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

	Instrument	Applications
1920-1980	Surface profilers	Measurement of surface texture, line profiles for step heights
1980-2000	Scanning probe instruments	Basic research, exploration, engineering, physical science, life science, process development
2001-?	Turnkey SPM	Micro- and nano-device manufacturing and process control

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].

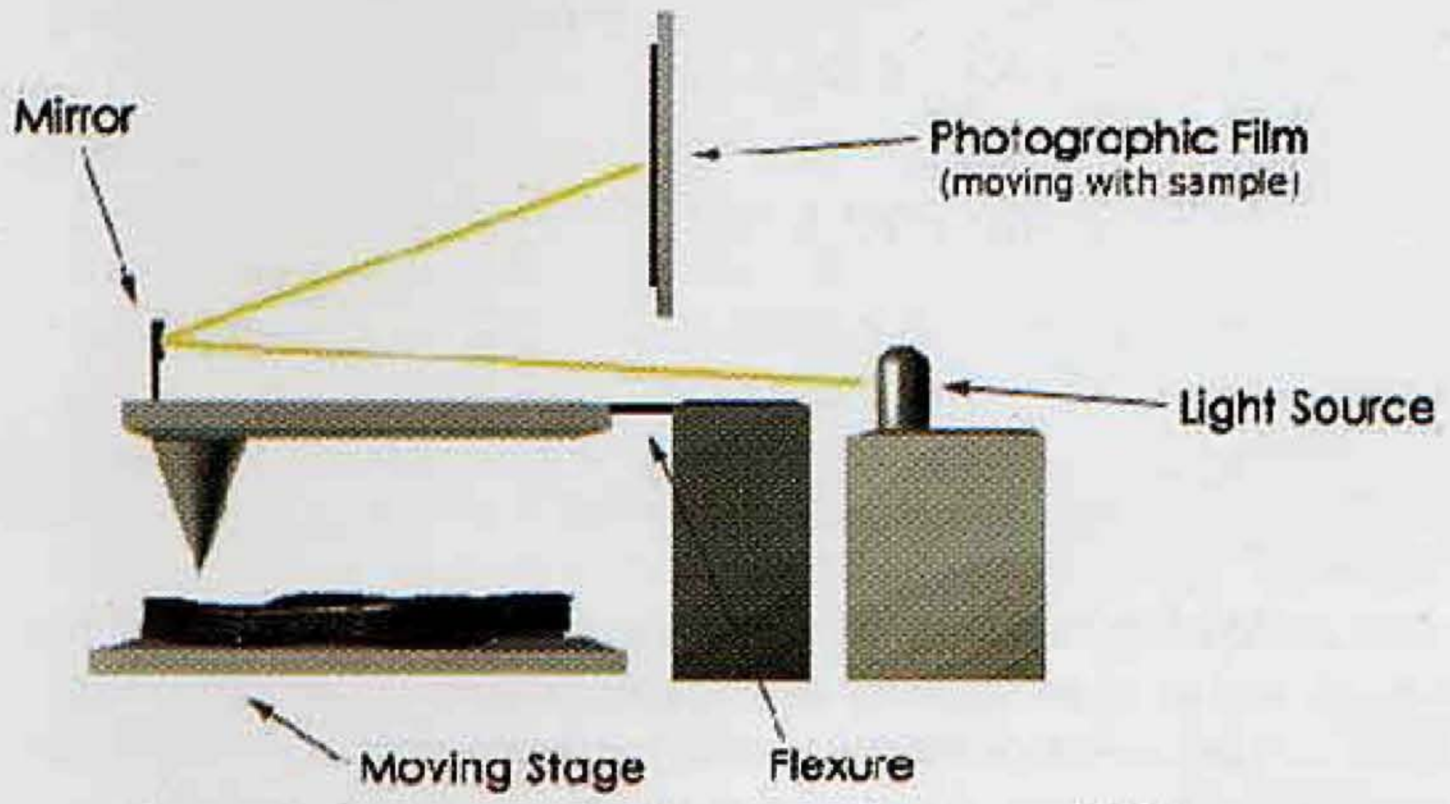

Figure 1. Schmalz optical-lever profilometer, 1929.

