

# Compact deposition system for device-based ultrathin crystalline film growth

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The study of hot electron excitation at surfaces requires the deposition of ultrathin metal films. To probe the role of particle bombardment in such film excitations, homogeneous, atomically ordered, and relatively defect free thin films must be deposited in the same ultrahigh vacuum system where they will be studied. With these constraints in mind, the authors designed a compact deposition chamber that allows for *in situ* growth and analysis of metal layers, which are only a few monolayers. This deposition chamber is attached to the commercial variable temperature scanning tunneling microscope (STM) and has an internal volume of 500 cm<sup>3</sup>. The target substrates for deposition are compatible with the STM design and are held in place in a specially designed clamping slot that enables low temperature growth. They used the custom built chamber to deposit Ag top layers with thicknesses between 8 and 15 nm on Si(100). Electronic and morphological characteristics of the prototype Ag/*n*-Si(100) devices are presented. © 2009 American Vacuum Society. [DOI: 10.1116/1.3097859]

## I. INTRODUCTION

Recent work has demonstrated the use of ultrathin film devices as detectors of chemical reactions and kinetic particle collisions at metal surfaces.<sup>1-4</sup> In these experiments, electrons are driven out of thermal equilibrium by the addition of energy to the lattice (i.e., chemisorption of incident atoms, kinetic energy transferred during a collision) and can propagate through a layer of the device, which is smaller than the elastic mean free path for electron-electron scattering. Metal-oxide-semiconductor, metal-insulator-metal, and metal-semiconductor (MS) ultrathin film devices provide experimental systems which facilitate the detection of hot charge carriers ballistically transporting through a thin metal layer. The investigation of hot electrons at surfaces requires devices with homogeneous and crystalline metal layers and minimal defects to be grown only a few monolayer thick. Furthermore, growth and characterization in ultrahigh vacuum (UHV) ensures minimum contamination.

Here we report on the design of a compact deposition chamber for the growth and characterization of ultrathin noble metal layers on solid crystalline substrates. Our design includes the capability to rapidly cool the surface during growth to change growth energetics. We chose to grow Ag/*n*-Si(100) Schottky diodes as a prototype MS device because it has been shown that Ag grows layer by layer and has domains of (111) orientation at deposition temperatures easily obtainable by liquid nitrogen cooling. Additionally, there is minimal chemical reactivity between the substrate and film at room temperature so no wetting layer is required and the metal-semiconductor interface is abrupt.<sup>5-7</sup>

After deposition, the morphological and electronic properties of the films were characterized. Noncontact atomic

force microscopy (AFM) was used to determine the thickness, and current-voltage (*J-V*) measurements were recorded. We also present ion scattering spectroscopy data as a means of probing the continuity of the top layer of the devices and investigating energy loss at a metallic thin film surface through hyperthermal particle bombardment.

## II. EXPERIMENT

In order to study clean surfaces and interfaces of as-grown films, we designed a novel compact deposition system that can be attached to the fast exchange load lock (FELL) of our Omicron variable temperature scanning tunneling microscope (VT-STM).<sup>8</sup> One goal of the design was to ensure that epitaxial metal layers could be deposited and characterized in ultrahigh vacuum. A schematic of our apparatus can be found in Fig. 1. The following subsections describe the custom built deposition system and a procedure for fabricating ultrathin film MS devices tailored to the detection of hot electrons by particle bombardment.

### A. Setup

The deposition system is attached to our Omicron FELL chamber and has an internal volume of 500 cm<sup>3</sup>. Both chambers are evacuated by a Pfeiffer TC600 turbo pump. A linear-rotary manipulator is attached to one end of the deposition system via a differentially pumped seal. The seal is made between two O rings at the joint where the arm comes out of vacuum and is evacuated with a Pfeiffer TMU071P. The base pressure of the deposition/FELL chambers is 10<sup>-8</sup> Torr measured by a Pfeiffer ionization gauge mounted opposite the linear-rotary manipulator in Fig. 1. We note that 10<sup>-8</sup> Torr is above the UHV pressure regime. However, the mean free path of evaporated metal atoms at 10<sup>-8</sup> Torr greatly exceeds the distance between the source and the substrate, and the

