

The Value of S/TEM: Matching Solutions, Applications, and Economics

Dominique Hubert,
FEI Company, Eindhoven, The Netherlands
dhubert@nl.feico.com

Introduction

A new generation of scanning/transmission electron microscopes (S/TEM) offers a broad range of capabilities and dramatic improvements in usability, challenging the conventional perception of these techniques as needlessly difficult and expensive. Improvements in usability address both the operation of the instrument and the interpretation of results, while the cost of the analysis must be evaluated relative to the value of the information it provides—a value that has appreciated significantly with the burgeoning growth of the nanotechnology sector. A million dollar instrument is a bargain if it solves a multi-million dollar problem. Moreover, the broad range of capabilities available in current generation instruments makes careful matching of the cost of the capability with the potential value of the solution the key to sound economic choices. This article reviews the factors that typically drive TEM costs and provides examples of valuable matches in a number of applications.

Cost/Value of Ownership

Cost-of-ownership (CoO) is a familiar concept referring to the total cost of a particular piece of equipment. It includes not only the initial acquisition cost but also all costs associated with operation. Likewise, value-of-ownership (VoO) refers to the total economic value of the benefits received in exchange. In its simplest form, VoO for a TEM is simply the value of the information derived from each analysis. A more detailed analysis will show that VoO is also influenced by other factors including productivity, throughput, and time to “smart” data—time to the exact information you need. Well designed and engineered hardware and optimized software that combine to deliver high levels of automation and ease-of-use also contribute to VoO. Finally, functional modularity that anticipates future developments and expanding capabilities is essential to maintaining VoO over the lifetime of the system.

Background

S/TEM is not new. The first electron microscope, built by Ernst Ruska at the University of Berlin in 1931, was a TEM, and the first STEM was demonstrated in 1969. However, the overwhelming majority of electron microscopes built and installed to date have been scanning electron microscopes (SEM), which have been not only less expensive but generally easier to use than their S/TEM brethren. Over the same period of time S/TEM developed principally as a research technique with its primary emphasis on pushing the limits of imaging and analytical capability, often at the expense of usability. The recent growth in demand generated by nanotechnology applications is now driving a renaissance in S/TEM, and the latest generation of instruments combines sub-Ångstrom resolution with dramatic improvements in convenience and usability. Previously difficult setup and operational procedures are now completely automated, yielding faster more reliable results and eliminating the need for expert operators. Equally important, advances in imaging technology have eliminated much of the difficulty previously associated with interpreting and understanding TEM images.

Although TEM and SEM are both electron microscopies, they are fundamentally different in the way they form images. TEMs focus electrons that have been transmitted through the sample into a real optical image. SEMs construct a virtual image from signals emitted when a finely focused beam of electrons scans the surface of a bulk specimen. SEM resolution is ultimately limited to about one nanometer by the scattering of beam electrons inside the bulk specimen.

STEM combines aspects of both SEM and TEM. As in SEM, it scans a finely focused beam of electrons over the sample surface. As in TEM, the sample is thin and the virtual image is constructed from transmitted electrons. The thin specimen greatly reduces beam spreading so that STEM resolution is limited primarily by the size of the electron probe. STEM may be performed on either an SEM or a TEM platform. TEMs generally offer better STEM performance because they operate at higher accelerating voltage, which permits the formation of smaller electron probes and increases electron transmission relative to sample thickness.

Cost Drivers

The final selection of any S/TEM must be made relative to the criteria that are important for a given application. In high volume manufacturing applications, the most important consideration may be the cost per analysis, while in a basic research application it may be highest possible resolution regardless of cost. Many aspects of TEM design and configuration drive the price and CoO of a TEM, and in some cases they may trade off against each other. For example, a 300 kV instrument is more expensive to acquire but that acquisition cost may be offset by allowing thicker samples that are less expensive to prepare, or more flexibility for different sample classes, or simply more detailed and critical information. An important consideration in eliminating unnecessary costs is design modularity—the ability to add capability to an existing platform—to address anticipated needs or incorporate unanticipated new technologies.

