APPARATUS FOR IMAGING LIQUID AND DIELECTRIC MATERIALS WITH SCANNING POLARIZATION FORCE MICROSCOPY

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BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates generally to imaging of liquids, liquid films, liquid droplets, weakly bound particles on surfaces, and surfaces of thick dielectric samples using scanning force microscopy (SFM).

2. Description of Related Art

Atomic force and scanning force microscopes have been used to image solid surfaces with regions of electrostatic charge or dielectric material using metal or metal-coated cantilever probe tips. However no technique has been successfully used, including scanning force microscopy and atomic force microscopy, to image the nanometer-scale structure of liquid films, surfaces, and adsorbates. It is important to understand the growth of thin films of water because water films alter the adhesion and lubricating properties of surfaces and the reactivity of solids with ambient gas molecules. Additionally, water is a common solvent and cleaning agent. A method to study the growth of thin films of water would be important for physics, chemistry, and biology. In chemistry, the contact angle of water is used to measure the chemical activity of the surface. In biological processes, water films are critical for ion transport. In the semiconductor industry, water is used to wash off surface residues including and similar to reactants and acids. It is very important to know of the presence of residual liquid residues on the surface. Several studies have been recently devoted to the layering and orientation of water molecules on surfaces (Q. Du, E. Freysz, Y. R. Shen, *Phys. Rev. Lett.* 72, 238, 1994; J. D. Porter and A. S. Zinn, *J. Phys. Chem.* 97, 1190, 1993; J. N. Israelachvili, *Chem. Scr.* 25, 7, 1985; *AC. Chem. Res.* 20, 415, 1987; J. Glosli and M. Philpott, *Proceedings of the Symposium on Microscopic Models of Electrode-Electrolyte Interface, Electrochem. Soc.* 93-5, 90, 1993). Ice-like structures have been predicted for the first layers of water molecules, but no experimental evidence is available to validate the predictions. A method and apparatus to image water surfaces with nanometer resolution would make it possible to study many basic aspects of wetting, including condensation, evaporation and chemical reactions. With such a tool, the validity of growth models and the structure of the first layers could be investigated at the molecular level. If this type of tool were available it would also enable people to map the location of liquid droplets in the nanometer range.

Modern scanning force microscopes (SFM) like the scanning tunneling microscope (STM) and the atomic force microscope (AFM) can be used to obtain atomic-scale resolution of solid surfaces. SFM uses a probe tip attached to a spring, normally in the form of a lever or cantilever. Any of a number deflection sensor systems is used to measure displacement of the spring from its rest position under the influence of external forces, typically coming from the sample to be imaged.

SFM cantilever probe tips are available in a range of force constants (k), typically in the range between about 0.01 and 100 Newtons per meter (N/m). The resonant frequency (f) of the probe tip can typically range from 1 to 200 kHz. Probe tips are generally available with radii (R) of about 5 to about 0.01 micrometers (μm). Many materials have been used for probe tips, including tungsten, platinum, other metal alloys, and semiconductors such as silicon or silicon nitride. Diamond has also been used as a SFM probe material. Conductive coatings have been applied to otherwise nonconducting probe tips.

Sometimes the SFM cantilever probe is used as a null displacement sensor, where a feed back loop in the SFM apparatus applies a compensating force to balance external forces from the sample. Operated in this mode, the SFM cantilever probe is maintained in a position as close to its rest position as possible. As the probe is rastered across the sample, the magnitude of the force required to prevent the probe from deviating from its rest position is measured and plotted as a function of probe location over the sample. The resulting two-dimensional plot is the image obtained under constant distance constraints.

More frequently, however, the SFM cantilever probe is allowed to deflect in response to external sample forces and, as the probe scans the sample, the magnitude of the deflection is measured and plotted as a function of location over the sample. This two-dimensional plot is the image. There are many types of sensor systems that can be used to measure the displacement of the cantilever. One of the most popular is the optical lever, using a segmented or differential photo diode (G. Meyer and N. M. Amer, App. Phys. Lett. V 53, p 1045, 1988; G. Meyer and N. M. Amer, App. Phys. Lett. V 56, p 2101, 1990). Other types of displacement sensor systems include an optical interferometer which may be based on optical fiber technology, electron tunneling sensors, capacitance based sensors, and piezoresistive sensors that directly detect lever deflection.

An SFM can be operated in a contact mode or in a non-contact mode. In the contact mode, the probe tip is pushed against the sample surface so the surface and probe tip are in close mechanical contact. In the contact mode, the lateral spatial resolution is approximately the width of the contact, about 1 nm. In the non-contact mode the probe tip is typically 5 nm to 1000 nm from the surface, a great distance compared to atomic bond distances. The probe tip and sample surface interact by long range forces such as van der Waals, electrostatic and magnetostatic forces. If the probe tip and/or sample are submerged in a liquid, they may interact via variations in chemical or electrochemical potentials. In air the lateral resolution for a probe tip of radius R that is separated from the sample surface by a distance D, is about $(2 D R)^{\sim 1/2}$ if D is smaller than R. If D is larger than R, the lateral spatial resolution is about equal to D.

Because liquid surfaces will be disturbed in a contact mode, liquids must be imaged in the non-contact mode. The SFM is operated in the non-contact mode by measuring any of several parameters. The deflection or displacement of a cantilever probe is one common measurement parameter. Another is modulation of an AC signal applied to the probe. Alternatively, the probe tip can be mechanically vibrated, and the effect of sample interaction with the vibration measured. Monitoring the change in mechanical vibration measures force gradients rather than magnitude. For solid samples, it is also possible to make measurements in a hybrid mode, sometimes referred to as a "tapping" mode, wherein parameters measured for contact and non-contact imaging are combined.

Van der Waals forces are always present and can be used for imaging. However, because they are very weak (the force