A miniature electron-beam evaporator for an ultrahigh-vacuum atom-probe field-ion microscope

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A miniature electron-beam evaporator (MEBE) has been fabricated and adapted to our ultrahigh-vacuum atom-probe field-ion microscope (APFIM). The MEBE allows for in situ vapor deposition—under ultrahigh-vacuum conditions ($< 4 \times 10^{-10}$ Torr)—of a wide range of elements, on the surface of an atomically clean FIM specimen; the surface is prepared via the field-evaporation process. The deposition rate of an evaporant from the MEBE is calibrated to give an accurate value of this quantity. Examples of the deposition—at $\approx 0.3$ nm min$^{-1}$—of silicon or titanium on tungsten FIM specimens are presented. And in the case of a Ti/W couple it is demonstrated that an interface between a tungsten substrate and a titanium overlayer is chemically sharp on an atomic scale; the titanium was vapor deposited at a substrate temperature of 77 K. Also a 20-kV electron-beam gun was adapted to our APFIM. This gun is useful for in situ electron-beam heating of bilayer couples, or the introduction of point defects in metal oxide or semiconductor overlayers via electronic mechanisms.

I. INTRODUCTION

This paper presents a technique for the in situ vapor deposition of an overlay—metal or semiconductor—on atom-probe field-ion microscope (APFIM) specimens, thus creating a bilayer couple consisting of a substrate material and an overlay. The approach we have taken involves in situ vapor deposition of evaporants via a miniature electron-beam evaporator (MEBE) at a controllable and accurate value of the deposition rate. Our APFIM employs an ultrahigh-vacuum (UHV) system$^1$ and therefore the MEBE must be completely compatible with it. Also the background pressure must be $< 5 \times 10^{-10}$ Torr during the deposition process, so that an atomically clean interface between a substrate, which is prepared by the field-evaporation process, and a vapor-deposited overlay remains atomically clean during vapor deposition. We have also adapted a 20-kV electron-beam gun for in situ heating of bilayer couples, or the introduction of point defects in metal oxide or semiconductors via electronic mechanisms.

The bilayer specimens created in this manner can be used to study the reaction between, for example, a silicon substrate—the FIM specimen—and a deposited metal layer to study the chemical composition of the very earliest stages of metal silicide formation$^{2,3}$ and also to study metal/metal solid-state reactions on the sub-nm scale.$^{4,5}$ It is furthermore possible to deposit germanium on a silicon FIM specimen to study the chemical composition at Si/Ge interfaces,$^4$ and to search for oscillatory segregation profiles, or the effects of arsenic on the growth morphology of epitaxial germanium films.$^7$

The deposition of a second element on FIM specimens has been accomplished generally by a resistive heating technique; i.e., a source material is attached to a tungsten filament and vaporized by electrically heating the filament. This technique has been used to study, e.g., epitaxial crystal growth$^{8,9}$ and interfacial chemical reactions.$^{10}$ In spite of the simplicity of this approach it is not satisfactory for our purposes, as (a) the thickness of the deposited layer can not be precisely controlled, and (b) a large charge of material can not be placed in the source and therefore it must be frequently changed.

In this paper the design, fabrication, and performance of an MEBE are discussed. Examples of its application to the deposition of silicon or titanium on tungsten FIM tips are presented. A chemical analysis, via the APFIM technique, of a Ti/W bilayer was performed, and this bilayer is shown to have an atomically sharp interface, from a chemical point of view, on the basis of APFIM analyses.

II. DESIGN AND FABRICATION OF A MINIATURE ELECTRON-BEAM EVAPORATOR

Figure 1 is a schematic diagram of the MEBE; see the Appendix for the electrical circuits. The principles of operation of this MEBE are as follows. A positive voltage is applied to the target, and it is heated by electrons that are thermionically emitted from a resistively heated tungsten filament (0.25 mm diam.). The latter is biased at a positive potential $V_a$ to prevent the grounded evaporator body—a
FIG. 1. A schematic diagram illustrating the principle of the construction of the miniature electron-beam evaporator and the arrangement of the power supplies with respect to it. The high-voltage dc power supply places the target at a positive potential and determines the energy of the electrons that bombard it. The tungsten filament is resistively heated using the filament power supply. The filament is biased at a potential of 60 V dc, with respect to the stainless-steel cylinder that contains the evaporator, employing the bias potential power supply.

