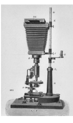
Transmission Electron Microscopy

HOW DID WE GET HERE?



First UV Microscope

For 300 years, the observation with microscopes was limited to objects bigger than the wavelength of the visible light. In 1904, Pr. August Köhler (Germany-1866) broke this barrier by introducing the first UV microscope while working for Carl Zeiss AG.

First TEM Prototype

In 1931, Ernst August Friedrich Ruska (right), (Germany-1906) collaborated with Max Knoll (left) at the Technical University of Berlin to create the first electron microscope then helped Siemens AG release the first commercial TEM in 1939. He received the Nobel prize in 1986 for his work on electron optics.

1673

1904

1924

1931

1997

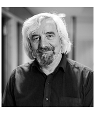
First Microscope

Microscopes had been around since 1595 but Antonie van Leeuwenhoek (Netherlands-1632) highly successful as a merchant is credited for developing the first practical microscope in 1673 to fulfill his passion for nature observation.

Moving Electron Wavelength

In his 1924 thesis, Louis de Broglie (France-1892) formalized the wave nature of the electron. His theory, proven in 1927, was rewarded by the 1929 Nobel prize. Knowing the wavelength of the electron (~1nm) would be the first step towards the development of electron microscopes.

Aberration-Corrected TEM



Following several years of research in Heidelberg, Maximilian Haider (Austria-1950) presented in 1997, with his colleagues Knut Urban and Harald Rose, the first pictures taken with a spherical aberration C_s-corrected TEM allowing the development of sub-ångström resolution TEM.

HOW DOES IT WORK?

Technical Background

There are only a handful of technologies that image the location of individual atoms which include atom probe tomography (APT), scanning tunneling microscopy (STM) and transmission electron microscopy (TEM). TEM is the most versatile tool that can deliver images and high quality elemental analysis with atomic precision. The TEM ecosystem is vast with customized sample holders and additional detectors all aligned to push the limit of resolution and increase the level of high technology. Join us as we highlight the key components in a modern TEM.

Microscopy uses small particles/waves, such as photons or electrons, to image larger objects. Light microscopes have benefited from brighter sources, polarizers and filters to make the beam as uniform as possible when it interacts with a specimen. In an analogous way, intense electron beams are aligned, filtered and collimated to generate high resolution images. The product of decades of development has pushed the resolution from light microscopes range of microns (10^{-6} m) to TEM range of Angstroms (10^{-10} m).

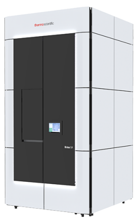


Figure 1. Modern TEM tools are easily larger than an average adult in height. Image from Thermo Scientific

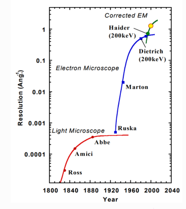


Figure 2. Yearly Limit on Microscope Resolution in Å (Angstrom 10^{-10} m). Image from Appalachian State University 2016

SEM VS TEM

Analogous to light microscopes, there are two orientations for the electron beam, reflection and transmission. Scanning electron microscopes (SEM) use reflected electron beams to gather information about the samples. The resolution of the SEM is on the order of 10^{-9} m which is less than that of TEM, but combined with the facile sample prep it has proven to be a cost-effective tool for measurements of relatively large features. For SEM, the sample needs only to be mildly compatible with vacuum and slightly conducting to be imaged. For TEM, the sample must be sectioned and thinned to under 10^{-7} m for imaging. This sample preparation is accomplished using a Focused Ion Beam (FIB). FIB preparation has kept pace with TEM's as the requirements continue to grow stricter for higher resolution.

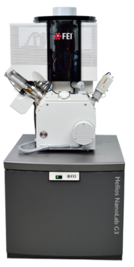


Figure 3. Focused Ion Beam (FIB) equipment used for TEM preparation. Image from Thermo Scientific.

