Contamination Free
TEM Specimen Preparation

TEM Specimen Requirements

- Thin
- Representative of the bulk
- Uniform thickness
- Flat
- Rugged
- Conducting
- Clean

Topics

- Electropolishing > Model 110 Twin Jet EP
- Disk cutting > Model 170 Ultrasonic Disk Cutter
- Sample grinding > Model 160 Specimen Grinder
- Dimple grinding > Model 150 Dimpling Grinder
- Ion milling > Model 1010 LAMP Ion Mill
- Plasma cleaning > Model 1020 Plasma Cleaner

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Mechanical preparation methods

Initial Thinning

- Cut a slice
  - Diamond saw
    - Damage ~ 100 μm
  - Acid saw
  - Wire saw
  - Cleaving as with silicon wafers

Ultrasonic Disk Cutting

- Site specific cutting of specimen disks
- Fast
- Cutting by mechanical grinding removal of material
- Rate dependent on abrasive (SiC, CBN)
- Automatic termination of cutting

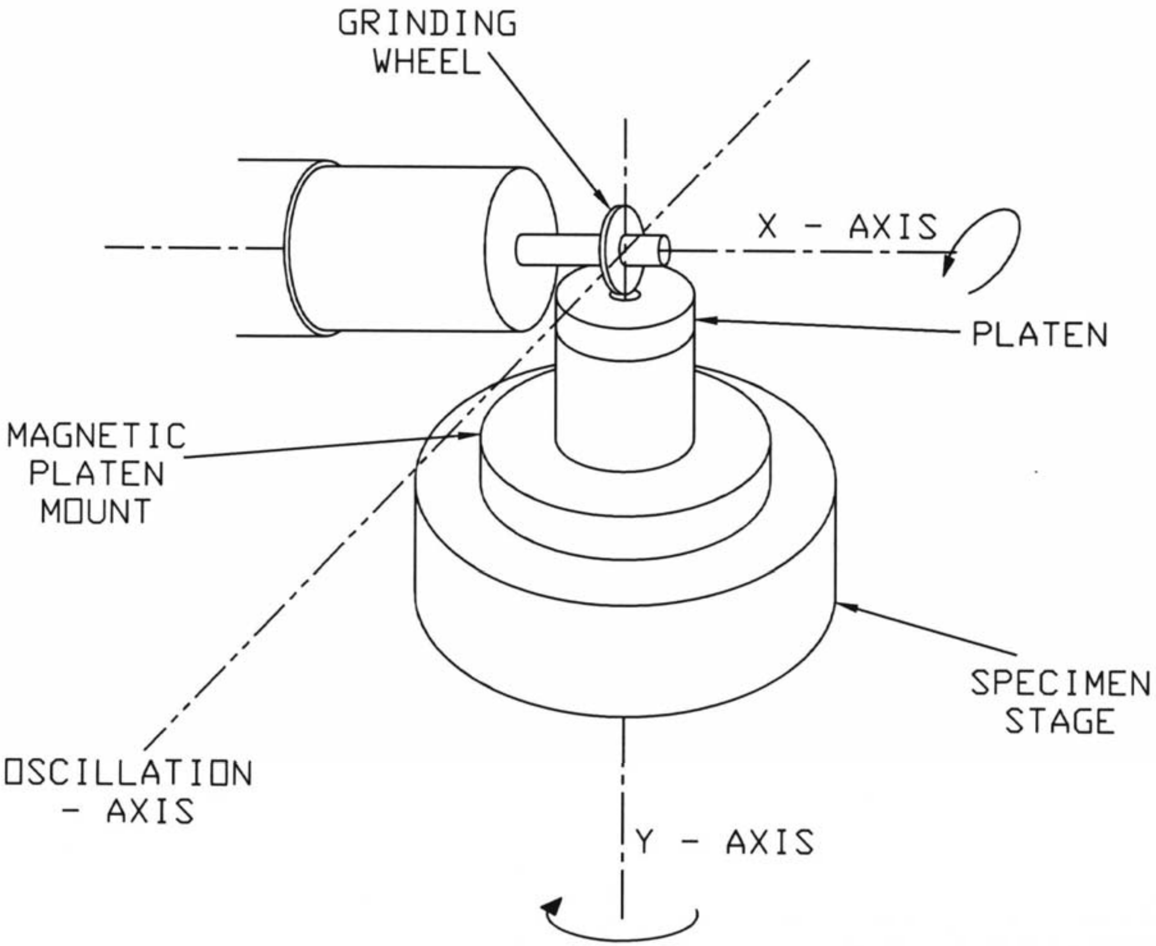
Specimen Grinding

- Take cut disk down to <100μm total thickness
- Important factors
  - Parallel top and bottom surfaces
  - Accurate measurement of specimen thickness
  - Final surface polish to be damage free - colloidal (0.05 μm) silica

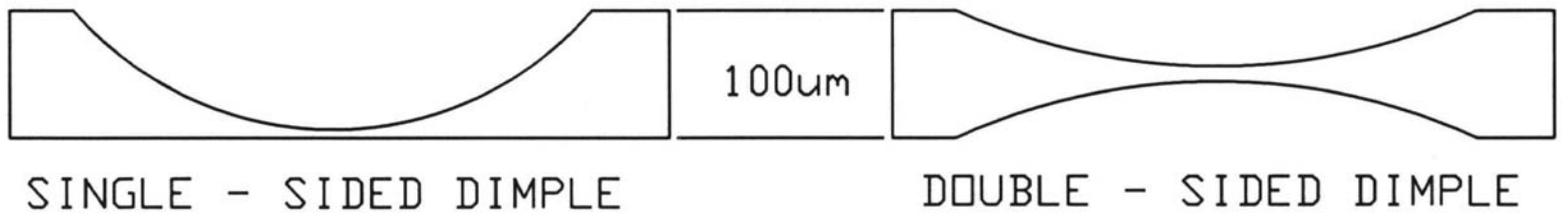
Dimple Grinding

- Location specific thinning
  - Selected location
- Reduces ion milling time
- Creates thin center – thick edge
  - Sample is more robust
  - Easier to handle
- Single or double sided dimpling

Dimple Grinding



Cross-Section of a Dimple



Important Dimpling Parameters

- Dimpling rate
  - Abrasive size
  - Lubricant
  - Wheel speed
  - Wheel pressure
- Dimple geometry
  - Round
  - Oblong

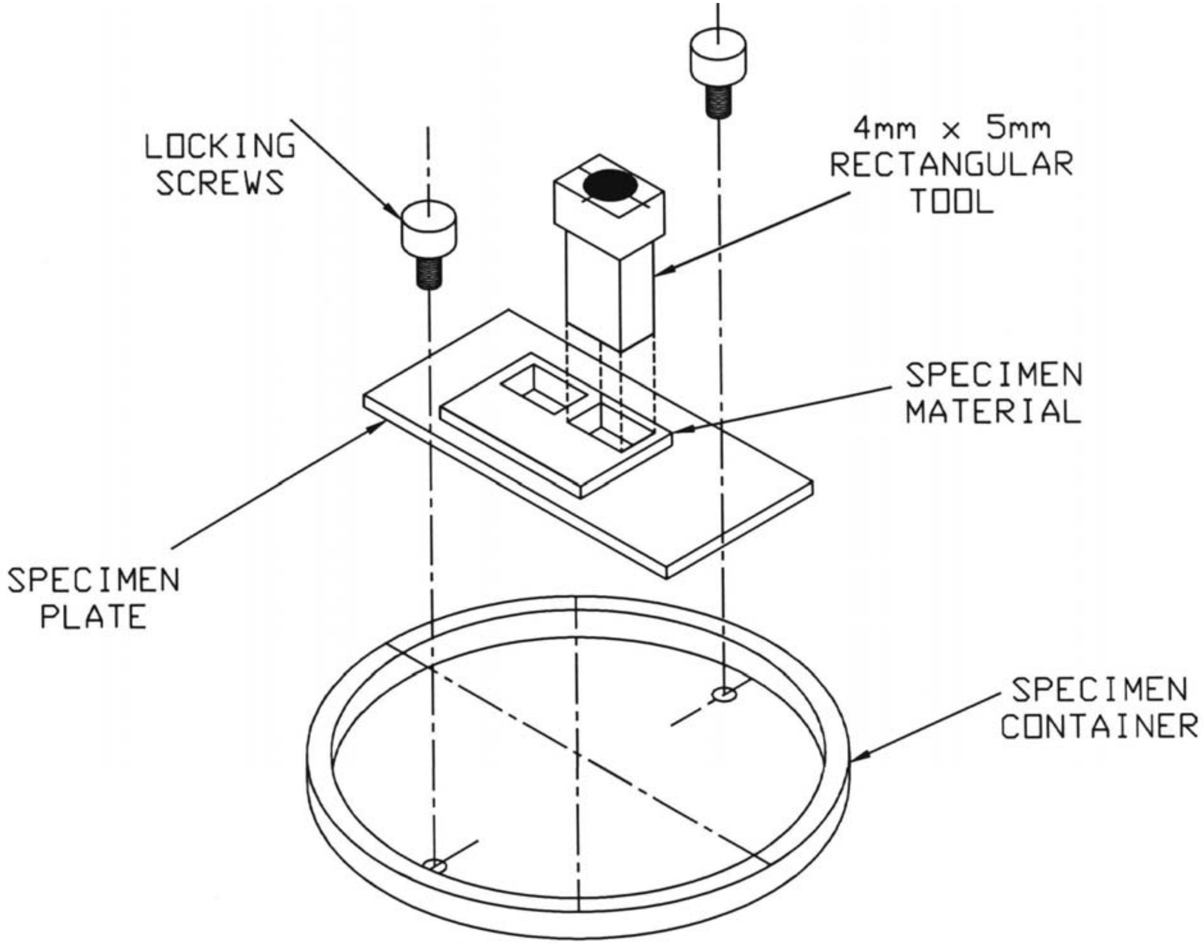
Cross-Sectional TEM (XTEM)

