Key to Midterm Examination for
"Characterization and Manipulation at Nanometer Scale"
April 14, 2009

ID Number: ________________ Name: _______________________

Q1. This is a problem related to the scanning tunneling microscopy (STM).

(a) Please describe how the scanning tunneling microscopy works and explain why scanning tunneling microscopy can achieve atomic resolution on the surface of a conducting substrate. (10%)

(b) List the impacts the STM brought to the advancement of nanoscience. (5%)

A: (a) The scanning tunneling microscopy operates based on the quantum phenomenon of tunneling. When two conductive objects (tip and sample) are brought within about 1 nm and a suitable bias voltage is applied, a tunneling current will flow. By controlling this current with a feedback circuitry during the two-dimensional scan of the tip, the surface topography of the sample can be imaged. In the tunneling regime, if the distance between the tip and the surface decreases by 1 Å, the tunneling increases by about one order of magnitude. Therefore, if an atom sticks out by 1 Å (or more) than other tip atoms, the major contribution of the tunneling would come from that outmost atom. The surface atom directly underneath that tip atom would be imaged. That is why STM can achieve atomic resolution easily.

(b) The potential impacts for the advent of the STM:

1. It employs a fine probe to obtain the imaging, which is different from the traditional beam-type microscopy.
2. Due to the interaction through a tangible probe, the sample surface can be modified or decorated, which opens up a new avenue for scientific research.
3. The advent of atomic force microscopy (AFM) and the subsequent whole family of scanning probe microscopy (SPM) is founded on the working principles of the STM.

Q2. This is a problem related to the atomic force microscopy (AFM).

(a) Please describe the differences in operation among the three common imaging modes, ie. contact, non-contact, and intermittent contact, of the atomic force microscopy, and the advantage and disadvantage of these modes. (10%)

(b) Describe how to operate the “lift mode”? and why is it necessary? (5%)

A: (a) 1. Contact mode: the probe is pushed against the sample surface in the repulsive force range.

Advantage: high imaging resolution can be achieved.

Disadvantage: the sample and probe are easily damaged.

2. Non-contact mode: the probe is brought toward the sample surface in the attractive force range and the imaging is performed with the probe vibrating with small amplitude.

Advantage: the interaction between the sample and probe is reduced and they can sustain for a longer lifetime of imaging.

Disadvantage: high resolution images are more difficult to obtain.

3. Intermittent contact mode: the probe vibrates with large amplitude so that it will make an intermittent contact with the sample.

Advantage: the probe is kept away from the sample for most of time, so the conditions of the probe and sample can be preserved longer meanwhile the high imaging resolution can be achieved.

(b) The “lift mode” is operated with scanning the sample twice at each line scan. One is taken at a close range of the sample to acquire the topographic information of the sample surface, e.g. using the tapping mode, and the data is recorded in the computer. Then, the second scan is performed at a distance from the sample (normally > 50nm) following the trajectory recorded in the first scan.

When a specific physical property, usually detectable in a longer range, is sought from a sample using the AFM, the geometric factor of the sample surface has to be eliminated in order to obtain the pure information being concerned. In a normal operation, both the AFM and property-defined images are displayed side by side for comparison.

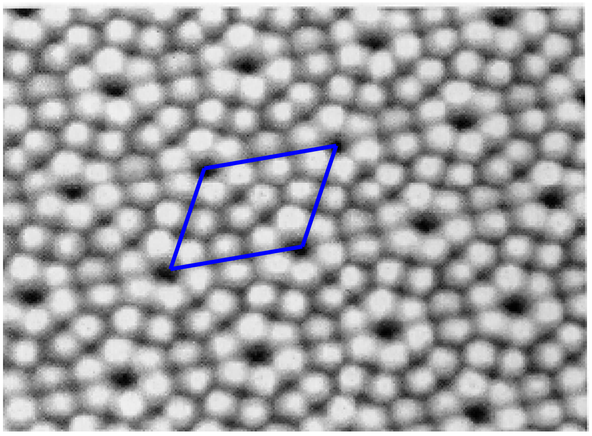
Q3. This is a problem related to the reconstruction of the Si (111) surface.

(a) The STM image of a reconstructed Si(111) surface is shown below. Please draw the unit cell of this reconstruction and name it with reference to the truncated (111) surface structure of the Si crystal. (5%)

(b) Name and describe the key features within a unit cell of this reconstruction. What is the driving force behind this complicated reconstruction? (10%)

A:

(a) The reconstruction is named Si(111)7x7 and the unit cell is drawn below:



(b) The main features in the unit cell of Si(111)7x7 reconstruction include:

i. Dimers: they are formed by two neighboring Si atoms and there are 15 pairs delineating the edges of the two triangular halves of the unit cell.

ii. Adatoms: there are twelve of them sitting on the top surface.

iii. Stacking faults: Along the (111) direction, a bulk Si crystal has a stacking sequence following a normal fcc crystal if each bi-layer is considered as the stacking unit. Below the top adatom layer of a reconstructed Si(111) surface, there lies a Si bi-layer. On the top one of this bi-layer, atoms in one of triangular half have occupied the positions that are seen as faults.