Sample Preparation

FIB or other ion milling techniques are essential to TEM sample preparation. The challenge faced by engineers is how to repeatably segment a sample with extreme precision. A sharp blade mounted to precision motion control is the instrument known as a microtome. The most advanced microtome systems, ultramicrotome, can segment samples on the order of 10^{-7} m, which is the range for TEM sampling. Microtome is a good approach when sectioning metals but it is much more difficult to section brittle materials and soft materials. This is where an FIB tool excels.

FIB tools perform 3 essential functions for TEM prep, imaging, cutting and depositing material. When performing any kind of precision work there is usually a means to magnify one's vision. The FIB tool has a built in SEM which allows for more precise sample handling. To segment materials, the FIB uses a highly charged beam of Ga ions. This works like a microscopic version of a sand blaster to remove paint. Much like the sand blaster, the voltage of the Ga beam can be controlled to increase or decrese the kintectic energy of the Ga beam. This allows for faster coarse cuts and slower precision cuts. As can be expected from the sand blaster comparision, higher power beams cause more damage to the sample surface. So the optimization is in preparing the highest quality sample without excessive instrument time.

There are two common configurations for preparing materials for TEM, cross section and plan view. Cross sections are used to visualize deposited layer thickness and lateral uniformity. Cross sections are prepared by digging large trenches in the sample to expose the layers of interest. Those regions are then removed from the sample in a process known as lift out. Lift out is where the newly exposed sliver of material known as the lamella, is fastened to a transfer arm by depositing carbon and is then cut away from the rest of the sample. After lift out, the sample can be reinforced by depositing metal to provide rigid support around the area to be imaged. After reinforcement, the lamella can finally be thinned then affixed to a TEM grid for imaging.

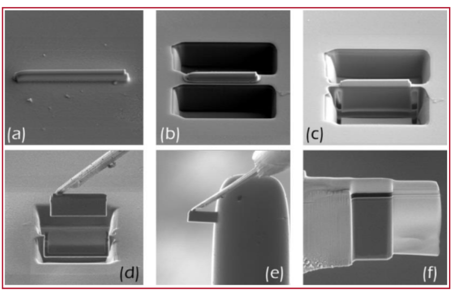


Figure 4. Preparation of TEM lamella. A) Depositing Pt, B) Trench milling, C) U-cut, D) Lift-out, E) Welding to TEM grid, F) Lamella thinning. Image from Regina Seidl, 2016

Plan view preparation is distinct from cross section views because in this case the surface is of most interest. This sample preparation is akin to exfoliating the top most layer of material. The challenge is to make sure that the top most layer remains structurally sound unlike most materials which scroll after exfoliation. By tilting the sample to nearly parallel with the ion beam, the top layer of material can be removed from the sample. As would be expected, because of the cutting angle the process has to be done using lower beam currents so it generally takes more time and precision to prepare a sample for plan view than for cross sectioning.

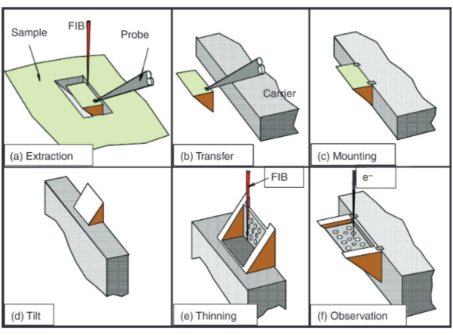
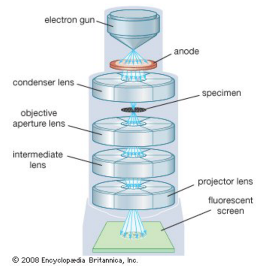


Figure 5. Method to prepare plan-view lamella for TEM. MRS Bulletin, Volume 32, May 2007

TEM Imaging Details

Once a sample has been appropriately thinned, it can be imaged by the TEM. The TEM contains collections of magnets to focus the electron beam. Light can be bent by interacting with lenses to change its direction. Electron beams are more easily manipulated by magnetic fields because of the Lorentz force which curves traveling electrons in magnetic fields. Magnets consist of a north and south end collectively referred to as a dipole. Arranging magnets symmetrically around the electron beams leads to additional common configurations. 2 dipoles create a quadrupole, 3 dipoles create a sextupole and 4 dipoles form an octupole. Lenses in the TEM are formed by 1 or 2 sets of quadrupoles and beam collimator are formed by sextupoles. Referring to the image, each lens will contain quadrupoles for manipulating the beam and sextupoles for keeping the beam vertically aligned. It is clear that there are numerous components that need to work flawlessly in unison to properly control the electron beam.



