BAL-TEC

Preparation to view information

RESSEM - SEM CONTROLLED BROAD ION BEAM MILLING/ETCHING

RES 120

The new world of electron microscopy sample preparation

Coating Cryo Preparation
Ion Etching Solid State Preparation
Accessories and more...

KEY FEATURES INCLUDE:

- Ion beam milling with in-situ SEM observation
- Full computer control
- TEM and SEM applications of highest flexibility
- Variable ion energies and milling angles
- Load lock system for permanent high vacuum
- Specimen observation while ion milling through BSE detection
- Integrated milling recipes
- Process evaluation through high resolution SEM imaging
- TEM process auto-termination through STEM imaging

RESSEM - SEM CONTROLLED BROAD ION BEAM MILLING/ETCHING

RES 120

TEM and SEM specimens of highest quality

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UNIT DESCRIPTION:

Over the last few years, ion milling devices have drastically improved in a variety of features which have made ion milling the most common sample preparation method in electron microscopy today. However, with the fast movement in semiconductor technology to continuously smaller feature sizes, a new limit in ion milling sample preparation has been reached – the process observation. By integrating a SEM (Scanning Electron Microscope) into the ion milling process, Bal-Tec moves ion beam sample preparation for electron microscopy to a level that allows accurate and efficient site specific sample preparation complementary to the current FIB (Focused Ion Beam) technique. With its high level of flexibility and the unrivalled integrated SEM monitoring system, the RES 120 is the ideal instrument to prepare specimens of all type of materials, for TEM (Transmission Electron Microscopy), SEM and LM (Light Microscopy) analysis such as:

- Metals
- Ceramics
- Multilayers
- Multi-component systems
- Thin Films
- Powders
- Semiconductor
- High Tc Semiconductors
- Diamond composite materials
- Glasses
- Minerals
- Dental material
- Bone implantation material
- Selected organic materials

IN-SITU SEM IMAGES OF A TYPICAL TEM SAMPLE PREPARATION PROCEDURE IN THE RES 120

RES 120

A good HRTEM specimen is only as good as its sample preparation process. This already starts with the mechanical preparation step. A low quality sample surface before ion milling will never lead to a high quality HRTEM sample. As a first preparation step, the SEM in the RES120 enables to examine the surface quality of a mechanically pre-prepared specimen (Figure 1) at a much higher precision than a light microscope. This provides the chance to go back and mechanically enhance the surface quality prior to ion milling (Figure 2) to ensure HRTEM samples of highest quality as ion milling end result (Figure 5). During the milling the smoothening of the specimen surface can be observed (Figure 3) and the final electron transparency determined in the split screen mode (Figure 4).

Set up: Quick Clamp Holder, double sided low angle ion milling, 7kV acceleration voltage, (3kV final thinning), sample rotation.