This model is also called DAS model, which is nowadays well accepted as the correct model to describe this complicated reconstruction.

Q4. This is a problem related to the spectroscopic measurements.

(a) When the electronic properties of a nanoparticle are measured by optical spectroscopy, both blue and red shifts, comparing to its bulk counterpart, can occur. Please explain the causes of the blue and red shifts, respectively. (10%)

(b) The vibrational properties of a nanoparticle, when measured by optical spectroscopy, can result in a blue shift in reference to its bulk counterpart. Please explain the cause. (5%)

A: (a) Blue shift: Due mainly to the energy-gap widening because of the size effect.

Red shift: Bond shortening resulted from surface tension causes more overlap between neighboring electron wavefunctions. Valence bands will be broadened and the gap becomes narrower. Therefore, the optical transition will shift toward the red band.

(b) Blue shift: Due mainly to the bond shortening resulted from surface tension.

Q5. This is a problem related to the transmission electron microscopy (TEM).

(a) Following the path of the flying electrons from the beginning to the end of their trip, name the parts (lens, apertures, etc.) in an electron optics column of a transmission electron microscope. (10%)

(b) Use diagrams and statement to explain how an electromagnetic lens works. (10%)

(c) Explain the basic principles of specimen preparation for transmission electron microscopy. In particular, explain the special considerations in preparing nanomaterial specimens. (10%)

A: (a) Electron gun (including filament, Wehnelt, anode), condenser lens (including condenser lens 1, condenser lens 2, condenser aperture), specimen (including specimen holder), objective lens, objective stigmator, objective aperture, select area aperture, intermediate lens (usually more than one), projector lens (one or more), fluorescence screen (or CCD camera or other detector).

(b) The electromagnetic lens consists of a coil of wire wounding and a pole piece made of paramagnetic alloy. Electric current in the coil produce a cylindrically symmetrical magnetic field. The pole piece, inserted in this magnetic field, is magnetized and make a strong magnetic field in a small region at the center of the pole piece. This strong magnetic field, still cylindrically symmetric and is the “lens” for electrons or other charged particles. Electrons moving into this magnetic field are accelerated according to the Lorentz force law

$$\mathbf{f} = q\, \mathbf{v} \times \mathbf{B},$$

where **f** is the force on the electron, q the charge of the electron ($q < 0$), **v** the velocity of the electron, and **B** is the magnetic field. With the magnetic field being cylindrically symmetrical to the axis which is the general direction of the electrons motion, electrons passing through this magnetic field are deviated and converge to a point on the exit side of the magnetic field, very much the same as the light rays converged to a focal point after passing a converging lens.

(c) Specimen for transmission electron microscopy examination must be made small, thin, dry, electrically conductive, and firmly attached to the specimen holder. The space in the pole piece of the objective lens is limited to only few mm and the standard grid for attaching specimen is a thin foil of 3 mm diameter. Normally used high energy electrons can penetrate few hundred nm of solid materials. So this is the limit of specimen thickness. Coating for good electric conductivity is generally not advisable in TEM as coating materials interfere with observation. But making the specimen thin allows most electrons to transmit through the specimen and not accumulated on the specimen. This in turn help the electric stability of the specimen. Evaporation of water or other organic vapor from the specimen is harmful to both the specimen and the instrument. This should be avoided by making the specimen dry before entering the microscope. Specimens should also be firmly attached to the grids so that they would not fall during the insertion or extracting processes. To make the bulk specimen thin, there are mechanical, chemical, or ion bombardment techniques: Ultamicrotomy is the technique of cutting thin slices using a glass or diamond blade. Metal or alloy specimens can be thinned in etching fluid with an electric voltage applied. Mechanical thinning followed by ion bombardment (ion milling) is another common employed method. As for nano-materials, they are already small and thin. These are generally treated as other powder specimens. They are deposited on the copper grid, usually with holey carbon grid attached, and then inserted into the TEM for observation without further treatment.

Q6. This is a problem related to the X-ray microscopy.

Please describe the limiting factors to the performance (resolution, contrast and applications) of X-ray microscopy in scanning and in full field imaging modes. (10%)

A: X-ray optics:

In scanning mode, the ability to generate tight focus.

Detecting system:

In scanning mode: x-ray detector and diffractomator, photo electron detector and energy analyzer, or illuminacence detection.

In full field imaging mode: phase contrast devices; effective conversion from X-ray to visible light or electron.

.. etc.,