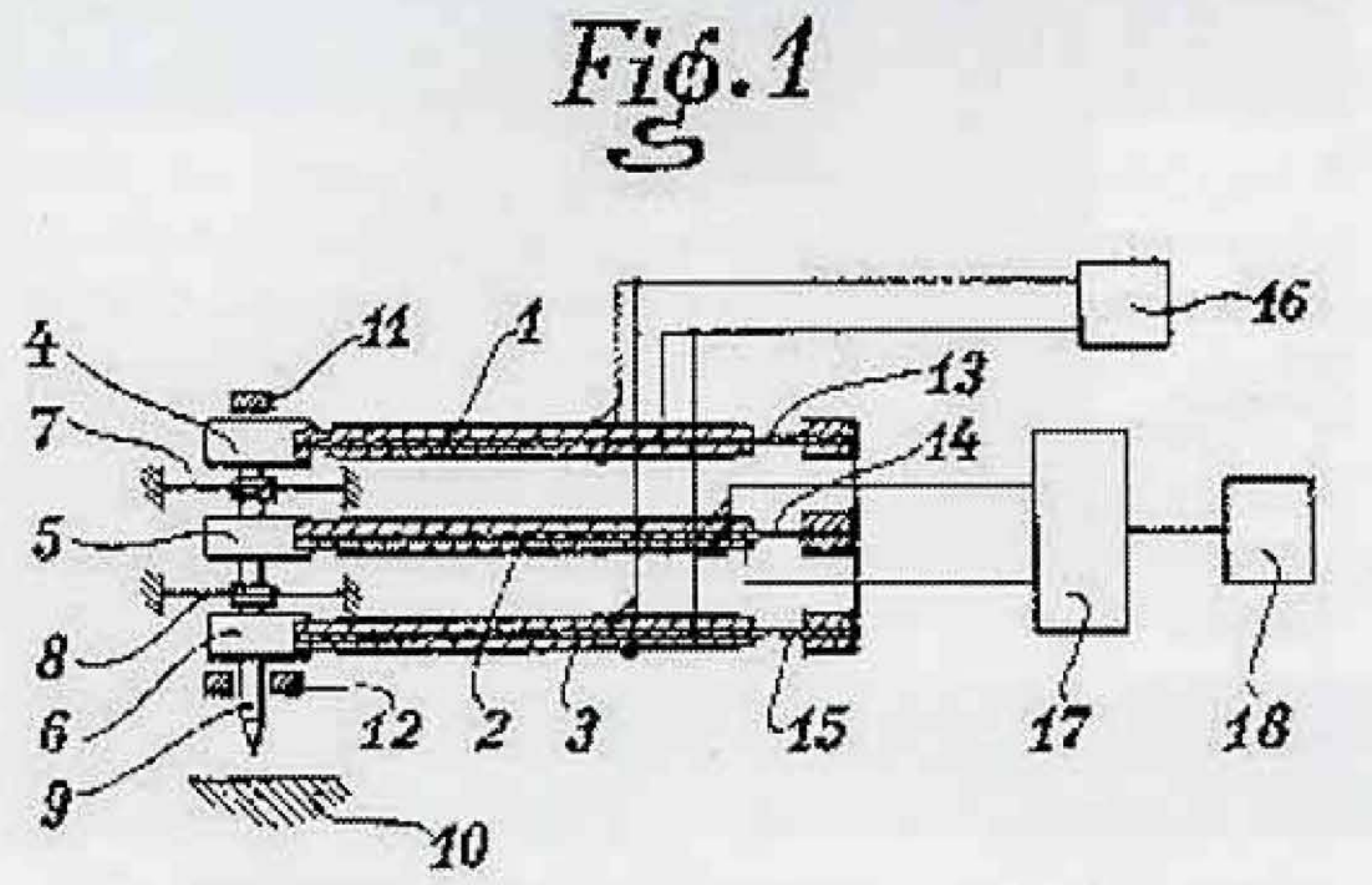

Figure 2. Figure from Becker's 1955 patent [2] for a vibrating probe controlled by piezoelectric elements 1 and 3 with signal output from the piezo element 2.

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 profiler 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.

