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Ultra high vacuum scanning tunneling microscopy to study thin film growth

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Rice University, 1994
Ultra High Vacuum Scanning Tunneling Microscopy to Study Thin Film Growth

by

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A THESIS SUBMITTED
IN PARTIAL FULFILLMENT OF THE
REQUIREMENTS FOR THE DEGREE
MASTER OF ARTS

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April, 1994
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ABSTRACT

An ultra high vacuum chamber was constructed to study thin film growth using scanning tunneling microscopy (STM). The sample stage is specially designed so that samples can be transferred in situ among various film preparation techniques. For first studies, Ru thin films were deposited at sub-monolayer coverages on C(0001) substrates. From our STM studies, it is found that Ru diffuses easily along the graphite surface, that Ru atoms tend to nucleate at defect sites, and that the first monolayer of Ru grows homogeneously on graphite. These results are in agreement with our recent Auger electron spectroscopy data.
Acknowledgments

I would like to thank and acknowledge all of the people who have helped me.

My advisor, Dr. Carl Rau for his guidance and eternal optimism.

Dr. Nanjiu Zheng for his guidance, patience, and scientific insight.

Dr. Rupert Pfundzelter and Gerold Steierl for their Ru/C(0001) results which motivated this project as well as their guidance and support.

Dr. Robert Cave for accepting me for who I am, believing in me, and always being willing to listen.

The Chemistry Department for allowing me to pursue this research project. Especially Dr. Brooks, Dr. Curl, Dr. Margrave, Dr. Hutchinson, and Julie Kudelka for all of their help and support.

My family for always believing in me.

And most of all to my friends whose support, encouragement and assistance have helped me to complete this project: Cass Sackett, Greg Odom, Mark Anderson, Sheila Lesser, Shahla Farhat, Ian McAlexander, Tish Poland, Jenifer Monks and Marie Villarba.
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Chapter 1
Introduction

With the recent revolution of information technology and the ever growing need for high density data storage, surface and interface magnetism in ultrathin films and multilayer structured magnetic materials has received worldwide attention. Reduced coordination numbers and slight changes in lattice constants, imposed by an adequate substrate surface, can cause deposited thin films to exhibit properties that are quite different from the properties of the bulk material. Many materials that are nonmagnetic in the bulk may become magnetic in the form of thin films. The overall goal is to be able to tailor the physical and chemical properties of new materials. In order to realize this goal, a deeper understanding about the interrelation between the magnetic and structural properties of materials is required.

Since these ultrathin films have been shown to exhibit properties that are very different from the bulk properties of the material, they provide a new type of system from which to gain more information about the interrelationship between structure and magnetism. This is especially useful because a lot of theoretical work has been done on two-dimensional (2D) magnetism. The 2D problem can be solved exactly for the Ising model[1, 2].

In the past, it was difficult to perform the experiments to test these theoretical predictions, but now, since film growth techniques have improved, it is possible to grow good quality ultrathin magnetic films on single crystal substrates and to measure their properties. As an example, Rau et al. found that a monolayer of vanadium on silver is ferromagnetic[3]. They were able to determine the critical exponent for the 2D phase transition. Their results agree quite well with the predicted value of 1/8 for the Ising model.

Theoretical work predicts that both Ru and Rh monolayers on Ag(001) or Au(001)
possess a ferromagnetic ground state[4, 5]. Experiments have been done for Rh on Ag(001) as well as Rh and Pd on Au(001), but no unambiguous evidence of ferromagnetism has been found[6, 7, 8]. Experimental evidence suggests that the discrepancy between theory and experiment is due to difficulties in growing the monolayer film. Schmitz et al. propose that the equilibrium structure of Rh on Ag(001) is a sandwich with a monolayer of Ag on top[9]. Mulhollan et al. find evidence that Rh can diffuse easily into the Ag substrate[7]. Other authors think that islanding may be an important issue[6, 7]. All of these "growth effects" are likely to destroy possible ferromagnetic order.

In order to further look into this, Pfandzelter, Steierl and Rau chose the graphite C(0001) surface as a substrate to avoid the growth problems associated with Ag(001)[10]. They selected graphite because it is atomically flat, relatively inert and should not interfere with the 4d electrons responsible for magnetism. Ru was chosen as the film material instead of Rh because it has a larger magnetic moment and a larger magnetic energy[5]. Also, Ru is hexagonal with an a lattice constant which is almost exactly double the a lattice constant of graphite, which means that epitaxial or pseudomorphic growth of Ru in a p(2x2) superstructure on graphite should be favored. Spin Polarized Secondary Electron Emission Spectroscopy (SPSEES) experiments were performed on the chosen system of Ru on C(0001), and the Ru monolayer was found to be ferromagnetic[10]. The growth mode was determined using Auger Electron Spectroscopy (AES), and it was found that the monolayer grows homogeneously on graphite. After the first layer is completed, it appears that islands are beginning to form.

The AES results used to determine the growth mode and structure of the films are not completely conclusive, so further studies of the growth modes and structure of ultrathin Ru films on graphite need to be done. Scanning Tunneling Microscopy (STM) was chosen for the further study because it provides direct, real-space, three-
dimensional topological information with atomic resolution as opposed to most other techniques, such as AES etc, which are indirect and provide information that is "averaged" over the sample surface. The STM technique also has the advantage that it can be used to study both the structure of the film as well as the growth mode. STM results have been obtained for the initial growth of Ru on C(0001).