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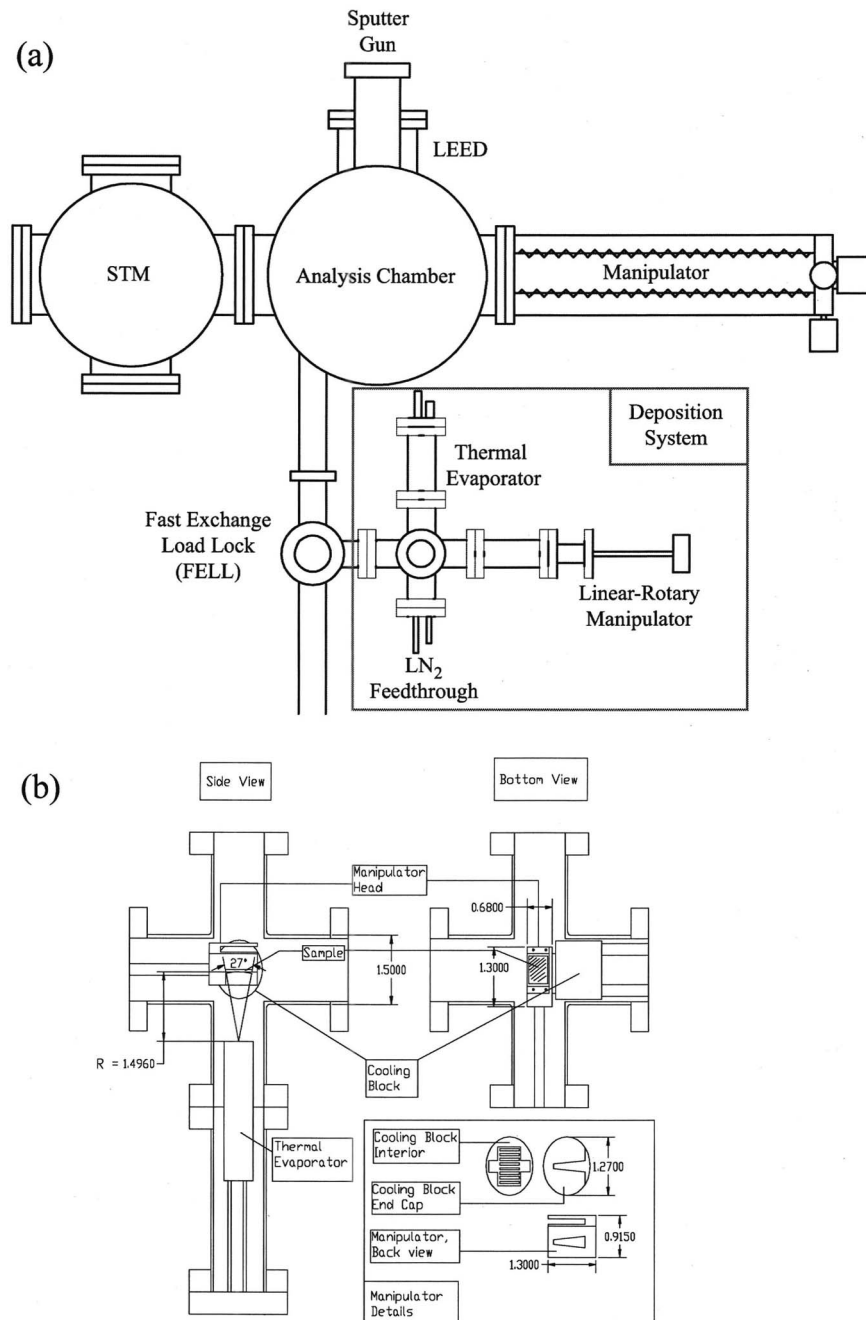


FIG. 1. (a) Top view of deposition and STM system. The compact thin film deposition system is attached to the FELL of a VT-STM by Omicron. Samples can be transferred to the analysis chamber and STM for *in situ* characterization. (b) Side and bottom closeup views of the deposition chamber with dimensions given in inches.

deposition/FELL chambers are directly connected to the VT-STM analysis chamber which has a base pressure of  $10^{-11}$  Torr.