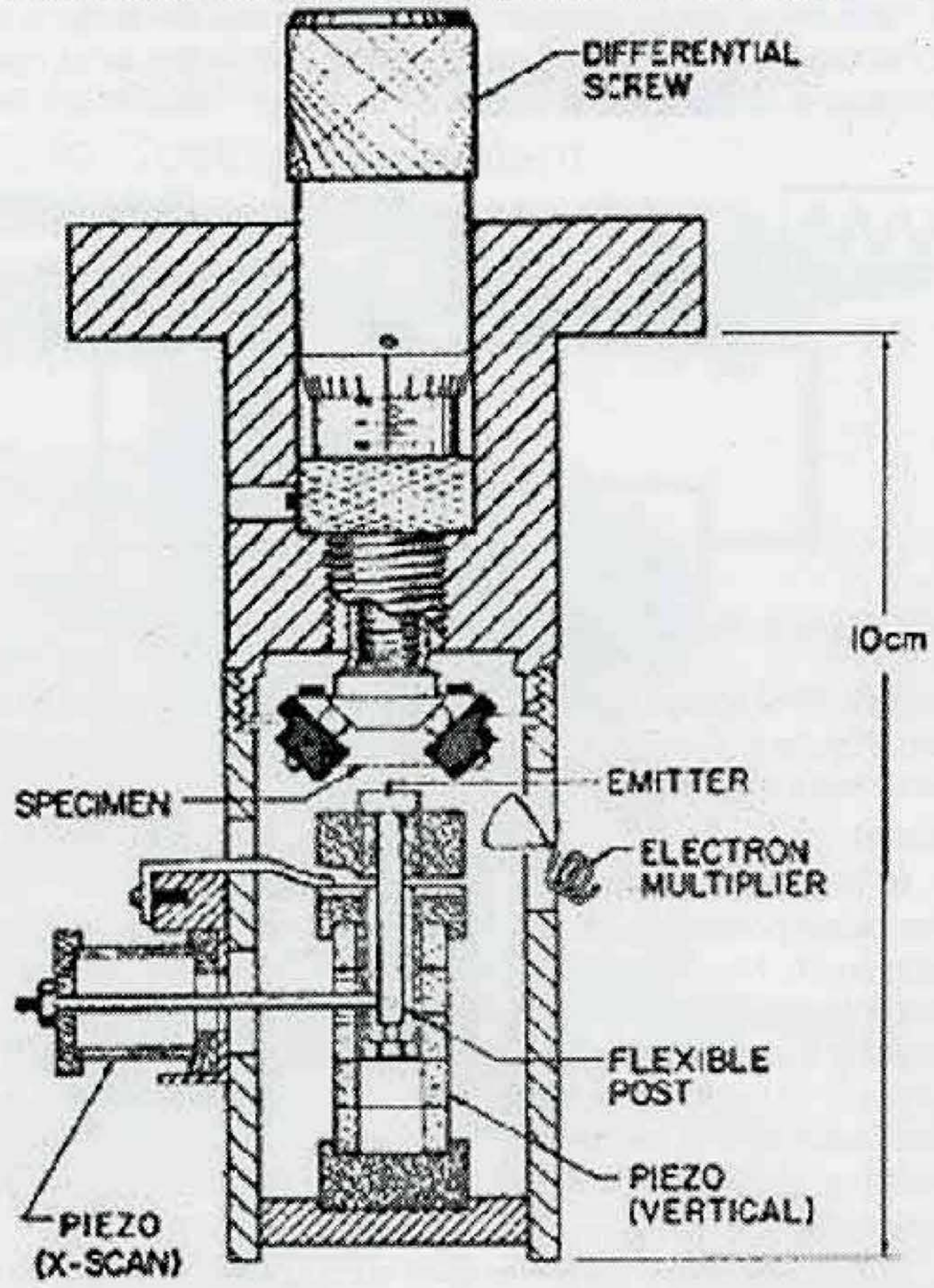

Figure 3. The topographiner (1972), a non-contact profiler with x-y-z piezo control and constant current feedback (Figure from [5]).