The following chapters discuss the ultra high vacuum (UHV) chamber designed and constructed to study thin film growth and structure using STM. A review of STM is presented in Chapter 2, followed by a brief review of thin film growth (Chapter 3). The experimental apparatus is discussed in Chapter 4. Preliminary results for the growth of Ru on C(0001) are presented and discussed in Chapter 5, including a discussion of tip effects. A summary and the conclusions are given in Chapter 6.
Chapter 2
Scanning Tunneling Microscopy (STM)

2.1 Introduction and Basic Principles

Scanning Tunneling Microscopy (STM) is an excellent technique to use to study thin film growth because it provides direct, real space, local, three-dimensional topological information with atomic resolution, while most other methods of structural determination are indirect and give results that are "averages" over the sample. STM was invented at the IBM Zürich Research Laboratory in Switzerland by Binnig, Rohrer and their coworkers in 1981[11]. In 1986, they were awarded the Nobel Prize in Physics for that work.

Since its invention, STM has developed into a very important surface science technique and has inspired a whole family of scanning probe microscopies. Its popularity is due to the fact that it gives geometric and electronic information about surface structures with a resolution of 1 Å laterally and 0.01 Å vertically.

The underlying principle of STM is electron tunneling. This principle can be illustrated by looking at a particle in a box model for two solids placed near each other, as shown in Figure 2.1[12, p. 50]. Figure 2.1(a) shows the interaction when the two solids are separated by a macroscopic distance. From quantum mechanics, it is known that the Fermi level electrons can be found outside of the confining potential. If the distance between the two solids is reduced and a bias voltage is applied (see Figure 2.1(b)), the Fermi electrons can tunnel through the barrier, and a tunneling current can be detected through the vacuum gap. This metal-vacuum-metal tunneling was first observed in 1971 at the National Bureau of Standards in Washington D.C.[13].

This one-dimensional barrier problem is easily solved. In the limit of small gap
Figure 2.1 Potential wells and Fermi level wave functions for electron tunneling [12 p. 50]

distance and low bias voltage, the equation for the tunneling current is given by

\[ I \propto \frac{V}{d} e^{x(-A\phi^{1/2}d)} \]

where \( A \) is \( 1.025(eV)^{-1/2} \AA^{-1} \) for a vacuum gap, \( \phi \) is the difference of the barrier heights between the two electrodes, \( V \) is the bias potential between the two electrodes, and \( d \) is the gap distance [14]. From this equation, it can be seen that for a 1 Å change in the gap distance there is an order of magnitude change in the tunneling current. This exponential dependence on the gap distance gives the STM its high resolution.

In order to obtain such high resolution images of surfaces, the stability of the tip-sample gap distance needs to be on the order of \( \delta z \approx 0.01 \) Å and \( \delta x, \delta y \approx 1 \) Å [15]. To obtain this level of stability, a vibrational isolation system is necessary. Several methods of vibrational isolation have been used. The first was superconducting magnetic levitation, but it is not very common anymore because it is rather complicated. Two more common methods are two-stage spring suspensions and stacked metal plates.
with Viton damping between each pair. The springs are the most efficient system, but they become complicated when it comes to sample transfer and tip exchange because the instrument is not very rigid. The metal plates are easier to build and more rigid which makes sample transfer and tip exchange easier. The trend now is towards smaller more rigid instruments because vibrational isolation becomes less important when the whole instrument experiences the same vibrations.

The STM is a relatively simple instrument. A schematic is shown in Figure 2.2[15]. The tip is attached to a piezoelectric tripod such that the position of the tip can be precisely controlled by applying electric fields to each of the legs. A bias voltage is applied and the tip is brought close to the sample until a tunneling current is detected. The tip can then be scanned across the sample as the tunneling current is being measured, and an image of the surface is obtained.

There are two ways to collect images – constant current mode and constant height mode. These two modes are illustrated in Figure 2.3[16]. In constant current mode, the tunneling current is fixed, and the height of the tip above the sample is adjusted via a feedback loop in order to maintain a constant current. The image results from plotting the height of the tip over the sample versus the distance along the surface. In constant height mode, the height of the tip is kept constant, and the tunneling current is measured at each point. In this mode, the image results from a plot of the tunneling current versus the distance along the surface. Constant current mode was used first and is still the most common, since it can be used to track surfaces that are not flat at the atomic level. Constant height mode is advantageous in some situations because flat surfaces can be scanned one hundred times faster in this mode as opposed to constant current mode since the tip height does not need to be adjusted.

There are some limitations on the types of samples that can be studied using STM. Samples need to be sufficiently electrically conductive, relatively flat, and should have limited mobility of the surface atoms or molecules. Samples can be studied in a wide
Figure 2.2  Schematic of a typical STM instrument[15]
Figure 2.3 Illustration of constant current and constant height modes[16]
variety of environments. Atomic resolution images have been reported in vacuum, air, water, liquid nitrogen, electrolytes, solvents, oils, and greases[17]. This is one of the major advantages of STM, which contributes to its versatility and allows it to be a useful technique applicable to many areas of research.

Initial STM work was largely focused on the study of semiconductors. Application to metal surfaces is more complicated. Semiconductors have large charge density corrugations ($\approx 1\text{Å}$) due to dangling bonds, while metal surfaces have a much smaller charge density corrugation of $\leq 0.1\text{Å}$ [18]. To study this low corrugation on metal surfaces, good vibrational stability is crucial, as well as very stable electronics. Although metal surfaces present a challenge, STM has already contributed greatly to the understanding of metal surfaces because it provides the ability to directly see real space surface atomic arrangements. STM has provided a wide variety of information about metal surfaces, such as surface structure, phase transitions, surface diffusion, adsorbate reaction and epitaxial growth. UHV STM is a very powerful technique for studying surfaces and overlayers especially when used along with other surface science techniques.

### 2.2 Interpretation of STM Images

Since STM images are obtained via tunneling between the tip and the sample, the resulting image must be related to the electronic structures of both. In order to understand the information that is contained in an STM image, it is useful to look at a theoretical model.