- Semiconductor industry
  - Study microstructure of each layer in an integrated circuit
  - Thin film thickness
- Computer hard drives
- Interface science
- Multiple sections in one XTEM specimen

XTEM Step-by-Step - I

- Cleave small section of wafer to work with
- Mount to ultrasonic disk cutter specimen plate
  - Face up (blank) or face down (with pattern)
  - Face up – cover surface with thin glass cover side to protect surface during cutting process
- Cut out desired sections
- Remove cover slips and discard. Remove sections from specimen plate

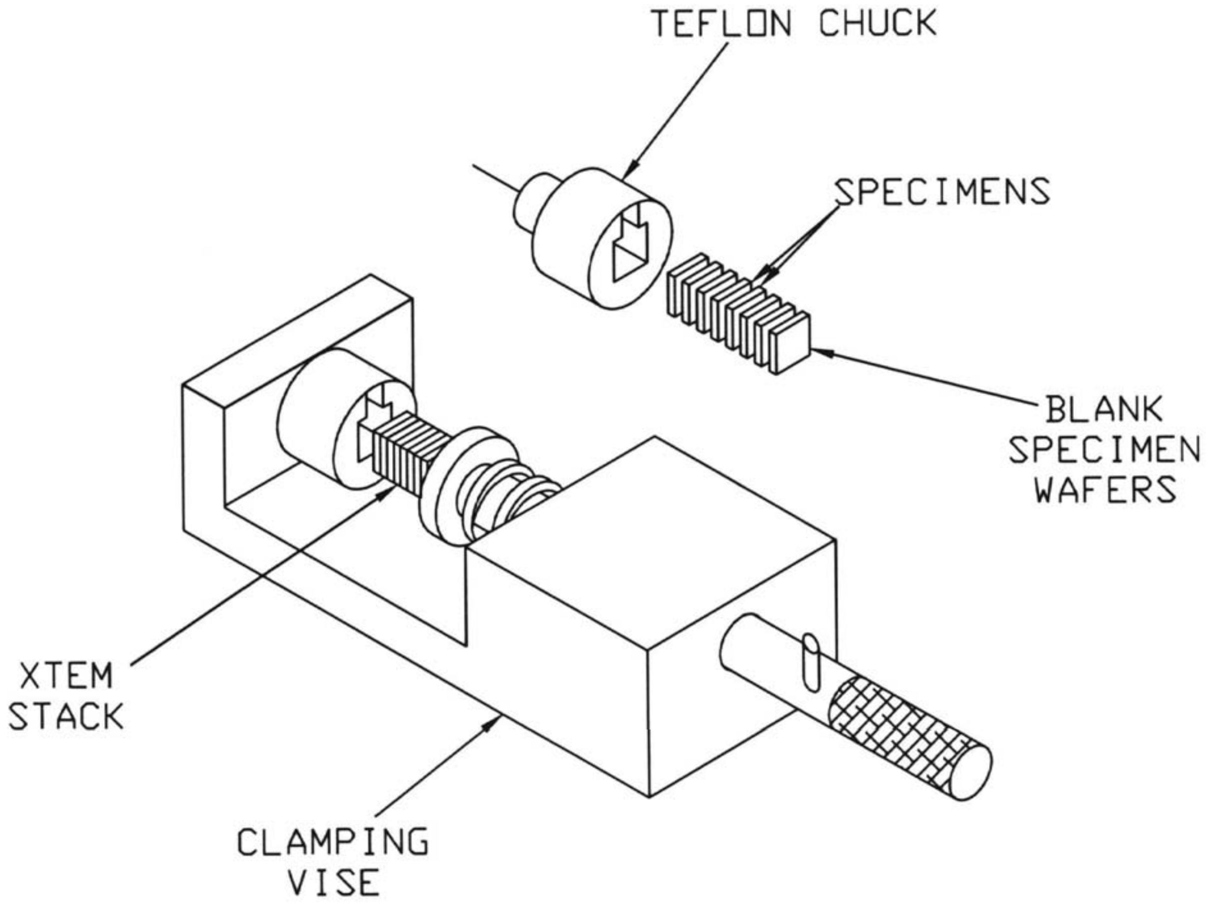
Wafer Mounting and Cutting



XTEM Step-by-Step - II

- Remove sections from specimen grinder and soak and rinse several times
- Create stack
  - Three thick dummy pieces on each end sandwiching sections in between. Innermost dummy pieces can be unthinned real material.
  - Place dummy pieces and sections into teflon chuck, placing droplet of epoxy on each piece
  - Clamp stack in vise and place on hot plate to cure