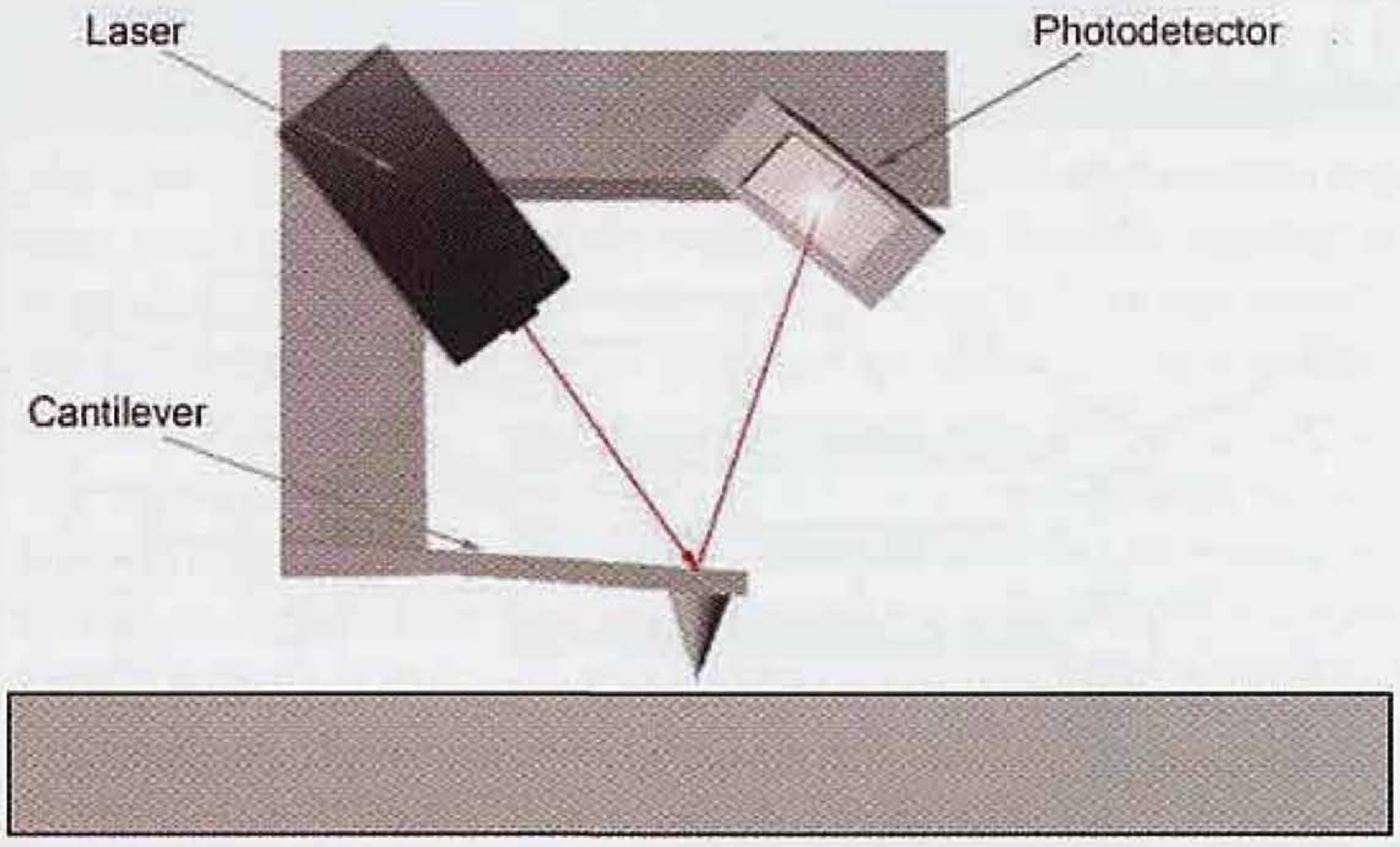

Figure 4. This figure illustrates the light lever sensor commonly used in atomic force microscopes [7].

In the fall of 1979 Gerd Binnig and Heinrich Rohrer at the IBM's Zurich Research Laboratory began experimenting with what became the scanning tunneling microscope that won them the Nobel Prize in 1986 [4]. Their instrument relied on the electron tunneling current flowing between a sharp conductive tip and the surface being scanned. They raster-scanned the probe to produce profiles that could be converted to a three-dimensional representation. In principle, the technique could provide atomic scale resolution, but required solving problems such as elimination of vibration and fabrication of an atomic-scale probe tip. They patented their device in 1982 [6].