The spherical tip model proposed by Tersoff et al. is presented here[19, 20]. The tip is assumed to be spherical at the end, as illustrated in Figure 2.4[19]. This assumption is valid if there is a single atom or a small cluster of atoms at the tip. The wave function for the tip is then written as an s-wave wavefunction given by the
Figure 2.4 Schematic of tunneling geometry. The tip is assumed to be spherical with a radius of curvature $R$ where it approaches the surface. The gap distance is $d$, and the center of curvature of the tip is labeled $r_0$[19].

The equation

$$\Psi_\mu(\vec{r}) = \Omega_t^{-1/2} e^{\kappa \vec{r} \cdot \vec{r}_0} \frac{\exp(-\kappa |\vec{r}|)}{\kappa |\vec{r}|},$$

where $\Omega_t$ is the tip volume and $\kappa = \frac{\sqrt{2m\phi}}{\hbar}$.

The sample is assumed to have two-dimensional periodicity in the vacuum region, so its wavefunction is given by the equation

$$\Psi_\mu(\vec{r}) = \Omega_s^{-1/2} \sum_G n_G \exp[-(\kappa^2 + |\kappa_G|^2)^{1/2}z] \exp(i\kappa_G \cdot \vec{z}),$$

where $z$ is the distance normal to the surface, $\vec{z}$ is the vector along the surface and $\kappa_G = \vec{k}_\parallel + \vec{G}$, the sum of the surface Bloch wave vector of state and the surface reciprocal lattice vector respectively. Note that the sample wavefunction decreases exponentially in the $z$ direction.

Using these wave functions, the tunneling current can be calculated and is given by the equation

$$I_t = \left(\frac{2\pi e}{\hbar}\right) \sum_{\mu\nu} f(\epsilon_\mu)[1 - f(\epsilon_\nu + eV_t)] |M_{\mu\nu}|^2 \delta(\epsilon_\mu - \epsilon_\nu),$$
where \( f(\epsilon) \) is the Fermi distribution, \( V_t \) is the bias voltage and \( | M_{\mu \nu} | \) is the transmission matrix element given by

\[
| M_{\mu \nu} | = \frac{\hbar}{2m} 4\pi \kappa^{-1} \Omega_t^{-1/2} \kappa R e^{\kappa R} \Psi_\nu(r_o).
\]

At \( r_o = R + d \), so the exponential dependence on the gap distance shown earlier using the one dimensional tunneling current equation is present here also and is contained in the \( \Psi_\nu(r_o) \) term.

In the limit of small voltages and at zero temperature, this equation simplifies to

\[
I_t = 32 \pi^3 \hbar^{-1} e^2 V_t \phi^2 D_t(\epsilon_f) R^2 \kappa^{-4} e^{2\kappa R} \rho(r_o, \epsilon_f).
\]

The density of states of the tip \( D_t(\epsilon_f) \) is assumed to be constant, so the only variable in the equation is \( \rho(r_o, \epsilon_f) \) which is the local density of states (LDOS) of the surface at the position of the tip. The LDOS of the surface is defined as

\[
\rho(r_o, \epsilon_f) \equiv \sum_\nu | \Psi_\nu(r_o) |^2 \delta(E_\nu - E).
\]

Since the tunneling current is proportional to the LDOS of the surface, STM images contain information about the electronic structure of the sample.

### 2.3 Scanning Tunneling Spectroscopy (STS)

Scanning Tunneling Spectroscopy (STS) is used to study the electronic states of the sample. In STS, different electronic states of the sample can be probed by varying the magnitude and direction of the applied bias voltage. This is because the only electrons that can tunnel are those lying in states between the Fermi energy of the tip and the Fermi energy of the sample. A diagram illustrating this is given in Figure 2.5[21]. As shown in Figure 2.5(a), if the sample is biased positive to the tip, electrons will tunnel from the filled states of the tip to the unfilled states in the sample. This probes the unoccupied density of states. If the bias is reversed, as in
Figure 2.5  Relative energy levels between the tip and the sample[21]
Figure 2.5(b), the sample is negative and electrons will tunnel from the filled states of the sample to the unfilled states of the tip, probing the occupied density of states.

This has been shown experimentally for the GaAs(110) surface by Feenstra et al.[22]. Voltage dependent STM images were obtained and are shown in Figure 2.6[22]. They find that, depending on the bias voltage, it is possible to image both the Ga and

![Image of STM images](image)

**Figure 2.6** Constant current STM images of GaAs acquired at (a)+1.9 Volts and (b) -1.9 Volts. The top view of the surface atoms is shown in (c). Ga atoms are shown as closed circles, and As atoms are shown as open circles. A unit cell is shown at identical locations in each image[22].

As atoms individually. With a bias voltage of +1.9 V, the Ga atoms are imaged as seen in Figure 2.6(a). When the bias is reversed to -1.9 V, the As atoms are imaged as shown in Figure 2.6(b). This corresponds to theoretical work which shows that the occupied density of states lies around the surface As atoms while the unoccupied
density of states lies around the Ga atoms\[20, 23].

Like the sign, the magnitude of the applied bias voltage can be adjusted to probe certain electronic states. By turning off the feedback loop and ramping the voltage at a position on the sample, current versus voltage curves, \( I(V) \), can be obtained. With this information it is possible to obtain electron density maps because

\[
\frac{\partial I}{\partial V} \propto n^{-1}
\]

where \( n \) is the surface density of states (SDOS)\[24].

Using this spectroscopic technique, Hamers et al. were able to obtain energy-resolved real space images of the filled and empty surface states of Si(111)-(7x7) surface states\[25]. Their images are shown in Figure 2.7\[25]. The top image is a conventional topograph. The second image shows a surface state at 0.35 eV below the Fermi level. The third image shows a surface state that is 0.8 eV below the Fermi level. The last image shows a state located 1.7 eV below the Fermi level. These states correspond to expectations obtained using the model of the Si(111)-(7x7) surface proposed by Takayanagi et al.\[26].