Accelerating Voltage for an S/TEM ranges typically between 100 and 300 kV. Electron optics generally offers better optical performance at higher voltages. Equally important, higher energy beam electrons will penetrate thicker samples—the likelihood of a beam electron being scattered by the sample decreases with increasing electron energy. Although scattering is desirable because it creates the contrast necessary for image formation, too much scattering degrades the image and eventually prevents transmission altogether. In the ideal case, most electrons that do scatter do so only once as they pass through the sample. Each scattered electron then contains specific information about that scattering event alone. Practical considerations regarding the choice of accelerating voltage include the anticipated sample thickness and sample composition. From a cost point of view, it is important to look at the accelerating voltage as an additional experimental parameter. A wide range of voltages permits analyses that are tuned to specific applications. Higher voltages provide higher resolution, better sample penetration, and higher currents for better analytics. Higher voltage can also help to improve sample stability by reducing ionization processes that degrade the sample. On the other hand, lowering the voltage improves contrast and reduces damage induced by knock-on processes. This flexibility is important in nanotechnology with its wide range of sample classes.

Electron Sources may be thermionic or field emission. Thermionic sources may be tungsten or LaB₆. Tungsten is least expensive. LaB₆ provides greater currents and longer lifetime at an increased cost. Field emission sources require much better vacuum and are significantly more expensive, but they are brighter (current density per unit solid angle), have narrower energy distributions, have smaller emitting regions, greater spatial coherence, and last longer. The best choice depends on application. For X-ray microanalysis, where signal strength is a direct function of beam current, total beam current may be most important. High resolution imaging using phase contrast requires good spatial coherence. Energy loss spectrometry benefits from narrow energy spread.

Objective Lens Configuration may be optimized for a specific purpose. In general, a smaller bore and gap will improve lens performance. However, the sample in a TEM must be located within the gap, so lens performance must be traded off against sample size and manipulation capabilities. Likewise, the lens configuration controls the ability to position other detectors, such as an X-ray detector, close to the sample.

Aberration Correctors have recently become available. In optical systems the term aberration refers to characteristics of the lenses that interfere with the system's ability to bring all of the electrons emanating from a point in the object to perfect focus at a corresponding point in the image. The most important aberration in electron optical systems is spherical aberration, which causes electrons distant from the optical axis to focus closer to the lens than electrons close to the axis. As a result, electrons from a point in the object form a disk in the image, and the disks from adjacent points overlap and blur the image. Spherical aberration has long been the limiting factor in S/TEM resolution.

Until the advent of spherical aberration correctors, two different types of resolution were needed to characterize TEM performance: image resolution (or point resolution) and information limit. Image resolution referred to the usable resolution that could be observed directly in an image and information limit referred to the finest spatial resolution that could be transferred by the optical system. While the image resolution is limited primarily by spherical aberration, the information limit is controlled by other factors including: chromatic aberration (due to the beam electron energy distribution), specimen drift and vibration (mechanical or EM fields), and certain characteristics of the source and the detector.

Spherical aberration correction eliminates a major cause of confusion and frustration among users and consumers of TEM results. An uncorrected image may not be at all what it appears to be. Image details at the spatial scale between the image resolution and the information limit cannot be directly interpreted. In fact, detail at this scale exhibits complete phase reversals in contrast as a function of focus conditions. Thus, we may hear about the research lab's new TEM with a sub-Ångstrom information limit only to learn that the best resolution we can really expect to see is two or three Ångstroms. Worse still, we may observe some detail that is just what we've been looking for, only to find that it's not what it looks like. With aberration correctors, what you see is what you get.

Chromatic aberration refers to the dependence of lens power on the energy of the electron. Lower energy electrons focus closer to the lens than higher energy electrons. There is always some energy distribution among beam electrons, and even if there were not, there would be after the beam passes through the sample. A monochro-

mator can reduce chromatic aberration by filtering beam electrons to narrow the energy distribution. However, in so doing it reduces the current available in the beam. Correctors for chromatic aberration are in development but not yet commercially available.

A final consideration with regard to aberration correction is the electrical and mechanical stability of the system. Correctors may be retrofitted to an existing column, or added to a new column, but if it has not been designed for correctors, the result can be disappointing. For example, sufficient mechanical stability may require a larger diameter column, clearly not a change that can be implemented retroactively.