Images:
Plan view Ti-alloy TEM sample

Fig 1: Surface of a dimpled specimen, too rough for HRTEM

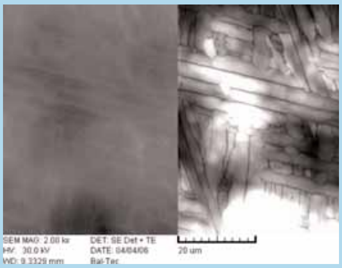


Fig 4: SE/TE Split Screen

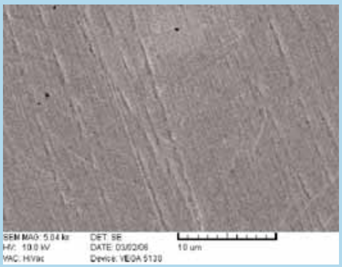


Fig 2: Surface enhancement through diamond polishing

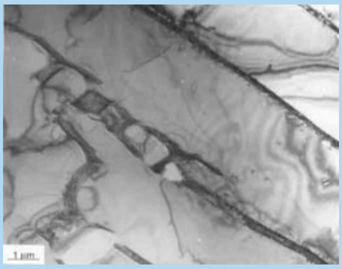


Fig 5: Corresponding TEM image

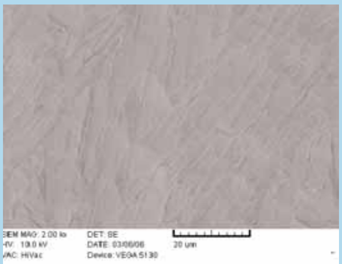
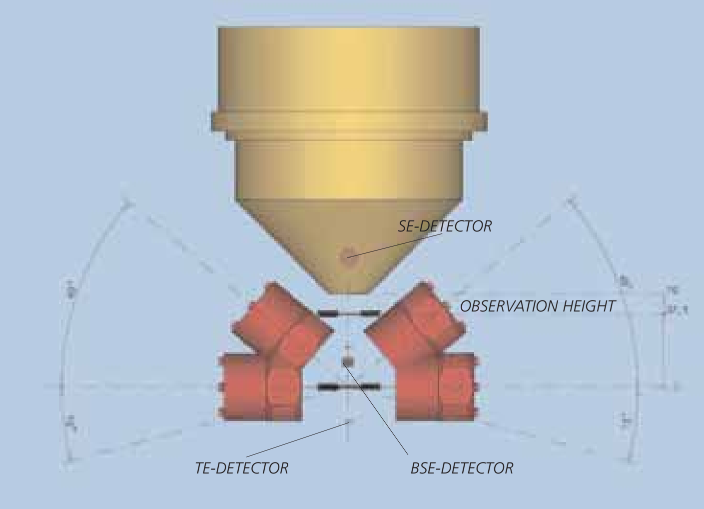


Fig 3: In-situ SEM image during ion milling

Fast and accurate automated target preparation

SEM (SPECIMEN OBSERVATION)

The RES 120 features a SEM equipped with a SE (Secondary Electron), a BSE (Back Scattered Electron) and a TE (Transmitted Electron) detector. This combines the advantages of conventional broad ion beam milling with the possibility to visualise and control the milling progress precisely to a specific specimen site. This eliminates over/under milling of specimens, resulting in a drastic increase of milling accuracy, specimen yield and throughput. Consequently this leads to a time and cost efficient sample preparation procedure with high ROI (Return on Investment).



The SEM as observation device enables optimal specimen watching and monitoring of preparation results (resolution approx. 50nm) while ion milling. In this so called ion milling mode, it is possible to thin a specific specimen area in the x-y-plane precisely to a desire depth in z-direction using BSE and/or TE detection. The TE detector is mounted right underneath the specimen and allows very sensitive process termination using STEM imaging. The split screen mode, that allows BSE and TE imaging at the same time, is a very helpful tool particularly during the final thinning procedure.

The SE detector is positioned to allow high resolution investigation (resolution <15nm), for which the ion milling process is stopped and the specimen temporarily 'transferred to the so called observation position.

There are two infrared cameras installed in the RES 120. One is used for general chamber observation, the other one for beam alignment. The visibility of the ion beam facilitates the alignment procedure and enables precise and accurate beam positioning.

Infrared camera for easy and accurate beam alignment

COMPUTER CONTROL

Continuing Bal-Tec's philosophy, the RES 120 is fully computer controlled. All parameter settings for the milling process and the SEM system are operated via one touch screen. The ability to program complete milling sequences, store application recipes for various user levels, the semi-automatic SEM operation as well as settings for advanced users enables successful and repeatable sample preparation for all levels of experience.

Once the RES 120 program is started, the PC controls the entire milling process including:

- the vacuum system
- the regulation of the gas inlet as well as the high voltage for the ion sources
- the complete movement of the ion sources and the 5-axis stage
- the vacuum load-lock for sample transfer.

This provides the following options:

- precise process control and parameter settings
- complete process automation through:
  - individually created milling programs
  - a program library, that allows precise preparation of reoccurring applications
  - sensitive auto-termination using TE detection
  - specimen control using BSE detection
  - process evaluation via high resolution SEM imaging
  - storage of all live images viewed with the SEM or b/w CCD cameras
  - split screen mode operation, that allows data collection from two detectors simultaneously.

DRY VACUUM SYSTEM AND GAS INLET

The vacuum system consists of a turbo molecular drag pump backed by a multistage diaphragm pump, creating an oil-free ultimate vacuum of <$2x10^{-6}$mbar. The innovative computer controlled gas inlet system guarantees stable operation of the ion sources at working pressures between $6x10^{-5}$mbar and $4x10^{-4}$mbar.