The next milestone in SPM evolution was the atomic force microscope (AFM) invented in 1986 by Binnig, Calvin Quate (Stanford) and Christoph Gerber (IBM) [7]. This instrument relied on the mechanical forces between the probe tip and the surface and did not require a conducting tip or sample. While the most useful applications of the STM required an ultra-high vacuum ambient,

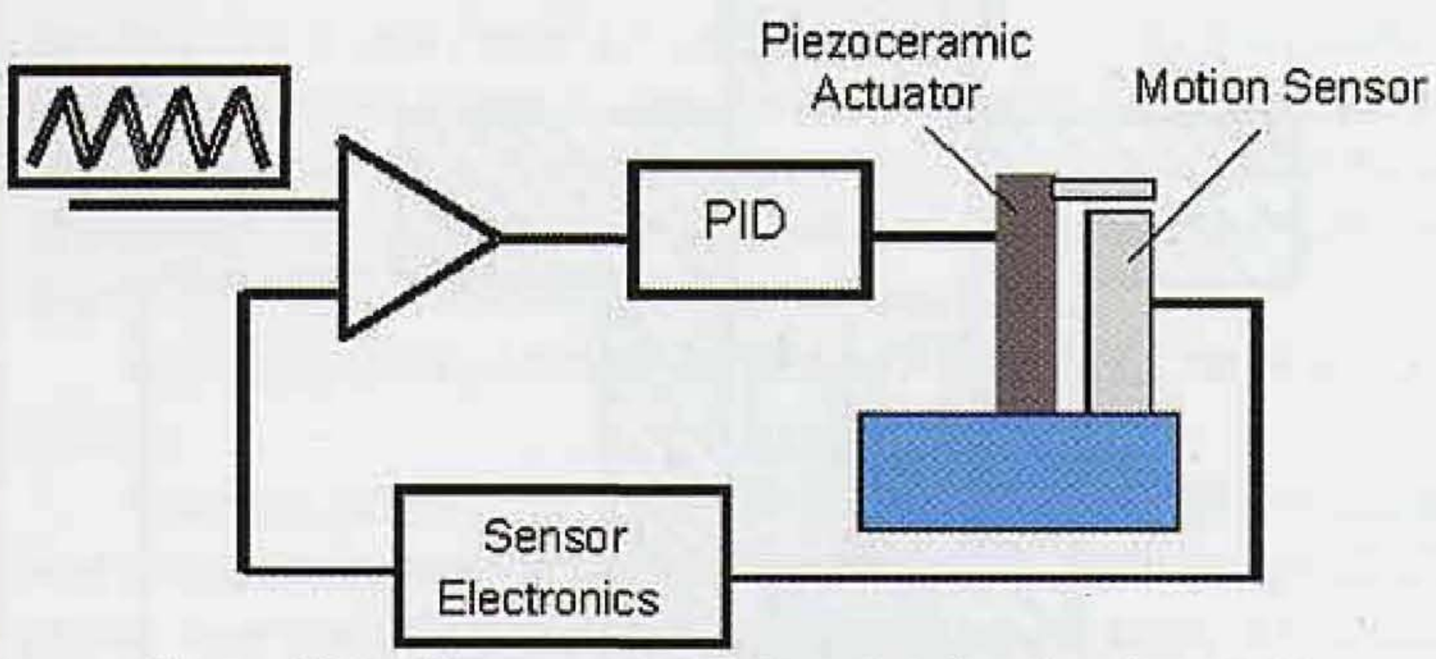

Figure 5. Position sensing and motion feedback control.

excellent AFM images could be obtained in air or even under a liquid. Figure 4 illustrates a light lever sensor commonly used in atomic force microscopes.

Present

SPMs became capable of accurate metrology in the 1990s 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