stainless-steel cylinder with a 15 mm diam and a 50 mm length—from being bombarded by the electron beam. The cylindrically shaped target is held in place by a 0.75-mm-diam tungsten wire. The dimensions of the target for the power input are such that a very thin surface layer on the side of the target facing the filament is molten, and the evaporation rate from this molten region is sufficient to obtain the desired deposition rate. In the case of Ti or Si, for example, a cylinder with a 6 mm diam and an 8 mm height yields a deposition rate of ≈0.3 nm min⁻¹ for a voltage of 2 kV dc between the filament and the target, an electron emission current of 10 or 19 mA for Ti or Si, respectively, and a bias potential of 60 V dc with respect to the body of the evaporator are employed. A conically shaped nozzle is used to focus the desired evaporant onto the FIM tip because it makes a larger solid angle with respect to the target than a cylinder, and therefore yields a large flux of evaporant at the tip of an FIM specimen. The diameter of the orifice at the end of the nozzle is ≈3 mm. This diameter is necessary to prevent the evaporant from being deposited on the sapphire electrical insulator that holds a FIM specimen or the channel electron multiplier array (CEMA).¹

The flux emanating from a MEBe is calibrated in a separate high-vacuum system before it is mounted on the APFIM chamber. Two different calibration procedures are used to determine an average value of the flux. The first one consists of evaporating onto a glass slide for a known period of time. The thickness of the layer is then measured employing a Tencor Instruments profilometer (Alpha-Step 200) with a resolution of 10 nm. The second method involves evaporating onto the apex of a tungsten tip for a given period of time and measuring the thickness of the deposited overlayer using a transmission electron microscope (TEM). For both calibration procedures the distance from an evaporation source in a MEBe to the glass slide or the tungsten tip is identical, and is the same value as in the APFIM during vapor deposition. The two methods yield the same value of the flux. Once an MEBe has been calibrated for a specified set of conditions, its rate varies very little for a large accumulated deposition thickness (several hundred nm), as only one surface of the target is evaporated and the volume of source material employed is rather large. The steady-state evaporation rate from a MEBe allows us to precisely control the thickness of a deposited layer.

An MEBe is attached to a linear motion feedthrough (Huntington Corporation, model number VF-107-6-A) via ceramic tubes, and the entire evaporator assembly is then mounted on the main chamber of the APFIM (Fig. 2). The linear motion feedthrough permits us to bring an MEBe to within <5 mm of an FIM tip during a deposition process, and to retract it at the completion of a deposition; the total travel of the MEBe is ≈25 mm. Our APFIM possesses a goniometer stage with two axes of tilt; they allow us to position precisely an FIM specimen with respect to the MEBe during vapor deposition.¹ The goniometer stage is attached to a continuous-flow liquid-helium cryostat, via a flexible gold braid, whose temperature can be varied between room temperature and ≈12 K (Ref. 1); thus we have a wide temperature range over which an FIM specimen can be maintained during a deposition. At the completion of a deposition the bilayer specimen can be irradiated with low-energy ions (50 V–3 kV) without having to change the temperature of the specimen,¹¹ or alternatively the bilayer can be reacted thermally at an elevated temperature.

The background vacuum in the APFIM is ≈4×10⁻¹⁰ Torr during a deposition. At this pressure the
flux of evaporant atoms hitting an FIM tip during the course of an in situ deposition is much greater than the flux of atoms from the background vacuum arriving at the tip. This point is significant because it implies that the surface of an atomically clean FIM tip, prepared by the field-evaporation process, can be maintained in this state during an in situ deposition. In this manner an atomically clean interface between the deposited overlay and a FIM tip is achieved.

III. PERFORMANCE OF A MINIATURE ELECTRON-BEAM EVAPORATOR

We have utilized the MEBE to deposit silicon or titanium atoms on field-evaporated FIM tungsten tips with the evaporation conditions stated in Sec. II. Solid-state reactions at Si/metal interfaces are of great technological interest. We therefore prepared a Si/W bilayer couple by vapor depositing Si on a tungsten FIM tip which was maintained at room temperature. An FIM micrograph of the Si layer is exhibited in Fig. 3(a) (the imaging gas is neon at a gauge pressure of $1 \times 10^{-5}$ Torr) at a specimen temperature of 45 K. The image gives the appearance of a random array of atoms, and thus the vapor-deposited silicon layer may be amorphous; we cannot, however, state that it is amorphous from this FIM image. There is experimental evidence that a silicon overlay deposited on a metal surface at room temperature is amorphous. Figure 3(b) is a transmission electron micrograph showing the silicon overlay; it was not possible to obtain a selected-area diffraction pattern from this overlay to ascertain whether it was amorphous or crystalline.