Substrates enter through the VT-STM FELL and are moved into the deposition system via the linear-rotary manipulator. The manipulator has been fitted with a custom built oxygen free high conductivity copper (OFHC) head that holds samples by friction fit and accommodates platens, which are compatible with the sample size constraints of the VT-STM.

For evaporations, samples are secured on an OFHC sample stage that allows for efficient cooling. The stage is hollow and directly connected to a liquid nitrogen feedthrough. The manipulator head fits into the top of the

sample stage through a dovetail design [Figs. 1(b) and 2(b)]. The head is held in place by atmospheric pressure on the manipulator arm. Sample temperature is monitored by a type K thermocouple placed at the top corner of the manipulator head. With this design the temperature of the substrate reaches 120 K in 30–35 min with a continuous flow of liquid nitrogen through the stage.

The deposition chamber is equipped with a thermal evaporation gun by McAllister Technical Services with high-voltage source and temperature gauge.<sup>9</sup> The gun is loaded with deposition charge out of vacuum and mounted on the chamber in a position  $r=38$  mm from the sample position. A water cooled stainless steel jacket contains a Ta filament contacted with a type C thermocouple so that temperature can be

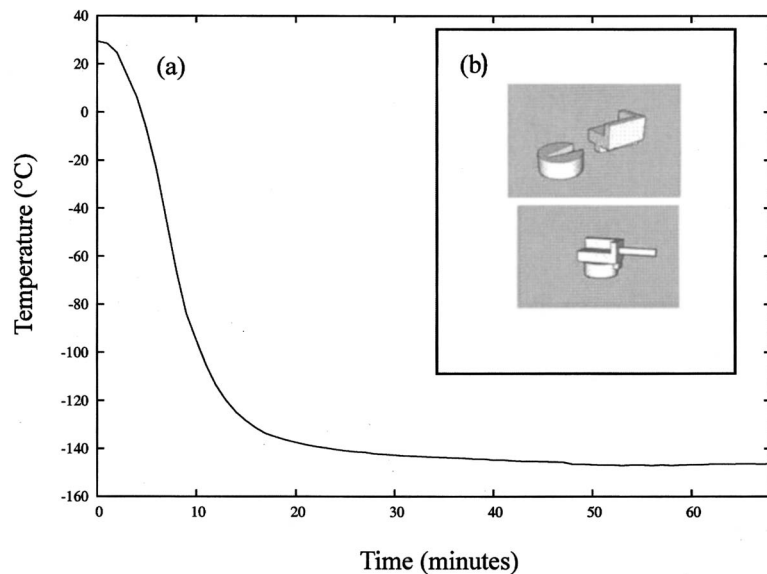


FIG. 2. (a) shows temperature data recorded from a thermocouple mounted in the manipulator head during liquid nitrogen cooling prior to the deposition. The stage and head assembly fit together with a dovetail design displayed in the inset (b). The cylindrical stage can be pre-cooled (top) and the manipulator head is pressed into the slot in the stage by atmospheric pressure on the manipulator arm during a deposition (bottom).

continuously monitored. The water cooled jacket prevents overheating and minimizes outgassing during each deposition run.

Following a thin film deposition, samples can be moved to the VT-STM analysis chamber which contains low energy electron diffraction and STM surface probes or be brought into the FELL for *ex situ* analysis.