IN-SITU SEM IMAGES OF CROSS SECTIONAL TEM SAMPLE PREPARATION USING THE WIRE SHADOWING TECHNIQUE

RES 120

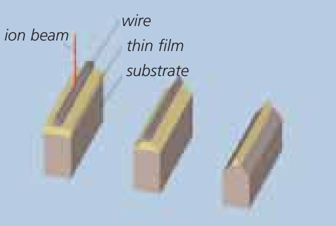


Fig 1; Principle of the Wire Shadowing Technique (developed by H. Bartsch, 1986)

A wire is glued on top of the sample right above the structure of interest. The wire and the uncovered part is then removed by ion milling with an ion beam perpendicular to the specimen surface (Figure 1 and 2). Due to the shadowing effect of the wire the specimen becomes wedge shaped (Figure 1). Continuing this process until the wire almost disappears results in a specimen that is highly electron transparent over an area of a few hundred microns in length underneath the remnant wire (Figure 4). With the SEM as observation device this is a very fast and precise method to produce cross sectional TEM specimens with an extremely large electron transparent area (Figure 5 and 6).

Set up: 90° angle with respect to the sample surface (Figure 1), 7kV acceleration voltage, sample oscillation.

Images:
Semiconductor TEM samples

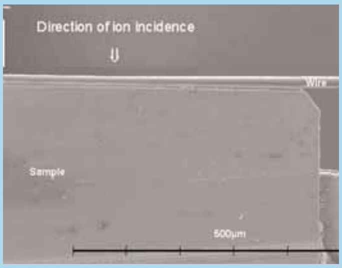


Fig 2: In-situ SEM image of the wire mounted on the specimen before ion milling

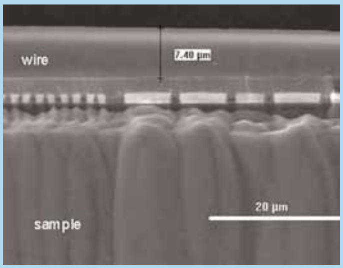


Fig 3: In-situ SEM image after 30 min of ion milling

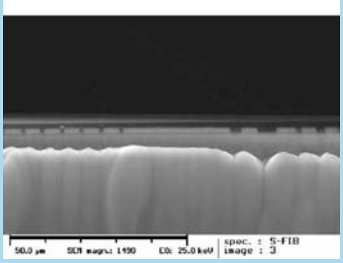


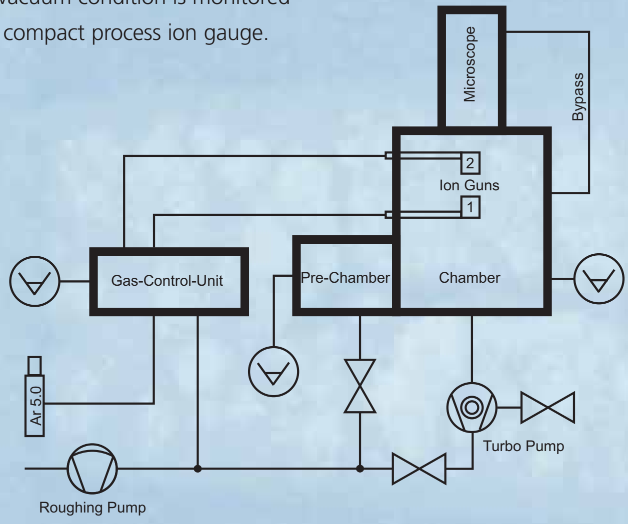
Fig 4: In-situ SEM of the electron transparent area underneath the remnant wire

Fig 5: Corresponding TEM image

Fig 6: Magnified area of the red box in Fig 5

Full SEM functionality

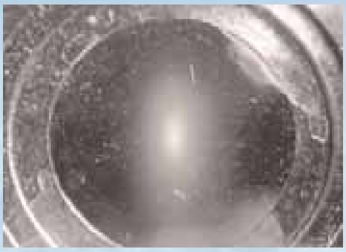
The vacuum condition is monitored by a compact process ion gauge.