2.4 Related Techniques

STM is constrained to conducting samples, so another similar device called the Atomic Force Microscope (AFM) was developed to study nonconducting samples as well as conducting samples. The AFM is designed to probe the forces acting between the tip and the sample. This is illustrated in Figure 2.9 along with some of the interactions that can be measured using AFM\[27]. The tip is attached to a spring and the deflections of the tip are measured as the tip is scanned across the sample. Different types of forces cause different interactions between the tip and the sample. Van der Waals forces reveal the topography of the surface. Magnetic or electrostatic forces will cause additional repulsions or attractions. Adhesion forces can show friction.
Figure 2.7  STM images of the topography and three electronic surface states of a Si(111)-(7x7) surface[25]
Elastic and plastic properties can be studied by indentation experiments. AFM can also be used to study thin film growth and structure for non-conducting materials.

Figure 2.8 The principle of AFM and the effects of different types of interactions on the measured signal[27]

Another related technique that is in development is spin polarized STM (SPSTM). This technique is based on spin polarized tunneling using a magnetic tip. This concept is illustrated in Figure 2.9[28]. In Figure 2.9(a), there are no available states near the Fermi energy in the down spin sub band of the tip. Therefore, all of the tunneling current is carried by spin up electrons, so the current is proportional to the density of
Figure 2.9  Concepts of spin-polarized tunneling[28]
states for spin up electrons. When the magnetization of the tip is reversed as shown in Figure 2.9(b), the tunneling current is carried by the spin down electrons, providing information about their density of states. Figure 2.9(c) shows the resulting tunneling current if the magnetization of the tip is reversed periodically with time. The same result occurs if the tip magnetization is kept constant and the magnetization of the sample is reversed. Since spin up and spin down electrons have different tunneling behavior, information about the spin states of the atoms or molecules on the surface can be obtained.

A study by Wiesendanger et al. was done on magnetite, Fe$_3$O$_4$, using an Fe tip[29]. Their results were the first demonstration of magnetic imaging at the atomic level. In their study, the differences in the spin states of the Fe$^{2+}$ and Fe$^{3+}$ sites could be seen in the STM images. Figure 2.10 shows a line scan from one of their STM images and how it corresponds to a structural model of the magnetite surface[29]. The intensity of the arrows represents the intensity of the magnetic moment of the ion (Fe$^{3+}$ ions have a larger magnetic moment since they have more unpaired electrons). The magnetic tip remained further away from the Fe$^{3+}$ sites as seen in the line scan. The line scan has a periodicity of 12 Å which corresponds to the repeat periodicity of the Fe$^{2+}$ and Fe$^{3+}$ ions shown in the model. When the sample is imaged with a non magnetic tungsten tip, the 12 Å periodicity is not found, confirming that the periodicity is a result of spin effects.

Magnetic imaging with SPSTM has not yet been fully realized, but work continues to be done in this area. It would be extremely useful in the study of magnetic materials to be able to obtain both magnetic and structural information directly at the atomic level using a single technique.

STM is a remarkably versatile and useful surface science technique. It has been applied in many areas and many similar techniques have been and continue to be developed. With all of its uses, there is still a gap in the understanding of the
Figure 2.10  Simplified picture for the origin of magnetic effects in an STM image of magnetite. A line scan from an STM image of magnetite obtained using an Fe tip is shown with a diagram of the surface atomic structure of magnetite. The circles with the light arrows represent the $Fe^{2+}$ ions, while the circles with the dark arrows represent the $Fe^{3+}$ ions[29].

tunneling process in STM. Obviously, the STM works very well, but the reasons why it works so well are still not completely understood, especially for images of non-conducting samples such as biological molecules[30]. STM remains a challenging and promising area of research.
Chapter 3
Thin Film Growth and Characterization

3.1 Thin Film Growth

Experimental results indicate the existence of three distinct epitaxial film growth modes: Frank-van der Merwe (FM) growth, Stranski-Kranstanov (SK) growth and Volmer-Weber (VW) growth[31]. Figure 3.1 shows a schematic representation of these types of growth[32]. Figure 3.1(a) shows Volmer-Weber growth which is referred to as

![Diagram of epitaxial growth modes]

(a) Volmer-Weber  (b) Stranski-Kranstanov  (c) Frank-van der Merwe

Figure 3.1 Epitaxial Growth Modes[32]

island growth. Clusters nucleate on the surface and grow to form islands. Eventually, after a large number of layers have been deposited, the islands will grow together. This type of growth occurs when the deposited atoms are more strongly bound to each other than to the substrate. It is the common growth mode for metals on insulators.
Stranski-Kranstanov growth, shown in Figure 3.1(b), is characterized by initial layer growth followed by island growth. It is not completely understood why the layer by layer growth becomes unfavorable after a few layers. Au on Si(111) is an example of this type of growth.

Frank-van der Merwe growth is shown in Figure 3.1(c). It is referred to as layer-by-layer growth. This type of growth occurs when the atoms of the film bind stronger to the substrate than to each other. This is common for the growth of noble gases on graphite and some metal on metal systems.

These growth modes occur when local surface equilibrium is achieved. In some cases, slow kinetics may lead to metastable growth which allows the formation of additional growth modes[32]. These growth modes are illustrated in Figure 3.2[32]. One such mode is simultaneous multilayer (SM) growth, which is often referred to as

---

![Diagram](image_url)

**Figure 3.2** Metastable Growth Modes[32]
Poisson growth. In this mode, the surface mobility of the atoms is negligible. Since there is no lateral motion, the atom sticks where it lands on the surface, so the layers are built up randomly. Monolayer plus simultaneous multilayer (MSM) growth may also occur. This type of growth occurs when the mobility of the atoms on the bare substrate is fast enough to produce a uniform layer, but the mobility for the atoms on top of the monolayer is negligible and SM growth starts.

