SEM and AFM: Complementary Techniques for Surface Investigations

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ABSTRACT
There are a wide range of analytical techniques which may be used for materials characterization depending on the type of information needed. For high resolution surface investigations, two commonly used techniques are Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM). Each of these techniques resolves surface structure down to the nanometer scale. However, the image formation mechanisms are quite different, resulting in different types of information about the surface structure. The occurrence of the SEM and AFM side-by-side has become common in many of today’s analytical laboratories. This article will compare and contrast the two techniques, describing their unique and complementary capabilities.

Although investigations that use both SEM and AFM to characterize a material are common, there are just a few studies that directly discuss the complementary nature of the techniques [1-8]. A comparison of these techniques will be conducted with respect to three factors: (1) surface structure, (2) composition, and (3) environment. The comparisons are presented for typical equipment configurations and operating procedures.

IMAGING MECHANISMS

Scanning electron microscopy
In the SEM a beam of electrons is directed from a filament to the sample in a vacuum environment ranging from 10^{-4} to 10^{-10} Torr. The electrons are guided to the sample by a series of electromagnetic lenses. The resolution and depth of field of the image are determined by the beam current and the final spot size, which are adjusted with one or more condenser lenses and the final, probe-forming objective lenses. The electrons interact with the sample within a few nanometers to several microns of the surface, depending on beam parameters and sample type. Electrons are emitted from the sample primarily as either backscattered electrons or secondary electrons. Once these electrons escape from the sample surface, they are typically detected by an Everhart-Thornley photomultiplier detector.

Secondary electrons are the most common signal used for investigations of surface morphology. They are produced as a result of interactions between beam electrons and weakly bound electrons in the conduction band of the sample which then escape from the sample surface as secondary electrons. Secondary electrons are low energy electrons (<50eV), so only those formed within the first few nanometers of the sample surface have enough energy to escape and be detected. High energy beam electrons which are scattered back out of the sample (backscattered electrons) can also form secondary electrons when they leave the surface. Since these electrons travel farther into the sample than the secondary electrons, they can emerge from the sample at a much larger distance away from the impact of the incident beam which makes their spatial distribution larger. The SEM image formed is the result of the intensity of the secondary electron emission from the sample at each x,y data point during the rastering of the electron beam across the surface.

Atomic force microscopy
AFM consists of scanning a sharp tip on the end of a flexible cantilever across a sample surface while maintaining a small, constant force. The tips typically have an end radius of 2nm to 20nm, depending on tip type. The scanning motion is conducted by a piezoelectric tube scanner which scans the tip in a raster pattern with respect to the sample (or scans to the sample with respect to the tip). The tip-sample interaction is monitored by reflecting a laser off the back of the cantilever into a split photodiode detector. By detecting the difference in the photodetector output voltages, changes in the cantilever deflection or oscillation amplitude are determined.

The two most commonly used modes of operation are contact-mode AFM and TappingMode™ AFM, which are conducted in air or liquid environments. Contact-mode AFM consists of scanning the probe across a sample surface while monitoring the change in cantilever deflection with the split photodiode detector. A feedback loop maintains a constant cantilever deflection by vertically moving the scanner to maintain a constant photodetector difference signal. The distance the scanner moves vertically at each x,y data point is stored by the computer to form the topographic image of the sample surface. This feedback loop maintains a constant force during imaging, which typically ranges between...
MONOATOMIC SILICON STEPS ON THE SURFACE AS

A common application is to determine changes in surface morphology due to variations in deposition parameters, such as temperature, pressure, and time. Figure 2 shows SEM and AFM images of a polycrystalline silicon thin-film deposition at various points during the growth process. The three-dimensional nature of the AFM can be used to calculate changes in roughness and surface area variations due to differences in deposition parameters. For the SEM, a large area (several mm²) view of the surface in the AFM image of the same area revealed all at once, whereas a 100 µm x 100 µm area is typically the largest area viewed by an AFM. These images are an example of “negative” polycrystalline films which are used as capacitors in memory devices. By making these films rough, the surface area is increased which makes it possible to hold more charge without increasing the lateral dimensions of the capacitors on the chips. By adjusting the deposition parameters and using the AFM to analyze the surface area of the films, the deposition parameters needed to produce a film with the maximum surface area were determined.

Another example of the difference between the two techniques is in interpreting subtle differences in height. In the SEM image, changes in slope can result in an increase in electron emission from the sample surface, producing a higher intensity in the image. However, it can sometimes be difficult to determine whether the feature is sloping up or down (Fig 3a). Since the AFM data contains the height information, determining whether a feature is a bump or pit is straightforward. As can be seen in Figs 3b and c, the features on this sample are bumps. This information was used in the study of the growth mechanisms of GaP on Si during chemical beam epitaxy deposition [11].