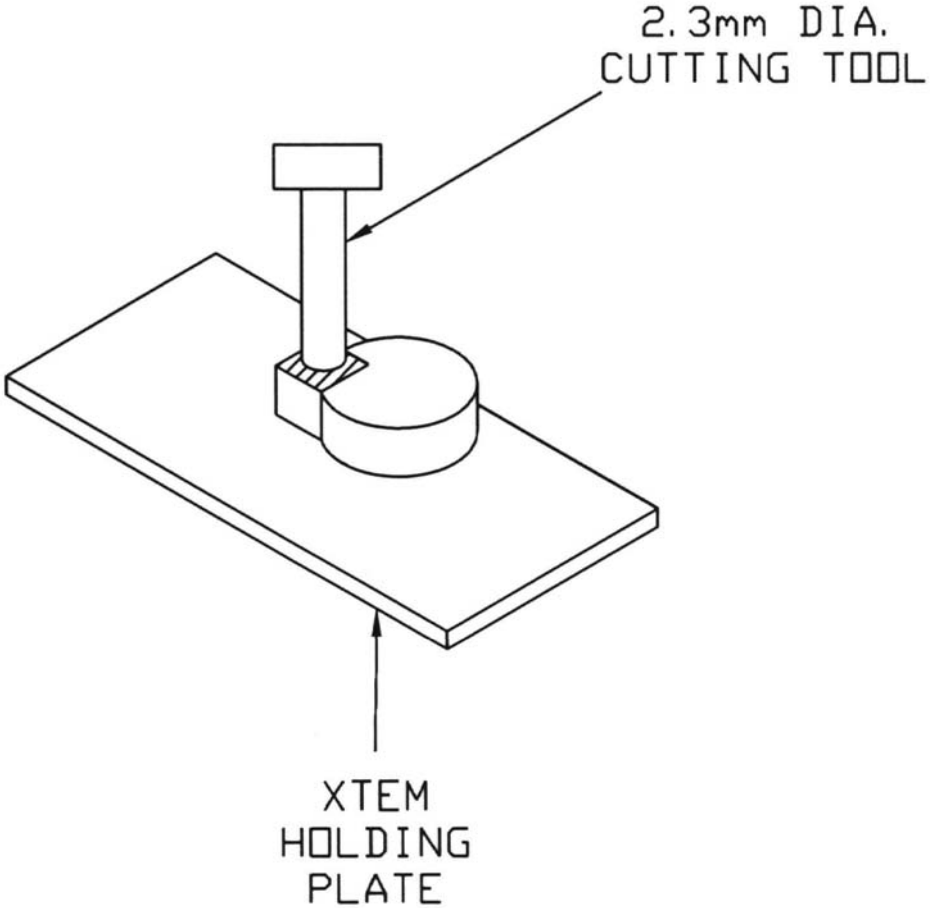
Create Stack



XTEM Step-by-Step - III

- Core stack
  - Ultrasonically cut 2.3 mm diameter cylinder from stack

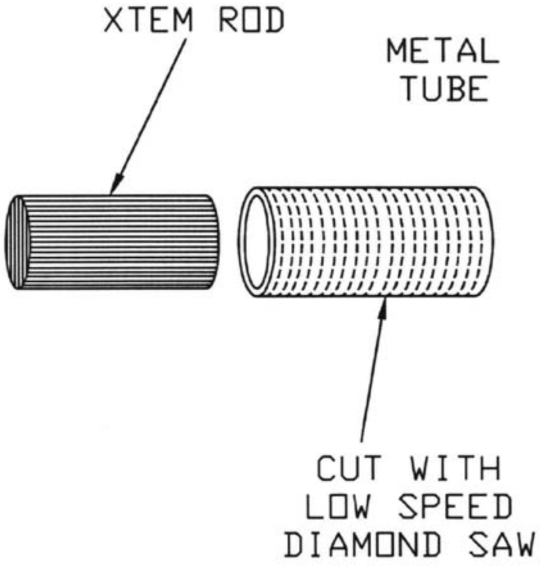
Core Stack



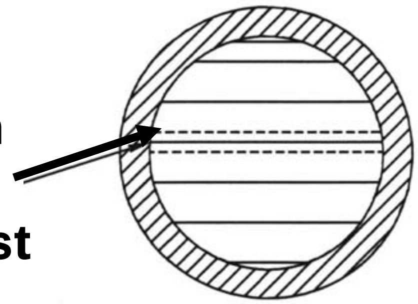
XTEM Step-by-Step - IV

- Place core into 3 mm brass tube, epoxy into place and cure
- Slice 3 mm disks from brass tube
- Place disks on specimen grinder and polish away damaged region from both sides of disk.
- Dimple cross-section
- Ion mill to electron transparency
- Plasma clean in Ar/25%O_2 (2 – 5 min.)

Place Stack in Tube & Slice



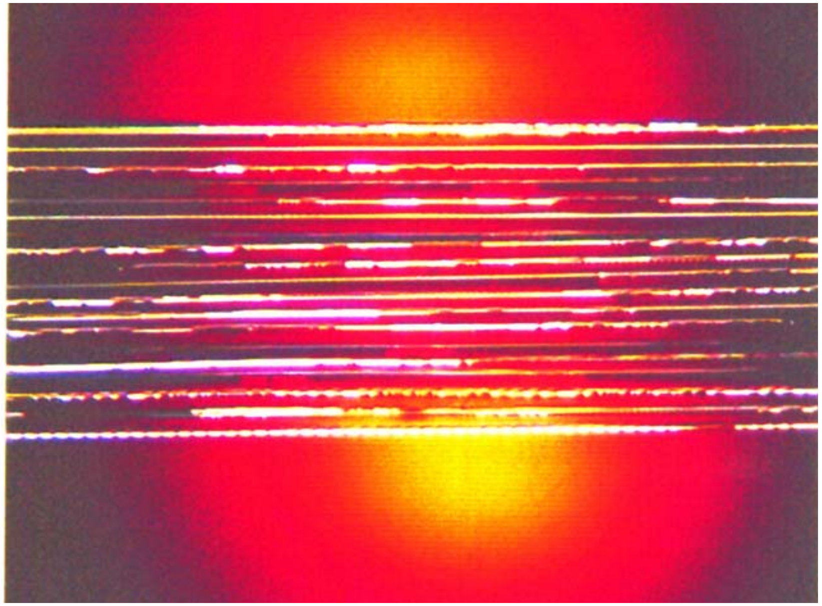
Region of Interest

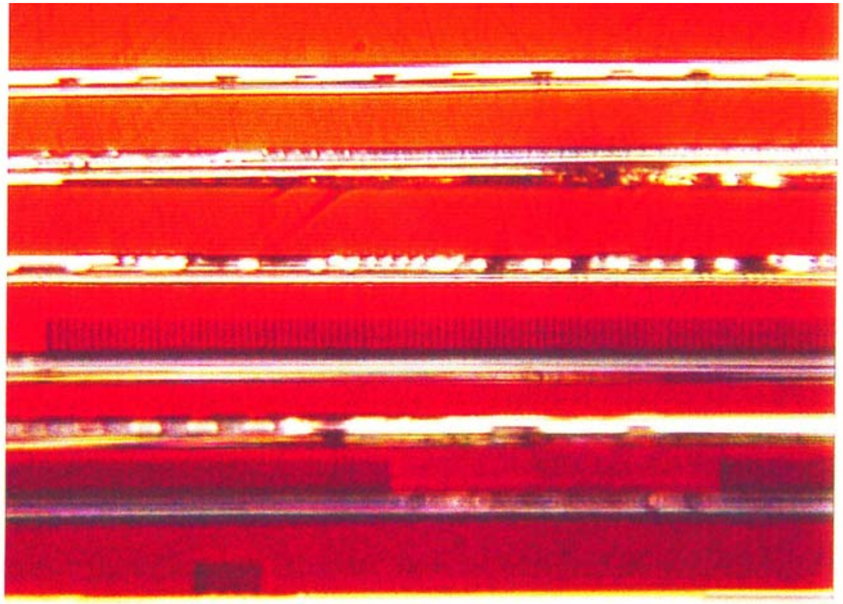


Grind 3mm Disk

- Rough grind with 600 grit SiC paper
- Step down grinding/polishing with diamond films
  - 30 μm, 15 μm, 9 μm, 6 μm, 3 μm, 1 μm, 0.5 μm
- Final polish with colloidal silica
- Flip over and repeat on second side, finishing with specimen < 100 μm thick

