Inspecting Surfaces With a Sharp Stick: Scanning Probe Microscopy – Past, Present, and Future
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With the growing emphasis on nanotechnology, scanning probe microscopy (SPM) is emerging from the surface science laboratories and becoming a mainstream inspection and metrology tool along side optical and SEM microscopes. Scanning probe instrumentation and applications evolved dramatically during the past quarter-century (Table I). By 1998 SPM-related papers were being published at the rate of nearly 5000 per year. Here we review the history of scanning probe microscopy, describe its current role as a critical enabler in nanotechnology, discuss why it has become a routine laboratory tool, and present a view of future directions for this advanced technology.

Table 1. Evolution of Scanning Probes and Applications

<table>
<thead>
<tr>
<th>Year</th>
<th>Instrument</th>
<th>Applications</th>
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</thead>
<tbody>
<tr>
<td>1920-1980</td>
<td>Surface profilers</td>
<td>Measurement of surface texture, line profiles for step heights</td>
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<tr>
<td>1980-2000</td>
<td>Scanning probe instruments</td>
<td>Basic research, exploration, engineering, physical science, life science, process development</td>
</tr>
<tr>
<td>2001-?</td>
<td>Turnkey SPM</td>
<td>Micro- and nano-device manufacturing and process control</td>
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The Past

Scanning probe microscopes (SPM) had their origins in optical-lever based mechanical profilers devised in 1929. This was enhanced by piezoelectric motion and detection in 1955, non-contact profilometry in 1972, culminating in the Nobel-prize-winning scanning tunneling microscope (STM) in 1982 that could actually produce images of atoms.

An optical-lever based mechanical profilometer was described by Gustav Schmalz (Germany) in 1929 [1]. His instrument, shown schematically in Figure 1, reflected a beam of light from a mirror on the sample probe mounted on a cantilever. The sample was on a moving stage and the reflected light traced a magnified image on photographic film moving in tandem with the sample. Profile magnifications up to 1000× could be achieved, but bending of the probe from collisions with large features on the surface presented a problem. In 1955 Helmut Becker, et al. (at Leitz in Germany) suggested oscillating the probe as it scanned the surface [2]. Figure 2 (from Becker’s patent) shows the probe tip, 9, with its motion controlled by piezoelectric elements 1 and 3. The middle piezo element, 2, produced the probe height signal. The vibrating probe concept was also described by David Lee and Raymond Harrison in 1977 [3].

As acknowledged by the inventors of the STM in their Nobel lecture [4], the STM was presaged by an instruments devised in 1972 by Russell Young, John Ward and Fredric Scire at the U.S. National Bureau of Standards (Figure 3) [5]. This instrument, which they called the topographiner, was a non-contact profilier that used piezo control in the x-, y- and z-directions and constant current feedback from electronic field emission between the probe tip and the surface. The vertical resolution was 30 Å and the horizontal resolution was about the same as an optical microscope.

Figure 1. Schmalz optical-lever profilometer, 1929.

Figure 2. Figure from Becker’s 1955 patent [2] for a vibrating probe controlled by piezoelectric elements 1 and 3 with signal output from the piezo element 2.

Figure 3. The topographiner (1972), a non-contact profilier with x-y-z piezo control and constant current feedback (Figure from [5]).
when probe position sensing and feedback control reduced image distortion [8]. Metrology step height standards of 18 nm and pitch standards of 0.5 μm are now available with accuracies of ±1%. Computer control and position feedback (Figure 5) has given us intuitive SPM instruments that rapidly provide precise and accurate measurements and can be operated with little or no training. Their laboratory and industrial use is becoming as routine as optical microscopy.

In 1982, Bennig and Rohrer used cutout patterns from their recorder trace data to construct a Plexiglas model of the arrangement of atoms on a silicon surface [4]. Today, software can simultaneously generate 3D images of topography and physical properties of the surface (Figure 6). The means of motion control for these instruments has become more precise and modern SPMs use calibrated sensors to control the motion. Probe tips have evolved from electrochemically-etched tungsten to chemically etched conical silicon to chemically deposited silicon nitride pyramids. Most recently, giant-molecule carbon nanotubes have been adapted as very high aspect ratio tips (Figure 7).

Various imaging modes are available including continuous, vibrating, step-and-repeat, and lateral-force scanning (Figure 8). Lateral force scanning measures the lateral or twisting deflections of the probe from forces parallel to the surface. A recent application is the study of the phase change of DNA molecules in response to tension and torsion [9]. In such applications, AFM cantilevers can measure angstrom-scale, millisecond events, and forces greater than 10 pN.

Applications have included basic research in surface science, and exploration in engineering, physical and life sciences, and nanoscale process development. More recent applications range from nanotechnology (including biotechnology) and nanoscience, to process development and process control.