Thin film growth involves many mechanisms and is controlled by both kinetic and thermodynamical factors. When a single atom reaches the surface, it can diffuse until one of several events occurs. A schematic of some of these processes is shown in Figure 3.3[33]. The atom may be re-evaporated and leave the surface. It may nucleate

![Diagram of nucleation and growth processes](image)

**Figure 3.3** Processes in nucleation and growth on surfaces[33]

by binding or by capture at a defect site such as a step. It may be captured by an existing cluster, or it may interdiffuse into the substrate. Diffusion effects are not limited to single atoms on the surface. Clusters themselves may have some mobility and larger clusters may rearrange after they grow together.

Another aspect of thin film growth that is important for heteroepitaxy, growth of a material on a substrate composed of a different material, is the compatibility of the lattice spacing between the materials. Some effects of this are shown in Figure 3.4. If the two compounds have similar lattice parameters, the film will grow easily
on the substrate, and the system is said to be lattice matched. If the mismatch is not too large, a stable film may occur where the interplanar lattice spacing of the film is different from that ordinarily adopted by the bulk material. This is called pseudomorphic growth and results in a strained film. If the difference in lattice spacing is substantial, the film material is not able to maintain the lattice constant of the substrate, so the film will relax to a more suitable structure, introducing misfit dislocations.

3.2 Methods of Thin Film Deposition

Thin film deposition can be done in several ways. The most common methods used to deposit metal films are sputtering, Metal-Organic Chemical Vapor Deposition (MOCVD), Molecular Beam Epitaxy (MBE), thermal evaporation and electron beam
evaporation (EBE).

Sputtered thin films are formed from atoms which are dislodged from the source via bombardment of a beam of an inert gas – usually argon. The sputtered atoms travel through a vacuum environment to a temperature controlled substrate. Sputtering is the method of choice in industrial applications where large and thick films are required at a reasonable cost. Because of the fact that the deposition rate is relatively low and the vacuum pressure is relatively high, sputtered films possess contaminations originating from the inert gas as well as from other impurities.

Another non-UHV technique for film deposition is Metal-Organic Chemical Vapor Deposition (MOCVD). Metal atoms are carried by large, easily dissociated organic molecules. The molecule sits on the substrate and the organic part can be easily dissociated by temperature or light, leaving the metal atom at the substrate surface. This method allows the creation of films with widely varying stoichiometry.

In order to obtain cleaner films, UHV deposition techniques must be used. One technique of doing this is Molecular Beam Epitaxy (MBE). MBE gives a highly controlled evaporation under UHV conditions. Molecular beams are released from temperature controlled effusion cells. These beams react on the substrate crystal. The cells are equipped with shutters so that the evaporation of different materials can be stopped and started to control the film composition. This is a very common method for producing semiconductor films and structured materials.

A simpler UHV deposition technique is thermal evaporation. Atoms are removed from the source via thermal means. A crucible containing the source material is resistively heated until atoms can effuse to the substrate. With thermal evaporation, there may be contamination from the crucible material, and the temperature that can be obtained is limited. This makes it difficult to evaporate films of high melting point materials.

Electron beam evaporation eliminates these problems. In this method, a heated
filament emits electrons which are accelerated toward the source using a bias voltage. The electrons heat part of the source material such that the effective crucible is the unmelted part of the source. The dislodged atoms travel through the vacuum to the substrate. The purity of the film is determined by the nature and level of impurities in the source, impurities from the heater or support materials and the pressure of the UHV environment. Electron beam evaporation is a widely used technique because it enables the evaporation of virtually all materials at any rate within a clean environment.

3.3 Methods of Thin Film Characterization

There is a wealth of information that can be gathered from a thin film, so there are many types of thin film characterization methods, each with its own unique capabilities. Our work has focused on the determination of growth modes and film structure, so techniques used for this type of characterization will be discussed. The three most common methods to determine growth modes are AES (Auger Electron Spectroscopy), RHEED (Reflection High Energy Electron Diffraction), and STM/AFM. The three most common methods used to determine film structure are Ion Scattering, LEED (Low Energy Electron Diffraction), and STM/AFM. One of the main advantages of using STM to study thin films is the fact that it can give simultaneous structural and growth mode information.

3.3.1 Growth Mode Determination

Auger Electron Spectroscopy is often used to determine growth mode. In this technique, a high energy electron beam is focused on the sample and induces secondary electrons. Sometimes sample atoms are ionized in an inner shell, which usually leads to the emission of an Auger electron. The kinetic energy of the outgoing electron depends only on the properties of the atom it came from, so the energy is assumed to be
a fingerprint of the atom. The characteristic Auger electron energies for the elements are well known and tabulated. The Auger signal for the material being deposited, as well as for the substrate, can be monitored. By watching how the signals change over time, information about the growth mode can be obtained. Figure 3.5 shows the types of signals expected for both the substrate and film materials for each type of growth[32]. The growth mode can be determined because the slope of the Auger

![Graphs](image)

**Figure 3.5** Expected AES signals during growth for the substrate and adsorbate materials, labelled S and A respectively. The expected results for Volmer-Weber mode are shown in (a), Stranski-Kranstanov mode in (b), and Frank-van der Merwe mode in (c)[32].

signal changes when a single layer is completed. No sharp changes in the slope are seen for island growth.

Reflection High Energy Electron Diffraction (RHEED) uses 5-100 keV electrons at grazing angles of incidence, so that the topmost atomic layers of the sample are probed. Because of the grazing angle incidence, RHEED can be done during film
growth. The intensity of the specular beam oscillates as layers are completed, so RHEED is a good technique to use to monitor layer-by-layer growth. This effect is shown in Figure 3.6[34, p 340]. There are no persistent oscillations in the RHEED signal for island film growth.