ION SOURCES:

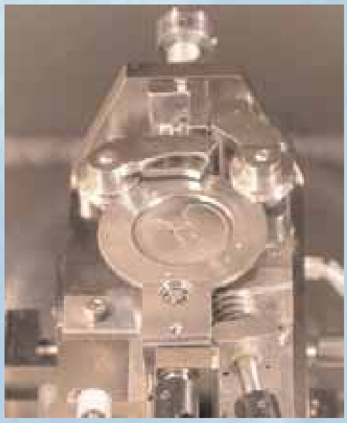
The RES 120 includes two individually controlled Saddle Field ion guns, which operate over a wide energy range. The ion accelerating voltage (0.8kV to 10kV) as well as the ion beam currents (0.5mA to 3.5mA) can be varied in any desired, programmable sequence and combination. This allows both, fast ion milling/etching processes with high milling rates as well as gentle low energy ion milling as the final preparation step to obtain specimens for High Resolution Transmission Electron Microscopy (HRTEM).

Since the ion beam diameter mainly depends on the acceleration voltage, it ranges from 0.8mm at 10kV up to approximately 2.5mm at 2kV. The angular adjustment of the ion sources and the ion beam alignment is realised using DC-motors with incremental encoders. The accuracy and repeatability of the angle settings reaches 0.1°. Using the integrated IR-camera as observation tool, ion beam alignment becomes a very precise and easy routine.



5-AXIS STAGE:

The RESSEM is equipped with a high precision 5 axis specimen stage allowing fast and accurate sample movement in X, Y and Z direction including sample tilt, rotation and oscillation, up to ±90° are feasible. All movements are realised using DC-motors with incremental encoders and are arbitrary programmable. As a result, an automated, precise and reproducible set up is possible. To simplify the process, there are three pre-defined stage positions:



- transfer position,
- milling position,
- observation position.

LOAD LOCK SYSTEM - quick specimen transfer and exchange

The RES 120 includes a load lock system, which is completely decoupled from the stage, avoiding any vibration during operation. The specimen initially mounted onto the specimen holder, is then placed into the load lock system. Once the load lock is evacuated, the specimen holder is transferred to the stage in the main chamber. Such a system allows save and rapid specimen transfer and exchange, without affecting the vacuum in the process chamber. Once the specimen is loaded, ion milling and/or specimen examination can start immediately.

FULLY AUTOMATED ION MILLING PROCEDURE

The computer controlled RES 120 can be programmed to run entire ion milling processes fully automatically. Each individual milling sequence can be programmed and combined to a complete ion milling recipe. These recipes can be stored in an application library and reused for reoccurring preparation tasks. The factory programmed library contains basic milling procedures and can be extended without software limitation.

In-situ evaluation of preparation results

BAL-TEC

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The RESSEM features an absolutely pioneering and extremely precise routine to automatically (or manually) terminate the ion milling process using the TE detector of the SEM in combination with advanced image processing software. In such a configuration auto-termination can now be applied to an unlimited range of TEM specimens, even delicate samples can be prepared, "unattended" and translucent materials do not require a Faraday Cup any more.

SITE SPECIFIC TEM PREPARATION OF A FIB SPECIMEN

RES 120

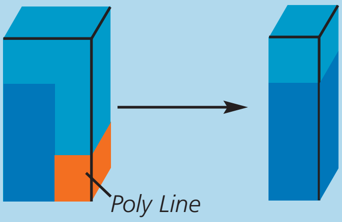


Fig. 1: Cross-section of a FIB sample with a contact structure (blue) and a 50 nm thick word line before and after removing the word line

The drastic shrinkage of semiconductor geometries suddenly let unexpected structures like word lines appear in the TEM image, that block the area of interest (Figure 1 and 3). A controlled removal of such a poly word line without over-etching the specimen requires the SEM monitoring system of the RES 120. It visualises the poly in form of a white line (Figure 2), and enables to follow the ion milling progress on the SEM monitor. After 16 minutes of ion etching a partial removal of the poly line is noticeable (Figure 4). Additional ion milling of 3 more minutes completely eliminates the poly line and makes the structure details of the defective contact evident (Figure 5,6, and 7).

Set up: FIB Cleaning Holder, single sided low angle ion milling , 2.5kV acceleration voltage, no sample movement.