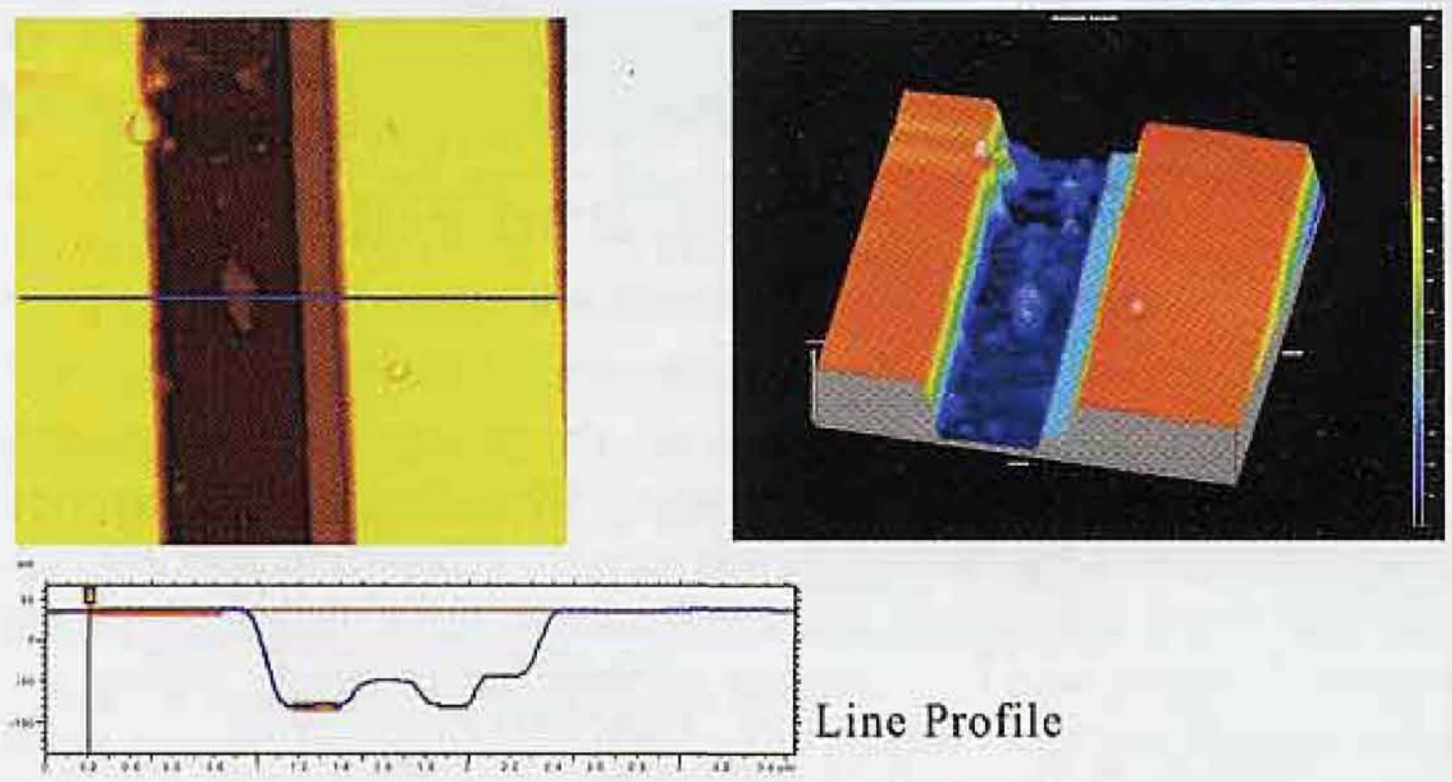

Figure 6. Modern software can convert scan data to a profile (lower left), 2D image (upper left) or a 3D image (right). (Source: Pacific Nanotechnology)

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).