Scanning Tunneling Microscopy is an extremely useful method for determining the growth modes of thin films because direct, real space images can be achieved. This means that local information is provided, instead of the statistically averaged information provided by most other techniques. This is very useful since different labs find different magnetic properties for samples grown under slightly different conditions[35]. STM provides access to the details of growth, such as the effect of individual defects,
so it should help provide a greater understanding of all of the variables involved in film growth. There is also the possibility to interrelate electronic properties to growth by using the spectroscopic capabilities of STM.

3.3.2 Structural Determination

Ion Scattering is often used to determine the structure of films. In this technique, $H^+$ or $He^+$ ions are scattered off the sample. Ion scattering is divided into three energy ranges based on the energy of the ions being scattered. High Energy Ion Scattering (HEIS) refers to energies of 200 keV-2 MeV. HEIS is sensitive to interplanar relaxations and requires an iterative procedure to determine the structure. Medium Energy Ion Scattering (MEIS) refers to energies of 20-200 keV. Low Energy Ion Scattering (LEIS) refers to energies of 1-20 keV. In the low energy case, there is a very short penetration depth so this method is useful to probe the structure of the surface layer.

Low Energy Electron Diffraction (LEED) is another popular technique used to determine surface structures. Electrons with energies of 20-500 eV incident normal to a crystal surface are backscattered. The elastically backscattered electrons can be seen as a pattern of illuminated dots (Laue or Bragg spots) on a fluorescent screen. The diffraction pattern that is produced is the Fourier transform of the atomic arrangement at the surface.

STM is very useful in structural determination since it provides real space, atomic resolution images, allowing the position of the atoms on the surface to be seen. Shortly after its invention, the STM was used to solve a long standing problem of the structure of the Si(111)-(7x7) surface. Several experiments had been done, and several models were proposed, but they were not in agreement. Binnig et al. used the STM to get the first real space determination of the structure of the (7x7) reconstruction of the Si(111) surface[36]. An image of the Si(111)-(7x7) surface obtained by Wiesendanger
et al. is shown in Figure 3.7[37].

Figure 3.7 STM image of the Si(111)-(7x7) surface reconstruction[37]
Chapter 4
Experimental Apparatus

4.1 Chamber Design and Capabilities

In order to conduct STM growth studies, an ultra-high vacuum (UHV) chamber was designed and constructed. A schematic of the chamber is shown in Figure 4.1. The chamber is capable of reaching pressures of $\leq 10^{-10}$ Torr. This is achieved by using a pumping system consisting of a rotary pump, a turbomolecular pump, an ion pump, and a titanium sublimation pump. Low pressures are reached after baking the chamber for twelve hours. Recently the entire chamber was placed on a large metal plate which is supported by six springs to reduce the effects of vibrations on the STM signal.

The chamber was designed so that samples can be transferred in situ between a sample preparation area and the STM. A sample holder, which can hold up to three samples, has been attached to a manipulator with $360^\circ$ of rotary motion and $18^\circ$ of linear motion. The sample holder is equipped with a heater so that the samples may be annealed up to temperatures of $600^\circ$C to $700^\circ$C. A thermocouple is used to monitor the temperature of the sample.

Sample preparation is done in the upper part of the chamber. This area of the chamber is equipped with an $Ar^+$ ion sputter gun that is used to clean metal substrates before films are deposited. This area also has two ports which can accommodate a variety of evaporation guns. There is a shutter which can be used to shield the sample from evaporation while the gun is warming up and after the desired film thickness has been achieved. Currently an electron beam evaporation gun, which is used to evaporate Ru, is attached to the rear of the chamber as shown in the figure. A quartz crystal thickness monitor allows the film thickness to be monitored.

Once a sample is prepared, it can be transferred into the STM to be imaged. The
STM was designed and built at Rice. A schematic of the STM is shown in Figure 4.2. The tip and the sample are attached to a sample stage which is supported by a two-stage spring suspension system. The sample stage can be locked into a stable position during sample transfer, and then unlocked so that it is suspended during imaging. The position of the tip is controlled by a piezoelectric tube scanner that allows positioning in three dimensions. The sample holder is attached to a commercial Burleigh Inchworm which is capable of both coarse and fine positioning of the sample. The speed can range from 0.002 micron/sec to greater than 1 mm/sec. The smallest step size is 0.2 Å [38].

The STM is interfaced to a 486 PC as well as the STM electronics and a Burleigh Inchworm controller. A schematic of the interface is shown in Figure 4.3.
Figure 4.3 Schematic of the STM Interface
An Advantech PCL-830 counter timer board is interfaced to the Burleigh Inchworm controller through a relay, allowing the movement of the Inchworm to be controlled by the PC. The relay enables the Inchworm to be run quickly at a constant speed determined by the frequency put out by the board, or slowly in a single step mode. A Real Time Devices DA700 digital to analog converter is used to set the desired tunneling current, bias voltage as well as the x, y, and z positions of the tube scanner. All of the settings can be adjusted using the computer. An Analogic LSDAS-16 data acquisition board is used for its analog to digital capabilities. It is used to read in the tunneling current signal, and to monitor the other settings.

An oscilloscope is used to monitor the tunneling current during the experiment because it provides a method of looking at the noise in the signal. A preamplifier, located as close as possible to the STM, is used to amplify the tunneling current signal. Images are usually collected in constant current mode, so the tip height data is collected by the computer. To reduce noise, the current is averaged several times at each point. As the image is being taken, a grey scale plot is shown on the monitor. The program has been written so that images can be taken of square areas with an edge length of 50 Å to 1000 Å. This can be adjusted up to ≈ 1 micron if it becomes necessary to study larger features. A single scan can be completed in a few minutes. Since the electronics were built in the lab, and the software was written in the lab, the system can be easily modified to meet future needs.