HIGH ASPECT RATIO STRUCTURES

Semiconductor processing commonly requires measurements of high aspect ratio structures such as trenches and via holes. In a SEM, these structures are typically measured in cross section by cleaving the wafer and imaging the sample end-on, as shown in Fig 4a. In contrast, the AFM image of a trench or via is made by scanning the sample surface. However, because AFM measures these structures non-destructively, the wafer can be returned to the production line after measurement. An AFM analysis of vias in photoresist is shown in Fig 4b. To image some higher aspect ratio structures, the proper tip shape is needed for the AFM to scan narrow openings and steep side-
which provides elemental analysis, as SEM is the only one of the two techniques dimensional Y
sional surface structure which shows how the range approaches 10um or larger. however, in some configurations the vertical information within a single image. A SEM image of non-woven polyethylene oxide fibers can be seen in Fig 5a. The depth of field and small beam size makes it possible to image the fibers far below the top layer. This ability also makes it possible to measure very rough surfaces over larger lateral areas as well. Although the AFM can measure vertical surface variations below 0.5Å, its ability to measure tall structures is limited. Standard scanners typically have 5 to 6µm of vertical range, however, in some configurations the vertical range approaches 10µm or larger.

Another example of a complex three-dimen-
sional surface structure which shows how the SEM and AFM can complement each other can be seen in Fig 5b. The convoluted three dimensional \( Y_2O_3 \) oxide crystal shown growing out of a relatively flat \( Y_2O_3 \) thin film on a Si substrate is easily imaged in the SEM (Fig 5b). Although the AFM would have problems imaging the obtuse angles and enclosed areas of this surface, the roughness of the \( Y_2O_3 \) film can be measured whereas in the SEM image the surface roughness is not evident. Therefore, the two techniques together give a more complete picture of the sample.

COMPOSITION
Both SEM and SPM provide compositional information through a variety of techniques. SEM is the only one of the two techniques which provides elemental analysis, as described above. However, both SEM and AFM are associated with techniques which can provide compositional information through analyzing materials and physical properties of the sample.

SEM
Two commonly used signals for compositional investigations are x-ray and backscattered electrons. X-ray signals provide elemental analysis by attachment of an energy-dispersive spectrometer (EDS) or wavelength-dispersive spectrometer (WDS) to the SEM system. X-ray emission results from ejection of inner-shell electrons in the sample and replacement of the lost electrons by outer shell electrons. This jump results in a change in energy that produces x-rays with energy corresponding to the change. A typical x-ray spectrum is shown in Fig 6.

Backscattered electrons are beam electrons scattered back out of the sample. Here, the electrons go much deeper into the sample than secondary electrons and still emerge from the sample to be detected. The percentage of beam electrons that are backscattered is dependent on the atomic number of the material, hence its utility for analysing material composition (Fig 7).

AFM
Although an AFM does not provide elemental analysis, it can supply compositional information by differentiating materials based on physical properties, such as stiffness, elasticity, compliance, friction, adhesion, magnetic and electrostatic fields, carrier concentration, temperature distribution, spreading resistance, and conductivity. Many of these techniques consist of looking simultaneously at another signal while performing standard AFM imaging. One of the most common techniques for mapping differences in materials properties is PhaseimagingTM which is conducted during TappingMode AFM operation by monitoring the phase lag between the oscillating drive signal used to drive the cantilever and the oscillating detection signal from the photodiode detector. This signal will indicate differences in viscoelasticity and/or adhesion across the imaged area. This technique is commonly applied to mapping the distribution of polymers in a heterogeneous system, or mapping the distribution of filler, such as silica or carbon black, in a polymer matrix. An example of

![Figure 5: SEM image of a non-woven textile sample of polyethylene oxide fibers. The large depth of field of the SEM makes it possible to image fibers which are 10's of µm's below the upper layer of fibers. Bar=10µm; (b) SEM image of \( Y_2O_3 \) crystal. Bar=6µm.](image)

![Figure 6: EDS X-ray spectrum of an AlGaN thin film on SiC substrate showing the presence of N, Ga, and Al.](image)

![Figure 7: Backscattered SEM image of an PdSn alloy showing contrast based on the atomic number of the two components. The brighter areas are Pd-rich. 5,000x, Scale bar=5µm.](image)

Phase imaging on a polyethylene film is shown in Fig 8. Other ways to get similar information are by force modulation AFM, which maps differences in friction across the sample surface, and lateral force microscopy (LFM), which maps differences in friction across the sample surface.

