ATOMIC FORCE MICROSCOPY

The Experiment: Film Growth and Nanostructure Formation

Thin films and nanostructures are used extensively in electronic device technologies from highspeed computer chips to solid state lasers and many kinds of sensors. Understanding the physics of thin film deposition and the way in which nanostructures are formed is therefore critical to much of modern microelectronics technology. In this experiment we will gain some insight into the basic mechanisms of film growth and nanostructure formation. We will use Atomic Force Microscopy (AFM), one of the most popular methods of scanning probe microscopy (SPM), to image the surfaces of thin films. This technique is capable of very high spatial resolution –all the way down to atomic dimensions under some conditions.

The growth of thin films can take various forms, depending on the deposition conditions (e.g., the temperature of the substrate, the rate of arrival of atoms on the growing surface, and the chemical bonding characteristics of the arriving atoms together with those of the substrate itself). Typically, film growth occurs under the following conditions: atoms or molecules impinge on substrate with thermal energies (~10's of millielectron volts per atom). The arriving atoms do not immediately bond with the substrate but diffuse across the substrate surface until they find a site that is favorable for chemical bonding. This might be at the corner of a step-edge (see Fig. 1) or on the edge of an "island" that has nucleated on the surface.



Fig.1: Schematic of step edge on a surface showing an adatom diffusing along the edge.

The initial stage of growth (the "nucleation stage") therefore consists of a collection of small (few nm) islands that are only a single atom in height. As more atoms arrive at the substrate surface these islands will grow in size until their boundaries start to intersect each other. This is known as "island coalescence" and corresponds to the completion of a single atomic "mono-layer". During this process, it is likely that atoms will start to form a second layer before the first one is completed (you will be familiar with this phenomenon if you have played the computer game "Tetris"!). The atoms on the second level have two choices: they can either attach to an edge on the second level or they can diffuse down to the first level and fill in an empty space there. Obviously, the second option ("layer-by-layer" growth) is preferable if one is trying to get a smooth film. However, there is usually a substantial energy barrier that inhibits atoms from

downward diffusion. The reason for this barrier is that an exposed edge site has a higher potential than a corner site because an atom sitting at an edge site has the least number of nearest neighbors (see Fig. 2). For tetrahedral bonding, as in Si, each atom likes to have 4 nearest neighbors, and for close packed metals like Au or Cu the preferred coordination is 12 nearest neighbors. The resulting barrier is called the Ehrlich-Schwoebel barrier and it can be as much as a few tenths of an eV (much bigger than the ambient thermal energy).



Fig. 2: Schematic of Ehrlich-Schwoebel potential barrier (lower panel) corresponding to a step edge (upper panel).

The presence of this barrier is what makes the physics of thin film growth interesting (and challenging for technological applications).

Depending on the relative height of the E-S barrier and the available thermal energy (supplied by the substrate temperature), and the rate of arrival of atoms, the growth mode of the film can proceed in one of three ways:

1. **Frank-Van der Merwe regime**. This is the ideal type of two-dimensional layer-by-layer growth to achieve atomically smooth films and is promoted by: low barrier or high substrate temperature, and low atomic flux

2. **Stranski-Krastanov growth**. The first few monolayers are layer-by-layer; then threedimensional islands begin to form. Conditions are: moderate barrier height, intermediate temperature and flux arrival rate. S-K is a popular regime for nanostructure formation, such as quantum dots.

3. Volmer-Weber growth. This is a "worst-case" scenario for film growth, when the barrier height is large, the substrate temperature is low and atom flux is high. Only three-dimensional clusters are formed. The clusters will be close together if the surface diffusion coefficient is small and/or the flux is high. Clearly this regime is also of interest for nanostructure formation.

These three growth modes are schematically illustrated in Fig. 3.



Fig. 3: The three most common modes of film growth (Zangwill, Physics at Surfaces, CUP).

Several aspects of film growth and nanostructure formation are therefore of interest:

- Which *mode* is the film growing in?
- What is the *surface morphology* of the film (steps, islands, clusters, etc.)?
- If nano-clusters are forming, how does their *lateral size* grow with deposition time? This is known as "coarsening".
- How does the roughness of the film evolve as a function of deposition time?
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These questions are still the subject of active research and, as yet, there is no complete theory describing the mechanism of film growth. However, considerable progress has been made during the past decade using a combination of analytical rate equations and computer simulations. A key concept is the idea that the films grow in a "self similar" geometry. For example, a micrograph of the film surface at time t_1 during growth would look qualitatively identical to an image of the surface at a later time t_2 , just scaled up by some magnification factor. This implies a scaling relationship such that the average island size, L, scales like t^{α} , where α is a scaling exponent, and the root mean square (RMS) roughness:

$$>^{1/2}$$
 scales like t^{β} .

In general the scaling exponents α and β are different, but can be related to each other.

Quantities to measure

In this experiment we will deposit gold on a smooth substrate of silicon, using a process known as "sputtering". Briefly, sputtering is a process whereby atoms are removed from a target by bombarding it with Argon ions. Since Argon is an inert gas it does not react with the target atoms or the substrate. In our case the sputtering target is gold and is placed in a background gas

of argon at \sim 50 mTorr pressure. If a DC voltage of about 5000V is applied between the target and ground (with the target as the cathode) a plasma of Argon ions forms above the target surface and argon ions are accelerated towards the target. At 5000 V potential, these ions have sufficient momentum to knock gold atoms out of the target, which are then deposited on the substrate at a rate of about 0.3 monolayers per second.

We will deposit films of gold for various times, chosen so that we cover the range of thicknesses from submonolayer to about 100 nm. [Question: how to choose the deposition times for each film? Hint: think about the scaling described above]. Do we want equally spaced times?