Images: Contact structure of a SRAM device (Courtesy of N. Wang, Cypress Semiconductor)

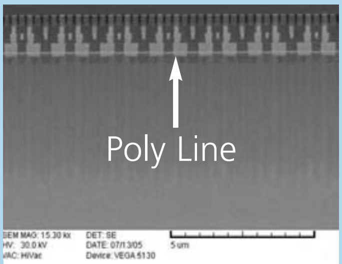


Fig 2: In-situ SEM image of the poly line before ion milling

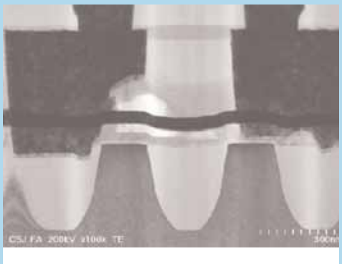


Fig 3: HSTEM image before ion milling

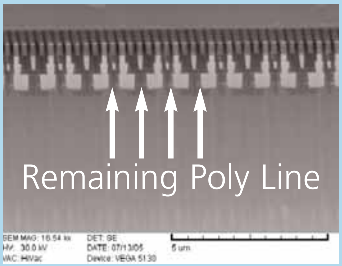


Fig 4: In-situ SEM image of the poly line after 16 min of ion milling

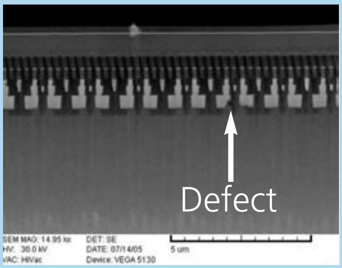


Fig 5: In-situ SEM image of the defect structure

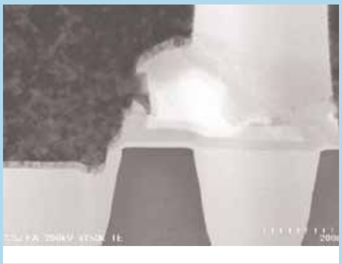


Fig 7: HSTEM image of the defect structure after ion milling

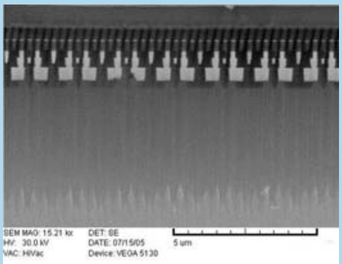


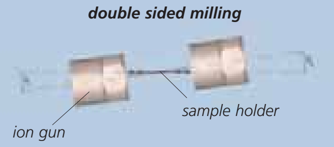
Fig 6: In Situ SEM image of the defect structure looking at the sample rear

Unparallel TEM endpoint detection through STEM imaging

UNLIMITED APPLICATION RANGE

The RESSEM distinguishes itself by its high flexibility in ion milling parameter settings. The combination of individual ion gun angular setting with the specimen tilt allows milling angles from 0°-90°. Together with the large ion gun energy range (0.8-10kV) an unlimited application range for TEM and SEM can be conducted in the RES 120 such as:

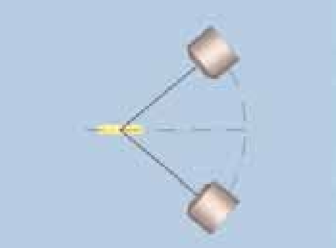
- Site specific preparation of plan view and cross-sectional TEM specimens using extremely low ion gun angular settings to



For cross sectional specimens

achieve high quality surfaces for HRTEM analysis.

- Additional thinning and cleaning of FIB samples removing the amorphous and damaged layers caused by the FIB process. A technique, where one of the ion guns continuously alternates between the two sides of the lamella to avoid recontamination.



FIB cleaning technique patent by BAL-TEC (Publication # of public application EP1 447 656A1, US2 004 164 242A1, JP2 004 245 841A)

SURFACE ENHANCEMENT OF MECHANICALLY PRE-POLISHED SOLDER BALLS BY Ar ION BEAM CLEANING AND POLISHING

RES 120

Like for TEM a good SEM specimen is only as good as its sample preparation process. This starts with the mechanical preparation step. A low quality sample surface before ion milling will never lead to a high quality SEM sample and will either provide no or very restricted surface information (Figure 1). Similar to the TEM preparation procedure the SEM in the RES 120 is used to evaluate the surface quality of a mechanical pre-prepared specimen prior to ion milling to ensure an ion milling end result of highest quality (Figure 3 and 4). Once the surface quality is approved ion milling can start. The process control via regular SEM imaging secures the visualisation of defects and voids without removing and/or destroying structure areas of interest (Figure 2 and 3).