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HR-TEMs

Any electron microscope that is in the transmission configuration can be called a TEM. When a TEM contains phase contrast and can image down to the atomic length scale at 10^{-9} m, then it is referred to as an HR-TEM. Most often instruments within the past 20 years will be HR-TEM but in more recent years a new version of the TEM with even higher resolution has emerged, the aberration corrected TEM (C_s-TEM).

Spherical Aberration:

Aberration usually refers to the optics concept that path length differences, when light passes through lensing, can create a loss in information. To form a sharp image, all changes in the monochromatic beam should be from sample interactions. In real life, lenses are not perfect so they can affect the light passing through them. Consider a fun house mirror, you yourself are not highly distorted but moving from one mirror to the next the imperfections in the glass will warp your image. Lenses can have the same effect albeit in a much more subtle way since most lenses' designers aim for perfection. When electrons enter the magnetic field lenses at the edge of the field, they take a slightly longer path than electrons entering at the center of the field. This path difference is what makes the image blurry and the main point that the aberration correction addresses.

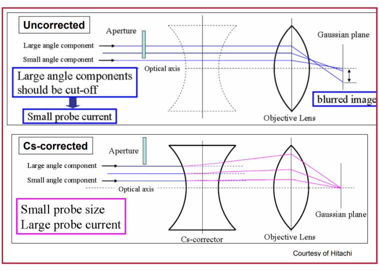

Figure 7. Ray diagram depicting high resolution HR-TEM and spherical aberration correction C_s-TEM. Image from Appalachian State University 2016

Spherical Aberration Corrected TEM (C_s-TEM)

At the end of this journey in lenses and collimators, a final image is produced. The result of the additional aberration correction components is a higher resolution image that more accurately depicts the atomic locations. This is demonstrated in the figure where the corrected image has additional visible diffraction planes and sharper resolution in the atomic locations. The Ga and N atoms are now also more clearly resolved in their orientation relative to each other.

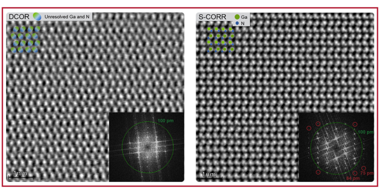

Figure 8. Difference between non corrected HR-TEM (Left image) and the aberration corrected C_s-TEM (Right image). Image from Thermo Scientific

Technologies for Composition Analysis

Scanning Tunneling Electron Microscopy (STEM)

Now for many engineers the location of atoms is good but insufficient. For elemental analysis there are 3 commonly used techniques with TEM. The first option is STEM, which is a Z contrast mode that uses inelastically scattered electrons along with detectors that are located far from the electron beam to be able to resolve the subtle differences in proton density between elements. STEM mode provides excellent contrast between light and heavy elements because the difference in nucleus size is the source of the contrast. This makes STEM the ideal mode for imaging thin interfaces that are sub nanometer in thickness. STEM works particularly well for thin samples, 40 to 60 * 10^{-9} m.

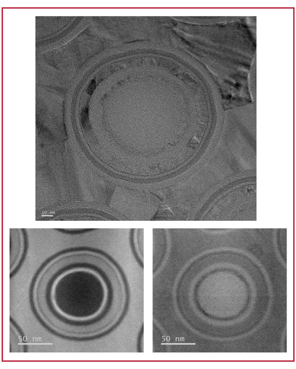

Figure 9. Example TEM image followed by two STEM images in dark and bright field modes. Elemental data is shown in figure 10. Images from Outermost Technology

Even though STEM is a powerful technique there are still additional ways to gain more information about a sample using the characteristic x-rays that the elements emit.

