Fabrication of High Quality S/TEM sample with Focused Ion Beam
Rich Technology Heritage – A History Of FIRSTS

1949  Philips introduces its first production TEM system
1966  Solid-State 5 Angstrom TEM delivered
1971  FEI Company founded
1977  High-Resolution SEM introduced
1986  Computerized CM TEM Series introduced
1989  World’s first Variable Pressure SEM introduced
1993  World’s First DualBeam™ (FIB/SEM) by FEI
1998  All-in-One Integrated Tecnai™ TEM
2000  World’s first small stage DualBeam
2003  World’s first Semiconductor FAB DualBeam
2004  FEI TEM breaks the 1 Angstrom barrier
2005  FEI’s all-new Titan™ S/TEM introduced with sub-Angstrom resolution
2006  First DOE TEAM project instruments shipped for 0.5 Angstrom resolution
2008  World’s First Extreme High Resolution SEM
2009  World’s Fastest Analytical S/TEM - Tecnai Osiris
2011  World’s First Integrated Light-Electron Microscope
20 years of DualBeam
What is a DualBeam?
Principle of a DualBeam – Helios NanoLab

- **Ga LMIS**
- **FIB Tomahawk**
- **SEM – Elstar**

Column and integration identical to the Magellan, even the final objective lens.

Beams Coincidence Point

52°

4mm

SPI Live imaging while milling
Dualbeam has an ion column to image, mill and deposit

**IMAGING**
- Emission of secondary electrons and ions
- **FIB imaging**
  - Secondary ions
  - Secondary electrons

**MILLING**
- Sputtering of substrate atoms
- **FIB milling**
  - Sputtered material

**DEPOSITION**
- Chemical interactions
- **FIB deposition**/enhanced etch
  - Volatile products
  - Gas molecules
  - Deposited material

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Ion channeling shows stronger contrast on a platinum aperture than is seen with electron beam imaging.

Milling removed the surface to expose the interior of lead-tin solder with integrated flux cores.

Platinum protective layer deposited easily on both conductive and non-conductive materials.
Important DualBeam Applications

- SEM imaging & analysis
- FIB imaging
- Site-specific cross section preparation
- Serial sectioning imaging/analysis=“3D” applications
- TEM sample preparation
- NanoPrototyping
- Device Edit (DE)=Circuit Edit (CE)=Microsurgery =Chip Repair
Benefits of a DualBeam FIB/SEM for TEM
Sample preparation

➢ Fast - prepare most samples in under 1 hour
➢ Site-specific – end-point accurately on sub 30nm features
➢ Orientation independent – choose the milling angle to match the required orientation of the lamella
➢ Possible to prepare multiple lamella from 1 bulk sample at multiple locations
➢ Sample independent – soft and hard materials are easily handled, as well as powders, large bulk samples etc.
➢ Easily study interfaces of materials with different milling rates.
Preparing highest quality thin samples
Example: preparation of an ultra-thin GaN sample

Step 1: Surface protection - Deposit a conductive layer (GIS)

Ellapsed Time: 0:16:00
Preparing highest quality thin samples
Example: preparation of an ultra-thin GaN sample

Step 2a: Lamella pre-preparation - trenching

Elapsed Time: 0:21:00
Preparing highest quality thin samples

Example: preparation of an ultra-thin GaN sample

Step 2b: Lamella pre-preparation - undercut

Elapsed Time: 0:25:00
Preparing highest quality thin samples
Example: preparation of an ultra-thin GaN sample

Step 3a: In Situ Lift Out – lamella welding

Elapsed Time: 0:30:00
Preparing highest quality thin samples
Example: preparation of an ultra-thin GaN sample

Step 3b: In Situ Lift Out – lamella cut free from bulk

Elapsed Time: 0:33:00
Preparing highest quality thin samples
Example: preparation of an ultra-thin GaN sample

Step 3c: In Situ Lift Out – lamella extraction

Ellapsed Time: 0:35:00
Preparing highest quality thin samples
Example: preparation of an ultra-thin GaN sample

Step 3d: In Situ Lift Out – lamella precise orientation (rotation)

Elapsed Time: 0:37:00
Preparing highest quality thin samples
Example: preparation of an ultra-thin GaN sample

Step 3d: In Situ Lift Out – Welding on TEM grid and cut free

Elapsed Time: 0:51:00
Preparing highest quality thin samples
Example: preparation of an ultra-thin GaN sample

Step 4: Final lamella thinning – 30 kV FIB polishing

Elapsed Time: 1:06:00
Preparing highest quality thin samples
Example: preparation of an ultra-thin GaN sample