19 Piece Cross Section

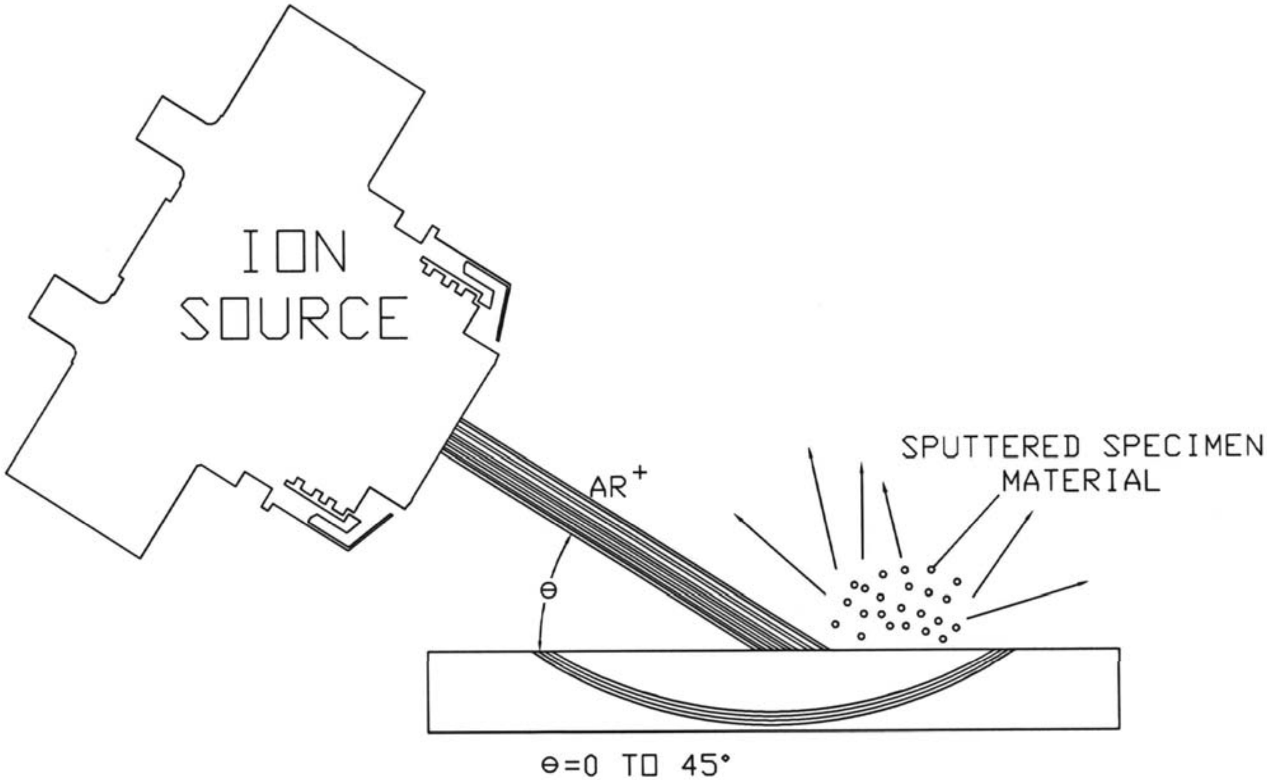




Ion Milling

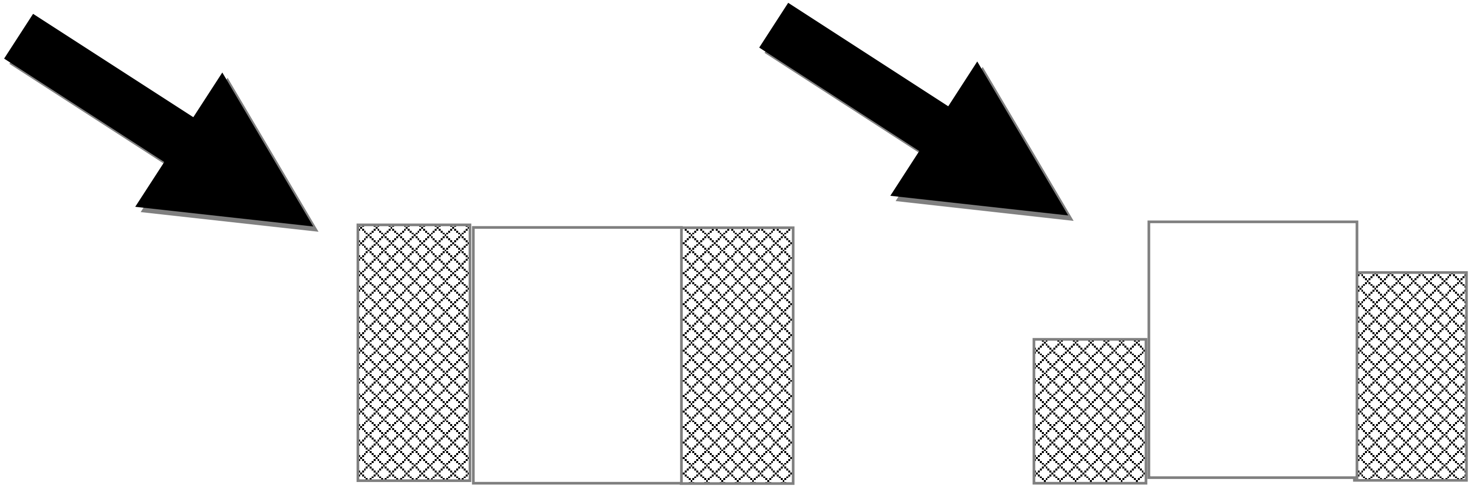
- Non-reactive ions directed at specimen
- Thinning by momentum transfer
- Thinning rate dependent upon:
  - Relative mass of thinning ion and specimen atom ion energy
  - Specimen crystal structure
  - Angle of incidence of ion beam

Ion Milling



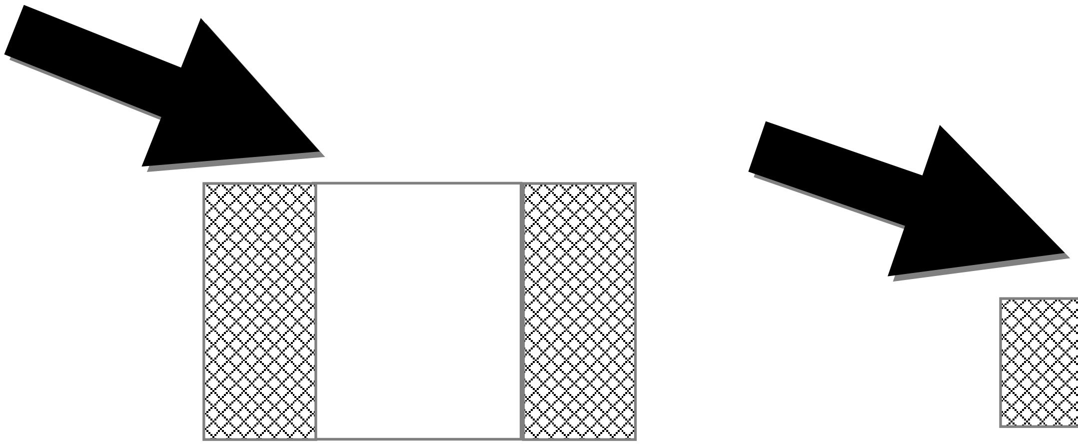
High Angles

- Angles >15º
  - Leads to surface topography as a function of atomic number
  - Rapid thinning



Low Angles

- Angles < 15º
  - Leads to flat surface but slower thinning



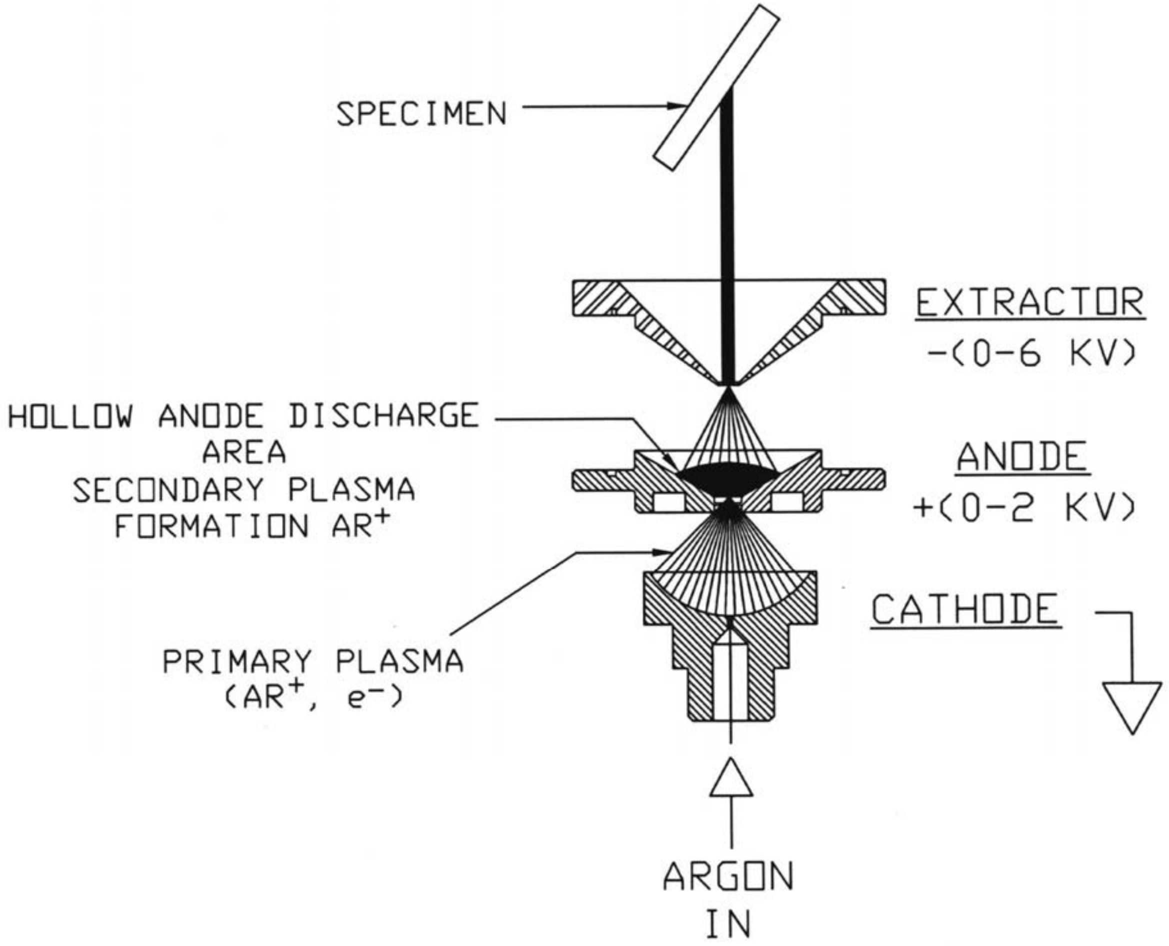
Energy

- Higher energies
  - Thinning rates decreased for some materials
  - Implantation
  - Amorphous layers
  - Point defects
  - Propagated artifacts
- Lower energies
  - Thinning rates more predictable
  - No / reduced damage to specimen