Energy Dispersive x-ray Spectroscopy (EDS)

EDS is an attachment to both an SEM and TEM that allows for the capture of x-rays emitted from the electron beam's interaction with the sample. These x-rays energies can be used to identify the particular atoms in the sample. There are some overlapping energies so the identification of elements is not 100% guaranteed. Overall, the ease of acquisition of EDS has led to its widespread use for elemental analysis. An example EDS shows fine changes in material in the different layers that comprise 3D flash memory. An important point is that EDS works better with thicker samples so 200 * 10^{-9} m gives much more counts than thin samples. Luckily there are dual EDS detectors which can increase the overall signal intensity by a factor of 10.

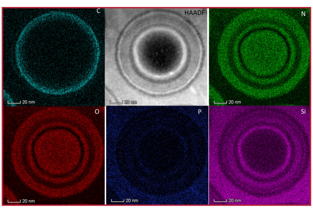

Figure 10. Showing changes in atomic concentration across atomic length scales in the inner section of 3D-Nand memory using C_s-TEM.

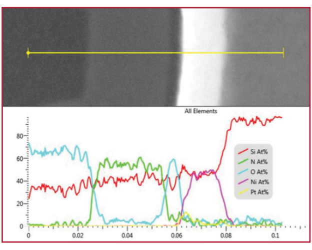

Figure 11. High resolution EDS line scan that shows the elemental concentration of a transistor gate structure. Image from Outermost Technology

The basis for contrast in STEM can be used for elemental analysis as a technique called EELS.

Electron Energy Loss Spectroscopy (EELS)

This brings us to the final elemental analysis technique EELS. EELS is the quantitative version of STEM, where specific energy peaks identify each element. Unlike in EDS, multiple peak locations are used to identify each element so it becomes much more improbably that an element will be misidentified. The drawback of EELS is that only a few elements can be scanned at the same time, which makes EELS the best choice for identifying known elements in a sample while EDS can be used to identify unexpected elemental impurities. There are also dual EELS detectors to further increase the signal intensity and improve the speed of the technique. EELS is the appropriate choice for very thin samples 40 * 10^{-9} m.

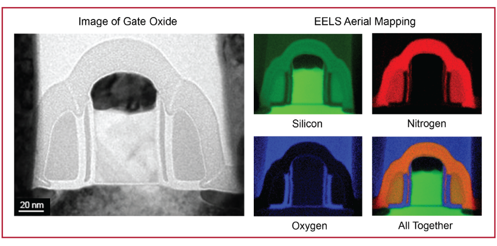

Figure 12. 10 nm scale bar demonstrating the strength of EELS for analysis of semiconductor gate structures with HR-TEM.

SUMMARY

Overall, we hope that you enjoyed our review of TEM and have a better appreciation for the usefulness of STEM, EDS and EELS. Outermost Technology is highly skilled in FIB and ion mill preparation which is why we consistently deliver high quality TEM images across a variety of materials. Outermost Technology has proven expertise in TEM imaging for semiconductor devices as shown by the 3D NAND images for both Si and Ga based devices. Please consider Outermost Technology for your next TEM job.

CUSTOMER SERVICE WORKFLOW

STEP 1
Consult with Our Technologists

STEP 2
Provide Your Samples

STEP 3
Receive In-Depth Report

STEP 4
Debrief with Our Technologists

WHY CHOOSE OUTERMOST TECHNOLOGY FOR TEM?

1. Access to a variety of TEM's. HR-TEM's for routine samples that need more resolution than SEM
2. Access to top of the line C_s-TEM's for very narrow features imaging
3. Access to twin EDS and twin EELS detectors with higher resolution and signal intensity than single detectors
4. Benefit from excellent FIB preparation skills applied to many semiconductor, metals and soft materials
5. Benefit from competitive pricing and in-depth analysis report

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*Valid until May 15 with a minimum quantity of 3 samples

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