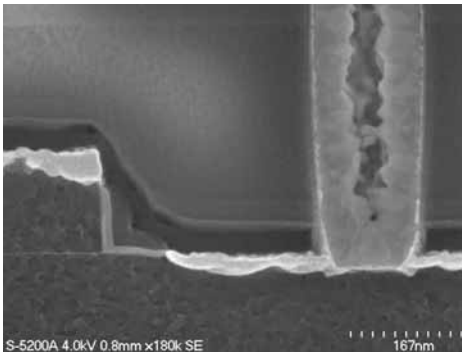
SE (2kV) image of a mechanically ground (with a 0.1 micron diamond finish) and oxidized cross-section of a Cu-based microelectronic material.



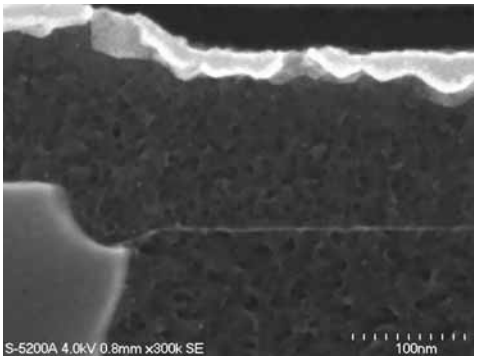
SE/BSE (2kV) image of a Cu-based microelectronic material following plasma cleaning, ion beam etching, reactive ion etching, and ion beam sputter coating. The sample was initially prepared using mechanical methods. It was then plasma cleaned for 2 minutes followed by argon ion beam etching (IBE) at a voltage of 4kV, a current of 5mA, and an incident milling angle of 10°. The sample was fully rotated (360°) during ion beam etching. Total IBE time was 7 minutes. Reactive ion etching (RIE) was done at 3 watts for 20 seconds. The sample was ion beam sputter coated (IBSC) with 1nm of platinum. Total processing time was less than 17 minutes.



SE/BSE (4kV) image of a Cu-based microelectronic material following plasma cleaning, ion beam etching, and reactive ion etching. The sample was plasma cleaned for 5 minutes followed by argon ion beam etching (IBE) at a voltage of 4kV, a current of 5mA, and an incident milling angle of 5°. The sample was fully rotated (360°) during ion beam etching. Total IBE time was 3 minutes. Reactive ion etching (RIE) was done at 5 watts for 20 seconds with a process gas mixture of CF_4 – 10%O_2. The W interconnects are etched consistently across the field of view.



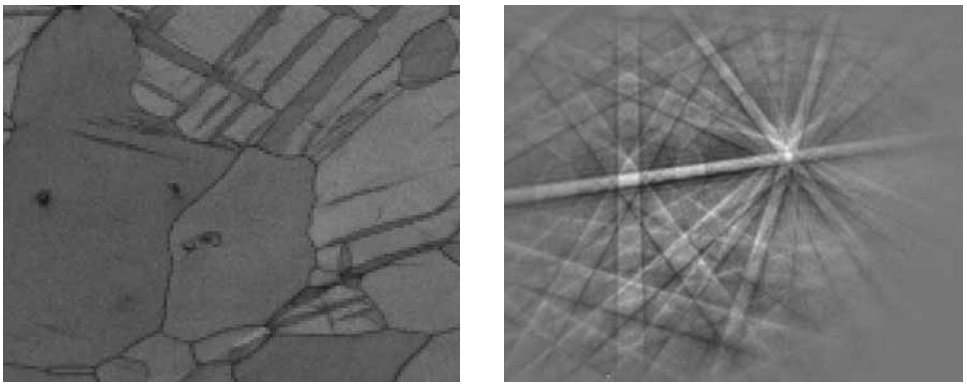
SE/BSE (4kV) image of a Cu-based microelectronic material at the transistor level following plasma cleaning, argon ion beam etching, and reactive ion etching. The sample was plasma cleaned for 5 minutes followed by argon ion beam etching (IBE) at a voltage of 4kV, a current of 5mA, and an incident milling angle of 5°. The sample was fully rotated (360°) during ion beam etching. Total IBE time was 3 minutes. Reactive ion etching (RIE) was done at 7 watts for 20 seconds with a process gas mixture of CF_4 – 10%O_2.



