CONDUCTING SCANNING PROBE MICROSCOPE WITH ENVIRONMENTAL CONTROL

BACKGROUND OF THE INVENTION

This invention relates to scanning probe microscopy, and, more particular to a conducting scanning probe microscope for the simultaneous acquisition of electrical and topographical information about a surface.

The use of a probe for the simultaneous acquisition of electrical and topographical data has been described by several workers, including Bryant et al, "Scanning Tunneling and Atomic Force Microscopy Performed with the Same Probe in One Unit," *J. Microscopy*, 152:871–875 (1988), who used a scanning tunneling microscope to detect the deflection of a conducting probe. Others have used optical means to detect the deflection of a flexible probe. To date, most measurements of electrical properties have been carried out in ambient air, using optical detection of the deflection of a flexible force-sensing cantilever which is either made from a conducting material or coated with one.

As illustrated in FIG. **1**, a beam of light from a laser **1** is reflected off the back of a flexible cantilever **2** and into a position sensitive detector **3**. The cantilever and probe tip are coated with a conducting material **4** and a potential difference **5** is applied between the coating **4** and a conductive sample **7**. A means for detecting current flow **6** is connected in series with the conductive coating **4** on the cantilever **2**. The cantilever probe tip is then scanned over the surface of the sample, in contact with it, to generate a current signal indicative of the local conductivity in addition to the usual atomic force microscope image. It has been shown that the force and current vary as a surface is approached.

Others, such as Martin et al, "High Resolution Capacitance Measurement and Potentiometry by Force Microscopy," *Appl. Phys. Lett*., 52:1103–1105 (1988) and Yasutake et al, U.S. Pat. No. 5,440,121, have used a non-contact method to map out local capacitance or charge on a surface. This method is illustrated in FIG. **2** where, in addition to the DC voltage **5** applied between the conducting sample **7** and conducting coating **4** on a cantilever **2**, an alternating voltage **9** is applied at a frequency f by means of a summing amplifier **8**. This causes a fluctuation in the cantilever tip to substrate voltage which, in turn, causes the electrostatic force on the tip to vary, resulting in an oscillatory deflection of the tip at a frequency f. The magnitude of the force, F, depends on the capacitance between the tip and sample according to

$$F = \frac{1}{2}V^2\frac{dC}{dz} \tag{1}$$

where V is the voltage applied between the tip and sample, and dC/dz is the capacitance gradient between the tip and sample. In a variation of this method, Tohda et al, U.S. Pat. No. 5,468,959, described a conducting probe microscope in which the variation in the electrical force while scanning is nulled by application of an opposing magnetic force. The measurements reported to date in the art have been carried out in ambient air.

It has long been recognized that contamination plays a major role in the local conductivity of a surface, as pointed out, for example, by Anselmetti et al, "Combined Scanning Tunneling and Force Microscopy," *J. Vac. Sci. Technol*. B12:1677–1680 (1994). For this reason, some workers have operated conducting-probe microscopes in ultrahigh vacuum. In this arrangement, shown in FIG. **3**, the laser **1** and detector **3** may be placed outside an ultra-high vacuum chamber **12** with the optical beams, **15**, **16** entering and leaving via a window **13**. The cantilever **2** and sample **7** are mounted inside vacuum chamber **12** and connected to a voltage source **5** and current sensor **6** via electrical feedthroughs **14**. The entire chamber is evacuated by means of a pumping system **17**.

While the use of ultrahigh vacuum can give reproducible results, it is extremely inconvenient to prepare and load samples for this environment. Even more limiting is the fact that certain important samples, such as biological material, cannot survive the vacuum environment. O'Shea et al, "Characterization of Tips for Conducting Atomic Force Microscopy," *Rev. Sci. Instrum*. 66:2508–2512 (1995) have suggested the use of a liquid as a simple means of protecting the sample and tip. Marrian et al, U.S. Pat. No. 5,504,338, have made the same suggestion for the operation of a device using a conducting probe to carry out lithography on a semiconductor surface.

While a liquid covering might well control the presence of water contamination layers that lead to spurious surface conduction, it does not, of itself, permit reliable recording of surface electrical properties. This is because of the inevitable presence of dissolved molecular oxygen which, in the presence of an electric field, is easily reduced to hydrogen peroxide. This process, of itself, gives rise to spurious current through the conducting probe. Even more problematic is the consequent oxidation of molecules and atoms at the sample surface. If this occurs, it results in non-reversible changes in the current-voltage characteristics at the surface because the oxidation changes the properties of the surface.

Furthermore, it is often desirable to examine a surface without a covering liquid in place. For example, in prior art methods in which motion of the cantilever tip is detected, the covering liquid would lead to significant damping of the motion of the tip, resulting in loss of sensitivity. Accordingly, there remains a need in this art for an instrument which can simultaneously acquire electrical and topographical information about a surface, but without the drawbacks and problems of prior art techniques.

SUMMARY OF THE INVENTION

The present invention meets that need by providing a conducting scanning probe microscope in which the atmosphere around the sample is controlled, but is not under vacuum. In this manner, molecular oxygen is eliminated from around the sample, preventing surface oxidation of the sample. Further, the thickness of any adsorbed water layer at the sample surface is controlled by controlling the relative humidity of the atmosphere around the sample, but otherwise the sample is maintained at or near ambient atmospheric pressure.

In accordance with one aspect of the present invention, a scanning probe microscope for measuring the characteristics of a surface of a sample is provided and includes a probe for scanning the surface of a sample to be measured and a sample stage which is adapted to position a sample in the microscope. In a preferred embodiment, the microscope is a conducting atomic force microscope. The microscope also includes a source of voltage in communication with the probe and the sample and a detector for measuring the electrical current to or from the probe and the sample. The probe and the sample are positioned within an enclosure which isolates the probe and the sample from the ambient environment, and the enclosure includes a gas inlet and a gas outlet for controlling the environment in the enclosure to