Surface Charging and Photoeffects on Semiconductor and Insulator Surfaces in AFM

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Having seen these effects often enough, and fielded questions on them from fellow AEM users, we'd like to share our speculations about surface charging effects on semiconductor and insulator surfaces. We do not claim the following to be authoritative, but just what we consider a reasonable explanation for some strange phenomena we have observed.

Q: Can charging have an appreciable effect on an Atomic Force Microscope (AFM) probe?

Yes! Of this we are absolutely sure. Marc Unger demonstrated this once by holding a wire connected to a Si sample while he dragged his foot across a carpet. His Si_3N_4 AFM probe (Au coated or not) would visibly leap toward or away from the sample surface, a deflection of tens of micrometers. (This is not recommend as a daily parlor trick as, if the cantilever comes too close to the sample, there will be an electrostatic discharge (spark) between the cantilever and the sample, which *does* damage the cantilever, and could damage the AFM laser! Applying a DC bias between the sample and tip also caused the cantilever to move. With the cantilever ~250 µm from the sample, a 50 VDC bias generated a 28 nN force on the cantilever. Charge-charge interactions are extremely long range, and they can often have an effect even before the probe is brought anywhere close to imaging a surface. This is all easily understood if the tip-sample interface is thought of as a capacitor, and a rough calculation is done of the number of charges expected for a given voltage.

Q: Can charging interfere with sample imaging?

When there is not a reasonably conductive electrical path from the sample surface to the tip of the AFM probe, and/or when something actively charges either of these two, large charge imbalances can certainly develop and we've seen them have major effects. Common symptoms include:

drifting tip deflection or oscillation magnitude at fixed height, even before engaging.

sudden changes in probe force, sometimes leading to disengagement during scanning, or premature engagement during approach.

an inability to engage the sample surface at all using "normal" engagement parameters.

excessive noise in the feedback-controlled deflection or oscillation signal.

With a Si_3N_4 probe close to a silicon sample, Marc Unger has observed "self-tapping" at between 0.1 and 10 Hz. The probe bends closer and closer to the sample, touches the sample, and springs back all at once. This is actually a pretty amazing demonstration of progressive charging. If the charge was not increasing, the cantilever position would be constant.

Q: What causes sample charging?

We suspect there are three common causes of sample charging, although we do not have experimental evidence for any of these. The first occurs on insulating samples, and mica is the classic example. When a sheet of mica is cleaved, the newly-exposed surface often seems strongly charged. As mica is an insulator, this charge can not escape.

A second possible source of charge is bleed-off from the 400-some volts applied to the AFM's piezos and from other electrical components in the microscope. Although generally built of conductive metal and grounded through their umbilical cords, AFM's may sometimes be able to pump energy into the sample or probe faster than they can bleed it off. On home-built systems, or systems optimized for STM, the microscope may be only partly grounded, or not grounded at all.

The third effect is seen on semiconducting samples, and while it is only supposition, it makes sense given the evidence we have. Most semiconductor to metal junctions are diodes: that is, they allow current to flow in one direction but not in the other. Additionally, small-bandgap semiconductors (Si and Ge, for example) are readily photoexcited by the red laser light used in most AFM systems, and almost all semiconductors are excited by the trickle of UV light emitted by fluorescent lamps. When both the rectification and photexcitation conditions are present, the semiconductor can act as a solar cell, developing a sizable, light-intensity dependent potential difference between itself and the microscope on which it is mounted. Note that this can also occur for semiconducting probes mounted in metallic probe mounts, and thus both sample and probe can have the same charge. Silicon probes used on silicon samples are the most common example of this.

Q: What can I do to prevent this?

When there's no active charging going on, tools from outside the science arsenal often seem to do the trick. Anti-static devices based on ionized gas and/or ionizing radiation can effectively neutralize the charge on an insulating surface. We suggest either a "Staticmaster" Po-210 α particle-emitting strip (~$35), available from major photography supply houses, or a "Zerostat" piezo-electric ionizing gun (~$7O), available from Aldrich Chemical Co. (Milwaukee, WI). "Staticmaster" devices are often most effective if taped or otherwise held in place over the aperture housing the sample and probe during imaging. Note that these fixes are only effective when the troubling charge is residual on the sample, and not actively generated by any component of the sample or microscope.

When up against active charging of any sort, the sure-fire way to solve it seems to be providing a low-resistance electrical path from the sample to the probe tip. This can be difficult with insulators, but shorting the sample stage to the probe mount should eliminate any active charging taking place in such systems.

When you have a solar cell on your hands, the easiest part to defeat is the diode formed where the semiconductor meets the microscope. In our experience, electrically insulating a semiconductor from the microscope only makes matters worse. Instead, using a carefully selected metal that forms an "ohmic" contact to the semiconductor you are using should be most effective. For Si, indium acts as an effective ohmic contact. An easy-to-use liquid 'glue' can be made by alloying indium with gallium. Simply touching a blob of indium to a small bead of Ga and allowing the two to form a eutectic over a few hours provides a conductive liquid alloy that effectively discharges any Si sample and also holds the Si in place on a sample puck or stage. However, be forewarned: the toxicity of indium and gallium are thought to be low but have not been thoroughly studied, and the eutectic is extremely difficult to remove from both sample and substrate once applied. Marc Unger reports success with Si using carbon conductive tape, also, though we can not vouch for this being an "ohmic" contact.

Another sure-fire solution is to image the sample under a liquid (although we suspect a polarizable liquid like water or alcohol may be needed to eliminate a particularly nasty problem). Liquids reduce the effective range of electrostatic interactions to the point where they are generally negligible. ■

A postscript:

ImageSXM: A Modified Version of NIH Image for SPM Applications

Those who like using NIH-Image or a Macintosh for image processing should by all means take advantage of the hard work and generosity of Dr. Steve Barrett. His freeware "ImageSXM" program effectively integrates a slew of handy microscopy features into NIH-Image, and makes importing images from almost any scanning microscopy technique a snap. To find out more about ImageSXM, or to download a copy of it, visit one of the following sites:

Liverpool, U.K. (Univ. of Liverpool): http//reg.ssci.liv.ac.uk/
Pasadena, CA, U.S.A. (Caltech): http//reality.caltech.edu/imagesxrn.htrn [Note: NIH-Image is a freeware image processing program available at: http://rsb.info.nih.gov/nih-image/ and there is the address for the NIH-Image mailserver in the FAQ--MT.j