SE/BSE (4kV) image of a Cu-based microelectronic material following plasma cleaning, ion beam etching, and reactive ion etching. The sample was plasma cleaned for 5 minutes followed by argon ion beam etching (IBE) at a voltage of 4kV, a current of 5mA, and an incident milling angle of 5°. The sample was fully rotated (360°) during ion beam etching. Total IBE time was 3 minutes. Reactive ion etching (RIE) was done at 7 watts for 20 seconds with a process gas mixture of CF_4 – 10%O_2. The gate oxide has been revealed by slightly over-etching the surrounding Si.

continued

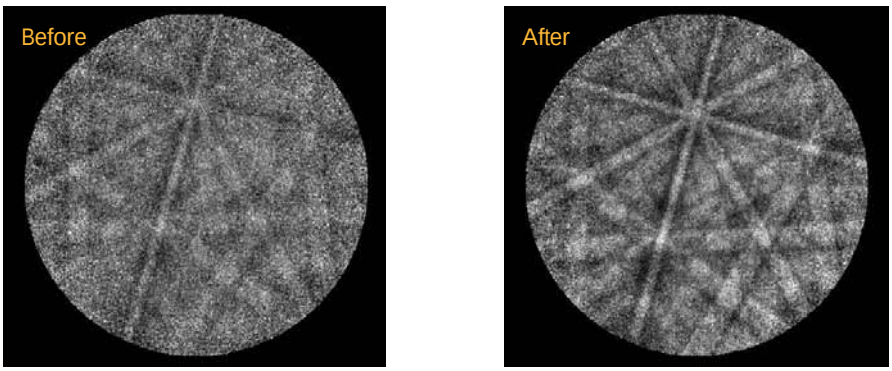
Results *(continued)*



Band contrast image (left) and EBSD pattern (right) of a Mg alloy following plasma cleaning and argon ion beam etching. After 3 minutes of plasma cleaning (PC), ion beam etching (IBE) was performed at a voltage of 3.5kV, a current of 5mA, and an incident milling angle of 8°; followed by a voltage of 2.5kV, a current of 5mA, and an incident milling angle of 7°; and then a voltage of 1.6kV, a current of 5mA, and an incident milling angle of 6°. The sample was fully rotated (360°) during each step.

Materials such as this Mg alloy are low in average atomic number and exhibit weak scattering during EBSD analysis, making patterning and indexing difficult. Low angle ion beam etching removed residual plastic deformation and oxidation. Twins could be identified in the band contrast image, and the individual electron backscattered diffraction patterns were sharp.

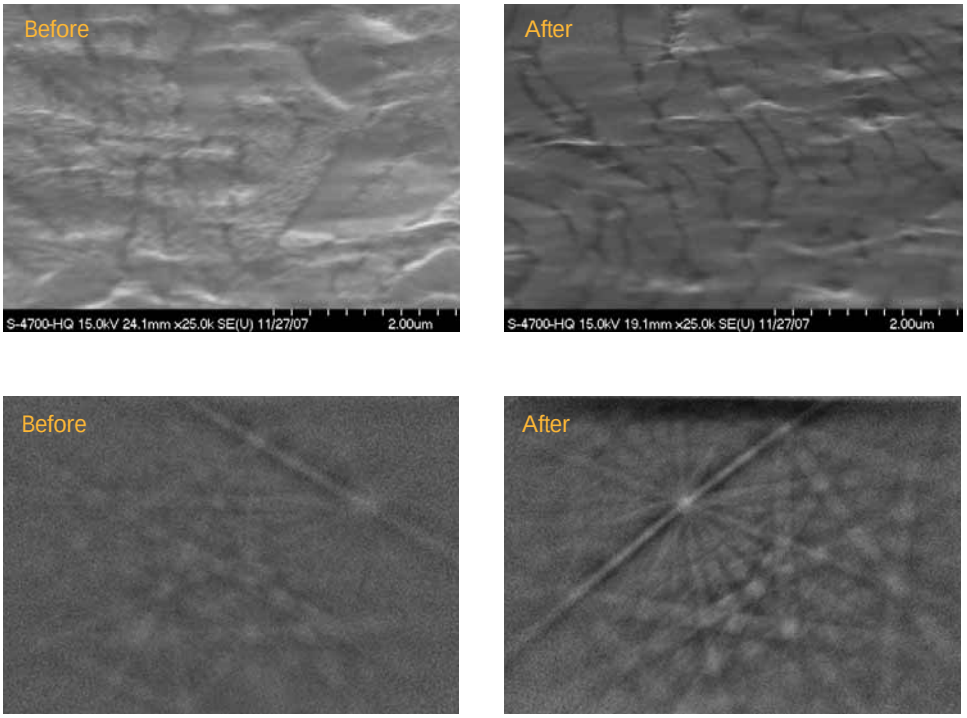
Images courtesy of Jennifer Cocle and Pierre Hovington, Institut de recherche d'Hydro-Québec and Raynald Gauvin, McGill University (Canada)



EBSD patterns of a low-carbon Martensitic steel sample before (left) and after (right) a combination of plasma cleaning and argon ion beam etching. After 5 minutes of plasma cleaning (PC), ion beam etching (IBE) was performed at a voltage of 5kV, a current of 5mA, and an incident milling angle of 5°. The sample was fully rotated (360°) during ion beam etching.