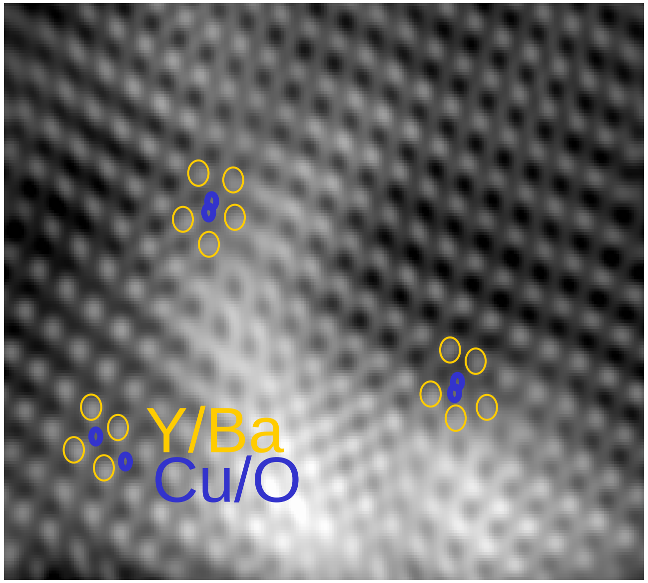
Temperature

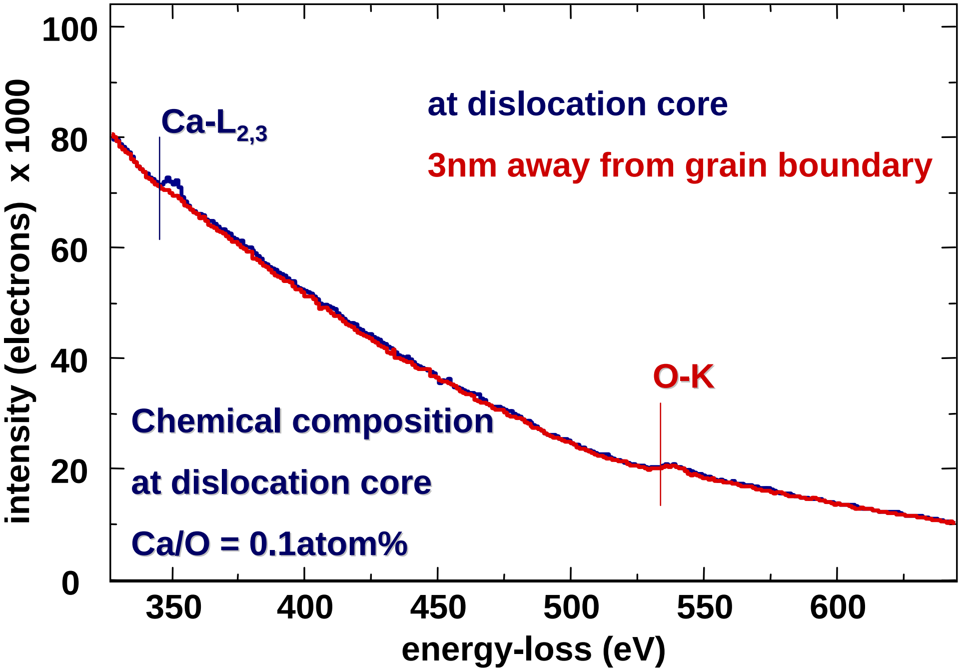
- Without cooling
  - Heat sample to as high as 200°C
    - Changes in phase chemistry
      - Changes in sample chemistry
      - Mobility of defects increases
      - Structural changes
  - Differential / preferential milling

Hollow Anode Discharge Ion Source



Ion Milled Ca doped YBaCuO (123) Bicrystals



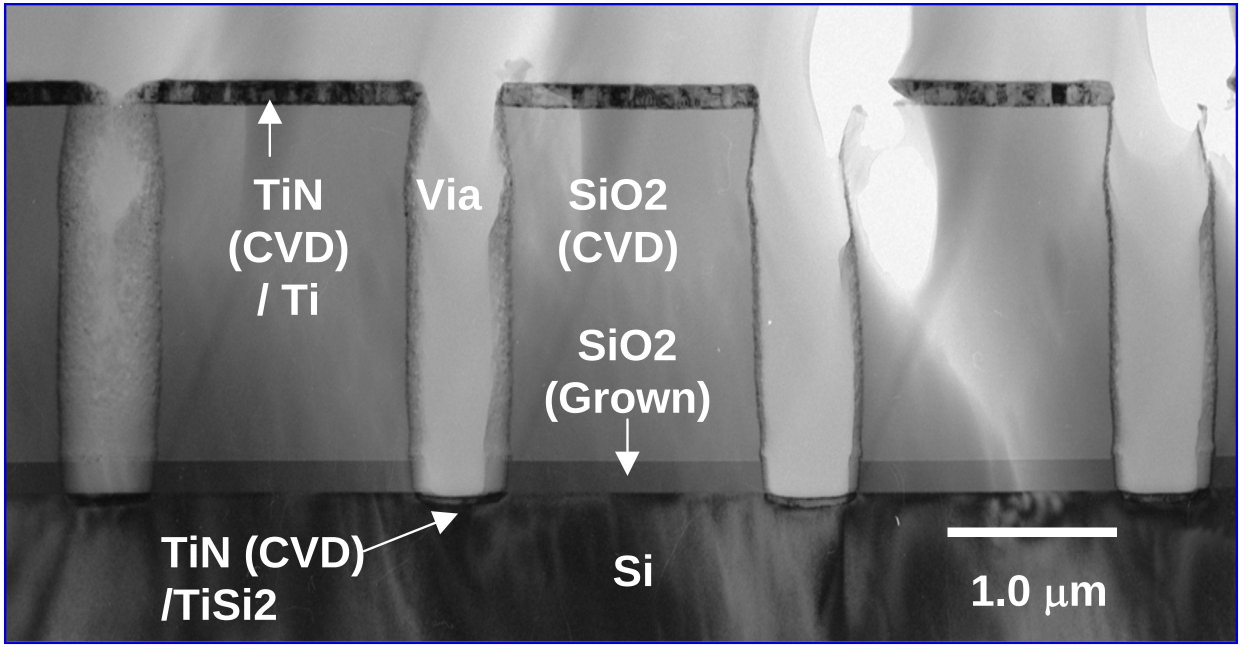


ADF STEM Image

EELS Spectra

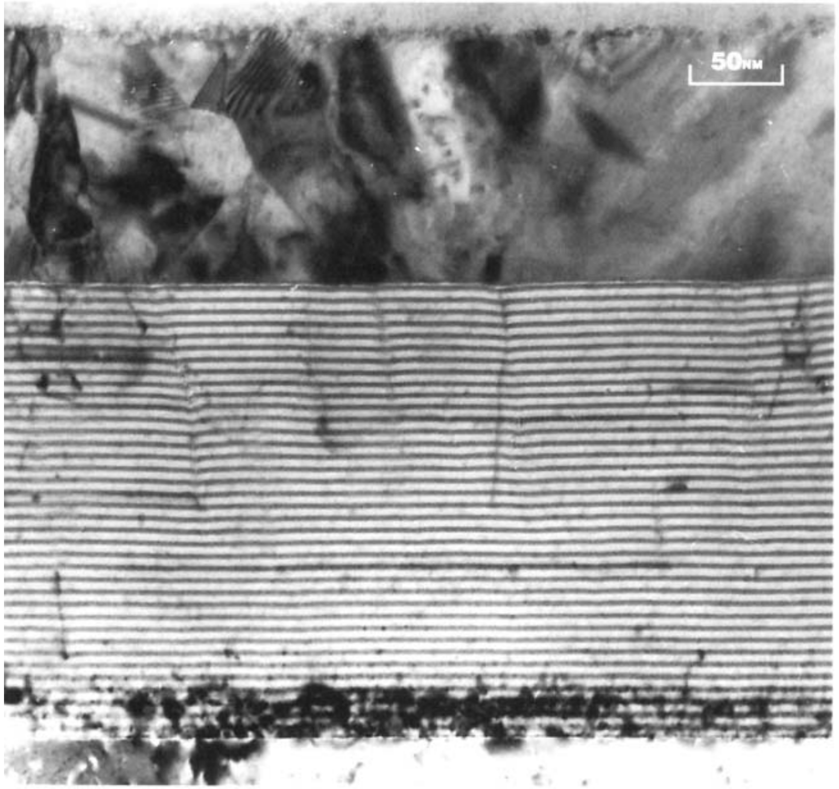
Courtesy of Dr. Gerd Duscher and Julia Luck, ONRL