There are also techniques that can be used to investigate long range forces across the imaged area. Magnetic force microscopy (MFM) and electric force microscopy (EFM) map the magnetic and electrostatic field gradients, respectively, which extend from the sample surface. These techniques are performed by using either a magnetic or conductive probe to map the attractive and repulsive forces between the tip and the sample. MFM is commonly used to detect the domain structure of magnetic bits written on magnetic media, to evaluate the performance of magnetic heads, and to investigate the magnetic structure of experimental materials [12]. This is conducted by a routine called LiftModeTM in which a TappingMode topographic image and a magnetic image are acquired over the same area. LiftMode consists of first collecting a line scan in TappingMode of the surface morphology. The tip is then lifted above the surface and a second scan is made over the same line using the saved topographic scan to maintain a constant tip-sample separation. The long-range magnetic forces shift the resonance frequency of the oscillating cantilever, which is detected to produce the magnetic image. An example of bits written on a textured hard disk is shown in Fig 9.

Although the AFM is applied so that it is nondestructive to the sample surface, it can be used to study differences in mechanical properties by performing nanoinduction to investigate hardness differences between materials. This technique uses a diamond tip mounted on a stiff, stainless steel cantilever. A TappingMode AFM image is collected with the probe to determine the area of interest for indentation, the nanoinduction is then made at a specified force, and an image is then collected of the indented area. Scratching and wear testing may also be conducted with this configuration to investigate adhesion and delamination of films under a small applied force.

ENVIRONMENT
One of the primary differences between these
two types of microscopy is the environment in which they are performed, i.e. SEM is conducted in a vacuum environment, and AFM is conducted in an ambient or fluid environment. There are several issues which make environment an important issue. First, there is a frequent need in fields such as biology and biomaterials to study hydrated samples. These two techniques compensate for this need by different means: an environmental chamber for a SEM, and a fluid cell for the AFM. Second, the SEM is required to work in a vacuum environment due to the nature of the technique which brings up the issues of vacuum compatibility of the sample, the conductivity of the surface, and vacuum maintenance. To image poorly conductive surfaces without sample charging may require conductive coatings or staining, which may alter or obscure the features of interest, or it may require low voltage operation, or an environmental chamber, which may sacrifice resolution.

For SEM, hydrated samples are addressed by placing a specimen in an environmental chamber with either an electron transparent window or a small aperture for the beam to enter the chamber. The chamber is typically flushed with an inert gas saturated with water vapor. Common applications are to either investigate hydrated surfaces to preserve their surface structure when hydrated, or to reduce charging on insulating samples. For the electron beam to interact with the surface in this configuration, it must go through an environment of gas and water vapor, which may result in the sacrifice of image quality and resolution. One of the primary advantages of the AFM is its ability to image insulating surfaces at high resolution in fluid. Imaging samples in a hydrated state with an AFM is commonly performed by enclosing the sample and probe in a fluid environment. Since AFM is not based on conductivity, the image and scanning mechanism is not disturbed by the presence of the fluid. Common applications for AFM investigations in fluid are in the biological sciences: biomaterials, crystal growth, force interaction studies, and for investigating processes in situ at a lateral resolution of 1 to 5nm and a vertical resolution down to 0.5Å without sample damage. (Fig 10). With the appropriate accessories, AFM can also be used in varied gaseous environments and at elevated temperature. The latter is particularly important for research and development of polymers.

RECENT AFM ADVANCES

AFM has experienced some recent technological advances that are worth noting. New technologies, for example the NanoScope IV controller (Digital Instruments, VMG), provide the following advantages relative to traditional designs:

• Faster scanning for increased throughput - Scanning at up to ten times the speed of traditional designs has opened doors to new applications that require faster turn around at the rate of seconds - rather than minutes - per scan.
• Higher lateral resolution - Higher data density increases productivity by allowing zooming into the finest details, even on large survey scans, without the need to perform repetitive smaller scans. This also provides the resolution required to characterize steep sidewalls on such samples as DVD bumpspits and semiconductors.
• Enhanced electronics - These new technologies allow quantification and improved sensitivity for quantifying the probe’s phase response/interaction, improving understanding of the nano-scale properties of materials.
• Enhanced sensitivity - Controlling the quality, or Q, factor of the probe offers better control of the forces between tip and sample and improves the sensitivity of measurements such as with Phaselinking and MFM.
• Nanomanipulation - New designs now provide the positioning accuracy and precision to move and manipulate objects at the nanometer scale.

DISCUSSION

Although SEM and AFM appear very different, they actually share a number of similarities. Both techniques raster a probe across the surface to detect some interaction with the surface to form an image. Both have a lateral resolution which is similar in scale (although under certain conditions AFM is superior). And both techniques have image artifacts that the operator is trained to identify. The SEM has had a much longer time to mature but the rapid adoption and implementation of AFM has resulted in a similar understanding of artifacts. Furthermore, by using two techniques which are complementary, one technique will often compensate for the imaging artifact of the other technique.

REFERENCES