This super-martensitic stainless steel (AISI 415) exhibited a relatively soft martensitic matrix with some retained austenite. The sample was initially prepared by electropolishing. Martensitic steel and similar materials, which are high in average Z and scatter strongly during EBSD analysis, benefit from ion beam etching. IBE was used to remove the last deformation layer. It also removed the surface roughness produced during electropolishing.

Images courtesy of Pierre Hovington, Institut de recherche d'Hydro-Québec (Canada)

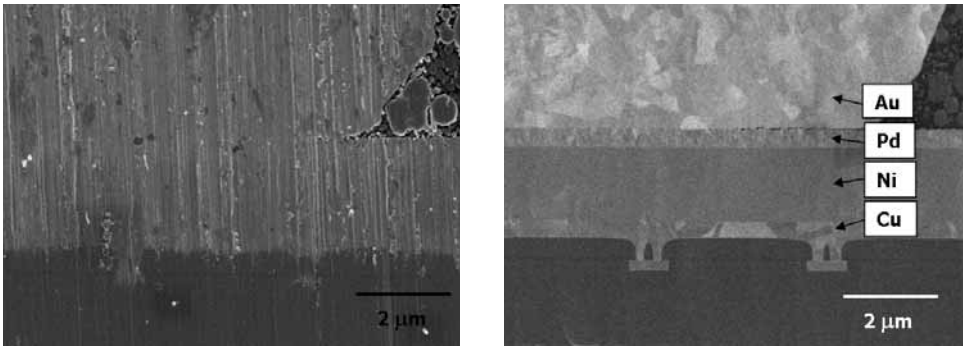


FESEM images (top row) and EBSD patterns (bottom row) of a Zr-Nb alloy before and after low-energy ion beam etching. The Zr-2.5Nb pressure tubes used in the CANDU (Canada Deuterium Uranium) reactor have a complex microstructure as a result of their extrusion process starting from the two-phase region and ending with a fair amount of cold work (~25%). As with other refractory metals, Zr-2.5Nb has low grinding and polishing removal rates, making the elimination of all polishing scratches and deformation difficult. This produces an important challenge for EBSD characterization which necessitates a sample without deformation from the preparation procedure. In addition, Zr is very reactive leading to the formation of a thin layer of oxides and hydroxides at the surface of the sample. The IBE process greatly decreases the plastic surface deformation and removes the roughness after polishing and etching, optimizing both FESEM imaging and EBSD mapping.

Images courtesy of Pierre Hovington, Institut de recherche d'Hydro-Québec (Canada)

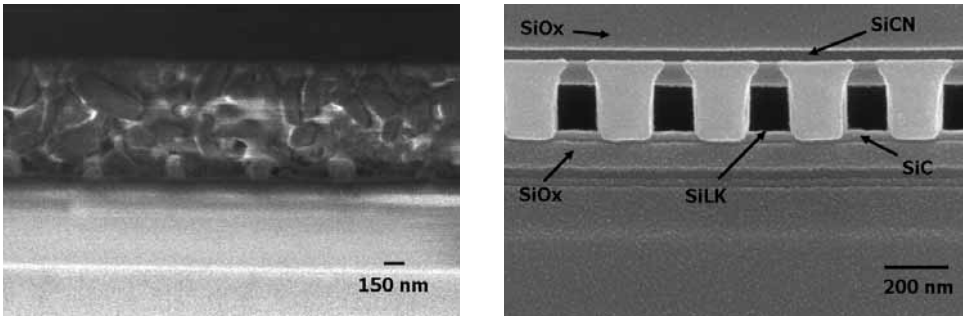
continued

Results *(continued)*



FESEM images (2kV) of the ball bond region of a multi-component system after mechanical grinding (left) and subsequent argon ion beam etching (right). The ion beam etching (IBE) process was performed starting at a voltage of 4kV with a current of 5mA and ending at a voltage of 1kV with a current of 5mA. An incident milling angle of 7° was used with a sample rocking angle of +/- 70°.