To make accurate distance measurements, the piezoelectric tube must be calibrated. The piezoelectric tube was calibrated by obtaining an atomic resolution image of graphite where the atomic spacing detected by the STM is well known. With this information, an accurate mapping of the voltage on the piezo to distance can be obtained. The tube is calibrated to have motion of 100 Å per Volt in the x and y directions, and 30 Å per Volt for the z direction.
4.2 Experimental Measurements

An STM study of the growth of Ru on C(0001) has been performed. A piece of ZYA grade polycrystalline highly oriented pyrolytic graphite (HOPG) is used as the substrate. The substrate is attached to a copper mount using silver paint. A very flat graphite substrate can be obtained by cleaving the surface, removing several layers. STM images of the substrate show that the surface is atomically flat along the surface at the micrometer scale.

STM tips are prepared by electrochemically etching a piece of 1 mm tungsten wire approximately 1 cm long in 1 M NaOH solution. A circular Ni or stainless steel electrode is placed in the solution. The tungsten wire is placed in the NaOH solution at the center of the electrode with approximately two-thirds of it immersed. A voltage of 15-20 V is applied for 1 min. The tip is lifted from the NaOH solution and is rinsed with distilled water. This etching procedure was optimized by varying the parameters and looking at the quality of the resulting tips under a microscope.

The freshly cleaved graphite sample is placed into the STM as well as the freshly etched tungsten tip. The chamber is closed and baked overnight. When it has cooled down, the titanium sublimation pump is used to achieve a pressure in the $10^{-10}$ Torr range. The substrate can be imaged if desired. Afterwards, the sample is transferred from the STM to the sample preparation area. Since graphite has an extremely low gas adsorption efficiency, the substrate does not need any further in situ cleaning.

Ruthenium is deposited using the same evaporation gun and approximately the same conditions as in the magnetism experiment conducted by Pfandzelter et al.[10]. Because the gun needs to be outgassed, the sample is initially turned 90° from the evaporation gun so that no Ru atoms can reach the substrate. To start the evaporation, the sample is turned towards the gun for the desired length of time. To end the evaporation the sample is once again turned 90° from the evaporation position, and the gun is turned off.
After the deposition is complete, the sample is transferred into the STM. Once the sample is in the STM, the sample is moved as close to the tip as possible using the coarse motion of the inchworm. The sample position is monitored through the windows in the vacuum chamber. Care must be taken not to crash the tip into the sample.

Once the tip is relatively close to the sample, the bias voltage and tunneling current are set. In this study a bias voltage of $\approx 0.2$ V and a tunneling current of $\approx 0.3$ nA are used. During the approach, the sample is slowly moved closer to the tip until a tunneling current is observed. Once tunneling is reached, the tip is moved along the sample surface while watching the signals on the oscilloscope. When an area of low noise is found, a large size scan of about 1000 Å x 1000 Å is performed. When an area of interest is found, smaller scans are done. The bias voltage and tunneling current can be adjusted and optimized to find the conditions resulting in the best tunneling current signal on the oscilloscope.

After imaging, the sample can be transferred back up to the sample preparation area, and more ruthenium can be deposited. This avoids opening the chamber to cleave the graphite substrate again in order to study higher coverages of Ru. The quality of the images after evaporating more Ru is very dependent on the vacuum conditions and the time lapse since the initial evaporation. Ru is known to become contaminated with CO, therefore it is eventually necessary to open the chamber and remove the graphite sample to cleave it again. Once a fresh graphite surface is prepared, the process is repeated to study different coverages of Ru.

Preliminary STM measurements have also been done on a Pd(111) single crystal sample. The Pd surface was cleaned in situ using $Ar^+$ ion sputtering for 20 minutes at 1.5 mA plasma current and 4 kV ion energy. The sample was annealed for 30 minutes at 490°C to restore the crystalline structure at the surface. The sample was imaged using a tunneling current of 0.3 nA and a bias voltage of 0.3 V. The Pd surface was
found to be quite rough – too rough to continue with a study of the growth of Co on Pd. The sample was removed so that it could be polished to achieve a flatter starting surface. Meanwhile, the Ru on C(0001) experiments were performed.
Chapter 5
Results and Discussion

5.1 Growth of Ru on C(0001)

Preliminary STM results have been obtained for the growth of Ru on C(0001). These results will be presented and discussed within the framework of the growth mode and structural determination of Pfandzelter et al.[10]. Figure 5.1 shows the Ru and C Auger data they obtained. A change in slope occurs in each Auger plot at \( \approx 0.25 \) ML, where 1 ML refers to the atomic density of the graphite layer. This implies that the atomic density of the Ru monolayer is 0.25 that of the graphite atomic density, which is in accordance with the proposed p(2x2) superstructure. The authors conclude that for the first monolayer Ru grows laterally on graphite. After the first monolayer is completed, islanding starts (Stranski-Kranstanov growth mode). This determination is not unambiguous, so the STM study was initiated to try to remove the ambiguity.

The graphite substrate was imaged with atomic resolution using the STM, and it was found to be flat and largely free from defects. Ruthenium was deposited at coverages of approximately .01, .04, .05 and .09 ML, where 1 ML refers to the atomic density of the HOPG graphite layer.
Figure 5.1 AES data for the growth of Ru on C(0001)[10]
Figure 5.2  STM image at less than 1 percent coverage of Ru on C(0001)

At very low coverage (less than 1 percent), STM images show that Ru adsorbs on the graphite substrate randomly as shown in Figure 5.2. This scan is 120 Å x 120 Å in size. The bright spots are Ru atoms or clusters located on the graphite substrate. The graphite substrate looks to be flat and free from defects. During imaging at this coverage, all of the bright spots appeared to be the same height and size, about 10 Å across and 4 Å tall, suggesting that single atoms are being imaged. This is not unreasonable for a single atom since the horizontal scale may be significantly distorted by the diameter of the tip, and images on graphite are known to have exaggerated heights due to deformation of the graphite substrate[39]. This deformation effect will be discussed further in the Tip Effects section later in this chapter. When an area containing a bright spot was imaged again at higher resolution, the bright spot was
not seen. This suggests that the atoms diffuse easily along the graphite surface.