Set up: Clamp Holder, single sided low angle ion milling, 5kV acceleration voltage, sample oscillation.

Images:
Mechanically pre-polished Pb free solder balls

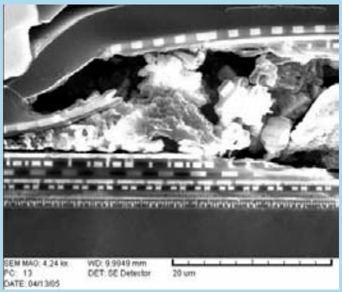


Fig 1: Early detection of interface damage due to large grit size polishing

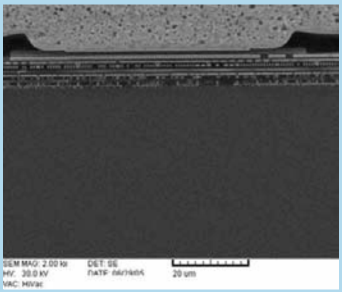


Fig 2: 3 hours of ion milling and the change of the mechanical pre-preparation procedure, leads to a clear interface surface (See also fig 3 at higher magnification)

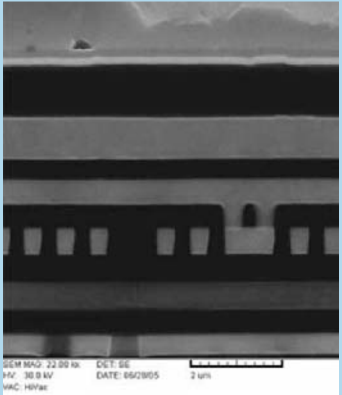


Fig 3: Solder ball interface at a higher magnification. It shows a very smooth surface and the visibility of voids.

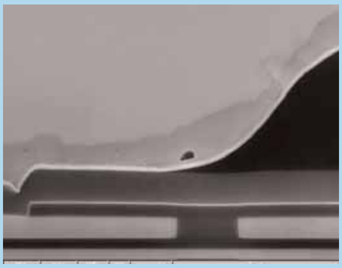


Fig 4: Corresponding FE-SEM image

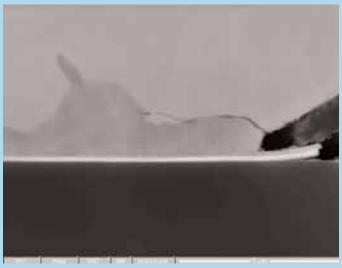


Fig 5: Corresponding FE-SEM image

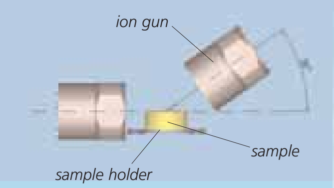
No preparation failure ...

- Cleaning and polishing of TEM and SEM specimens, to remove surface contamination (cleaning) and irregularities (polishing) working with milling angles between 6° (polishing) and 15° (cleaning). A specimen movement in x-direction enables to uniformly process large areas up to 25mm in diameter.

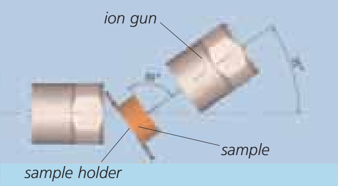
single sided milling

Single sided low angle ion milling for plan view TEM specimens or any polishing procedure

- Contrast enhancement of SEM specimens, to visualise layer boundaries, grain structures and structures in general.



Contrast enhancment by 35° ion milling with one ion gun



Contrast enhancement by 90° ion milling with a 35° tilt ion gun and a 55° tilt specimen.

To enhance the contrast a selective milling procedure is used that is based on the individual grain removal rates in a polycrystalline material, and works with steep milling angles in the range of 35°-90°.

- Ion beam slope cutting of SEM specimens, a fast method to prepare cross-sectional and angled samples with a very large cutting area of 3-4mm in length and several hundred microns in depth. 35° or 90° cuts can be performed.