The image after mechanical preparation shows the common artifacts remaining after the grinding of the soft solder. Planarization by IBE yields grain structure information in both the Au and Pd based phases.



FESEM image (2kV) of Cu/P-SiLK*Y single damascene wafer after cleaving and plasma cleaning (left). The sample was plasma cleaned (PC) for 5 minutes using a process gas mixture of 25% O2 with a balance of argon.

FESEM image (2kV) of Cu/P-SiLK*Y single damascene cross-section after plasma cleaning, ion beam etching, reactive ion etching and plasma cleaning followed by coating with Cr (right). To prepare this sample, 5 minutes of plasma cleaning (PC) was followed by ion beam etching (IBE) at a voltage of 4kV, a current of 5mA, and an incident milling angle of 9° for 20 minutes. The sample was fully rotated (360°) during IBE. Reactive ion etching (RIE) was done with a process gas of Ar – CHF_3 – CF_4 for 1.5 minutes. A final two minutes of PC and 6nm of Cr coating were added prior to FESEM imaging.

Removal of the damage from cleaving this Cu-based microelectronic material containing a low-k dielectric resulted in the characterization of all metallic and nonmetallic phases.

Sample courtesy of the Inter-University Microelectronics Center (IMEC) (Belgium)

Images courtesy of E.R. Beach, C.J. Wood, S.J. Rozeveld, J. Waeterloos, The Dow Chemical Company (U.S.A.)

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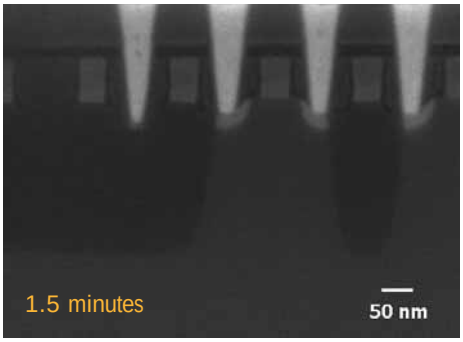
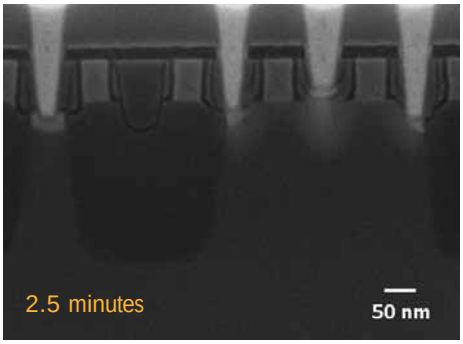
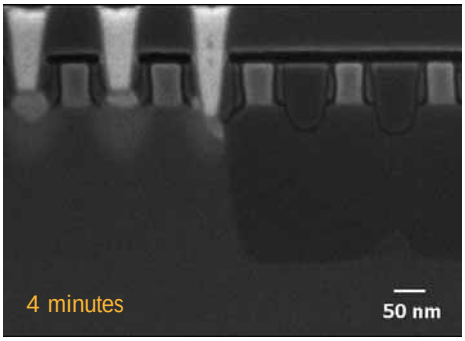




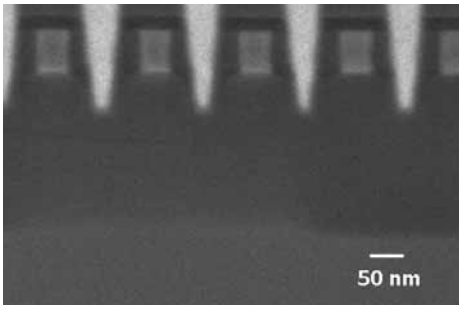


FESEM images (5kV) yield details of a 45nm-scale microelectronic device. Ion beam etching (IBE) was performed starting at a voltage of 4kV with a current of 5mA and ending at a voltage of 1kV with a current of 5mA. An incident milling angle of 7° was used with a sample rocking angle of +/- 70°. Reactive ion etching (RIE) was done with a process gas of CF_4 – 10%O_2 at a gas flow rate of 40sccm and 150mT pressure.

Rapid optimization of process recipes is possible using a single cross section of a 45nm - scale microelectronic material. Repetitive planarization and reactive ion etching allowed the definition of process parameters. A reactive etch with chemistry selective to the Si-rich phases was used. The bulk Si, strained Si (Si-Ge), and Si nitride were etched back to various depths with respect to the planarized surface.