Sample preparation is an important cost consideration in any TEM operation. In order to transmit electrons, samples must be very thin, typically less than 100 nm, and thinner is almost always better. Conventional sample preparation techniques involve careful polishing, perhaps with final thinning done in a broad beam ion mill. More recently, focused ion beam (FIB) milling has provided a means to extract location-specific samples, such as a previously detected defect or a particular circuit structure from an in-process silicon wafer. After initial extraction, continued milling thins the sample to electron transparency. Automated FIB systems configured specifically for sample preparation can reduce preparation time from days to minutes. Their unique ability to extract a sample at a precise location is invaluable in semiconductor manufacturing applications.

Biological samples are often best viewed under cryogenic conditions. Of course, the TEM must be capable of maintaining the sample in its frozen state, but consistent execution of the initial freezing preparation is equally important. This process, known as vitrification, freezes the sample so rapidly that ice crystals, which would destroy delicate biological structures, cannot form. Robotic vitrification systems are available that automate the process for improved repeatability and reliability. Vitrification provides artifact-free samples in near-natural state.

Tomography provides a means to acquire information in the third dimension, the direction of the beam. Conventional TEM images are two dimensional projections of three dimensional objects. Tomography reconstructs a 3D model of the sample from multiple 2D images acquired from different perspectives. Automated image acquisition and reconstruction make tomography fast and easy.

Analytical systems provide elemental and chemical information about the sample. Two of the most important are X-ray and Electron Energy Loss Spectrometry (EELS). Both techniques are optimally performed in STEM mode. X-ray spectrometry measures the energy of X-rays emitted by sample atoms as they relax after being ionized by beam electrons. The energy of each X-ray indicates the element from which it originated. EELS examines the energy of transmitted beam electrons that have interacted with sample atoms. The amount of energy lost by the electron through that interaction carries specific information about the identity and chemical state of the sample atom.

Application Examples

The discussion that follows describes applications in three different areas—nanobiology, nanoresearch, and nanoelectronics—in each case offering examples at three levels of system capability. **Basic** instruments offer full TEM capability and may include STEM. They typically provide imaging resolution of about a half nanometer (4–5

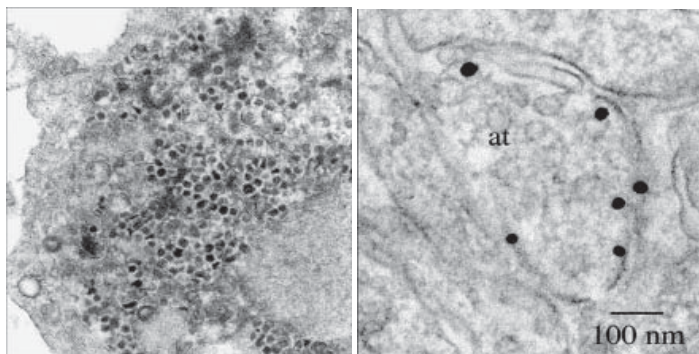


Figure 1

figure 2

Ångstroms). They are often customized/optimized for dedicated use in routine commercial or industrial applications. **Research** grade instruments are the next level up. They offer imaging resolution of 2-3 Å, roughly a quarter nanometer. They emphasize flexibility and capability and may be configured to optimize some particular technique, such as X-ray analysis, sample size and tilt, or cryo operation. The final category is **Advanced** S/TEMs. These instruments typically include aberration corrected optics and deliver sub-Ångstrom imaging resolution.

Nanobiology

Biological samples are usually composed of lighter elements and contain substantial amounts of water. The limited range of elements generates little contrast, and the water, if introduced into the vacuum environment of the TEM, would vaporize rapidly, potentially damaging both the sample and the instrument. Biological samples may be “fixed” to allow them to tolerate the vacuum, and stained with heavy elements to enhance image contrast. Alternatively, the samples may be frozen to preserve (as nearly as possible) their natural state and prevent vaporization. Immunologically targeted markers, such as gold or silver nanoparticles and quantum dots, may be used to identify specific structures or molecules.

Fig 1. **Basic TEM in NanoBiology** – Clinical Pathology.