Ion Milled XTEM of Ti-Si Optoelectronic



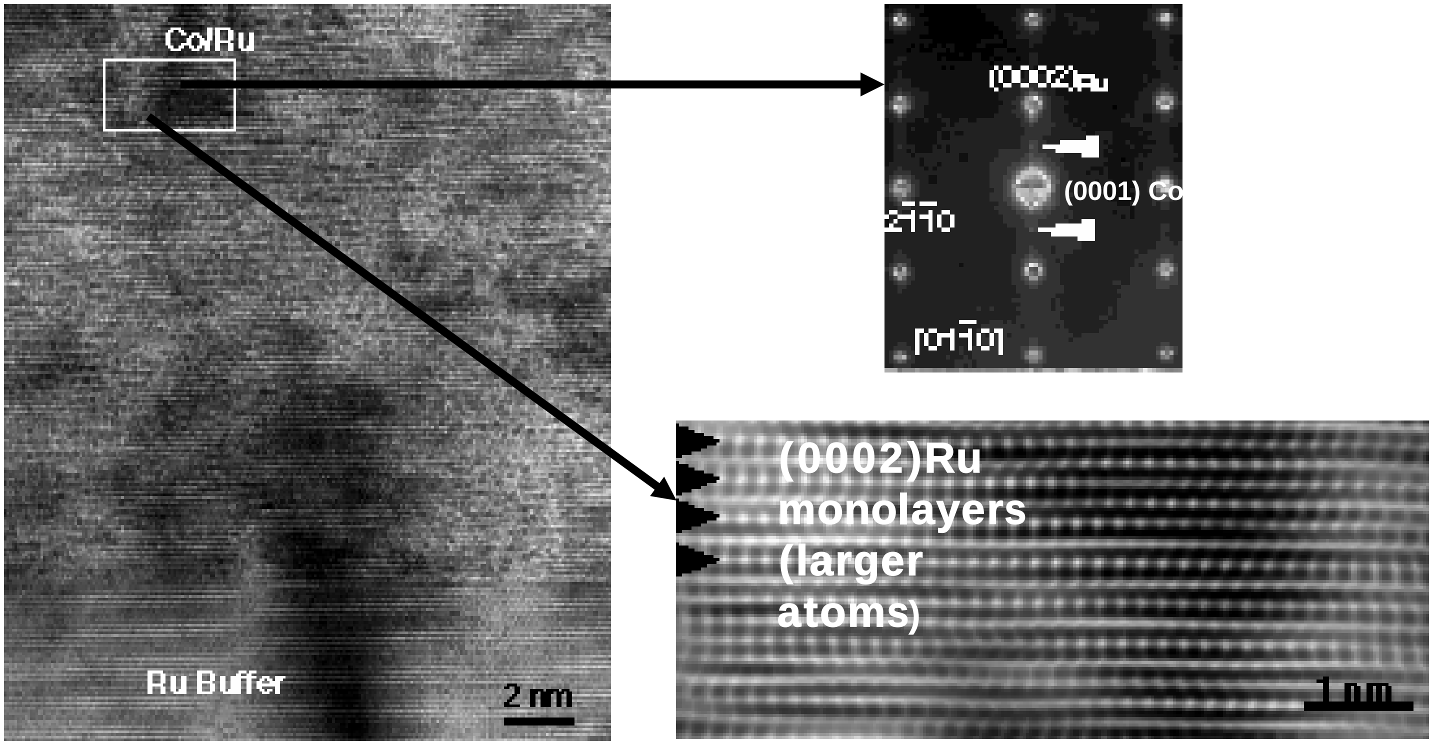
Courtesy of Ivan Petrov and Young Kim, University of Illinois

Ion Milled X-TEM Sample



Polycrystalline copper film on epitaxial $Ti_{0.5}W_{0.5}$ N/TiN superlattice on MgO (001)

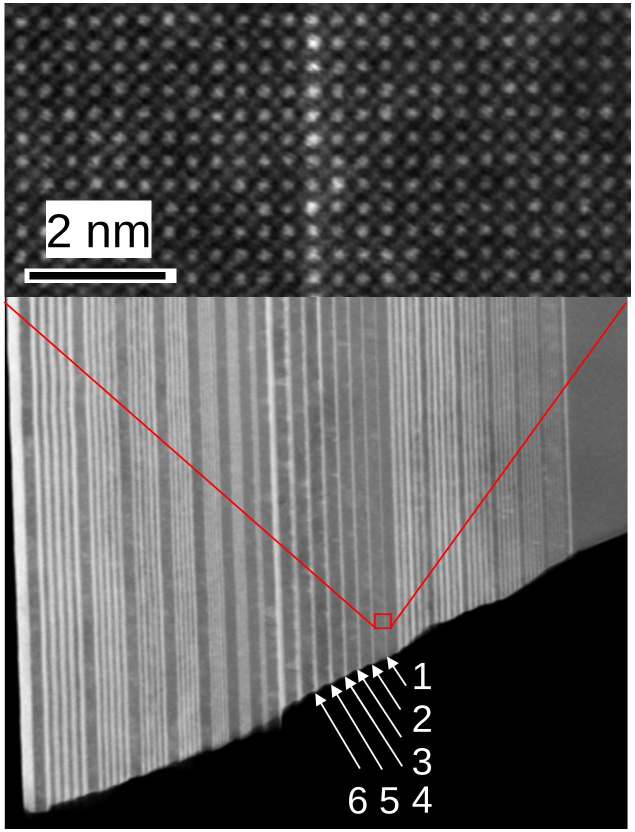
Ion Milled XTEM of Co/Ru Lattices



HREM image showing alternating monolayers of Co and Ru within the Co / Ru ordered superlattice

Courtesy by Kazuhiro Hono & D. H. Ping, National Research Institute for Metals

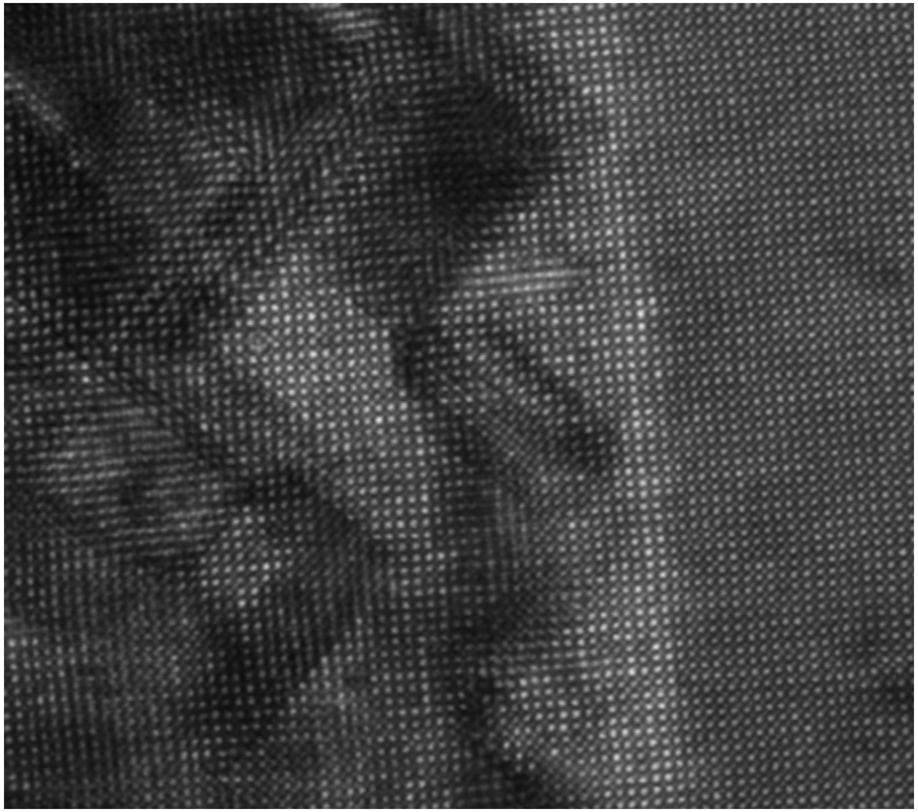
Imaging of LaTiO3 Multilayers



- Wedge polishing, followed by IBE at low incident angle, low ion energy and low temperature
- $LaTiO_3$ multilayers in $SrTiO_3$ imaged in a FE TEM
- Material (in box) thin enough for ADF lattice imaging over 0.4 μm field of view