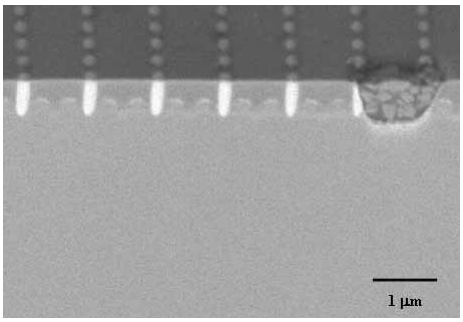
continued

Results *(continued)*



FESEM image (2kV) gives details of a 45nm scale microelectronic device. Ion beam etching (IBE) was performed starting at a voltage of 4kV with a current of 5mA and ending at a voltage of 1kV with a current of 5mA. An incident milling angle of 9° was used with a sample rocking angle of +/- 65°. Reactive ion etching (RIE) was done with a process gas of CF_4 – 27%O_2 at a gas flow rate of 40 sccm and 150mT pressure.

After planarization by argon ion beam etching, a reactive ion etch with chemistry selective to Ti-based phases was used. This process decorated the TiAl and TiAlO phases, as well as the gate oxide containing Hf and Zr.



FESEM image (2kV) yields details of a site-specific, cross-section sample following argon ion beam etching. Ion beam etching (IBE) was terminated at a plane that bisected the pre-configured FIB marks placed on this microelectronic material. The IBE process parameters were a voltage of 2kV, a current of 5mA, and an incident milling angle of 4° until the target was reached.

Model 1030 specifications

Sample size	Accommodates samples up to 1.0 in. (25 mm) diameter and 1.0 in. (25 mm) thick
Chamber/Stage	Single vacuum chamber
	Uses oil-free vacuum system
	5-axis stage for sample manipulation
	Automated load lock facilitates sample exchange
Plasma cleaning	High frequency (HF) 13.56 MHz capacitively coupled plasma
	Auto tuning and matching of RF power supply
	Ion energies of less than 12 eV
Ion beam etching (IBE)	Ion source parameters:
	Variable voltage (0.5 to 6.0 kV) continuously adjustable
	Variable current 1 mA to 8 mA continuously adjustable
	Milling angle 0° to 90° adjustable in 1° increments.
	360° sample rotation or rocking with 1° increments adjustable from 1° to 179°
Reactive ion etching (RIE)	Adjustable RF power, chamber pressure, gas flow rate(s), plate distance, and sample cooling
	Auto tuning and matching of RF power supply
	Six process gas inlets
Ion beam sputter coating (IBSC)	Coating thickness uniformity on a 1 cm sample
	Amorphous coating
	Six user-selectable targets
	Target carousel interchangeable through load lock
Supporting hardware	Load lock pump down time <20 seconds
	Auto sample height detection
	Auto sample positioning at individual processing locations
	In situ sample imaging with a microscope and CCD camera (1,000X)
	Diagnostics maintenance software
Safety specifications	SEMI S2; CE Complaint
Dimensions	39" (991 mm) W x 57" (1,448 mm) H x 31" (788 mm) D
Weight	646 lb (293kg)
Power	100/110/220/240 VAC, 50/60 Hz, 1,000W

continued

Model 1030 operating parameters

Operation	Process parameter
Plasma cleaning (PC)	Time
Ion beam etching (IBE)	Gas – argon Ion voltage Ionization current Milling angle Sample rotation or rocking angle Time
Reactive ion etching (RIE)	Process gas flow rate and chamber pressure (up to six gases) RF Power Temperature Time Distance separating sample and plate electrode
Ion beam sputter coating (IBSC)	Target selection (one of six) Coating thickness Coating rate Sample rotation or rocking angle Distance separating sample and target

FISCHIONE INSTRUMENTS

EXCELLENCE...MAGNIFIED

Cover image: SE/BSE (4kV) image of a Cu-based microelectronic material following plasma cleaning, ion beam etching, and reactive ion etching. The sample was plasma cleaned for 5 minutes followed by argon ion beam etching (IBE) at a voltage of 4kV, a current of 5mA, and an incident milling angle of 5°. The sample was fully rotated (360°) during ion beam etching. Total IBE time was 3 minutes. Reactive ion etching (RIE) was done at 5 watts for 20 seconds with a process gas mixture of CF_4 – 10%O_2. The contrast levels of the doped vs. undoped SiO_2, polysilicon vs. silicon, as well as that of the nitrides, oxides, and silicides are apparent.

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