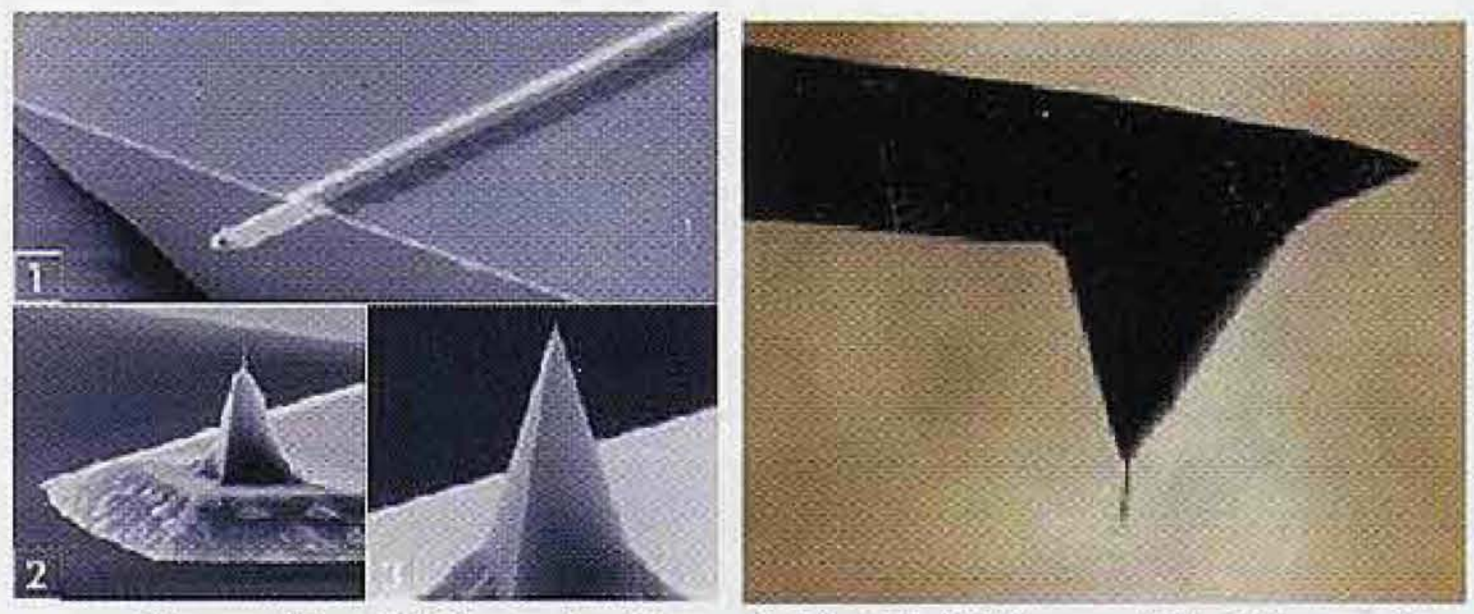

Figure 7. SPM probe tips. Left: (1) Silicon; (2) Silicon tip sharpened by focused ion beam machining; (3) Silicon tip sharpened by chemical etching (Source: NanoWorld). Right: carbon nanotube probe tip (Source: nPoint, Inc.).

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 of 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 SiO_2/Si

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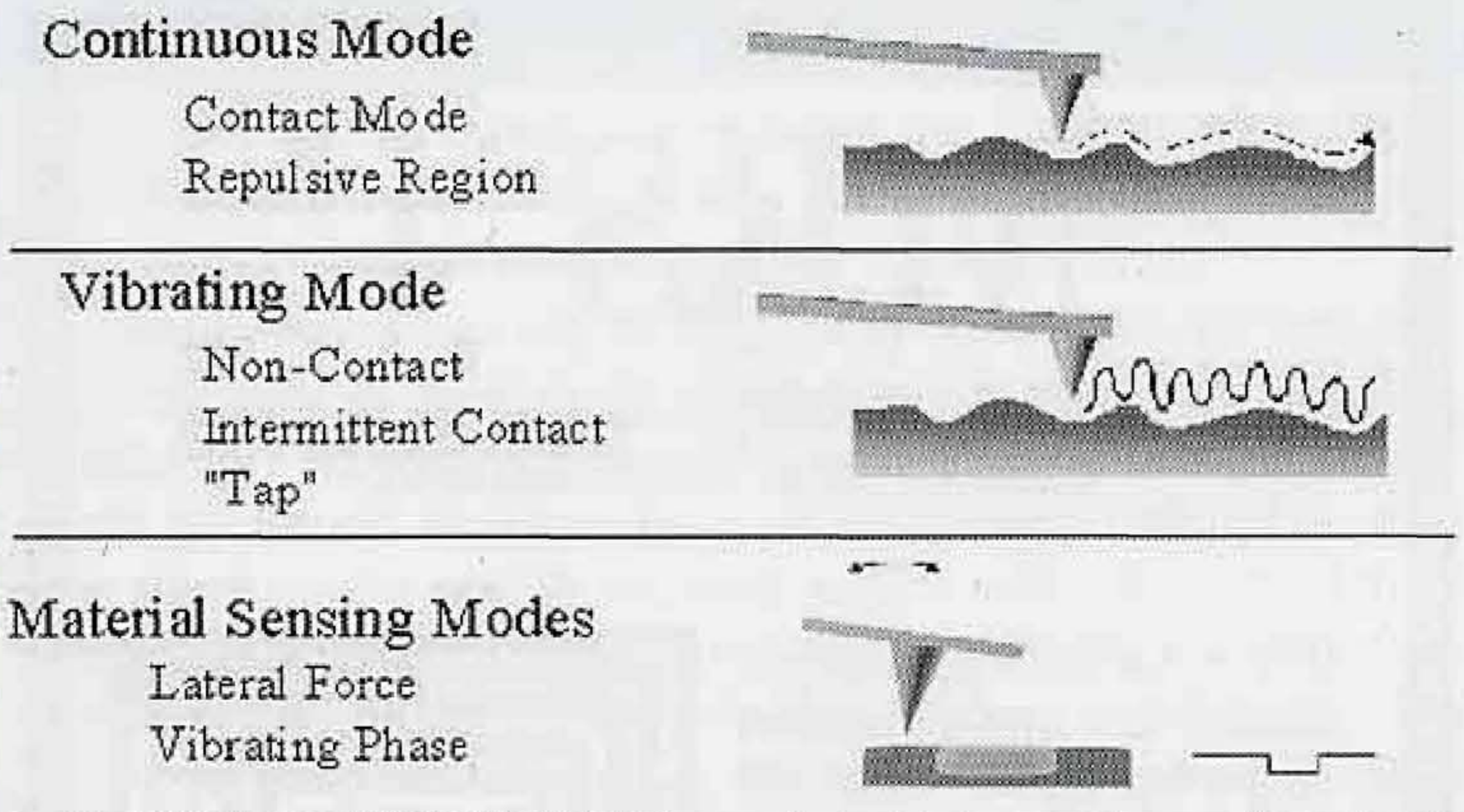