Medical researchers and clinicians have long used TEM for routine pathology and diagnosis. In this example TEM was used to detect the presence of SARS virus in a patient biopsy. For this application ease-of-use and the ability to acquire and store data in digital format are important considerations. (Courtesy: Queen Elisabeth II Hospital Hong Kong)

Fig 2 **Research TEM in NanoBiology** – Protein Localization

Determining the location of proteins and



Figure 3

other biomolecules relative to cellular structures can often provide vital insights into the function of both the cellular structure and the molecule. In this example a protein, dopamine transporter, has been immunologically labeled with gold particles (black dots). It is clear from the image that the labeled molecules are localized on the cytoplasmic side of the axon terminal (at) membrane. Previous studies using more conventional staining and amplification techniques (immunoperoxidase) had been able to localize the protein within the terminal but not demonstrate the close association with the membrane. (Image courtesy of Hong Yi, Emory University and Jan Leunissen, Arion,¹).

Fig 3. **Advanced TEM in NanoBiology** - Cryo tomography

While the completion of the human genome project has made available the peptide sequence of every protein in the human body, the structure and function of only a few are known. Existing methods for structural analysis, though they offer atomic level resolution, are limited in the types and size of molecules they can analyze. TEM lacks atomic resolution in biological materials but can directly visualize the overall shape of protein molecules and large multi-molecular protein complexes. Most importantly, it can examine bio-molecules *in situ*, in their natural associations with other molecules. Figure 3 shows neurotrophin receptor p75, a membrane protein (red), and other cell proteins (grey) and gold labeled antibodies (yellow and blue). The structure of the molecule was derived using electron tomography, an advanced technique that combines many images taken from different perspectives to create a three dimensional model of the sample. (Image courtesy of Sidec Technologies²).

NanoResearch

Much of the work in the nanoresearch sector is materials research. Ultimately the properties of all materials are determined at the atomic or molecular level. Electron diffraction in the TEM can provide complete crystallographic characterization of crystalline materials. Aberration corrected TEM can image atomic-scale detail at interfaces and edges.

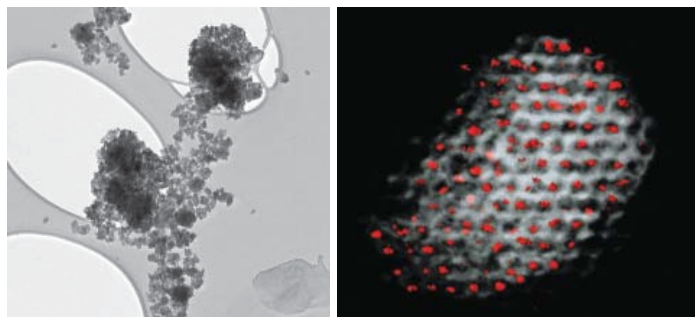


Figure 4

Figure 5

Fig 4 **Basic TEM in NanoResearch** – Particle Size Distribution

Nano-scale particles play an increasingly important role in many industrial products. Characterizing the particle size distribution is a fundamental requirement for controlling many manufacturing processes. Light microscopy is not capable of accurately measuring particles smaller than about one micrometer. Although other particle analysis techniques, such as laser scattering, can characterize particle populations, only image based analysis provides direct observation of individual particles and permits evaluation of particle shape, agglomeration, and other critical process parameters.

The particles shown in this image are pigment particles used to color paint. (Image courtesy of Dr. Kupers and Dr. Zilse, Sachtleben Chemie, Duisburg, Germany.)

Fig 5. Research TEM in NanoResearch – Catalysts

Electron tomography can reveal structure, homogeneity and particle size distribution information that are critical to the performance of catalysts, and difficult or impossible to obtain with any other method. In this example, the three dimensional capability provided by tomographic analysis makes it possible to tell whether the Ru₁₀Pt₂ nanoparticles (red) are on the surface or distributed throughout the porous silica support structure. (Tomography by T.J.V. Yates, Dr. P.A. Midgley, Cambridge University, Dr. M. Weyland, Cornell University and Dr. Lydia Laffont, Université de Picardie; Specimen provided by Prof. J.M Thomas and Dr. R. Raja Cambridge University³).