SPMs have also been used for nanolithography, nanopatterning, and nano-construction. One example of nanolithography is the formation of an etch-resistant material by the action of an SPM tip. Nanopatterning can be accomplished directly by several methods. Direct action of a conducting AFM tip or STM tip can form an oxide pattern on metal by anodic oxidation or a metal pattern by chemical vapor deposition. Patterns with dimensions less than 10 nm can be achieved. A patterning technique called dip-pen nanolithography uses an AFM tip to deliver molecules to a surface via a solvent meniscus, just as in an ordinary dip ink pen. Molecular or biomolecular ‘inks’ can be used to write on a variety of substrates such as metals, semiconductors, and functional monolayers. Field evaporation in an AFM has been used to form nm-sized gold dots on an SiO₂/Si
COMING EVENTS

- GATAN 2003 Microscopy Training Schools
  April 9-10, TEM Digital Microscopy
  April 14-17, EELS Imaging & Analysis
  May 5-7, TEM Specimen Preparation
  May 8-9, SEM Specimen Preparation
  Pleasanton, CA
  mritter@gatan.com or www.gatan.com/training

- Histochemical Society Annual Meeting 2003
  April 11-15, 2003, San Diego, CA
  johc@u.washington.edu

- Experimental Biology
  April 12-16, 2003, San Diego, CA
  seb@sebiology.org

- Scanning 2003
  May 3-5, 2003, San Diego, CA
  www.scanning.org

- American Society for Microbiology
  May 18-22, 2003, Washington, DC
  www.asmusa.org

- Microscopical Society of Canada
  June 4-6, 2003, Vancouver, BC, Canada
  ech@unbkg.ubc.ca

- Lehigh Microscopy Schools
  June 8, Introduction to SEM & EDS
  June 9-13, SEM & X-ray Microanalysis
  June 16-20, Advanced SEM
  June 16-20, Quantitative X-ray Microanalysis
  June 16-19, Analytical TEM
  June 16-19, Characterization of Nanostructures
  June 16-19, Particle Characterization
  June 17-19, TEM Specimen Preparation
  June 17-20 Atomic Force Microscopy
  sharon.coe@lehigh.edu

- Materials Research Society
  April 21-25, 2003, San Francisco, CA
  info@mrs.org

- Microscopy and Microanalysis 2003
  August 3-7, 2003, San Antonio, TX
  www.msa.microscopy.com

- Society for Neuroscience
  November 8-13, 2003, New Orleans, LA
  web.sf.n.org

- American Society for Cell Biology 2003
  December 13-17, 2003, San Francisco, CA
  www.ascb.org

- Materials Research Society
  December 1-5, 2003, Boston, MA
  info@mrs.org

- Microscopy and Microanalysis 2004
  August 1-5, 2004, Savannah, GA
  www.msa.microscopy.com

- EMC 2004 (former EUREM)
  August 22-27, 2004, Antwerp, Belgium
  www.emc2004.be

- Microscopy and Microanalysis 2005
  July 31-August 4, 2005, Honolulu, HA
  www.msa.microscopy.com

Please check the "Calendar of Meetings and Courses" in the MSA journal "Microscopy and Microanalysis" for more details and a much larger listing of meetings and courses.
from amorphous to crystalline

Material Sensing Modes

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<tr>
<th>Mode</th>
<th>Description</th>
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<tbody>
<tr>
<td>Lateral Force</td>
<td>Use lateral force or vibrating phase to measure surface properties</td>
</tr>
<tr>
<td>Vibrating Phase</td>
<td></td>
</tr>
</tbody>
</table>

Figure 8. SPM scanning modes. Top: continuous, contact mode in repulsive region; Middle: vibrating mode, non-contact or intermittent contact ("tapping"). Bottom: material sensing modes that use lateral force or vibrating phase to measure surface properties such as friction.

substrate. Local heating by a near-field scanning optical microscope (NSOM) can change the phase of a GeSbTe optical recording film from amorphous to crystalline [10]. In nano-construction an STM tip is used to physically position adsorbed atoms to form a structure or pattern [11].

Future

Applications that were in the exploratory research phase in 1999 are now entering the marketplace. These include dip-pen lithography and "virtual reality" force-feedback nano-manipulation. The future will give us easy to use non-optical sensor systems that will enable new AFM applications. Force-feedback control will permit "feeling" the shape of surfaces, molecules and surface atoms. Probe materials will include glass or plastic, and we will be able to automatically sense when a probe is broken. Atomic-scale reference standards will be available. Fast data acquisition may be achieved by parallel scanning using arrays of multiple probes fabricated by MEMS techniques.

The ability to manipulate individual atoms will ultimately lead to practical computer memory with single-atom memory cells.

Conclusion

Scanning tunneling instruments have evolved into many offshoots, the first being the atomic force microscope, which have been used to observe nearly every physical property of a surface that is possible to measure. Applications have included measurement of surface texture and step heights, and after 1980, scanning probe microscopes have been applied to basic research in surface science and exploration in engineering, physical and life sciences, and nanoscale process development. Today, computer control and position feedback has given us intuitive SPM instruments that rapidly provide precise and accurate measurements and can be operated with little or no training (Figure 9). The laboratory and industrial use of SPMs is becoming as routine as optical microscopy.

References


Figure 9. tabletop atomic force microscope (Pacific Nanotechnology).
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