![STM image at 1 percent coverage of Ru](image)

**Figure 5.3** STM image at 1 percent coverage of Ru

At a Ru coverage of 1 percent, most of the graphite substrate was bare. One area contained two large islands. An 915 Å x 915 Å image of this area is shown in Figure 5.3. Two large islands of about 200 Å can be seen. These islands are located on the upper level of a step edge. This also suggests that Ru atoms easily diffuse along the graphite surface until they reach an island or defect where they nucleate. The islands have a height of 15 Å. Although the height is greater than that expected for a monolayer island, the possibility cannot be ruled out because of the deformation of the graphite substrate mentioned earlier.
Upon further increase in the Ru coverage up to 4 percent, STM images show the formation of more islands as shown in Figure 5.4. This scan is 230 Å x 230 Å. There are several large islands approximately 30 Å across and 15-20 Å tall. The background graphite area looks to be rough. The fact that several islands have grown in this area suggests that Ru nucleates at defect sites.
The same film was imaged again after 12 hours. One of the resulting images is shown in Figure 5.5(b) next to an image of similar size from the initial imaging. The image in (a) is 92 Å x 92 Å and the image in (b) is 82 Å x 82 Å. After twelve hours, the islands are larger with a size of approximately 40 Å as opposed to the 30 Å seen in the initial imaging. This again suggests that surface diffusion of Ru on graphite is an important factor.
A 152 Å x 152 Å image at 5 percent coverage is shown in Figure 5.6. At this coverage, smaller islands were seen on rather flat areas of the substrate. When smaller size scans were attempted to locate the small peaks again, they could not be found. Most likely this is due to diffusion of the Ru atoms along the flat part of the substrate until they are nucleated at a defect or a cluster of Ru atoms. At this coverage, the island heights were 4-6 Å which is closer to the expected height of $\approx 2$ Å for a monolayer island of Ru. Since this experiment was conducted with a lower pressure in the vacuum chamber than any of the others, the heights are probably more accurate. It is important to note that all features appear twice in this image. This is the result of a double tip, which is another tip effect that will be discussed in more detail later in this chapter.
Figure 5.7  STM image at 9 percent coverage of Ru

The experiment done at a Ru coverage of 9 percent had a much higher vacuum pressure than all of the other experiments. As can be seen in the 710 Å x 710 Å image in Figure 5.7, there are many adsorbates on the graphite substrate. The surface appears very rough, and almost entirely covered. Most likely there are a lot of other adsorbates besides Ru.

Thus far, atomic resolution images of a Ru island which could confirm the p(2x2) structure have not been obtained, but the initial formation of islands has been seen. At this time, no definite conclusion can be reached about which growth mode best describes the growth of Ru on C(0001). Further work needs to be done, especially at higher coverages to see how the initial islands grow together.
5.2 Tip Effects

Sometimes STM tips undergo minor changes during imaging that can alter the appearance of the images. It is important to have some understanding of what kinds of effects there may be and how they will influence the images, so that these effects may be taken into account so as not to interfere with the results.

In this study, the most common effect is that the tip picks up Ru atoms during imaging. This occurs because the sticking coefficient of Ru to the tungsten tip is higher than to the graphite substrate. When the tip comes near a Ru atom, the atom may be picked up by the tip. If the tip picks up an additional atom, it has to move further away from the sample to maintain the same tunneling current, so the area following the addition of the atom appears brighter than the preceding area in the image. Once atoms are picked up, they may eventually become dislodged, and the reverse effect happens. These effects can be removed from the data by using Fast Fourier Transforms with Y-notch filtering. In order to reduce the likelihood of the tip picking up atoms, imaging can be done at higher bias voltage settings and/or lower current settings to keep the tip further away from the sample.

In the 5 percent coverage images, the double tip effect is seen. In these images all of the features appear twice, always at the same angle and distance from each other. Since all of the features are repeated, it is easy to recognize that this is occurring and to realize that only half of the features are real. The double tip effect appears differently depending on the spacing between the two tips. If the distance between the two tips is larger than the scanning distance, features from two different parts of the sample will be superimposed on each other. If the distance is smaller than the scanning distance, the result is imaging the same feature twice, as occurred in this case. If the tips are very close together, a variety of asymmetric atomic resolution images are obtained.

Another prevalent tip effect for studies done on graphite is that the apparent
heights of features in the image are larger than expected. This is because of deformation of the graphite substrate due to contamination or oxide on the tip[39]. In order to reach the desired tunneling current, the tip has to touch the graphite surface, pushing it down. Then, when the tip moves up to go over an adsorbate, it has to travel further than the actual height of the adsorbate. This leads to exaggerated heights in the images. This effect can be corrected for if atomic resolution is obtained for the graphite background in the image. The atomic corrugation for graphite is known to be 0.9 Å, so the image heights can all be scaled to this value in order to get more accurate values for the heights of the adsorbates[40].
Chapter 6  
Summary and Conclusions

A sophisticated UHV-STM apparatus has been designed and constructed for the study of thin film growth. Refined growth studies of Ru on C(0001) have been performed. From our results, we can conclude that Ru diffuses easily along the graphite substrate and tends to nucleate at defects. It appears that the first monolayer of Ru grows homogeneously on graphite. Further study is needed to check the growth mode reported by R. Pfandzelter et al.[10]. Our results, thus far, show that the experimental equipment that was built is capable of being used to study thin film growth, and it can now be used to study a variety of other interesting systems.
Bibliography