Courtesy of D. A. Muller, J. Grazul, A. Ohtomo, H. Hwang, Bell Labs, Lucent Technologies, NJ (USA)

ADF Imaging of LaTiO3 / SrTiO3 Interface



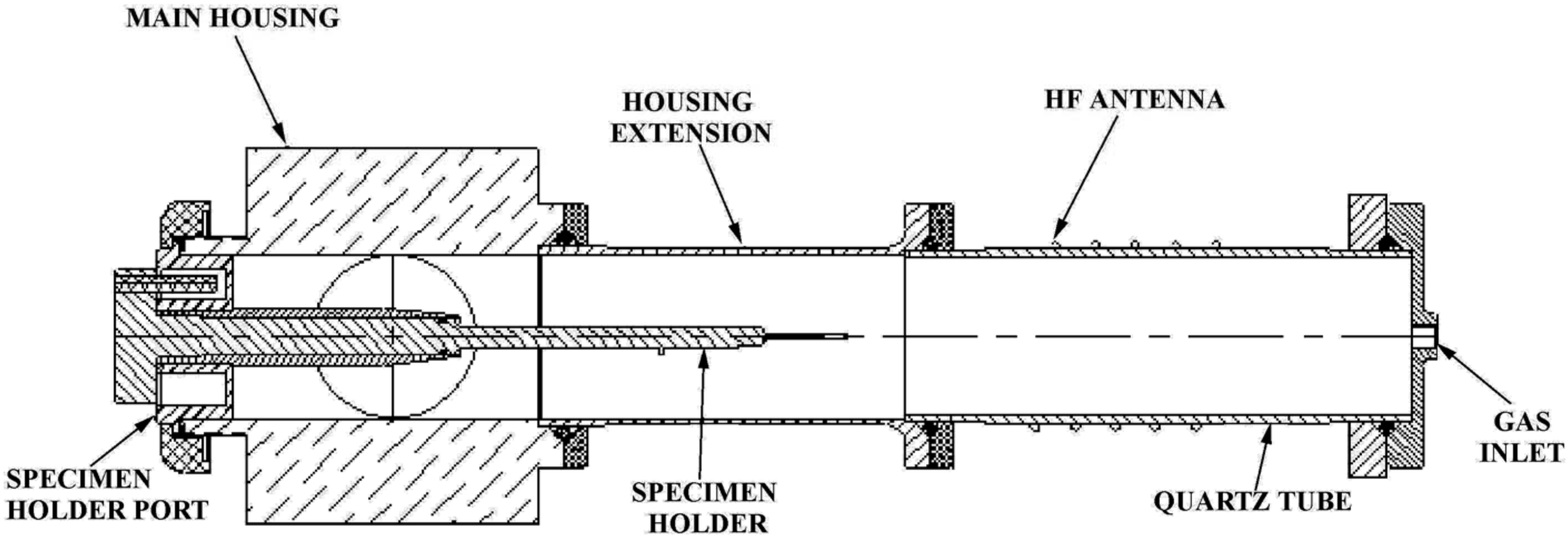
- Imaged in a FE TEM
- 1K X 1K image, 128 μs / pixel
- Gaussian smoothed
- La brighter (left)
- Sr darker (right)
- Resolution of Sr vs. Ti is 1.9 Angstroms

Plasma Cleaning

- Theory
  - Chemical reaction (oxygen plasma)
    - Plasma disassociates oxygen
    - Disassociated oxygen reacts with hydrocarbon on specimen holder to form CO, CO_2, and H_2O
    - These species are pumped away