### B. Procedure

To fabricate Ag/*n*-Si(100) MS devices, Sb-doped *n*-Si(100) substrates were first annealed at 900 K for 2 h to outgas the samples and to remove native oxide and then allowed to return to room temperature inside the VT-STM analysis chamber. During the Si cool down, liquid nitrogen was pumped through the OFHC sample stage in the deposition chamber. Precooling the stage without the sample inserted limited the cryotrapping of residual gas onto the substrate. The Si substrate was then moved into the deposition manipulator head and slid into the cooling stage. After the head and stage equilibrated to 120 K, the Ta filament in the thermal evaporator crucible was heated to 770 K for several seconds by ramping the evaporator source voltage. The volume of Ag (99.99% pure<sup>10</sup>) used per deposition varied from 0.736 to 1.96 mm<sup>3</sup> for the devices presented in this article. After deposition, liquid nitrogen flow was stopped and the Ag film was annealed at room temperature.

## III. CHARACTERIZATION

Fabricated Ag/*n*-Si(100) MS devices were characterized to determine thickness, electronic behavior, and mechanical stability for beam induced hot electron studies. Noncontact AFM, current-voltage, and ion scattering energy loss measurements are presented in the following subsections.

### A. Atomic force microscopy

Bare *n*-Si(100) substrates were masked at their centers with a metal shim prior to deposition. After film growth, AFM micrographs and surface profiles were recorded as in Fig. 3 using a Digital Instruments Nanoscope III in the non-contact mode. Mean profiles across the mask boundary were used to calculate the thickness of the Ag top layer. Film thickness data are shown in Fig. 4.

In order to understand the thickness of our devices as a function of the charge evaporated, we employed an empirical model which assumed an isotropic distribution of metal atoms evaporating from a point source,

$$t = \frac{\epsilon v}{4\pi r^2}. \quad (1)$$

The thickness  $t$  of the film is described by the volume  $v$  of the evaporated metal divided by the surface area of a spherical shell  $A$  at a distance  $r$  between the source and a point on the substrate. This linear relationship between volume of evaporated metal and film thickness was used to fit our AFM measurements (Fig. 4). The efficiency factor  $\epsilon$  is introduced to account for the fact that evaporation of the metal happens within a crucible with a small opening and that not all evaporated atoms are deposited at the surface. The AFM measurements also confirmed that the metal layer was continuous and that variations in thickness near the mask were of the order of  $\pm 1$  nm.

### B. *J-V* measurements

Current-voltage measurements were recorded *ex situ* with a Micromanipulator 7000-LTE interfaced to an HP 4156B parameter analyzer (Fig. 5). To determine the Schottky barrier height  $\phi_b$  of our MS devices at zero bias, thermionic emission theory was used.<sup>11</sup> Under forward bias, current density from thermionic emission can be written as,