Ti/W bilayer couples were fabricated by depositing titanium atoms on a field-evaporated tungsten surface at a specimen temperature of 77 K. Figure 4 exhibits three transmission electron micrographs of a Ti/W bilayer couple taken at three different rotation angles ($+30^\circ$, $0^\circ$, $-30^\circ$) about the long axis—$\approx[011]$-type direction, of the FIM specimen. This specimen was examined employing a radically modified version of a double-tilt stage for an Hitachi 700 200-kV TEM; the tilt stage accepts one cm long FIM specimens, and thus it is possible to re-examine a specimen in the APFIM after it has been examined by TEM. The micrographs show that the Ti layer thickness is $\approx 25$ nm, and from the three different views of the tip it is clear that the thickness is fairly uniform.

The as-prepared specimen was examined in the APFIM before being removed for TEM observation. The FIM images of the titanium surface are similar to those of a Si/W specimen (Fig. 3) and this indicates that the titanium is in a highly disordered state; it may, in fact, be in an amorphous state but we do not have a quantitative way of
assessing this fact. During APFIM analyses the specimen was maintained at 45 K and the ratio of the pulsed field-evaporation voltage \( (V_f) \) to the steady-state imaging voltage \( (V_{dc}) \) was \( V_f/V_{dc} = 0.1 \).

Figure 5 is an integral depth profile (cumulative number of events versus the cumulative number of field-evaporation voltage pulses applied to the tip) of a cylinder of material that is normal to the plane of the Ti/W interface. This integral profile demonstrates conclusively that this Ti/W interface, created at 77 K, is extremely sharp on an atomic scale from a chemical point of view. Initially only titanium ions are detected and the last Ti ion to be collected is at 17,500 pulses. The next species to be collected is tungsten, and once this happens no titanium ions are collected. This result demonstrates unequivocally that the transition from titanium to tungsten occurs within one interplanar spacing along the direction being analyzed. Since the titanium image appears random there is no way to index the plane of the region being analyzed in the titanium overlay. In the tungsten specimen the axis of the cylinder of metal analyzed is the \( \approx [011] \)-type direction.

IV. 20-kV ELECTRON-BEAM GUN

An electron-beam gun is utilized for the irradiation of bilayer couples. A 20-kV electron gun, manufactured by Superior Electronics Corp., U.S.A., was mounted on a 2.75-in.-dia Conflat flange for attaching it to our APFIM (see Fig. 2). This electron gun has both \( x \) and \( y \) deflection plates so that the electron beam can be rastered as well as focused; a current of 10 mA at 20 kV can be obtained from this gun (see the Appendix). During an electron bombardment of an FIM specimen it is possible to maintain the tip either at earth potential or at a positive potential; a positive potential prevents positively charged ions from adsorbing on the surface of a tip during an irradiation. This gun is useful for a number of experimental situations, e.g., (a) electron-beam heating to cause interdiffusion of a bilayer couple, (b) electron-irradiation of metal oxide/metal interfaces to create point defects in the metal oxide overlayer via electronic processes, and (c) the creation of point defects in semiconductor overlayers via electronic processes.

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APPENDIX

The high-voltage dc power supply constructed is a simple full-wave bridge rectifier with each arm composed of eight diodes (1N4007) and four 2.2-M\( \Omega \) resistors. One ammeter is used to measure the emission current of the filament, and a second ammeter is used to measure the high-voltage output.

Figure 6 is a circuit diagram for the filament power supply circuit. The voltages \( V_{fo} \) and \( V_{fb} \) are connected to the filament, and \( V_f \) is the bias voltage. This power supply can be used in either an unregulated or a regulated mode;
a mode is chosen via the switch SW3. In the regulated mode the emission current of the filament is stabilized through a regulator circuit that controls the power to the filament and hence the filament temperature; the emission current is, of course, a function of the filament temperature.

Figure 7 is a schematic diagram of the specially fabricated circuit used with this electron gun. This circuit was designed for operating at a maximum voltage of 20 kV and it provides an input power of 200 W at the FIM tip for an electron emission current from the gun of 10 mA. The filament current, emission current, and focusing voltages are controlled by the potentiometers labeled PO1, PO2, and PO3, respectively.