We will take AFM images of each film, making sure that the resolution and the range of the scans are sufficient to measure the relevant quantities such as island size and rms roughness. Image processing software (both that provided by the AFM manufacturer and freeware such as NIH Image) is useful for extracting quantitative information from the AFM images.

The analysis will consist of comparing our AFM data with what is expected from theory, in terms of the growth mode and in terms of the film's coarsening and kinetic roughness. We will measure the scaling exponents and compare them with estimates from current theories and simulations. Some references listed below contain more details on the current theoretical situation.

Use of the Scanning Probe Microscope/Other Samples

Our microscope was made by Quesant Instrument Corporation. You should study the manual and discuss the procedures with Ramon before attempting to use the microscope. Much useful information can be found on the Quesant.com web site. The manipulation of the microscope can be challenging and the software is somewhat buggy.

A calibration sample is available and this should be looked at first. Other interesting samples are a hologram from a credit card, a solar cell, a floppy disc. The gold samples discussed above may not be available. You are invited to investigate some samples that you find of interest. The gallery at Quesant.com will suggest some ideas. Freshly cleaved mica is a good substrate for AFM samples and mica is available in the lab. You may be able to resolve the steps in the mica lattice. Preparing suitable samples is something of an art, and you should do some research on sample preparation.

The scans you make can be saved and representative ones should be included in your lab report.

Introduction to Scanning Probe Microscopy (SPM)

(Thanks to J.W. Cross © 2003)

Scanning probe microscopy covers several related technologies for imaging and measuring surfaces on a fine scale, down to the level of molecules and groups of atoms. The development of SPM techniques, starting in the early 1980's, has revolutionized surface science and has had a huge impact on device technologies such as microelectronics, DVDs, and high-density magnetic recording.

SPM techniques share the concept of scanning an extremely sharp tip (3-50 nm radius of curvature) across the object surface. The tip is mounted on a flexible cantilever, allowing the tip to follow the surface profile (see Fig. 4).

When the tip moves in proximity to the investigated object, forces of interaction between the tip

and the surface influence the movement of the cantilever. These movements are detected by selective sensors. Various interactions can be studied depending on the mechanics of the probe

Probe Techniques

The three most common scanning probe techniques are:

Atomic Force Microscopy (AFM) measures the interaction force between the tip and surface. The tip may be dragged across the surface, or may vibrate as it moves. The interaction force will depend on the nature of the sample, the probe tip and the distance between them.

Scanning Tunneling Microscopy (STM) measures a **weak electrical current** flowing between tip and sample as they are held a very distance apart.

Near-Field Scanning Optical Microscopy

(NSOM) scans a very small light source very close to the sample. Detection of this light energy forms the image. NSOM can provide resolution below that of the conventional light mi-



Fig. 4: Above: A probe used for atomic force microscopy.

Below: How a probe tip scans over a sample (not to scale).



croscope. There are numerous variations on AFM techniques; AFM may operate in several modes that differ according to the force between the tip and surface.

Mode of Operation	Force of Interaction
Non-contact mode	weak (attractive) - vibrating probe
Intermittent contact mode	strong (repulsive) - vibrating probe
Lateral force mode	frictional forces exert a torque on the scanning cantilever
Magnetic force	the magnetic character of the surface is imaged
Thermal scanning	the distribution of thermal conductivity is imaged

Scanning. The probe (or the sample under a stationary probe) generally is moved by a piezoelectric tube (see Fig. 5). Such scanners are designed to be moved precisely in any of the three perpendicular axes (x,y,z). By following a raster pattern, the sensor data forms an image of the probe-surface interaction. Feedback from the sensor is used to maintain the probe at a constant force or distance from the object surface. For atomic force microscopy the sensor is a positionsensitive photodetector that records the angle of reflection from a laser bean focused on the top of the cantilever:



Fig. 5: Schematic of AFM system showing the optical method of measuring cantilever deflection.

<u>Probe Tip</u>

The sharpness of probe tips that are used in AFM is a key factor determining the lateral resolution. Single wall carbon nanotubes (see Fig. 6) have been used to achieve some of the highest resolution AFM images at the atomic scale.



Fig. 6: Structure of single wall carbon nanotube. The diameter of the nanotube illustrated on the right is about 1nm.

An example of an AFM image is shown in Fig. 7. The object is the surface of a stamper used in the mass production of CD's. Note that the vertical scale (about 200nm) is greatly magnified relative to the lateral scale (about 10 microns).



Fig. 7: AFM image of a stainless steel CD stamper.

Further reading:

SPM Inventors. The first scanning probe microscope was the scanning tunneling microscope (STM) of Binnig and Rohrer (Binnig, G., Rohrer, H., *et al.*, Phys. Rev. Lett., **49**, 57 (1982). Gerd Binnig and Heinrich Rohrer were awarded half of the 1986 Nobel Prize in Physics for their design of the scanning tunneling microscope. Ivan Amato's 1997 article, "*Atomic Imaging: Candid Cameras for the Nanoworld*" (Science **276** (5321):1982-1985), recounts the history of STM and AFM development.

Review of SPM techniques: J.W. Cross: Scanning Probe Microscopy (web article: http://www.mobot.org/jwcross/spm/).

Physics of Surfaces and Thin Films: A. Zangwill, *Physics at Surfaces*, Cambridge University Press (1992); John A. Venables, *Introduction to Surface and Thin Film Processes*, Cambridge University Press (2000).

Studies of surface roughening of metal films: Temperature and orientation dependence of kinetic roughening during homoepitaxy: a quantitative x-ray scattering study of Ag, W.C. Elliott, P.F. Miceli, T. Tse, and P.W. Stephens, Phys. Rev. B **54**, 17938 (1996).