Kavli Workshop for Journalists

June 13th, 2007

CNF Cleanroom Activities
Seeing nm-sized Objects with an SEM

Lab experience: Scanning Electron Microscopy

Equipment: Zeiss Supra 55VP

Scanning electron microscopes (SEM) can be used to image objects that cannot be seen with the eye or even with an optical microscope. An SEM can magnify objects up to 1,000,000 times, opening up a world that is otherwise too small to see. Using an SEM, we can see the individual eyes of a fly, red blood cells, or even an HIV virus. The image is generated by the interaction of an accelerated electron beam with the object. The beam itself is only a few nanometers in diameter. When it reaches the object to be imaged it generates a number of signals. One such signal comes from secondary electrons that are collected by a detector. Since the secondary electrons are emitted from a thin layer of the specimen surface, they give information about the topography of the object. Other signals such as those generated by backscattered electrons allow us to see compositional differences of the object’s material such as grain boundaries. Learning to take images on an SEM might only take an hour or so, but learning how to quickly get a really good image takes months of experience.

In addition to imaging with electron beams, researchers also use them to write structures. These can be as small as 10 nm and are first written onto a radiation sensitive layer. The written pattern then serves as a masking layer for subsequent processing steps. These might be the modification of the underlying substrate with ions, the addition of another nanolayer such as a thin silicon dioxide layer, or the removal of minute amounts of substrate material via dry etching with reactive gases. To construct a working device (like a transistor, or a sensor), researchers repeat this process several times. The resulting device is so small that it requires the SEM to see it.
The formation of films only a few nm thin is a phenomenon that occurs in the environment every day. An example of such a thin film (nanolayer) is the layer of rust that begins to form on a metal. In nanofabrication we control the electrical and mechanical properties of thin films by growing them under controlled conditions. The combination of supplied gases, the temperature, and the pressure determine the electrical properties of the film, rendering it conductive, semiconductive, or insulating. We can also modify the growth conditions so that the films exhibit low stress or high stress. Often, the film will have properties different from those of the underlying substrate. It can be part of the fabrication process or it can serve as an integral component of the finished nanoscale device.

A first estimate of the thickness of an oxide nanolayer deposited on a silicon substrate can be obtained by the interference color of the layer (for examples see the table below). A more accurate method to measure the thickness of nanolayers is interferometry. A light with a specific wavelength is bounced off the nanolayer-substrate stack. Since the layers are semitransparent, the light is reflected not only from the surface of the thin film, but also from the underlying substrate. The distance between the surface of the thin film and the surface of the substrate (which equals the thickness of the layer) causes the two interference patterns of the reflected light beams to differ. A detector measures this difference and compares it with a standard. Using a suitable model, the thickness of the film can be extracted from these sensitive measurements.

Creating nm-sized Objects Using Nanolayers (Thin Films)

Lab experience: Low Pressure Chemical Vapor Deposition (LPCVD) and Interferometry

Equipment: LPCVD furnaces, FilMetrics F20 / F50

The oscillator consists of a 500 nm thick silicon nitride layer. It was structured in the horizontal dimension via photolithography and in the vertical dimension via dry etching. The membrane is suspended over a silicon substrate.

Round oscillator:

Interference colors for silicon dioxide films:

<table>
<thead>
<tr>
<th>Color</th>
<th>Thickness (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Red-violet</td>
<td>270</td>
</tr>
<tr>
<td>Blue</td>
<td>310</td>
</tr>
<tr>
<td>Blue-green</td>
<td>320</td>
</tr>
<tr>
<td>Light green</td>
<td>340</td>
</tr>
<tr>
<td>Yellow</td>
<td>390</td>
</tr>
<tr>
<td>Light Orange</td>
<td>410</td>
</tr>
<tr>
<td>Carnation Pink</td>
<td>420</td>
</tr>
<tr>
<td>Violet-red</td>
<td>440</td>
</tr>
<tr>
<td>Violet</td>
<td>470</td>
</tr>
</tbody>
</table>
Creating nm-sized Objects
Using Lithography

Lab experience: Photolithography

Equipment: EVG 620 Contact Aligner

The process of creating nm-sized devices starts with “writing” their horizontal features into a thin radiation-sensitive coating on a silicon substrate (wafer). This writing process is called lithography and a light, x-ray, electron, or an ion beam can serve as the “pen”. We distinguish between pixel-by-pixel-writing techniques and techniques that can write entire patterns in one flash. Often a master copy (called mask) is created by an accurate pixel-by-pixel writing technique. The pattern inscribed on the mask is then replicated onto a wafer and reduced in size via projection photolithography. The most straightforward and least expensive method to replicate a pattern, though, is contact photolithography. Here, the light generated by a mercury arc lamp is directed through a set of filters and the mask onto a coated substrate, which is held in direct contact or in close proximity to the mask. With this method, the pattern is replicated 1:1 on the wafer. The light alters the coating on the wafer so that it becomes susceptible to dissolution in alkaline solutions such as NaOH, or KOH. Because of the possibility of mobile ion contamination in semiconducting and insulating substrates, metal ion free developers are often used. A developer we frequently use is tetra-methyl ammonium hydroxide (TMAH). After developing the coating, the underlying substrate becomes exposed. The pattern can now be transferred down into the substrate by one of the pattern transfer techniques: addition of a nanolayer material, the alteration of the exposed areas with ions, or the removal of small amounts of substrate material. To construct a nanodevice, the entire process is repeated several times. In industry, a typical process can comprise up to 30 steps of lithography each followed by pattern transfer. Each step is called a layer and all of them need to be aligned to each other.

A typical fabrication sequence: Photolithography structures the resist layer, which subsequently acts as a lift off layer for a deposition step, or alternatively, as a protective layer for a following etching step.
Seeing nm-sized Objects with an AFM

Lab experience: Scanning Probe Microscopy

Equipment: Digital Instruments Atomic Force Microscope (AFM)

The development of scanning probe tools such as the atomic force microscope (AFM) and the scanning tunneling microscope (STM) was essential to the advancement of nanotechnology. They enable researchers to image surfaces of objects with atomic resolution. Scanning over the cell wall of a bacterium, for example, these tools reveal the shape and arrangements of clusters of proteins that constitute the wall. An AFM uses a flexible cantilever with a nanostructured tip as an imaging probe. The cantilever holding the tip is vibrated near its resonant frequency while it scans across an object. When the tip comes within close proximity to the surface, an atomic force is felt and the motion of the cantilever is dampened. The interaction of the tip with the surface is monitored with a laser spot reflected off the back of the cantilever. The AFM is used to measure nanometer scale topography and characteristics of surfaces. Using specialty tips together with the appropriate modes of operation, it can also be used to measure conductivity, magnetic field strength, and thermal conductivity of the surface with nm spatial precision. Some scanning probe microscopes let researchers manipulating the surface of a sample with the tip. This technique can even be used to write small horizontal features on a substrate.

AFM image of a lens written with an electron beam tool. (Ganesh Srinivasan, Principal investigator: Kenneth Foster.)

Carbon nanotubes imaged with an AFM.