IN-SITU OBSERVATION OF A BROAD ION BEAM SLOPE CUTTING PROCEDURE THROUGH Ag SOLDER BALLS

RES 120

In general, when preparing solder balls for SEM investigation the area of interest is the interface. Ag is a very soft material, which makes it nearly impossible to apply mechanical treatment without destroying the interface area due to the share forces that are applied during mechanical polishing. Thus slope cutting is the ideal method to prepare cross sectional SEM specimens for such delicate materials. The SEM observation device in the RES 120 allows to continuously observe the cut through the material ensuring a precise stop at the right location, avoiding to cut to deep or not deep enough. (Figure 1-5).

Set up: 90°- Slope Cut, 6kV acceleration voltage, sample oscillation ± 60°

Images:
Silver Solder Balls

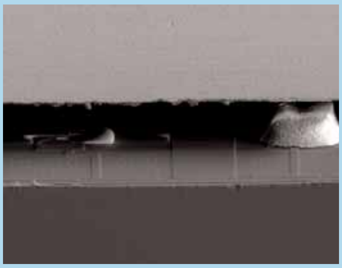


Fig 1; In-situ SEM image after 30 minutes cutting.

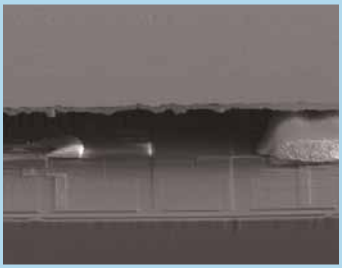


Fig 2; In-situ SEM image after 1h 30 minutes cutting time.

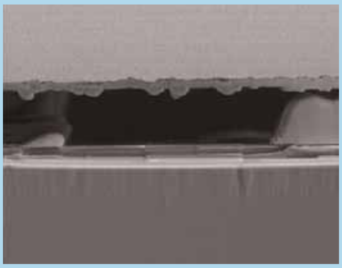


Fig 3; Completed cut after 3h of ion milling

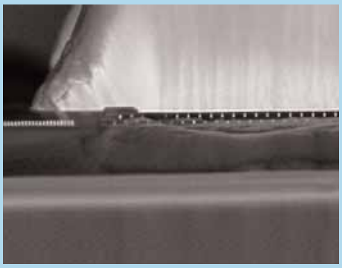


Fig 4: Completed cut at higher magnification shows a very smooth surface.

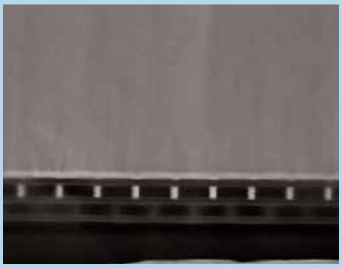


Fig 5: Same area like Fig 4 at an higher magnification, demonstrating surface smoothness and a fully intact interface.

ACCESSORIES

To support the large application spectrum, the RES 120 can be equipped with a variety of accessories:

ACCESSORIES FOR TEM APPLICATIONS

Quick Clamp Holder SET

for single and double sided low angle ion milling down to 6°. The set contains the Quick Clamp Holder with the loading stage and beam adjustment discs.

LZ 04039 VN

Graphite Specimen Holder Set

for single and double sided low angle ion milling down to 0°. The set contains the holder and beam adjustment discs.

LZ 00135 VN

FIB Cleaning Holder Set

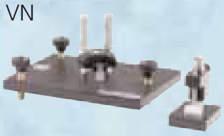
to reduce the lamella thickness and damaged surface region of typical FIB specimens. The cleaning set contains the holder, a loading stage, a specimen alignment device as well as beam adjustment discs.

DZ 00001 VN

X-TEM Kit

for polishing TEM cross sectional samples. The kit contains a tripod with calibration stage for fine adjustment of the tripod, a specimen press for face to face gluing of the specimen and a variety of consumables for X-TEM sample preparation and polishing.

LZ 03497 VN



ACCESSORIES FOR SEM APPLICATIONS

SEM Holder Set

for cleaning, polishing and contrast enhancement of SEM samples, up to 25mm in diameter and 12mm in height. The set contains the holder, an adjustment tool, the loading stage as well as an adapter for SEM stubs

LZ 04040 VN

Clamp Holder

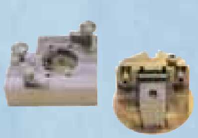
to mill cleaved and/or polished semiconductor wafer samples in a maximum size of
5 mm (H) x 7 mm (W) x 2 mm (D).