Step 4: Final lamella thinning – 5 kV FIB polishing

Ellapsed Time: 1:14:00
Preparing highest quality thin samples
Example: preparation of an ultra-thin GaN sample

Step 4: Final lamella thinning – 2 kV FIB polishing

Elapsed Time: 1:22:00
Preparing highest quality thin samples
Example: preparation of an ultra-thin GaN sample

Step 4: Final lamella thinning – 1kV FIB polishing

Ellapsed Time: 1:30:00
Controllably preparing ultra-thin samples
With FEI’s fully integrated in situ lift-out (INLO) solution

**FEI EasyLift™**

- Integrated & intuitive controls
- Stable & reliable operation
- Precise and repeatable motion
- Hitachi’s INLO IP licensing provided
Preparing highest quality thin samples
Example: preparation of an ultra-thin GaN sample

Step 5: In Situ STEM inspection: BF, DF, HAADF
Sample Preparation

Enabling technologies
Fast, reliable S/TEM Sample Preparation

Make the highest quality sample surfaces with low voltage cleaning

Amorphous layer measurements on samples prepared at 5kV, 2kV, and 500V

30kV CCS + 5kV Box

30kV CCS + 5kV + 2kV Box

Titan HRTEM ~ 500V Cleaned Surface
Sample Preparation for HR-STEM: GaN <211>

Preparation method:
- In-situ lift-out in Helios NanoLab
- Final polishing performed using 1kV FIB
- Total preparation time: 1 hour including lift out

STEM Resolution
Measured with the TEAM 0.5 Column

GaN <211> power spectrum transfers till 49pm

Z contrast image (raw data)

Z contrast image (filtered)
Consistently preparing high quality samples
With FEI’s EasyLift™ - fully integrated in-situ lift-out (INLO) solution

FEI EasyLift™
• Integrated & intuitive controls
• Stable & reliable operation
• Precise and repeatable motion

Dedicated holders
• UMB to flexibly configure the holder configuration
• Pre-tilted holder to allow for sample prep + FIB polishing + STEM without breaking the vacuum
Consistently preparing high quality samples
Precisely monitoring the process & endpointing

**Fast Monitoring**
Real time SEM imaging during FIB milling (SPI)
Info: iSE+eSE/BSE, 52° tilt

**Highest resolution, precise monitoring**
Automated, rapid switching between FIB and SEM (iSPI)
Info: pure eSE/BSE, 52° tilt

**Endpointing with strong materials contrast & surface sensitivity**
live (synchronized) FIB imaging while FIB milling (iRTM)
Info: iSE, top-down
Guided S/TEM Sample Preparation
AutoTEM 4 Software – Intuitive User Interface

- Site list
- Template selection
- Position in bulk
- TEM grid position
- Workflow steps
- Run/Stop
- Progress bar
AutoTEM 4 Software – Automation Modes

- Automatic
- Automatic with manual recovery
- Guided

- Automatic
- Automatic with manual recovery
- Guided

- Automatic
- Automatic with manual recovery
- Guided
Robust, predictable results for a wide range of materials
Continued innovation with 3 new DualBeam launches in 2017

- **Scios 2**
- **Helios G4**
- **Helios PFIB G4**
SCIOS
Scios

- Highest contrast and fast imaging with the unique Trinity detection scheme.

- Fastest and most reliable integrated S/TEM sample preparation with Sidewinder™ HT and Easylift™

- Instant productivity for inexperienced operators with unique User Guidance for common DualBeam activities.

- Highest throughput 3D characterization
Helios NanoLab G4
Pushing the limits of extreme high resolution, sample preparation and prototyping

• Highest resolution with most precise contrast
• Accurately create varied nanoscale functional prototype devices
• Prepare the highest quality ultra-thin samples for HR S/TEM or atom probe
• Access high resolution, multiscale and 3D information
Helios G4 PFIB DualBeam

- **Access high resolution, large volume**, multi-scale and multi-modal subsurface and 3D information
- **Fast and easy preparation** of high quality Ga⁺ free TEM and APT samples
- Unique **guided TEM sample preparation** workflow
- **Extreme high resolution imaging** with the most precise contrast
- **Fast precise micromachining** of complex structures for prototyping and *in situ* mechanical testing
Dualbeams offer a fast and flexible alternative for TEM sample preparation:

- Large range of samples can be prepared
- Site specific with high resolution SEM/FIB imaging
- Monitor the preparation process in real time
- Fast
- Automation for increased speed
- Reliable
- Able to produce HR-(S)TEM quality samples
- Easy to use

Thank You!