Fig 6 Advanced TEM in Nanoresearch – Catalyst Particle Surface

The catalytic activity of many catalysts occurs at the particle surface. Prior the availability of aberration correctors, delocalization and blurring caused by spherical aberration made this region difficult to image. As this image shows, aberration correction clearly reveals the atomic-scale detail of the surface. In this case, the kinks and dislocations at the surface were hypothesized to constitute the active catalytic sites. (Image courtesy of Prof. Dr. Robert Schlögl, Fritz-Haber-Institut der Max-Planck-Gesellschaft, Berlin, Germany).

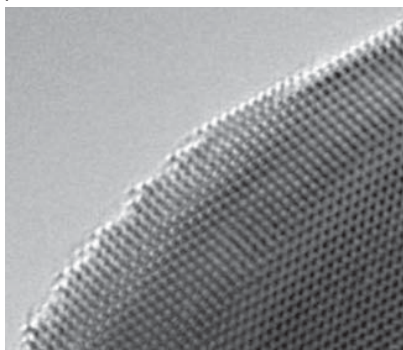


Figure 6

NanoElectronics

Resolution as Function of Use

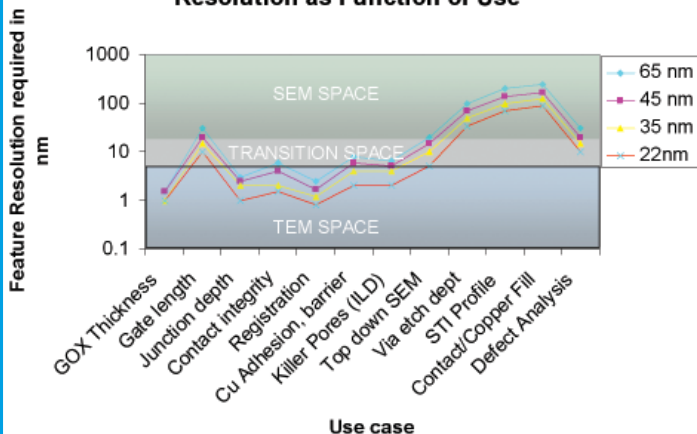


Fig 7 TEM application in semiconductor manufacturing.

Semiconductor device manufacturers have used microscopes to understand and control their manufacturing processes since the invention of the integrated circuit. As feature sizes shrank beyond the resolution capabilities of light microscopes, they were quick to embrace SEM. As Fig 7 shows, many of their critical processes are now approaching or have already exceeded the capabilities of SEM, requiring a transition to TEM for routine process control.

More than many industries, IC manufacturers clearly understand the detailed economics of their production processes. They adopt a new technology when and only when it can make a demonstrable contribution to the bottom line. In the case of TEM they have been quick to explore new techniques that maximize the efficiency and utilization of the instrument, such as automated, FIB-based sample preparation. In high volume applications automated sample preparation can reduce the cost-per-sample for TEM analysis to a level competitive with the cost of a typical SEM analysis. In at least one facility, engineers have sustained average TEM throughputs of 400 to 500 samples per week, a level that would have been unheard of only a few years ago.

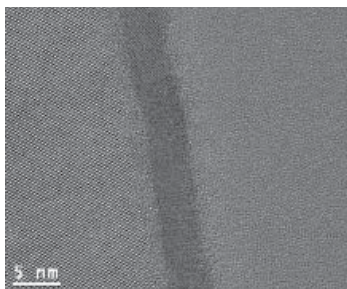


Figure 8

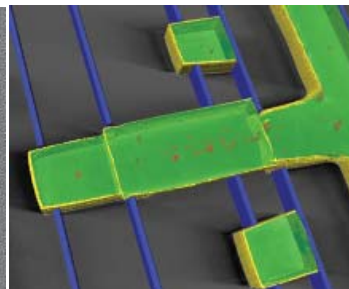


Figure 9

Nanoelectronic applications include process development, high-volume process control, and failure analysis, and run the gamut from simple automated metrology to sophisticated high-resolution analysis.