LZ 03471 VN

Slope Cutting Holder Set

to produce 35° and 90° angled cross-sections for the investigation of the vertical structure of the sample in the Scanning Electron Microscope. The kit contains a loading stage, 35° and 90° specimen carriers to perform 35° and 90° slope cuts, as well as spare masks.

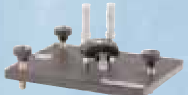
DZ 00036 VN



X-SEM Kit

for polishing SEM cross sectional samples. The kit contains a tripod with calibration stage for fine adjustment of the tripod as well as consumables for sample polishing.

LZ 03344 VNT



Specific accessories for specific applications

TECHNICAL SPECIFICATIONS:

Basic unit RES 120	RES 120 GvN

CONTROL

Computer	Control System Panel PC with touch screen video card for CCD camera SEM controller card LAN
Software	BT ion milling and SEM control software MS Win-dows™

ION SOURCES

Type:	Two Saddle Field ion sources:
Ion energy:	1keV to 10keV
Source current:	up to 3.5 mA (per ion source)
Ion current density:	10mA/cm2 (per ion source)
Beam diameter:	0.8mm (at 10kV) 2.5mm (at 2kV)
Gas:	Argon (other gases possible)
Gas flow:	< 1sccm / ion source with automatic control
Milling angle:	0° to 90°

STAGE

Specimen stage	Five axis stage with	
	x-direction: ± 5mm	
	y-direction: ± 5mm	
	z-direction:	Milling position: SEM working distance: 40mm ± 5mm Observation position: SEM working distance: 10mm
	Tilting:	0°-180° depending on holder
	Rotation:	0.5-10rpm
	Oscillation:	up to ± 90°

DETECTORS

SEM detectors

SE detector:	Everhart-Thornley detector for high resolution imaging
BSE detector:	Semiconductor detector for specimen observation during the ion milling/etching process
TE detector:	Semiconductor detector for process observation and auto-termination

Chamber observation devices	Two b/w CCD cameras for chamber observation and beam alignment
Illumination	IR LED's for b/w CCD camera
Auto-Termination	• TE detector for sensitive termination of TEM sample preparation • Timer

Vacuum

Pumping System	Turbo molecular drag pump backed by an oil-free multi stage diaphragm pump
Ultimate Vacuum	• 10^{-4}Pa (10^{-6}mbar)
Working pressure	• 10^{-2}Pa (10^{-4}mbar)

SEM SPECIFICATION

Resolution	<15nm in observation mode (SE) 50nm in milling mode (BSE/ TE)
Magnification	1 to 100 000 x (SE @ 30kV/10mm 1 to 20 000 x (BSE, TE @ 30kV/10mm
Acceleration voltage	500V to 30kV
Electron gun	Tungsten heated cathode
Probe current	1pA to 2mA
Scanning speed:	from 600ns to 10ms per pixel adjustable in steps or continuously
Focus window	Shape, size and position continuously adjustable
Image size	Up to 4 096 x 4 096 pixels, adjustable separately for live image (in 4 steps) and for stored images (9 steps), selectable square or 3:4 rectangle
Microscope control	All microscope functions are PC controlled using MS Windows platforms
Automatic procedures	Vacuum control, Filament heating, Gun Alignment, Compensation for kV, Probe Current optimised for Spot Size, Spot Size optimised for Magnification, Scanning Speed, Contrast & Brightness,Focus & Stigmator, Look up Table

Detailed specimen observation
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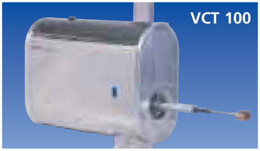


ION BEAM MILLING

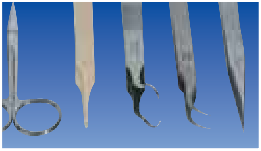
• ION MILLING/COATING FOR TEM, SEM AND LM

MEMS PROCESSING

• CRITICAL POINT DRYERS FOR 4", 6" AND 8" WAFERS

CONSUMABLES

• FULL RANGE FOR SEM AND TEM



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