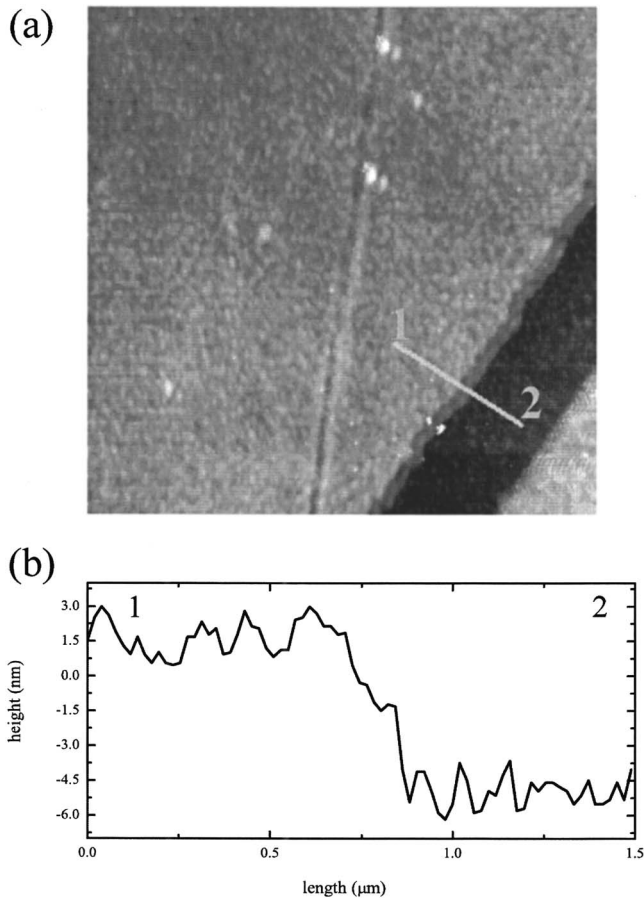


FIG. 3. Noncontact AFM image of the Ag layer and Si substrate with  $(5 \mu\text{m})^2$  area is displayed in (a) with a single height profile in (b) displaying approximate thickness  $t=8 \text{ nm}$ . Several height profiles across the mask boundary were averaged to determine the thickness for each sample.

$$J = J_0 \exp\left[\frac{eV}{nkT}\right] \left(1 - \exp\left[-\frac{eV}{kT}\right]\right), \quad (2)$$

where

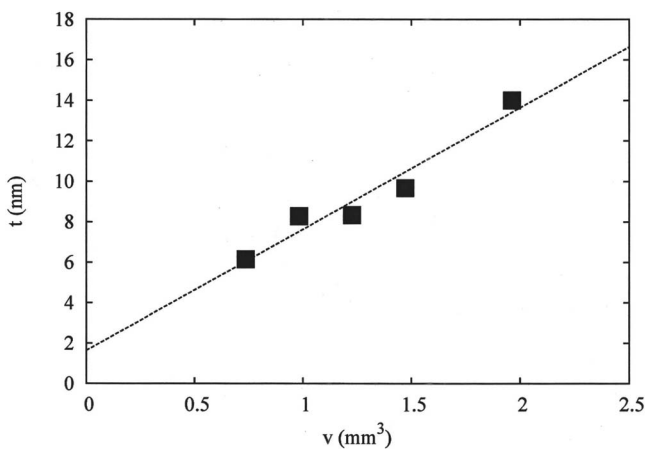


FIG. 4. Film thickness as a function of volume  $v$  of metal wire evaporated. A typical error bar for a point on the plot is  $\pm 1 \text{ nm}$ . The data were fit to an empirical model Eq. (1) which assumes an isotropic distribution of evaporated metal atoms from a point source. An efficiency factor of  $\epsilon=0.1$  was extracted.

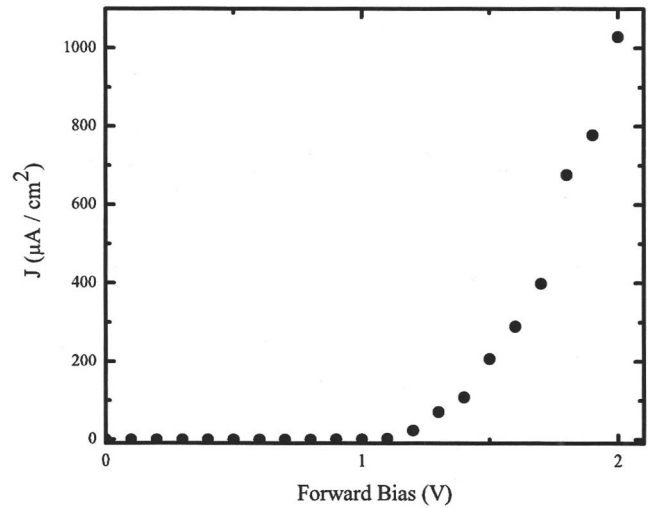


FIG. 5.  $J$ - $V$  curve of an Ag/ $n$ -Si(100) ultrathin film device with an 8 nm thick top layer under forward bias condition. The device displays a diodelike behavior with a barrier height near  $\phi_e=1.0 \text{ V}$  determined using thermionic emission theory.

$$J_0 = A^{**} T^2 \exp\left[-\frac{e\phi_e}{kT}\right]. \quad (3)$$

For an 8 nm Ag/ $n$ -Si(100) device at room temperature, a barrier height of approximately  $\phi_e=1.0 \text{ V}$  was obtained using the standard value for the effective Richardson constant  $A^{**}=120 \text{ A cm}^{-2} \text{ K}^{-2}$ . For this measurement, the ideality factor  $n$  was taken as unity. The presence of a Schottky barrier is important as it provides an internal energetic filter for detecting hot electrons excited in a MS device.