Fig 8 Basic TEM in Nanoelectronics – gate oxide metrology

The gate oxide prevents current from flowing between the gate electrode and the gate region of a CMOS transistor. For advanced processes (65 nm) the oxide is little more than 10 nm thick and both thickness and continuity are critical to device performance. TEM analysis is already used routinely to measure and control oxide thickness. Automated measurement routines acquire the image, detect the feature boundaries and report thickness. Figure 8 shows an advanced gate stack. The darker region is HfO_x. The region to the left is Silicon. The HfO_x is separated from the silicon by a very thin layer of SiO₂

Fig 9 – Research TEM in Nanoelectronics – interconnect inspection and metrology

The introduction of copper to connect transistors in integrated circuits greatly increased the complexity of the interconnect process. In addition to the plating process that deposits bulk copper, the conducting pathways must first be lined with a barrier layer to prevent copper diffusion into the surrounding material, and a seed layer to promote the plating process. Both of these layers are only a few nanometers thick and are critical to device performance and reliability. Layer continuity is particularly difficult to assess from a single planar cross section. Electron tomography offers a solution by allowing engineers to visualize the entire layer in three dimensions. Figure 9 is a tomograph of a copper interconnect structure. Bulk copper is green and barrier/seed is yellow. (Sample courtesy of Hans-Jurgen Engelmann, AMD Saxony)

Fig 10 Advanced TEM in Nanoelectronics – Advanced high-resolution analysis.

As described above, gate oxides are a critical part of CMOS transistor structure. Already they are no longer simple oxide layers. The complexity of the gate stack is expected to increase as we

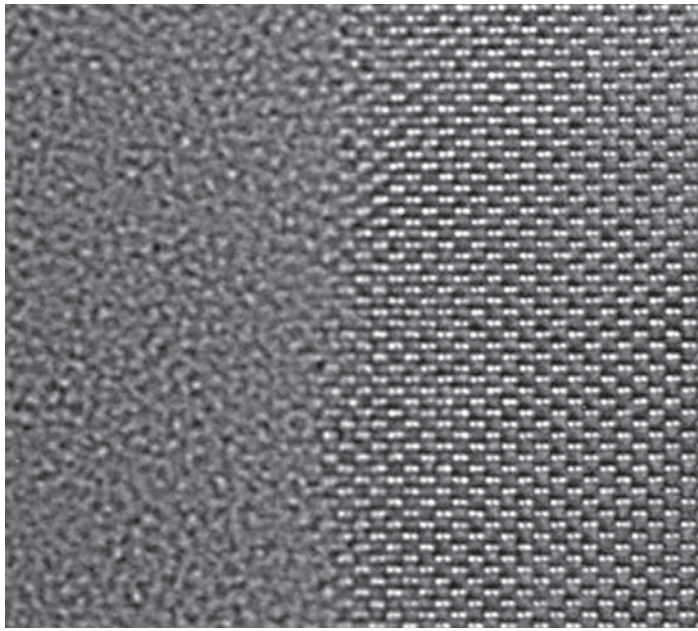


Figure 10

move to metal gate electrodes, and high k gate materials. The local composition of each layer, as well as the interfaces between the various materials in the stack, will be critical. Only aberration corrected S/TEM has the ability to image the interfaces and provide high resolution analysis. Figure 10 shows the interface between the gate oxide (left) and silicon (right).

Conclusion

TEM has been regarded as a complex and expensive technique relegated largely to the research laboratory and requiring highly trained personnel to operate the instrument and interpret the results. The recent explosion in nano scale research has resulted in greatly increased demand for imaging and analytical capability with sub-nanometer resolution. A new generation of S/TEMs provides the necessary resolution and also offers dramatic improvements in usability. As demand grows and volume increases, manufacturing efficiencies may permit lower system prices. Likewise, as important applications are identified, manufacturers will develop dedicated systems for their specific needs. For now, flexibility and modularity are key considerations in optimizing long term value. Embedding—the tight integration of specific capabilities with the base platform—also enhances value by providing a fast pathway to the specific information required for a given analysis. Judicious matching of system capabilities with application requirements maximizes the value to cost ratio. Most importantly, the cost of TEM must be viewed in the context of the increasing value of the information it provides. ■

References

- 1) J Elands *et al.*, "Subcellular protein localization with high resolution: the next step in proteomics," American Biotechnology Laboratory, October 2005
- 2) J Elands *et al.*, "CryoEM as a complement to current techniques in protein structural analysis," Current Drug Discovery, October 2004
- 3) T Fliervoet, "Automated electron tomography goes mainstream," G.I.T. Imaging and Microscopy, February 2005