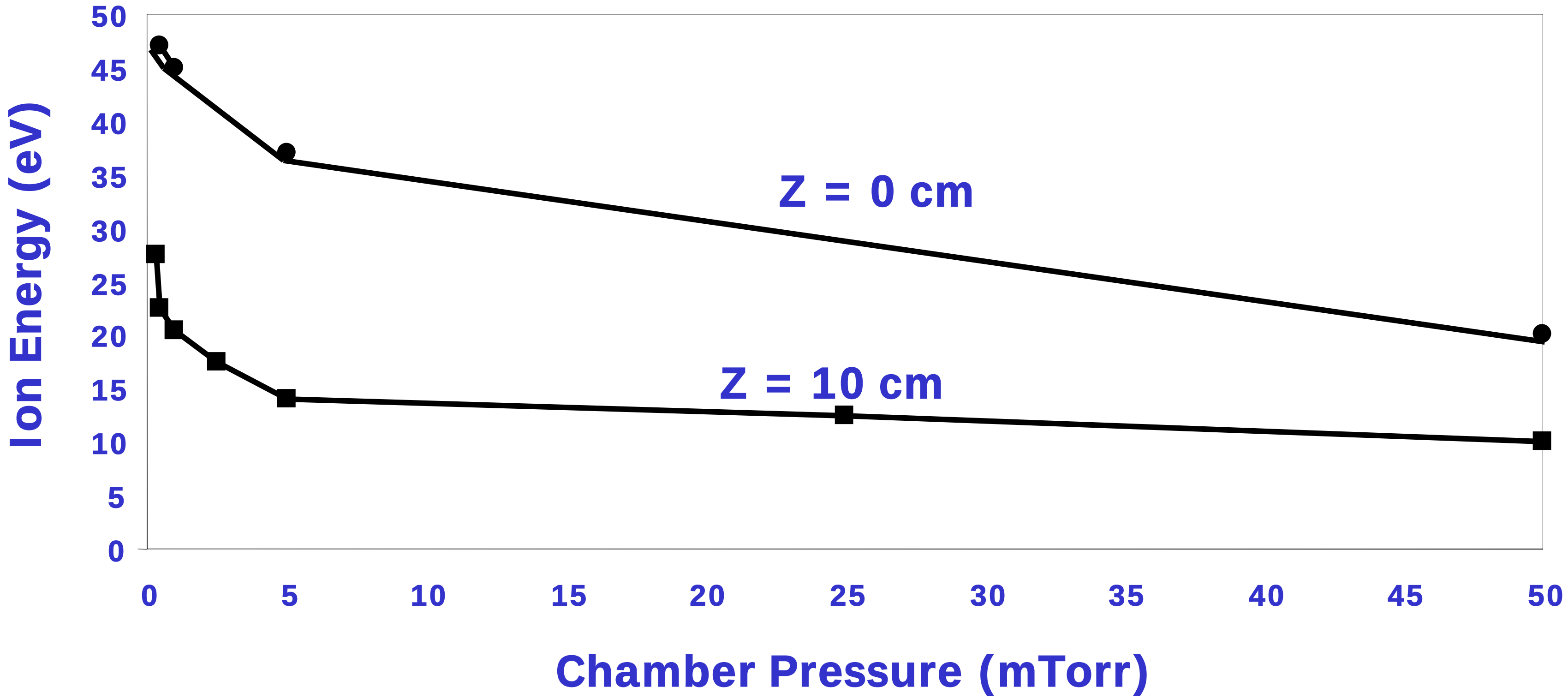
Design

Inductively Coupled HF Plasma

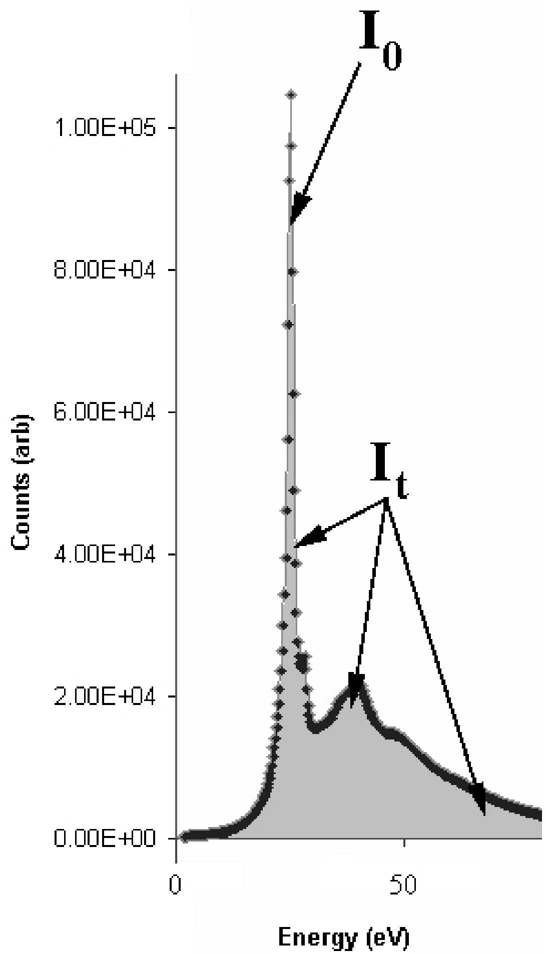


Plasma Chamber

Ion Impingement Energies

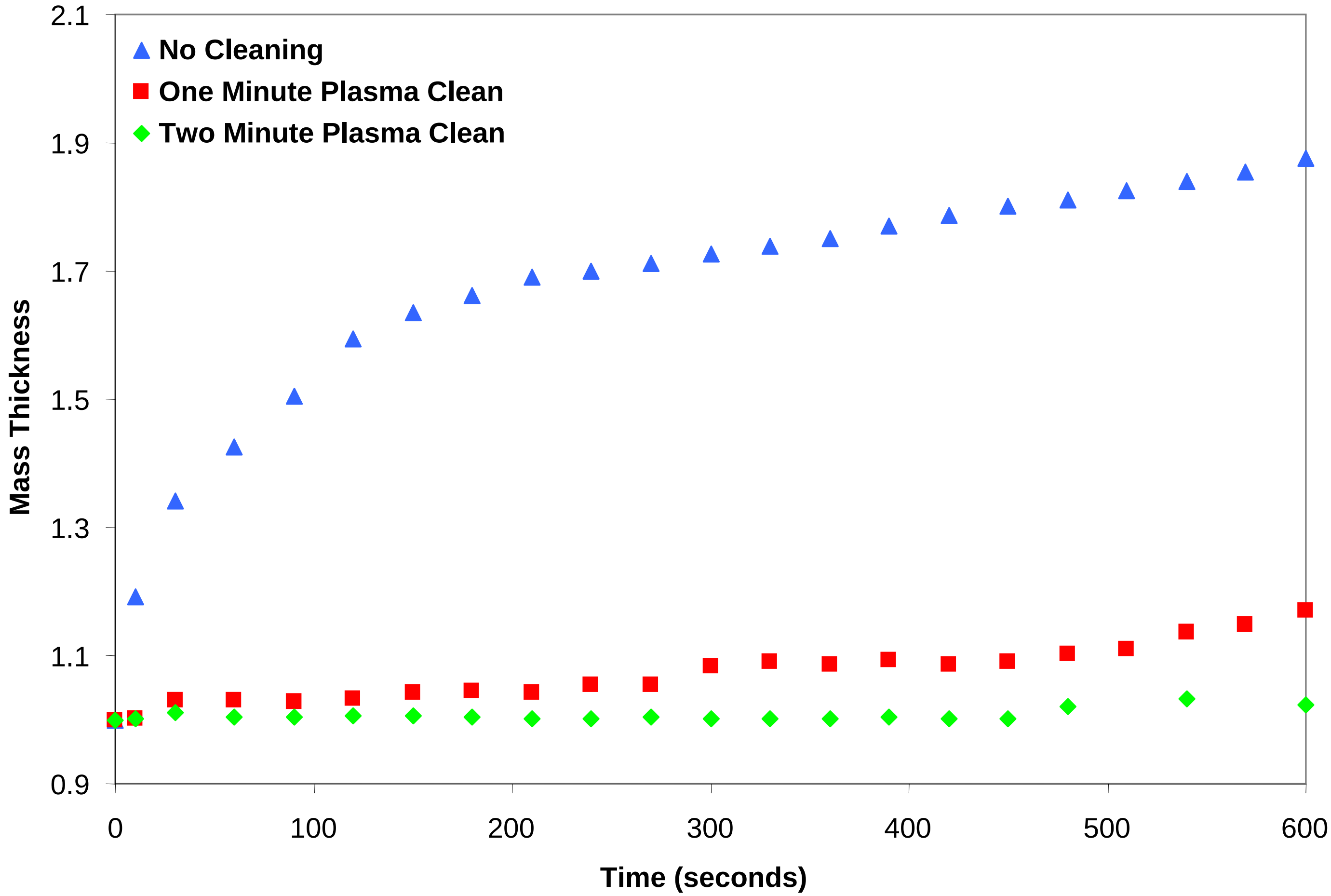


EELS for Contamination Thickness Determination

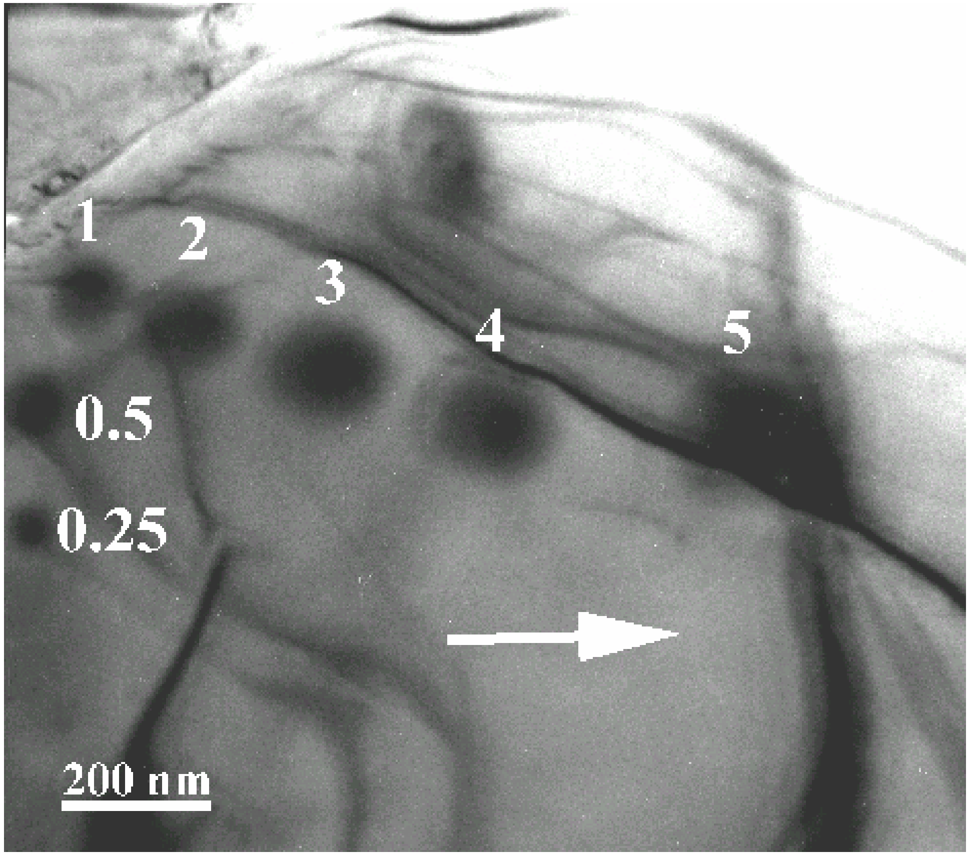


- I_0 - Intensity Under Zero Loss Peak
- I_t - Total Intensity in Spectrum
- $t = l \ln(I_t/I_0)$
- t/l - Mass Thickness

Contamination Thickness vs. Time SrTiO3



Plasma Cleaning of 304 Stainless Steel



- 1 – Minute
- 2 – Minutes
- 3 – Minutes
- 4 – Minutes
- 5 – Minutes
- Arrow
  - After plasma cleaning
  - 5 minute spot

Concluding Remarks

- Integrated approach using instruments in series
- Plan view or XTEM specimens ready for transfer to and analysis by FE TEM
- Resulting electron transparent regions are free of artifacts and contamination