### C. Ion scattering spectroscopy

In order to ensure that the top metal layer was continuous, ion scattering spectroscopy measurements were obtained with the apparatus and procedure described in Ref. 12. Hyperthermal energy ion scattering was used as a spectroscopic technique to evaluate the composition of the deposited top metal layer. Energy loss of ions scattered from the surface in this regime can be predicted according to the sequential binary collision approximation (SBCA) where ions scatter elastically from individual surface atoms. Distinct peaks in the energy spectrum of the scattered ion beam correspond to specific collision trajectories and depend on the mass of the surface atom.

Hyperthermal energy ion scattering spectroscopy was done by impinging 1 keV  $\text{K}^+$  ions at an incident angle of  $45^\circ$ . Energy loss spectra were recorded at a specular scattering angle and are presented in Fig. 6. The trajectories of ions scattered from the surface were analyzed in terms of the SBCA. The most prominent feature in the spectrum was a quasisingle (QS) scattering event occurring between the  $\text{K}^+$  ion and single atom on the Ag surface explained by a classical kinematic factor.<sup>13</sup>

Additionally, there was significant intensity near the quasideouble (QD) position which corresponds to two sequen-

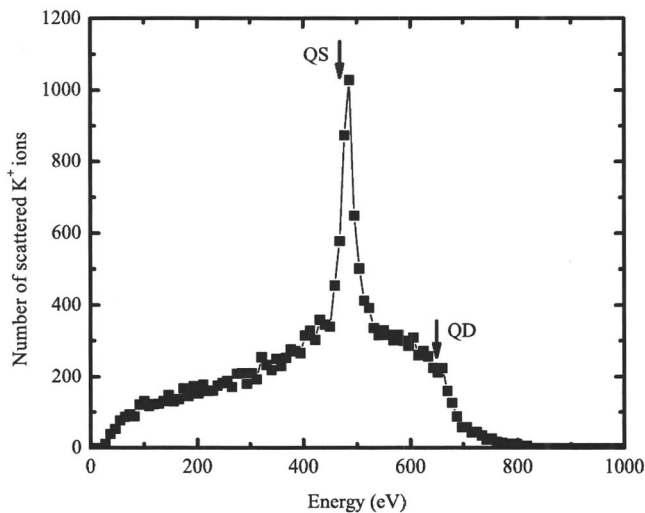


FIG. 6. Energy loss spectra for  $K^+$  1 keV  $45^\circ$  specular ion scattering. We observe a strong QS peak which displays energy loss described by a classical kinematic factor and is consistent with predicted  $K^+$ -Ag interaction. Intensity at the QD position corresponds to two sequential binary collisions between  $K^+$  and Ag surface atoms. A line is drawn through the data to guide the eye.

tial binary collisions between the ion and Ag surface atoms. The low energy tail is a mixture of complex scattering trajectories. Auger electron spectroscopy measurements before and after ion scattering also displayed a strong Ag peak and confirmed that ion scattering did not significantly sputter the top layer of the devices.

#### IV. CONCLUSION

We designed a thin film deposition system, which is compatible with a VT-STM that allows low temperature epitaxial growth and *in situ* analysis of fabricated ultrathin film devices. The system was used to grow and characterize

Ag/*n*-Si(100) ultrathin metal-semiconductor devices. AFM, *J*-*V*, and ion scattering spectroscopy measurements of the devices confirm that the metal layers that we have grown are continuous and display a diodelike behavior. Our compact chamber design has made the VT-STM system more effective for measurements of as-grown surfaces and interfaces suited to the investigation of internal emission of hot charge carriers induced by hyperthermal energy particle bombardment.

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