Figure 8. SPM scanning modes. Top: continuous, contact mode in repulsive region; Middle: vibrating mode, non-contact or intermittent contact ("tapping"). Bottom: material sensing modes that use lateral force or vibrating phase to measure surface properties such as friction.

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 it 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

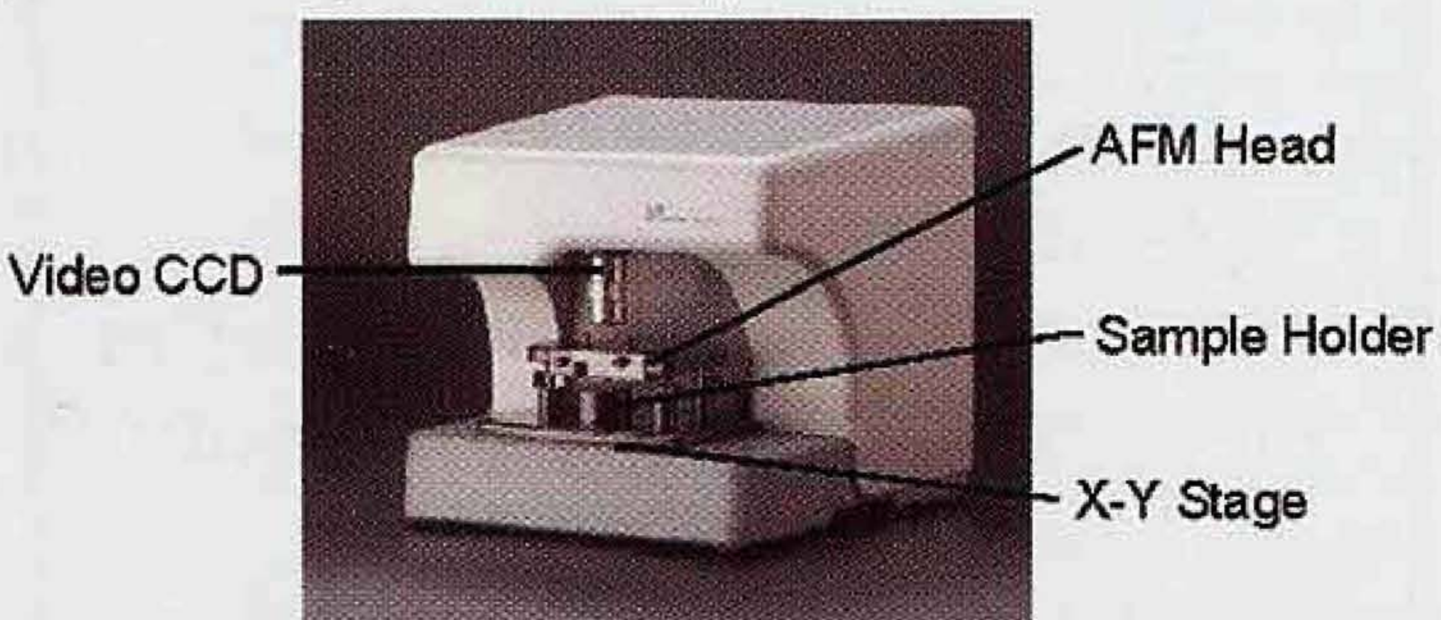

Figure 9. Tabletop atomic force microscope (Pacific Nanotechnology).

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. ■

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